# University of Nottingham

## School of Civil Engineering

# Investigating the Potential for Incorporating Tin Slag in Road Pavements

By

**Mohd Abd Wahab Yusof**

Thesis submitted to the University of Nottingham

for the degree of Doctor of Philosophy

March 2005

***To My Family and  My Mother***

***and***

***In Memory of My Father***

# TABLE OF CONTENTS

**CHAPTER 4 – INVESTIGATING THE POTENTIAL USE OF TIN SLAG AS UNBOUND GRANULAR MATERIAL**



**CHAPTER 5 – ASSESSMENT OF SLAG IN HYDRAULICALLY BOUND MIXTURES**

# ABSTRACT

The demand for and utilisation of industrial by-products and wastes in road pavements is increasing and becoming more important. Government policies and public awareness is also contributing to the enhancement of both this demand and utilisation. Ferrous slags such as blastfurnace and steel slags have been widely used in road pavements. On the other hand, non-ferrous slags such as tin, copper and phosphoric slags have potential and may also be utilised if technical feasibility can be proven. Hence, the principal objective of this research was to assess the possibility of using tin slag in road pavements.

A thorough laboratory evaluation of tin slag was performed. The physical and mechanical properties of the tin slag particulates were initially assessed in accordance with the UK specification for Highways Works. Based on these results, the tin slag was subsequently further assessed for its suitability for use as unbound granular material, as a pozzolanic binder, as cement bound material and as an asphalt. Conventional laboratory tests including repeated load triaxial, Marshall testing and Nottingham Asphalt Testing was performed. The durability of the mixtures was also evaluated in terms of ageing and water sensitivity. The environmental, including radiological, effects of using tin slag in a road pavement were also assessed using tank leaching and compliance leaching tests. The results showed that the slag had potential in all types of pavement materials except as a pozzolan.

The application of tin slag in road pavements was evaluated using the Nottingham Pavement Design Method and Shell's Bituminous Stress Analysis in Roads (BISAR) computer programme. The analysis demonstrated that the tin slag has potential for use in both asphalt layers (although, preferably, not as a wearing course) and unbound granular base layers.

# ACKNOWLEDGMENTS

I would like to express my gratitude to all persons and organisations who contributed directly or indirectly to this thesis with their support, encouragement and advice, thus helping towards its timely conclusions.

Acknowledgement is due first and foremost to the Government of Malaysia who generously sponsored this research project and without whose financial support it would not have been viable. I also wish to express my appreciation and thanks to Mr. Andrew R. Dawson and Dr. Salah E. Zoorob for their continued guidance, advice and invaluable insight throughout this research.

Special thanks are also to the Nottingham Centre for Pavement Engineering (NCPE), School of Civil Engineering for providing all the necessary facilities. My thanks are also extended to the laboratory staff and all members of the staff and postgraduate students at NCPE for all their help, support, teatime discussions and their friendship.

Finally, special thanks to my wife, Noorsiah, my children, Najwa Atheerah, Mohammad Darwish, Muhammad Ammar and Nabilah and my parents for their unconditional patient and continued support throughout the research period.

# DECLARATION

The work described in this thesis was conducted at the University of Nottingham, School of Civil Engineering between October 2001 and October 2004. I declare that the work is my own and has not been submitted for a degree of another university

# LIST OF SYMBOLS

| | |
|---|---|
| $\Delta$ | Increment |
| $\sigma$ | Normal stress |
| $\sigma_1$ | Total axial stress |
| $\sigma_3$ | Confining pressure |
| $\sigma_d$ | Deviator stress |
| $\sigma_{avg}$ | average value of compressive stress under the moving wheel |
| $\sigma_{x\,max}$ | Maximum tensile stress at the centre of specimen |
| $\varepsilon_{x\,max}$ | Maximum horizontal tensile strain |
| $\varepsilon_{1r}$ | Maximum resilient axial strain |
| $\varepsilon_z$ | Maximum vertical (compressive) strain |
| $\varepsilon_t$ | Tensile strain |
| $fr$ | Rut factor |
| ø | Angle of internal friction |
| $\nu$ | Poisson's ratio |
| $\eta$ | Viscosity of bitumen |
| $\omega$ | water content |
| $\tau_f$ | Shear strength |
| $c$ | Apparent cohesion |
| P | Total percentage by mass passing sieve size d |
| $C_2$ | Concentration of a particular constituent in the eluate |
| $C_8$ | Concentration of a particular constituent in the eluate from the second extraction |
| $C_i$ | total activity in the material per unit mass of radionuclide *i* |
| $C_{li}$ | Clearance level of radionuclide *l* |
| $C_m$ | dynamic effect correction factor |
| $G_b$ | Specific gravity of binder |
| $G_a$ | Specific gravity of mixed aggregate |
| CBM | Cement bound material |
| $E_{gb}$ | Stiffness modulus of unbound granular sub-base |
| $E_{sb}$ | Stiffness modulus of sub-grade |

| | |
|---|---|
| ITSMR | Indirect tensile stiffness modulus ratio |
| ITSR | Indirect tensile strength ratio |
| L2 | Volume of leachant used in first extraction |
| L8 | Volume of leachant applied in the second extraction |
| L/S | Liquid to solid ratio |
| LTOA | Long term oven ageing |
| $M_A$ | Aggregate content per cent mass of total mix |
| $M_B$ | Binder content per cent by mass of total mix |
| $M_r$ | Resilient modulus |
| $N_f$ | fatigue life |
| NAA | Neutron activation analysis |
| NAT | Nottingham asphalt tester |
| OMC | Optimum moisture content |
| OBM | Optimum binder content |
| $P_i$ | Mean compressive strength of immersed specimens |
| $P_c$ | Mean compressive strength of control specimens |
| $R_c$ | Compressive strength |
| $R_d$ | Rut depth |
| $R_i$ | Compressive strength of the immersed in water |
| $S_m$ | Stiffness modulus |
| $S_{mix}$ | Stiffness modulus of mixture |
| $S_{bit}$ | Stiffness modulus of bitumen |
| $SP_i$ | Initial softening point of binder |
| $T_w$ | loading time of one load pulse |
| TS | Tin slag mixture without any alteration on gradation |
| TSS | Tin slag mixture with sand added |
| TB | Tin slag mixture added aggregate with size between 10mm and 20mm |
| $V_{E1}$ | volume of eluate recovered from the first extraction |
| $V_b$ | Volumetric proportion of binder |
| VMA | Voids in mineral aggregate |
| $V_v$ | Volume of voids |

# LIST OF TABLES

**CHAPTER 5**



**CHAPTER 6**

# LIST OF FIGURES

**CHAPTER 2**



**CHAPTER 3**



**CHAPTER 4**

**CHAPTER 5**

**CHAPTER 6**

Chapter
1

# INTRODUCTION

## 1.1 BACKGROUND

The utilization of industrial by-products and recycled materials in road construction as secondary and alternative materials has gained widespread acceptance and is becoming more important. The demands for industrial by-products and recycled materials are increasing every year. In the United Kingdom (UK), the sale of secondary aggregate was 9.47 million tonnes (Mt) in 1997 compared with 5.16 Mt in 1993 (British Geological Survey, 2001). Several European countries have also set targets to use industrial by-products and recycled materials as alternative materials. The UK, for example, has set targets to increase the use of alternative materials (secondary aggregates and recycled aggregates) from 30 million tons per annum (Mtpa) in 1998 to 55 Mtpa by 2006 (British Geological Survey, 2001).

There are many factors that influence the success of recycling of these materials. The relative importance varies from country to country but typically includes the following: -

- The public is becoming more concerned about the environment.
- Lack of virgin aggregates.
- Public opposition to aggregate quarrying.
- Public opposition to land-filling.
- Government policy to minimize the use of natural aggregate and promote the use of recycled materials as alternative aggregates.
- Introduction of aggregate taxes to encourage recycling and utilization of secondary material.
- Introduction of landfill taxation to stimulate waste reduction and re-use.

- Introduction of climate change taxes by government.

With regard to the utilization of wastes or recycled materials, the European Commission, in the Community Strategy for Waste Management 1996, had set out general strategies on waste management as follows:

- Prevention of production of wastes;
- Recycling and reuse of by-products or wastes; and
- Optimisation of the final disposal of wastes and improved monitoring.

The use of industrial by-products and recycled materials in road construction falls under the second of these strategies. However, any proposal to incorporate a waste or by-product material into a pavement structure requires, in addition to an engineering evaluation, an evaluation of environmental, occupational safety and health, recyclability, economic and implementability issues. All of these issues need to be addressed prior to determining the acceptability of the material. The other issues which should be addressed are standardization and guidance. At present, there is no such overall standard protocol and guidance to be followed during the construction of a road using secondary aggregates.

There exists a wide range of wastes and by-products, which have potential to be used in a road environment. Some of the examples of these materials are china clay sand, blastfurnace slag, steel slags, pulverised fuel ash, gypsum and cement kiln dust [Nunes, 1997; Atkinson et *al*., 1999; Sherwood, 2001]. Granulated blast furnace slag and steel slag have been widely used in road construction in European countries such as France, Holland and Germany as an alternative material (Robinson and Acock, 1999). Other metallurgical slags which have potential to be used in road pavements are tin slag, lead slag, phosphorus slag and zinc slag.

Although the use of secondary aggregates in road construction is increasing, it is still very limited if comparison is made with the use of primary aggregate. In England, the total consumption of primary aggregate in 1997 was 164.9 million tonnes which is 155.2

million tonnes more than the amount of secondary aggregate used (British Geological Department, 2001). Some of the factors or barriers responsible for the limited use of secondary materials are described as follows (Nunes, 1997 and Reids, 2001):

- Relative abundance, ready availability and low cost high quality primary granular materials;
- Concerns about the uncertainty and unreliability of mechanical and durability performance of secondary aggregates;
- Low cost of landfill disposal;
- Inability to meet highway authority requirements for the materials to be used in road construction;
- Inadequacy of existing specifications for direct applications of secondary aggregates;
- Transportation costs from source to areas of major demand;
- The lack of information on the environmental effects of using secondary materials in road construction.

At present there are no specific restrictions on incorporating non-ferrous slags such as tin slag, zinc slag, copper slag and phosphoric slag in road pavements. The only barrier, which challenges the use of such slags in road pavements, is a technical barrier. This is illustrated by the following British Standards quotation:

> " Other slags may be suitable aggregates for rolled asphalt
> but are outside of the scope…"
>
> [BS 594-1:1992 and BS 4987-1:1993]

Therefore, the utilisation of non-ferrous slags in bitumen bound surfacings, base-courses and granular sub-bases of flexible pavements construction may be allowed if technical feasibility can be proven.

## 1.2 THE NEED FOR THIS RESEARCH

Malaysia is one of the major tin producers in the world with the biggest tin smelter being situated at Butterworth, in the State of Penang, on the northern part of the Malaysian Peninsula. Apart from the smelting of domestic mine production, the plant also imports between 50,000 and 60,000 tonnes of concentrates and residues for processing annually. The principal product is tin metal (in the form of ingot), and gangue material (the earthy or stony matter in a mineral deposit or the matrix in which an ore is found) which is received with the tin concentrates and accumulates as waste slag. The annual production of tin slag in Malaysia is between 20,000 and 24,000 tonnes. However, this depends on the quantity of the feed processed and the grade of the tin concentrates received. At present the slag is stored at the smelter premises. The situation is critical as the stockpiling of the slag continues indefinitely. At present, there is no solution to this problem. Disposal or recycling of the slag is currently not allowed until there is sufficient evidence to show that the slag will not pose any danger to the public or the environment.

The feed materials contain trace quantities of heavy metals and naturally occurring radioactive materials (NORM). In the smelting process, these materials partition to the slag phase, and ultimately combine with the gangue minerals, ash from the reductant, and flux additives in the waste slag.

Like other metallurgy slags, tin slag has the potential to be used as a secondary aggregate in road construction. However, this is not a simple decision to be made since many factors need to be considered. The evaluation of this material, before it can be used, should be based on technical, economic and environmental factors. In terms of technical adequacy, tin slag must satisfy the test requirements that are imposed on naturally occurring aggregates used for the same purpose.

Assessment of the suitability in terms of physical, mechanical and chemical properties of materials is required in order to maintain appropriate standards of quality and performance in road construction. However, this is not entirely true because some recycled materials, although failing against some standard tests, can still provide very good performance when used in the field. In addition to physical, mechanical and chemical properties, the materials used in pavements must also not generate any potential adverse effects on the environment whether during the construction of the road or throughout the whole life of the pavement. At present, there are very limited data for, and research that has been carried out on the application of tin slag in road pavements. For this reason, there is a need to assess real risks of this slag whether to the public or the environment and its mechanical capacity so it can be beneficially used in the future.

## 1.3 OBJECTIVES

The principal objectives of this research are to determine the possible application of tin slag in road pavements and to assess the environmental consequences when used in such an application. In more detail, the objectives of this research are as follows:

- To study the basic physical and mechanical properties of tin slag in order to contribute to a better knowledge of its properties.
- To investigate the potential use of tin slag as unbound granular materials.
- To assess the potential use of tin slag in hydraulically bound materials, including slag bound and cement bound materials.
- To investigate the potential for incorporating tin slag in bituminous bound materials.
- To evaluate the environmental issues surrounding the use of tin slag in road pavements.

## 1.4 BENEFITS OF THIS RESEARCH

There are many benefits that can be expected from this research. However, in the case of this investigation, the likely benefits of this research can be divided into an immediate and long-term benefit.

### 1.4.1 Immediate benefit

The results of this research will contribute to a greater understanding of the possibility for using tin slag in road construction as an alternative material, especially in terms of laboratory performance-based tests.

### 1.4.2 Long Term Benefits

The long-term benefits resulting from the use of tin slag in road construction are:

- The dramatic reduction in the stockpiling of tin slag.
- The reduction in the demand for primary aggregates in nearby areas allowing the preservation of finite resources and reducing the indirect environmental impact caused by quarrying activities.
- The reduction of costs related to the quarrying and transportation of conventional aggregate.
- The reduction of economic problems associated with storage and dumping.
- The reduction of the environmental problems associated with stockpiling.

## 1.5 THESIS ORGANISATION

The thesis is organized in nine chapters. After this introductory chapter, Chapter 2 provides an initial overview of the use of various slags in road pavements. The review covers the production processes, the physical and mechanical properties of several types of slags. It also describes the use of various types of slags in road construction as well as the problems arising from the use of these slags.

Chapter 3 presents more specific data on tin slag properties. Tin slag was assessed against current specifications regarding its physical and mechanical properties. The chapter also explains why certain tests were not performed. Chapter 4 is an investigation into the potential for using tin slag as unbound materials. The performance of tin slag as unbound granular material was assessed and is discussed in terms of its shear strength and stiffness modulus.

The pozzolanic property of tin slag was assessed and is discussed in Chapter 5. Details of the methodology and the possibility of using ground tin slag in slag bound materials was also discussed. This chapter also describes an assessment of the possibility of using tin slag in cement bound materials. Two mixtures, tin slag and tin slag/sand mixtures were evaluated to determine the suitability of incorporating tin slag into cement bound materials.

Chapter 6 focuses on the investigation of the potential for incorporating tin slag in bituminous bound materials. It presents the procedures used to design asphalt mixtures. A thorough investigation using a range of tests including the Marshall stability and flow test, the indirect tensile strength (ITS) test, the indirect tensile stiffness modulus (ITSM) test, the indirect tensile fatigue (ITFT) test and the repeated load axial test (RLAT) was performed and analyses of the results shown. The evaluation of tin slag asphalt mixture durability was also carried out using long-term oven aging (LTOA) and water sensitivity tests and is reported in this chapter.

The Environmental effects should be assessed if tin slag is to be used in road pavements and this aspect is discussed in Chapter 7. The heavy metals and radioactivity levels were thoroughly assessed using Tank leaching and Compliance test methods and the results were compared with World Health Organisation (WHO) reference values.

Many of the results from the assessment of tin slag presented in Chapters 4 and 6 were then used in pavement structural designs which are discussed in Chapter 8. The Shell Bitumen Stress Analysis in Roads (BISAR) computer model and the Nottingham Pavement Design Method were used to analyse the stresses and strains in hypothetical pavement structures incorporating the materials investigated earlier. This chapter shows design examples of how tin slag can be used as a granular base and in an asphalt layer. The final chapter, Chapter 9, details the conclusions and recommendations for further work.

**Chapter 2**

# SLAGS IN ROAD CONSTRUCTION
## A REVIEW

## 2.1 AN INTRODUCTION TO PAVEMENT STRUCTURE

A pavement is a part of the road, subjected to stresses imposed by vehicular loading applied, as well as to deterioration from the effects of weather and the abrading action of moving traffic. A satisfactory pavement design is one that is able to withstand these effects for a required period of time. A pavement consists of a multi-layer system, which is formed of a number of layers of compacted unbound aggregates and/or bound materials.

There are three main components of a road pavement; foundation, base and surfacing. Figure 2.1 illustrates some typical road pavement structures. The foundation comprises subgrade soil (cut or fill), capping and sub-base. The foundation is designed to provide a certain standard quality of support for the higher layers. The base is the main structural layer of the pavement. Meanwhile an asphalt surfacing comprises a surface course and a binder course. The function of the surfacing is to enable good ride quality to be combined with the appropriate resistance to skidding and resistance to crack formation. In the case of a concrete road, the surfacing and roadbase are combined to form a single layer.

Each of the layers in a pavement has its own function, which are summarised below (BACMI 1992).

- Subgrade

  Subgrade is not formally a pavement layer. However, its properties and function must be fully understood in order to design and construct a satisfactory pavement over it. Subgrade is the natural soil or made-up ground (fill) on which the pavement is built. The function of the subgrade is to support the load of the whole pavement.

- Capping

  Over a weak subgrade a capping may be provided to act as a subgrade improvement layer. It can also be used as a working platform and prevents deterioration of the subgrade. This layer usually uses a relatively low quality, cheap, locally available aggregate. Some recycled materials are also used as an alternative material to make a capping. Occasionally, a stabilizer such as cement or lime is incorporated into the upper part of the subgrade to improve the strength and bearing capacity of the subgrade soil. The surface of the subgrade or capping layer, where present, i.e. the level on top of which the sub-base is placed is known as the formation.

- Sub-base

  A sub-base is the main structural foundation layer. It must be able to resist the stresses transmitted to it via the road base and also must be stronger than the subgrade soil or capping beneath it. One of its main functions is to act as a platform upon which pavement construction can take place without damage to the subgrade. It also acts as a final load distribution layer and provides a frost-resistant layer.

- Base

  A base is the main structural layer of pavement, which provides the major part of the strength and load-distributing properties of the pavement so that the underlying materials are not over-stressed. It is normally designed to be very dense so as to be stiff, spread the loading well and is highly stable so that it resists permanent deformation and fatigue cracking due to repeated loading.

- Binder Course

  The main functions of the binder course layer are to distribute some of the traffic loading and to regulate the underlying layer to provide an even profile on which to lay relatively thin surface course layer.

- Surface Course

  The surface course is a uniform carriageway surface upon which vehicles run. It provides the initial load distribution. It also provides a weatherproof finish layer that gives skid-resistance and resistance to polishing and abrasion by traffic, an even running surface and one that will rapidly shed surface water. A good surface course should minimize traffic noise, resist cracking and rutting, protect the underlying road structure and be durable.

In a rigid pavement, the concrete slab is used as a substitute to the base and surfacing. The slab is composed of pavement-quality concrete with considerable flexural strength. This enables it to act as a beam and bridge over any minor irregularities in the surface of the layer beneath.

## 2.2 TYPES OF PAVEMENTS

There are four different types of pavement construction stipulated in the UK Manual on Highways and Bridge (Highways Agency 1998):

- Flexible

  In which the construction often comprises bituminous surfacing and roadbase layers placed over crushed stone or similar unbound sub-base or roadbase materials.

- Flexible Composite

  In which the surfacing and upper base (if used) are bound with bituminous binder on a base or lower base of cement-bound material.

- Rigid

  Pavement quality concrete is used for the combined surfacing and base. The concrete can be: -

  Jointed unreinforced concrete (URC)

Jointed reinforced concrete (JRC)

Continuously reinforced concrete slab (CRCP)

- Rigid Composite

  Continuously reinforced concrete base (CRCR) with bituminous surfacing.

Rigid pavements are seldom constructed in the UK.

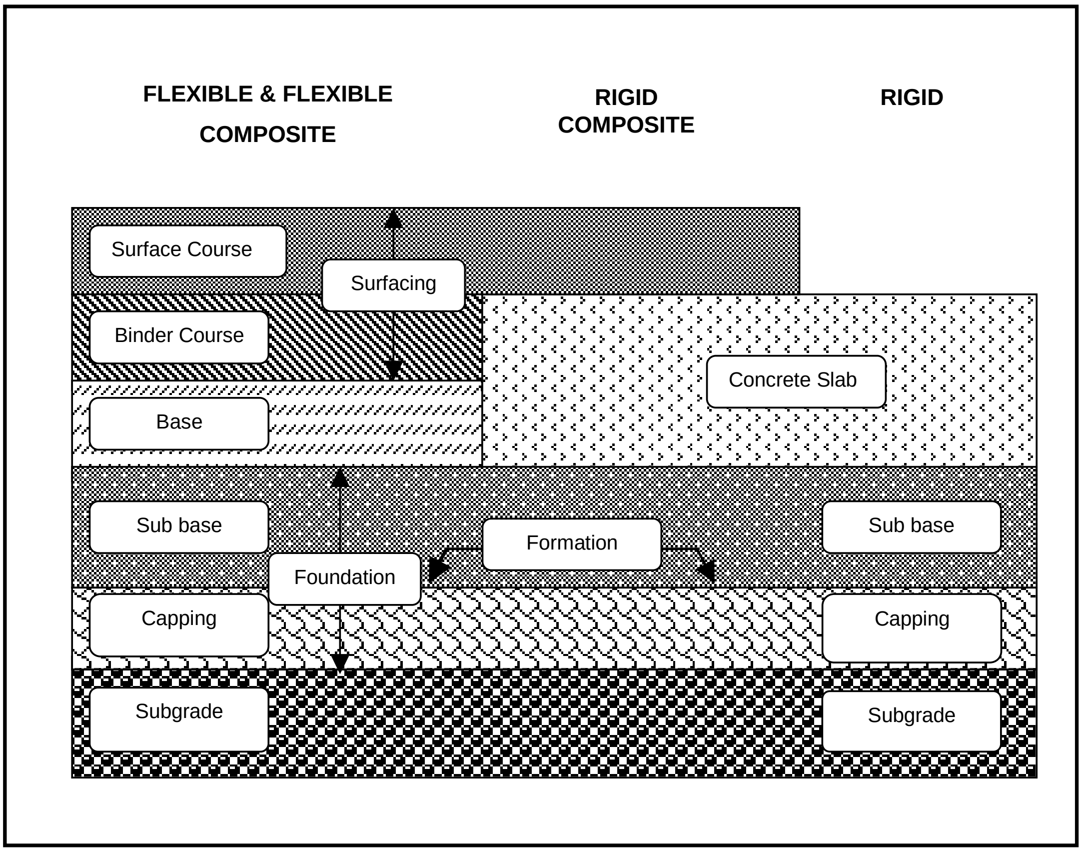



**Figure 2.1: Typical pavement structure (not to scale)**

## 2.3 GENERAL OVERVIEW ON THE USE OF SLAG IN ROAD CONSTRUCTION

Slag has been used in construction activities for a very long time. Much slag was used for road and wall making during the Roman period (Pawluk, 2004). In the early 19th century, slag was used to produce slag cement. The slag has also been used as rail ballast and for building bridges. During that time also, the casting of molten slag into blocks, the production of slag sand, the production of bricks and mortar and the manufacture of slag wool were introduced from blast furnace slag (Lee, 1974).

In general, slag can be divided into two categories, non-ferrous and ferrous slag. The various types of slag products have different properties and characteristics. The properties and characteristics of slag are very dependant on how they are processed during the making of the main products. Ferrous slag is produced during the refining of ferrous metal such as iron and steel. Blast furnace slag arises during the production of iron while steel slag is produced during the production of steel. Blastfurnace slag and steel slag can be produced in granulated, pelletised or air-cooled form. Blastfurnace slag has been used for many years and in many areas. For example, blast furnace slag is used as a high quality material in the cement industry, in glass making, concrete block manufacture and for road-stone application (Baldwin et. al., 1997). On the other hand, steel slag has more limited application.

Non-ferrous slag is produced during the recovery and processing of non-ferrous metal from natural ore. The slag is a molten by-product of high temperature processes that is primarily used to separate the metal and non-metal constituents contained in the bulk ore. When cooled, the molten slag converts to a rocklike or granular material. Examples of non-ferrous slag are copper, tin, nickel, lead, zinc and phosphorus slags.

Approximately 3.6 million metric tons each of copper and phosphorus slag are produced each year in the United States, while the annual production of nickel, lead and zinc slag is estimated to be in the range of 0.45 to 0.9 million metric tons (Collins and Ciesielski 1994). Most tin slag is produced in Malaysia, Thailand, China, and Indonesia which are the major tin producers in the world. In Malaysia, the production of tin slag is between 20,000 and 24,000 tonnes annually (MSC, 2001).

In the transportation industry, the slags are used for the construction and maintenance of roads as an alternative aggregate. Slags are also used widely as aggregate for concrete, the basis of Portland blast furnace cement, in sand blasting and as railway ballast. The Specification for Highway Works (SHW) also permits some slags to be used in place of natural aggregates. However when the specifications refer to individual materials, this may be taken to imply that the other materials not specified by name might be inherently unsuitable in some ways.

The Highways Agency of the UK has published guidance on the use of secondary aggregates in road construction. Under the present UK's Specification for Highway Works (Highways Agency, 2001) there are only two types of slag permitted to be used in road construction. Table 2.1 shows the possible application of blast furnace slag and steel slag within each series of SHW. However, in order that the slag can be used, it shall be well weathered and conforming to the requirements of BS 1047 (BSI, 1983) such as bulk density, stability (iron unsoundness and 'falling' or dicalcium silicate' unsoundness) sulphur content, water absorption, flakiness index, mechanical properties and grading.

## 2.4 BLAST FURNACE SLAG

### 2.4.1 Slag Production

Blast furnace slag is a non-metallic by-product produced in the process of iron production by chemical reduction in a blast furnace. It consists primarily of calcium silicates, aluminosilicates, and calcium-alumina-silicates. The molten slag comprises about twenty percent by mass of the iron produced.

In the production of iron, the blast furnace is charged with iron ore, fluxing agents (e.g. limestone and dolomite) and coke as fuel and the reducing agent. The iron ore is a mixture of iron oxides, silica and alumina. From this and the added fluxing agents, alkaline earth carbonates, molten slag and iron are formed. Oxygen in the preheated air is blown into the furnace where it combines with the carbon in the coke to produce the needed heat and carbon monoxide. At the same time, the iron ores are reduced to iron, mainly through the reaction of the carbon monoxide with iron oxide to yield carbon dioxide ($CO_2$) and metallic iron. The fluxing agents dissociate into calcium and magnesium oxides and carbon dioxide. The oxides of magnesium combine with silica and alumina to form slag (blast furnace slag). Figure 2.2 shows the production of iron and blast furnace slag. Typically, for ore feed with 60% to 65% iron, blast furnace slag production ranges from about 220 to 370 kilograms per metric ton of pig iron produced (Kalyoncu 2000). Lower grade ores yield much higher slag fractions.

The principal constituents of blast furnace slag are silica ($SiO_2$), alumina ($Al_2O_3$), calcia (CaO) and magnesia (MgO). Minor elements include manganese, iron, and sulphur compounds as well as trace amounts of several other elements. Depending on the method used to cool the molten slag, three different forms of slag product are usually produced namely air-cooled blast furnace slag, expended or foamed slag and pelletized slag or granulated slag.

Air-cooled blast furnace slag is produced when the liquid slag is poured into beds and slowly cooled under ambient conditions in an open pit. It has a crystalline and vesicular structure and is hard but can be crushed and screened. The vesicular texture gives the slag a greater surface area than smoother aggregate of equal volume. This will provide excellent bond with Portland cement as well as high stability in asphalt mixtures.

On the other hand if the molten slag is cooled and solidified by adding controlled quantities of water, air, or steam, the process of cooling and solidification can be accelerated, increasing the cellular nature of the slag and producing a lightweight expanded or foamed product. This product is called foamed blast furnace slag and can be distinguished from air-cooled blast furnace slag by its relatively high porosity and low bulk density. It is suitable to use in concrete.

If the molten slag is cooled and solidified by rapid water quenching, this prevents the crystallization of the constituent minerals, thus resulting in a granular, glassy aggregate. This process results in the formation of sand size fragments, usually with some friable clinker-like material and the slag, which is produced, is called granulated blast furnace slag. The physical structure and gradation of granulated slag depend on the chemical composition of the slag, its temperature at the time of water quenching and the method of production. When crushed, milled and screened to very fine cement-sized particles, granulated blast furnace slag has pozzolanic properties (hydraulic cementitious properties) which make a suitable partial replacement for, or additives to, Portland cement.

Figure 2.2 summaries the manufacturing process of both blast furnace and steel slags. About 70% of the blast furnace slag produced is air-cooled, which when crushed can be used as an excellent substitute for natural aggregates, while 25% is granulated slag and the rest is pelletized and foamed.

**Table 2.1: The application of blast furnace slag and steel slag in road construction (Highways Agency, 2001)**

| Application | General Fill | Capping | Unbound Sub-base | Bitumen Bound Layers | Cement Bound Layers | Cement Bound Roadbase | Pavement Quality Concrete |
|---|---|---|---|---|---|---|---|
| SHW Series | 600 | 600 | 800 | 900 | 1000 | 1000 | 1000 |
| Blast furnace Slag | √ | √ | √ | √ | √ | √ | √ |
| Steel Slag | √ | √ | √ | √ | X | X | X |
| √<br>X | Permitted<br>Not permitted | | | | | | |

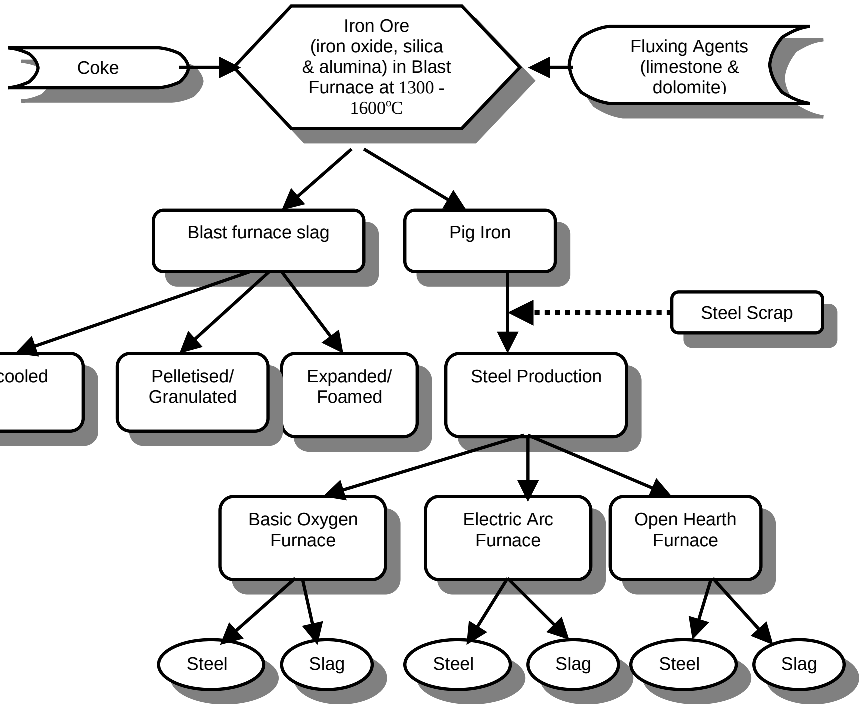



**Figure 2.2 The process of blast furnace and steel slag production (Sherwood, 2001)**

### 2.4.2 Slag Properties

Blast-furnace slag is consistent in its physical properties as would be expected for a particular source of natural aggregate. The principal constituents of blast furnace slag are silica ($SiO_2$), alumina ($Al_2O_3$), calcia (CaO) and magnesia (MgO). Minor elements include manganese, iron, and sulphur compounds as well as trace amounts of several others. When air-cooled blast furnace slag is crushed and screened, it produces an aggregate with a rough surface texture and relatively high porosity. This gives its good characteristics when used with both cement and bituminous binders.

#### 2.4.2.1 Physical Properties

The physical characteristics of blast furnace slag such as density, porosity and particle size are affected by the cooling rate (i.e. cooling method) of the slag and its chemical composition. Air-cooled blast furnace slag is angular, roughly cubical and has textures ranging from rough, vesicular (porous) surfaces to glassy surfaces with conchoidal fractures. When it is crushed and screened, its physical properties make it particularly suitable as an aggregate, both coated and uncoated. It has a rough surface texture giving good frictional properties and good adhesion to bituminous and cement binders, a low coefficient of thermal expansion and a high fire resistance. It also has relatively high water absorption due largely to its porosity.

**Table 2.2: Typical physical properties of air-cooled blast furnace slag (Lee 1974)**

| Property | Value |
|---|---|
| Specific gravity | 2.38 - 2.76 |
| Bulk density, $kg/m^3$ | 1150 - 1440 |
| Aggregate crushing value (ACV) | 25 – 35 |
| Aggregate impact value (AIV) | 21 - 42 |
| Aggregate abrasion value (AAV) | 5 - 31 |
| Water absorption (% by mass) | 1.5 - 6 |
| Polished stone value (PSV) | 50 - 63 |

### 2.4.2.2 Chemical Properties

The general chemical compositions for all types of blast furnace slags are shown in Table 2.2. The principal constituents of blast furnace slag are silica ($SiO_2$), alumina ($Al_2O_3$), calcia (CaO) and magnesia (MgO) which make up 95% of the composition (Kalyoncu 2000). According to Sherwood (1995), blast furnace slag is not a pozzolan, nor in itself cementitious, but it possesses latent hydraulic properties, which can be developed by the addition of an activator such as lime or another alkaline material. However, when ground to a proper fineness, the chemical compositions and glassy nature of vitrified slag are such that when combined with water, the vitrified slag reacts to form a pozzolanic product (by cementitious hydration). The magnitude of these cementitious reactions depends on the chemical composition, glass content and fineness of the slag. Blast furnace slag is mildly alkaline and exhibits a pH in solution in the range of 8 to 10.

**Table 2.3: Typical chemical properties of blast-furnace slag (National Slag Association, 1985)**

| Properties | Value (%) |
|---|---|
| Calcium oxide (CaO) | 34 - 43 |
| Silicon dioxide ($SiO_2$) | 27 – 38 |
| Aluminium oxide ($Al_2O_3$) | 7 - 12 |
| Magnesium oxide (MgO) | 7 – 15 |
| Iron (FeO or Fe2O3) | 0.2 – 1.6 |
| Manganese oxide (MnO) | 0.15 – 0.76 |
| Sulfur (S) | 1.0 – 1.9 |

### 2.4.2.3 Mechanical Properties

Air-cooled blast furnace slag is most commonly used as an aggregate material compared with other blast furnace slags. The slag, when processed properly, exhibits favourable mechanical properties for aggregate use, including good abrasion resistance, good soundness characteristics and high bearing strength (TFHRC 2001). Table 2.4 shows typical mechanical properties of air-cooled blast furnace slag.

**Table 2.4: Typical mechanical properties of air-cooled blast-furnace slag (Noureldin and McDaniel, from TFHRC 2001)**

| Properties | Value |
|---|---|
| Los Angeles Value (ASTM C131) | 35 – 45 % |
| Sodium Sulfate Soundness Loss (ASTM C88) | 12 % |
| Angle of Internal Friction ($\phi$) | 40 - 45 |
| Hardness (measured by Moh's scale of mineral hardness)* | 5 - 6 |
| California Bearing Ratio (CBR), max size 19 mm (3/4 in)** | Up to 250% |

*Hardness of dolomite measured on same scale is 3 to 4

** Typical CBR value for crushed limestone is 100%

## 2.4.3 The Use of Slag in Road Construction

Blast-furnace slag has been used in many fields such as Portland cement concrete, granular base, asphalt pavement and embankment, fill materials, concrete aggregate, mineral wool, and railroad ballast. Because there is no significant risk, the EC has eliminated blast-furnace slag from its waste schedule.

For quality control of blast furnace slag production, the British Standard Institution has produced some standards such as BS 1047 (1983) and BS 3797 (1990) to be followed. The UK Highway Agency has allowed the use of blast furnace slag in bulk fill, capping, unbound sub-base, bitumen bound layers and cement bound layers, cement. However, the use of blast furnace slag in road construction shall follow further specifications written in the Design Manual for Roads and Bridges (Highways Agencies et al (as amended 2001)) and also the slag to be used must meet the requirement of BS 1047. Sherwood (1995) has proposed some of the specifications to be followed when using blast furnace slag in construction as listed below:

- Total sulphur content must be not greater than 2% (as S) for concrete aggregate.
- Sulphate content must be not more than 0.7% (as $SO_3$)
- Water-soluble sulphate content must not exceed 2.0 g/l for unbound material.
- Blast furnace slag must not have absorption of more than 10%.
- Flakiness index (FI) of coarse aggregate must not exceed 35.
- Ten percent fines value must be not less than 50 kN.
- Bulk density of compacted blast furnace slag in the range 10 to 14 mm must be not be lower than 1.1 $Mg/m^3$.
- Stability against two forms of unsoundness - There are two forms of unsoundness, iron unsoundness and dicalcium silicate (also known as ‘falling’) unsoundness. Slag, which develops no cracking, disintegration, shaling or flaking during the storage period, is regarded as free from iron unsoundness. The slag is regarded as free from 'falling' when:

  $CaO + 0.8MgO \leq 1.2SiO_2 + 0.4Al_2O_3 + 1.75S$

  or/and

  $CaO \leq 0.9SiO_2 + 0.6Al_2O_3 + 1.75S$.

In asphalt paving applications, blast-furnace slag (air-cooled) is considered to be a conventional aggregate and can replace both coarse and fine aggregate. Hot mix asphalt containing properly selected and processed air-cooled blast furnace slag aggregates shows good frictional resistance in pavement surfaces, good stripping resistance, and high stability. However, bitumen absorbency may be greater than conventional aggregates due to bitumen being absorbed into the vesicles in the slag. Such vesicles are not found in most conventional aggregate.

Surface treatment applications incorporating air-cooled blast furnace slag aggregate demonstrate good friction resistance, good resistance to stripping, and fair wear resistance. However, the resistance of air-cooled blast furnace slag to impact is not very high and the material can break down under heavy traffic conditions (TFHRC, 2001). Some of the engineering properties of air-cooled blast furnace slag that are of particular interest when it is used as an aggregate in asphalt concrete include gradation, compacted density, absorption, abrasion and freeze-thaw resistance.

Reid et al. (2001) reported that air-cooled blast furnace slag has been used satisfactorily as an unbound sub-base in Sweden. They also found no significant influence on the surrounding environment was encountered with this material. Figure 2.3 shows the use of blast furnace slags in Europe in 2000. It shows that 39.5% of the blast furnace slag produced in Europe is used in road construction (Motz, 2003).

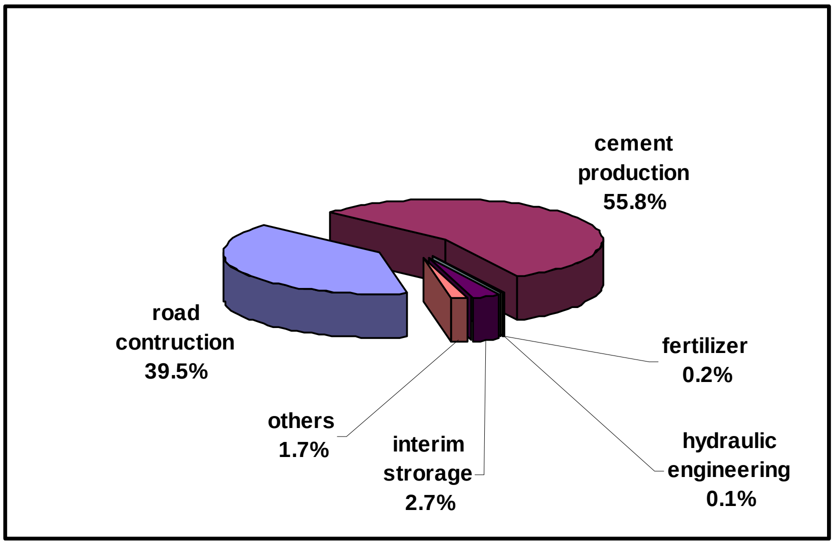



**Figure 2.3: The use of blastfurnace slag in Europe in 2000 (Motz, 2003)**

In the United States of America, air-cooled blast furnace slag has been used in asphaltic concrete aggregate, concrete aggregate, concrete products, fill mineral wool, railroad ballast and road bases. Figure 2.4 shows the use of air-cooled blast furnace slag in the US for the year 2000 (Kalyoncu, 2000).

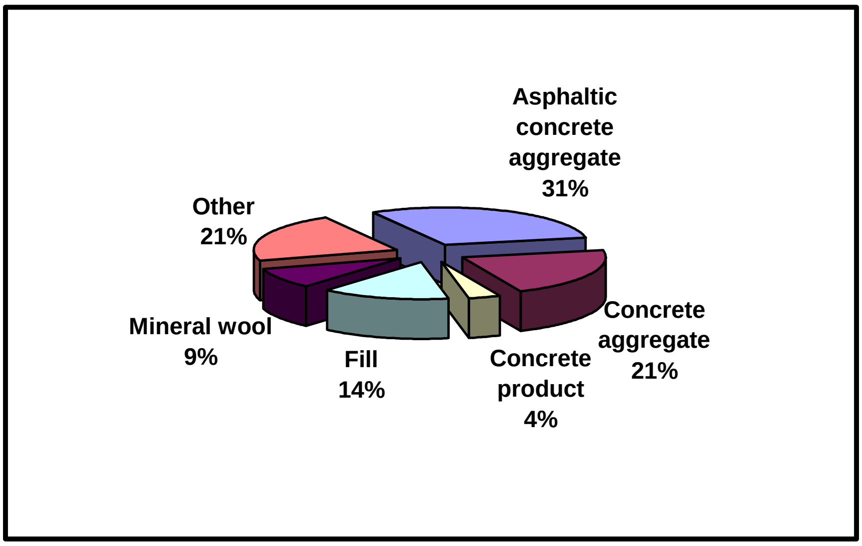



**Figure 2.4: Air-cooled blast furnace slag used in the United States of America in Year 2000 (Kalyoncu, 2000)**

### 2.4.4 Problems Arising from the Use of Slag in Road Construction

Although air-cooled blast furnace slag has been successfully used in many areas, there is still a need to evaluate factors that contribute to the lack of consistency in the physical properties (gradation, specific gravity, absorption and angularity) of the slag aggregate produced among individual slag producers. Besides that, it is also important to study its effect on performance problems such as flushing and ravelling in hot mix asphalt. In addition, formal quality control procedures should be instituted to monitor the quality of specific air-cooled blast furnace slag sources to assist in mitigating these problems.

The most likely form of instability in blast furnace slag is when a significant proportion of the sulphur oxidises to sulphate as a result of weathering. Chemical reactions may take place resulting in the formation of a sulphoaluminate hydrate phase, apparently similar to that taking place in sulphate attack on concrete. The result is volumetric expansion and disruption of the mass (Thomas Research, 2003). To counter this problem, weathering of blast furnace slag is essential before it can be used.

## 2.5 STEEL SLAG

### 2.5.1 Slag Production

Steel slag is a by-product of steel making. It is produced during the separation of the molten steel from impurities in furnaces. The slag occurs as a molten liquid melt and is a complex solution of silicates and oxides that are solidified upon cooling.

All steels are now made in integrated steel plants using a version of the basic oxygen process or in speciality steel plants using the electric arc furnace process. In the basic oxygen process, hot liquid blast furnace metal, scrap, and fluxes, which consist of lime (CaO) and dolomitic lime (CaO.MgO or “dolime”) are charged to a converter (furnace). A lance is lowered into the converter and high-pressure oxygen is injected. The oxygen combines with and removes the impurities in the charge. These impurities consist of carbon as gaseous carbon monoxide, and silicon, manganese, phosphorus and some iron as liquid oxides, which combine with lime and dolomite to form the steel slag. This type of slag is sometimes called basic oxygen steel slag (BOS). At the end of the refining operation the liquid steel is tapped (poured) into a ladle while the steel slag is retained in the vessel and subsequently tapped into a separate slag pot.

The properties of steel slag are very dependent on the grade of produced steel and can change significantly with each steel grade. Steel grade can be classified into three; high, medium and low grade. The manufacture of high-grade steel involves the removal, from the iron, of the excess quantity of carbon content by oxidation. This requires greater amounts of oxygen to reduce the carbon and high levels of lime and dolime for the removal of impurities, and so there is increased slag formation.

There are two types of steel slag from basic oxygen process, furnace slag and ladle slag. The furnace slag is produced during the primary stage of steel production. This is a major slag source of steel slag aggregate. Ladle slag is unsuitable for processing into aggregates and is usually sent to landfill for disposal.

Another steel-making furnace that can be used to produce steel is an electric arc furnace (EAF). In this furnace, slag is produced during the production of more specialist steels from the refining of scrap iron.

## 2.5.2 Slag Properties

### 2.5.2.1 Physical Properties

Steel slag aggregates are angular in shape and have a rough surface texture. They have higher values for specific activity, bulk density and polished stone value. On the other hand, they have moderate water absorption and low values for aggregate crushing value, impact and abrasion. Table 2.5 lists typical physical properties of steel slag.

**Table 2.5: Typical physical properties of steel slag (Lee 1974)**

| Property | Value |
|---|---|
| Aggregate crushing value (ACV) | 12 – 25 |
| Aggregate impact value (AIV) | 18 – 24 |
| Bulk density, kg/m$^3$ | 1600 – 1920 |
| Specific gravity | 3.1 – 3.5 |
| Water absorption (percent by mass) | 0.2 – 2 |
| Polished stone value (PSV) | 53 – 72 |
| Aggregate abrasion value (AAV) | 3 - 4 |

### 2.5.2.2 Chemical Properties

The chemical properties of steel slag are usually expressed in terms of simple oxides, which can be determined by x-ray fluorescence analysis. Table 2.6 lists a range of compounds present in steel slag from typical Basic Oxygen furnace (BOS) and from Electric Arc furnace slags (EAF). The mineralogical form of the slag is highly dependent on the rate of slag cooling in the steel-making process. The slag consists primarily of dicalcium silicate, tricalcium silicate, dicalcium ferrite, merwinite, calcium aluminate, calcium-magnesium iron oxide and some free lime and magnesia. The content of free lime and magnesia is the most important component for the utilisation of steel slags for civil engineering purposes with regard to their volume stability.

Steel slag is mildly alkaline with a solution pH generally in the range of 8 to 10. However, the pH of leachate from steel slag can exceed 11 which can be corrosive to some metals such as aluminium, if in direct contact with the slag.

**Table 2.6: Typical BOS and EAF slags chemical composition (Motz, and Geiseler 2001; Coventry et al., 1999)**

| Component | CaO | $SiO_2$ | $Al_2O_3$ | MgO | MnO | $P_2O_2$ | $Fe_{total}$ | $CaO_{free}$ |
|---|---|---|---|---|---|---|---|---|
| BOF slag | | | | | | | | |
| low MgO content | 45-55 | 12-18 | <3 | <3 | <5 | <2 | 14-20 | <10 |
| BOF slag | | | | | | | | |
| high MgO content | 42-50 | 12-15 | <3 | 5-8 | <5 | <2 | 15-20 | <10 |
| EAF slag | | | | | | | 1 | |
| low MgO content | 30-40 | 12-17 | 4-7 | 4-8 | <6 | <1.5 | 8-28 | <3 |
| EAF slag | | | | | | | | |
| High MgO content | 25-35 | 10-15 | 4-7 | 8-15 | <6 | <1.5 | 20-29 | <3 |
| Open hearth | 33-51 | 9-19 | 0.5-3 | 0.5-4 | 3-10 | - | 24-45 | - |

### 2.5.2.3 Mechanical Properties

Processed steel slag has favourable mechanical properties for aggregate use, including good abrasion resistance and high bearing strength. Table 2.7 lists some typical mechanical properties of steel slag.

**Table 2.7: Typical mechanical properties of steel slag (Noureldin and McDaniel, 1990)**

| Properties | Value |
|---|---|
| Los Angeles Value (ASTM C131), % | 20 - 25 |
| Sodium Sulfate Soundness Loss (ASTM C88%) | <12 |
| Angle of internal friction resistance (ϕ) | $40^o$ - $50^o$ |
| Hardness (Measured by Moh's scale of mineral hardness) | 6 - 7 |
| California Bearing Ratio (CBR) % max size 19 mm | Up to 30% |

### 2.5.3 The Use of Slag in Road Construction

Every year, nearly 12 million tons of steel slags are produced in Europe. However, 65% of the produced slags are used and the remaining 35% of these slags are dumped (Motz and Geiseler 2001). In 2000, 5.1 million tons of steel slags were used in the United States of America. Major uses are road construction, asphaltic concrete, Portland cement manufacturers and various concrete products. Table 2.8 shows the quantity of steel slag used in 2000 in the USA.

Experience has shown that steel slag can be used as granular base for roads in the above grades if properly selected, processed, aged and tested. Some of the important properties of steel slag which are very important for it to be used as an aggregate in granular bases are gradation, specific gravity, stability, durability, corrosivity, volumetric stability, drainage and tufa formation (TFHRC 2001).

**Table 2.8: Quantity of steel slag used in 2000 in the USA (Kalyoncu, 2000)**

| Use | Quantity (Million tons) |
|---|---|
| Asphaltic concrete aggregate | 0.9 |
| Fill | 1.0 |
| Railroad ballast | 0.4 |
| Road bases | 1.7 |
| Other | 1.1 |
| **Total** | **5.1** |

Steel slag can also be used in asphalt pavements. The slag is processed into a coarse or fine aggregate material for use in dense and open-graded hot mix asphalt concrete pavements and in cold mix or surface treatment applications (Turner-Fairbank Highway Research Center, 2001). Hunt and Boyle (2000), in their study on the use of steel slag in hot mix asphalt concrete, concluded that asphalt concrete can be produced and the pavement constructed readily when crushed steel slag is used as a proportion of the aggregate.

In the United Kingdom, the latest edition (May, 2001) of the Specification for Highway Works contains clauses permitting the use of steel slag in road construction such as general fill, capping, unbound sub-base, and bitumen bound layers (Highway Agency, 2001).

In Germany, at present, 93% of steel slags have been utilised, either in road construction or waterway construction. This country has 25 years practical experience of using steel slag on test roads and the following results may be achieved by using this experience (Motz and Geiseler 2001):

- The crushed and rough surface of processed steel slag aggregate mixtures provides a stronger bearing capacity directly after compaction and higher than when using other aggregates.
- There is no influence of heavy rain on the bearing capacity of unbound layers built with steel slag mixtures.
- The carbonatic solidification leads to an increase of bearing capacity.
- The aggregate mixtures are permanently stable if the requirements for the volume stability have been fulfilled.
- The asphaltic surface layers remain permanently plain even under heavy traffic.
- The resistance to polishing of asphaltic surface layers remains at a high level over a long term period
- Roads built with steel slags as unbound or bituminous bound aggregate do not influence the environment by leaching.

### 2.5.4 Problems that Arise from the Use of Slag in Road Construction

The minerals content of steel slag is highly dependent on the rate of slag cooling in the steel making process. Free lime (CaO) and free periclase (MgO), which are not completely consumed, still exist in the slag. Both free lime and free periclase in the presence of water in a humid environment will ultimately hydrate to portlandite [$Ca(OH)_2$] and brucite [$Mg(OH)_2$]. The formation of these hydrates is largely responsible for massive expansion. This is one of the reasons why steel slag aggregates may not be suitable for use in Portland cement concrete or as compacted fill beneath a concrete slab. Verhasselt and Choquet (1989) have also discussed in detail the use of steel slag as unbound aggregate in road construction. They also agreed that the free calcium oxide (CaO) contained in steel slag constitutes a factor of instability by its transformation into calcium hydroxide where its volume is doubled. However, research on the long term behaviour of hydraulic construction made from steel slags regarding the volume stability, the effects of leaching and the effects on the river fauna in Germany has shown that the properties of steel slag armourstones are comparable to those of established natural stones like basalt (Motz and Geiseler 2001).

Tufa-like formation, resulting from the exposure of steel slag aggregate to both water and atmosphere, is another problem when using steel slag in road construction. Tufa is a white powdery precipitate that consists primarily of calcium carbonate ($CaCO_3$). When free lime in steel slag combines with water, it will produce calcium hydroxide [$Ca(OH)_2$] solution. Upon exposure to atmospheric carbon dioxide, calcite ($CaCO_3$) is precipitated in the form of surficial tufa and powdery sediment on surface water. Tufa precipitates can clog up the exits of highway underdrain systems (TFHRC, 2001)

At present there are no specifications or standards relating to the use of steel slag in road construction either in the European countries or in the United States. Thus steel slag should be used with caution and strict specifications should be developed for its use in road construction. In this respect, Verhasselt and Choquet (1989) have put some recommendations for the safe use of steel slags in unbound crushed stone bases and sub-bases as follows:

- Steel slag should not have a free lime (CaO) content of more than 4.5% at the time of production.
- Before use the slag should be weathered at least for one year. This is to allow the phase change expansion to take place.
- The maximum particle size should be restricted to 20-25 mm.
- The interposition of a sand layer between steel slags and the overlaying layers makes it possible to greatly reduce or even eliminate the risks of localised surface deterioration due to expansion. Its thickness, with a minimum of about 15-20 cm, should be a function of the thickness of the layer of steel slag.
- Before use, the volumetric stability should be checked by a volumetric swelling test.

## 2.6 COPPER SLAG AND NICKEL SLAG

### 2.6.1 Slag Production

In the early days of copper mining, high-grade oxidized ores were smelted directly in blast furnaces. However, the copper content of the slag was quite high while the produced copper was impure and required further refining. Eventually, the practice of smelting in blast furnaces was abandoned. Currently, the production of copper slag and also nickel slag do not differ much. Both of them are produced by three operations (see Figure 2.5):

- Roasting, in which sulphur in the ore is eliminated as sulphur dioxide ($SO_2$);
- Smelting, in which the roasted product is melted in a siliceous flux and the metal is reduced; and
- Converting, where the melt is desulfurized with lime flux, iron ore, or a basic slag and then oxygen-lanced to remove other impurities.

## 2.6.2 Slag Properties

### 2.6.2.1 Physical Properties

Air-cooled copper slag has a dull black colour and a glassy appearance. Granulated copper slag is more vesicular and porous and therefore has lower specific gravity and higher absorption than air-cooled cooper slag. In general, the specific gravity of copper slag will vary with its iron content. The unit weight of copper slag is somewhat higher than that of conventional aggregate. The absorption of the material is typically very low. Table 2.9 presents the typical physical properties of copper slag.

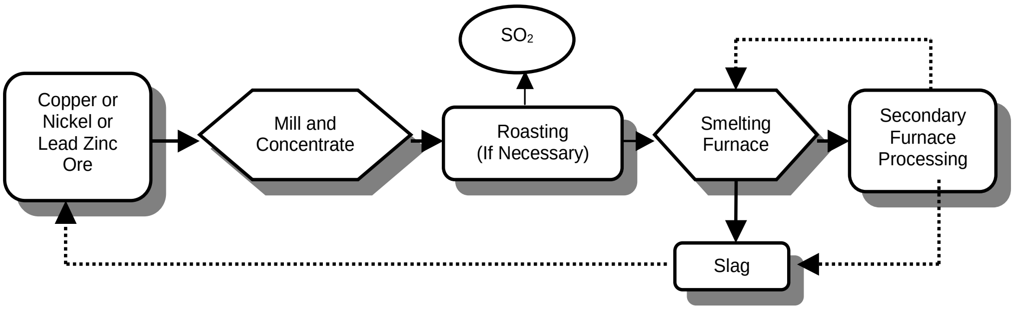



**Figure 2.5: General manufacturing process for copper, nickel and lead-zinc slag production (after TFHRC 2001)**

**Table 2.9: Typical physical properties of copper slag (Mineral Research & Recovery Inc. 2001)**

| Properties | Value |
|---|---|
| Specific gravity | 3.4 – 3.6 |
| Bulk density, kg/m$^3$ | 1762 - 1922 |
| Water absorption (% by mass) | 0.13 |

### 2.6.2.2 Chemical Properties

Copper slag is essentially ferrous silicate. Table 2.10 lists typical chemical compositions of copper slag.

**Table 2.10: Typical chemical properties of Copper slag (Mineral Research & Recovery Inc. 2001)**

| Properties | Value (%) |
|---|---|
| Calcium oxide (CaO) | 5.94 |
| Silicon dioxide ($SiO_2$) | 38.92 |
| Aluminium oxide ($Al_2O_3$) | 9.45 |
| Magnesium oxide (MgO) | 0.13 |
| Iron (FeO or $Fe_2O_3$) | 32.11 |
| Manganese oxide (MnO) | 0.13 |
| Sulfur (S) | 2.27 |
| Sodium oxide ($Na_2O$) | 0.13 |
| Potassium oxide ($K_2O$) | 0.17 |
| Free silica | <0.1 |

### 2.6.3 The Use of Slag in Road Construction

At an early stage the use of Copper slag and Nickel slag aggregate as a granular base was took place in the mining road (Emery, 1982) where this slag was produced. It has demonstrated satisfactory performance in what are generally considered to be very severe traffic and operating conditions. Cooper and Nickel slags have been used as granular base and embankment materials, aggregate substitutes in hot mix asphalt, mine backfill material, railway ballast material, grit ballast abrasives, roofing granule material, and in the manufacture of blended cements (Emery, 1982).

Copper slag is considered to be a conventional aggregate in Michigan, and covered by state specifications for granular base. It is used as a granular base because of the high stability and good drainage characteristics (Das et *al.*, 1993; Feasby, 1975) as well as good resistance to mechanical degradation due to freeze-thaw exposure (Feasby, 1975 and TFHRC, 2001). However, in El Paso (Texas), the use of copper slags with asphaltic and Portland cement concrete has been discontinued as a result of environmental concerns. The discontinuation has nothing to due with the materials' workability or strengths. There are very limited toxicity testing data which indicate the quality of the leachate from specific copper slag. In Canada approximately 45% of available copper slag is used in base construction, rail road ballast and engineered fill.

Copper slag has also been used for cement material, sand blasting and reclamation. It is also possible to use a copper slag as a concrete aggregate (Ayano *et al.* 2000) although there are some problems which still require solutions from researchers.

### 2.6.4 Problems that Arise from the Use of Slag in Road Construction

The most pressing issue that needs to be resolved when using the slag is the environmental suitability of copper slag in road construction application. Materials from each source must be assessed for heavy metals content and leachability.

There also are some problems in using copper slag as a concrete aggregate. One of the problems is excess bleeding attributed to the glassy surface of the copper slag. Another problem is the delay of setting time of concrete with copper slag where in some cases it takes more than one week. However the delay of setting time does not have a negative influence on the durability of concrete with copper slag (Ayano *et al*, 2000).

## 2.7 PHOSPHORUS SLAG

### 2.7.1 Slag Production

As for the other slags, phosphorus slag is a by-product of the elemental phosphorus refining process. The phosphorus is separated from phosphate-bearing rock in an electrical arc furnace, with silica and carbon added as flux materials to remove impurities during the slagging process (see Figure 2.6). Iron, which is added to the furnace charge, combines with phosphorus to form ferrophosphorus, which can be tapped off. The slag, which remains after removal of elemental phosphorus and/or ferrophosphorus, is also tapped off.

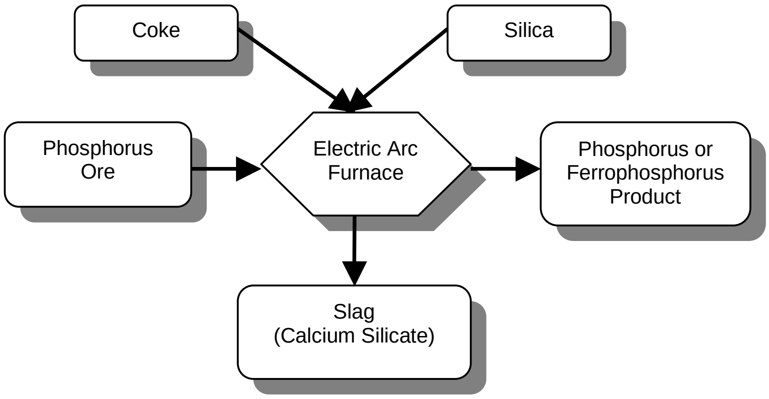



**Figure 2.6: General manufacturing process for phosphorus slag production**

## 2.7.2 Slag Properties

### 2.7.2.1 Physical Properties

There are two types of phosphorus slag, air-cooled phosphorus slag and granulated phosphorus slag. Air-cooled phosphorus slag tends to be black to dark grey, vitreous and of irregular shape while granulated phosphorus slag is made up of regular shape and angular particles. Granulated phosphorus slag is more porous than air-cooled slag and has lower specific gravity and higher water absorption. Table 2.11 presents the typical physical properties of phosphorus slag.

**Table 2.11: Typical physical properties of phosphorus slag (Lee 1974)**

| Properties | Value |
|---|---|
| Specific Gravity (oven dried) | 2.7 |
| Bulk Density * (kg/m$^3$) | 1490 |
| Aggregate Crushing Value | 32 |
| Aggregate Abrasion Value | 17 |
| Water Absorption (percent by mass) | 1.1 |
| Polished Stone Value | 44 |

* 14 mm single size

#### 2.7.2.2 Chemical Properties

There is very limited data available on chemical properties of phosphorus slag due to limited research on the slag (see Table 2.12).

**Table 2.12 Chemical properties of phosphorus slag (Lee 1974)**

| Properties | Value (%) |
|---|---|
| Lime (CaO) | 48 |
| Silica ($SiO_2$) | 41 |
| Alumina ($Al_2O_3$) | 3 |

### 2.7.3 The Use of Slag in Road Construction

Phosphorus slag has been used as an aggregate substitute in hot mix asphalt, as a lightweight masonry aggregate, and as a cement kiln feed. In the United Kingdom, the slag was produced and used mainly in the construction and maintenance of roads (Lee 1974).

The shape and hardness of the phosphorus slag coarse aggregate leads to a good initial mechanical interlock and the grading of the total mixture provides a dense matrix which, subject to a suitable moisture content and good compaction, will ultimately harden to maximum strength and minimum voids (Walsh 1999).

Kent County Council, in the United Kingdom, is the only county council that has carried out a very extensive trial using blended phosphorus slag and blast furnace slag material as both road-base and sub-base. In 1987, a full-scale trial was laid on a major by-pass near Pembury. The mix was used to replace both sub-base and road base. Extensive monitoring has shown the material has demonstrated excellent performance above expectations with good structural strength and no reflective cracking. A blended phosphorus slag and blast furnace slag was again used to build Hale Street by-pass in

1997 and Wainscott by-pass in 1997/98. In using phosphorus slag, Walsh (1999) has concluded that:

- Phosphoric slag with its angular aggregate structure could be trafficked immediately.
- The materials are very dependent upon density for strength.

### 2.7.4 Problems that Arise from the Use of the Slag in Road Construction

As a non-ferrous slag, phosphorus slag also has the same problem as faced by copper slag, the environmental suitability of the slag in road construction applications. Beside assessment of heavy metals content and leachability properties, radioactivity concerns should also be investigated for phosphorus slag. Furthermore, there is a need to establish standard methods and clear guidelines to assess the suitability of phosphorus slag that may be in contact with groundwater or watercourses.

As phosphoric slag is very dependent upon density for strength, this makes it difficult to determine whether the material or its compaction is the cause of any low values. It is important to standardise vibratory compaction to be used by careful reference to an appropriate standard (Walsh 1999).

## 2.8 LEAD, LEAD-ZINC AND ZINC SLAG

### 2.8.1 Slag Production

The production of lead, lead-zinc and zinc slags are similar to copper and nickel slag production, involving roasting, smelting and converting. Figure 2.2 shows the general view of the process of the slag's production.

### 2.8.2 Slag Properties

The zinc slag composition varies according to the particular process used in refining.

Distillation is required when the highest purity slag is required. The slag grade is characterized by a high content of ferrous iron and silica.

### 2.8.3 The Use of Slag in Road Construction

Some zinc slags have reportedly been used in the manufacture of ceramic tiles and as an aggregate substitute in hot mix asphalt. Lead slag has been used in a cement based stabilization/solidification process for the manufacture of concrete blocks (Cioffi *et al*. 2000).

### 2.8.4 Problems that Arise from the Use of Slag in Road Construction

Few studies have been done on using of zinc slag in road construction.

## 2.9 TIN SLAG

### 2.9.1 Slag Production

Tin slag is by-product of tin smelting. In tin processing, cassiterite ($SnO_2$) is reduced to tin metal by heating with carbon at 1200$^o$C to 1300$^o$C. Reverberatory furnaces are used to smelt tin concentrate and to re-smelt slag (the waste left after the ore has been smelted) for additional tin recovery. A furnace charge consists of cassiterite, a carbon-reducing agent, and limestone and silica fluxes. Smelting takes 10 to 12 hours. The molten batch is tapped into a settler from which the slag overflows. After tapping from the furnace the molten slag is granulated in water and solidifies into glass-like material. Tin slag contains lime, iron, silica and small amount of heavy metals. The slag also contains naturally occurring radioactive materials such as uranium and thorium and their decay products (MSC, 2001).

### 2.9.2 Slag Properties

Tin slag is a fused glassy like material containing lime, iron and silica and small amounts of heavy metals.

#### 2.9.2.1 Physical Properties

At present there is little or no literature explaining the properties of tin slag

#### 2.9.2.2 Chemical properties

The typical chemical composition of tin slag is presented in Table 2.13.

**Table 2.13 Typical chemical composition of tin slag (MSC, 2001)**

| Properties | Value (%) |
|---|---|
| Calcium oxide (CaO) | 16 - 20 |
| Silicon dioxide ($SiO_2$) | 28 – 32 |
| Aluminium oxide ($Al_2O_3$) | 10 - 13 |
| Magnesium oxide (MgO) | 2 - 4 |
| Iron (FeO or Fe2O3) | 13 – 18 |
| Wolfram Oxide (WO) | 2.0 |
| Arsenic (As) | <0.01 |
| Lead (Pb) | <0.01 |
| Cadmium (Cd) | <0.0001 |
| Titanium Dioxide ($TiO_2$) | 4 – 5 |
| Zinc (Zn) | <0.3 |
| Tin (Sn) | <1.4 |

### 2.9.3 The Use of the Slag in Road Construction

Little is known of the potential uses of tin slag in road construction. Tin slag has never reportedly been used as an aggregate substitute. However some of the slag is used as a shot blasting material. No research has been reported on using tin slag in road pavements.

### 2.9.4 Problems that Arise from the Use of Slag in Road Construction

Tin slag has never reportedly been used in road construction anywhere in the world. However, as a non-ferrous slag, tin slag also has the same problem facing other non-ferrous slags, the environmental suitability of the slag in road construction applications. Beside assessment of heavy metal content and leachability properties, radioactivity concerns should also be investigated for this slag. Furthermore, there is a need to establish standard methods and clear guidelines to assess the suitability of the slag that may be in contact with groundwater or watercourses.

## 2.10 DISCUSSION

Much research has been performed on ferrous slags. Blastfurnace slag has been successfully used in road construction. Another slag, steel slag, has very limited use in road construction although much research on it is being done. Blastfurnace slag has excellent resistance to binder stripping in asphalt mixtures caused by the combined action of water and traffic. It has relatively low density, providing good surface coverage with the associated economic benefit. Steel slag from the basic oxygen process is particularly well suited to the new generation of 'quiet' thin surfacing asphalts because of its high abrasion resistance. The aggregate shape that contributes to surface texture gives high-speed skidding resistance. The most likely problem with steel slag is its expansion properties which can be solved by allowing for proper weathering. Steel slag is particularly suitable as an aggregate for surface dressing in asphalts and macadams or for asphalt chippings on heavily trafficked roads.

On the other hand the use of non-ferrous slags in road pavements is very limited. Only phosphorus and copper slags are reported as having been used in road construction. Research on these materials is also not very intensive. There are some problems which need to be resolved by researchers and regulatory authorities, which currently prevent the use of slags in road construction. The following are some of these problems;

- There are no standard procedures, guidelines or specifications on using slag as a secondary material in road construction. This is most critical for non-ferrous slags.
- There is no extensive database on using slag in road construction.
- No comprehensive research has been done on the radiological impact of naturally occurring radioactive material contained in the slag if used in road construction.
- Government policy which does not always encourage recycling.

Table 2.14 shows an example of the possible uses of slags in road construction and the problems faced which need to be settled by researchers.

**Table 2.14: Summary the use of slags in road construction and problems faced**

| Type of Slag | Use in Road Construction | Problem Faced |
|---|---|---|
| Blast furnace slag | Fill, embankment, capping, unbound sub-base, bitumen bound layers, bitumen bound sub-base, cement bound roadbase and PQ concrete | 'Falling slag' |
| Steel Slag | Unbound sub-base, bitumen bound layer, cement bound sub-base, Hot mix asphalt | Expansion<br>Tufa formation<br>Gradation |
| Copper slag | Hot mix asphalt, | Suitability as alternative aggregate<br>Environmental suitability |
| Phosphorus slag | Roadbase and sub-base | Suitability as alternative aggregate<br>Environmental suitability<br>Radioactivity |
| Zinc, Lead Zinc and Lead Slag | Hot mix asphalt | Suitability as alternative aggregate<br>Environmental suitability |
| Tin Slag | Never been reported | Suitability as alternative aggregate<br>Environmental suitability<br>Radioactivity |

Chapter

3

# BASIC PHYSICAL AND MECHANICAL PROPERTIES OF TIN SLAG

## 3.1 INTRODUCTION

One of the main objectives of this research is to assess the possibility of using tin slag in road construction. To satisfy this objective, and since there are no specifications especially related to tin slag in road construction, the tin slag has to be classified and must meet specification requirements in the same way that a classification system and specifications have been drawn up for natural aggregates which are currently in use. In general, before tin slag can be adopted for use as an alternative aggregate source in road construction, it should be subjected to a series of tests in the laboratory and field. Examination of physical and mechanical properties of tin slag should be performed so as to determine the suitability, reliability and durability of this material.

In addition to tin slag, there is a wide range of slags available which can be used as alternative materials in various fields of road construction as discussed in Chapter 2. Some of the slags have been used in road construction for many years and are very well accepted such as steel slag and blast furnace slag, while others are still only used on a trial basis, such as non-ferrous slags. There has been very little study carried out on using non-ferrous slag, especially tin slag, in road constructions. Some of the reasons are:

- The production of non-ferrous slags is not as big as other waste materials such as ferrous slags or power station ashes. Therefore researchers have tended to focus their investigations on the larger volume waste materials.
- Most of the tin slag is produced in third world countries. Most of these countries

have relatively abundant, readily available and low cost high quality primary aggregates. Therefore, conventional aggregates are preferred rather than recycled materials.

- In general, there are no clear policies to promote the use of slags as a recycled material.

As a result, most of these slags become waste. Some of these materials have been stored on producer premises for a long time.

## 3.2 AGGREGATES ROLE IN PAVEMENT PERFORMANCE

In both flexible and rigid pavement, poor performance of aggregate materials contributes to reduced life and costly maintenance. Failures in flexible pavements such as rutting, fatigue cracking, longitudinal cracking, depressions, corrugations and frost heave result from poor performance of granular materials, while in rigid pavements, poor performance of granular bases contributes to pumping, faulting, cracking and corner breaks. Similarly, the performance of unbound bases and sub-bases in pavement systems depends on the properties of the aggregate materials.

Harvey and Monismith (1993) demonstrated the important influence of aggregate grading on the stiffness of asphalt mixtures (which will influence their load spreading ability and tends to attract stress to themselves in turn affecting the rutting response of lower layers and the fatigue response of the asphalt material itself). Roberts et al. (1996) found that it is very important to optimise the grading so as to ensure adequate interlock between the mineral aggregate particles. The authors indicated that gradation in hot mix asphalts helps to determine almost every important property including stiffness, stability, durability, permeability, workability, fatigue resistance, frictional resistance and resistance to moisture damage. Reasonable gradation with adequate voids in the mineral aggregate provides improved resistance to degradation during construction and under traffic and improved resistance to fatigue cracking when used in thick pavements.

Aggregate shape plays a very important role on the performance of a bituminous mix. Hard, smooth, rounded aggregate gives a very workable mix but one that has low stability. Very flaky and elongated particles have a larger specific surface and therefore a higher bitumen demand and they are very difficult to compact properly. Angular, cuboidal aggregates with a medium texture give optimum performance (Broadhead and Hills, 1990).

Moisture content has been recognized by many researchers as having the potential to adversely affect the strength and useful life of pavements. The presence of water in unbound aggregates might reduce the inter-particle friction where it may lubricate the faces of the aggregate particles. This reduces the resilient modulus and increases the deformation in the aggregate assemblage (Tien et al, 1998). Moisture content also affects the shear strength of granular materials, so that as the moisture content deviates from its optimum value, the cohesion is decreased. The above effects would eventually lead to severe rutting and/or cracking of the surface, displacement and shear failure of the base course and pavement failure. For asphalt mixtures, the presence of water affects the adhesion between the mineral aggregates and bitumen.

In general, environmental evaluation of primary aggregate is not very important because the aggregate itself comes from the natural environment. For industrial by-products or wastes such as slags or pulverised fly ash (pfa), it is necessary to conduct environmental tests before such materials can be used for road construction. The primary objective of performing environmental tests on slag aggregate used in road construction is to enable an assessment of potential or actual impacts of this material on the environment or human health. The results of the tests may be used to develop a rationale for the setting of criteria for material quality. It also may be used for quality control purposes to ensure that materials to be used in road constructions comply with regulations and criteria imposed by the authority (ies).

Secondary aggregates may contain varying concentrations of contaminants, such as heavy metals, which could potentially cause pollution of surface water or ground water. There may also be concerns with dust and fumes during the placing of these aggregates, but these aspects generally can be controlled by appropriate handling, restrictions and methods. The contamination of surface water and underground water is of greater concern.

**N**aturally **O**ccurring **R**adioactive **M**aterial (NORM) can originate from many different types of industrial production processes and is found, on occasion, in slags, fly ash and by-product gypsum. The recycling of these by-products, which contain NORM residues as pavement material in road construction and landscape construction projects is one of the main recycling options, especially for NORM residues generated from coal firing plants and from the primary (blast furnace) iron and steel manufacturing industry.

Although the slag is successfully used in road construction, no study has been performed on the effect of naturally occurring radioactive materials on humans or on the environment. Exposure pathways and parameters of using slags in road construction should be established. The radiological consequences during construction, repair and demolition work should be taken into account. Chapter 7 discusses in some detail environmental evaluation of using the tin slag in road construction.

## 3.3 MATERIALS ASSESSMENT

An evaluation of the quality of aggregates to be used in road constructions, in terms of both physical and mechanical properties, is very important. Adequate performance of the aggregates in a road environment is essential. A good road making aggregate should be durable, dense, angular, non-flaky, and preferably medium textured and resistant to crushing, impact, abrasion, polishing and stripping (Broadhead and Hill, 1990). There are many standards that can be used to evaluate the performance of the aggregates. On the other hand, there is no specific standard that can be used to assess the performance of the slag to be used in road pavements. With this regard, the slag should be assessed on the same basis as natural aggregates with appropriate allowance being made for inherent differences. As far as possible slags should meet the specification requirements and be classified in the same way that classification systems and specifications have been drawn up for road-making materials already in use.

### 3.3.1 Geometrical Properties of Aggregate

#### 3.3.1.1 Particle Size Distribution - Gradation

The determination of particle size distribution is performed in accordance with BS EN 933-1:1997 which is carried out by sieve analysis, in which the sample is passed through a stack of standard sieves of diminishing mesh size. The distribution of particle sizes is expressed as percent of total weight.

#### 3.3.1.2 Particle Shape – Flakiness

An aggregate particle is said to be flaky when it has one dimension significantly less than its other two dimensions. This shape factor is numerically standardized in the flakiness index test of BS EN 933-3:1997 by defining a flaky particle as one whose smallest dimension is less than 0.6 times the arithmetic mean of the aperture sizes of the two sieves that define the size fraction of the particle. A special flaky sieve with slots is used to determine the proportions of flaky particles.

The Flakiness Index is determined by the following procedure (BS EN 933-3:1997):

- First, using test sieves, the sample is separated into various particles size fractions. Each of the particle size fractions is then sieved using bar sieves which have parallel slots. Weigh and discard all particles passing the 4mm sieve and retained on 80mm sieve.
- The overall flakiness index is calculated as the total mass of particles passing the bar

sieves expressed as a percentage of the total dry mass of particles tested.

- If required, the flakiness index of each particle size fraction is calculated as the mass of particles passing the corresponding bar sieve, expressed as a percentage by mass of that particle size fraction.

### 3.3.1.3 Particle Shape – Elongation Index

The elongation index test is not greatly used. Elongated particles, like flaky particles, can break under compaction. An aggregate is elongated when it has one dimension significantly greater than its other two dimensions. BS 812-105.2:1990 defines an elongated particle as one whose greatest dimension is more than 1.8 times the arithmetic mean of the aperture sizes of the two test sieves that limit the size fraction of the particle (BSI, 1990). However, the test is not applicable to material passing a 6.30mm test sieve or retained on a 50.0mm test sieve.

BS 812-105.2:1990 provides a method of determining the elongation index of an aggregate sample. A summary of the method is as follows:

- The aggregate retained on 50mm and passing 6.3mm test sieves is discarded.
- The aggregates are sieved into the size fractions and the fractions of less than 5% by mass of the test portion are discarded.
- Test each remaining fraction separately by gauging by hand each particle against its appropriate length gauge which effectively checks for the 1.8 factor described above.

The elongation index can be determined using the equation 3.1.

$$\textit{Elongation index } (I_E) = \frac{\textit{Total mass of particles refused by gauges}}{\textit{Total mass of particles tested}} \times 100 \qquad \ldots(3.1)$$

### 3.3.1.4 Particle Shape – Shape Index

In natural aggregate, the shape is a function of the petrology of the rock and the quarrying and aggregates production process. However, this is not the case for alternative aggregates. The shape of recycled materials or waste by-products is dependent on the process of their production. Shape index is determined according to the BS EN 933-4 (BSI, 2000). The shape index is determined using a particle slide gauge as shown in Figure 3.1

In principle, particles in a sample of coarse aggregate are classified on the basis of the ratio of their length L, to thickness E using a particle slide gauge. The shape index is then calculated as the mass of particles with a ratio of dimensions L/E more than three expressed as a percentage of the total dry mass of particle tested. The test is applicable to particles passing 63mm and retained on 4mm test sieve.

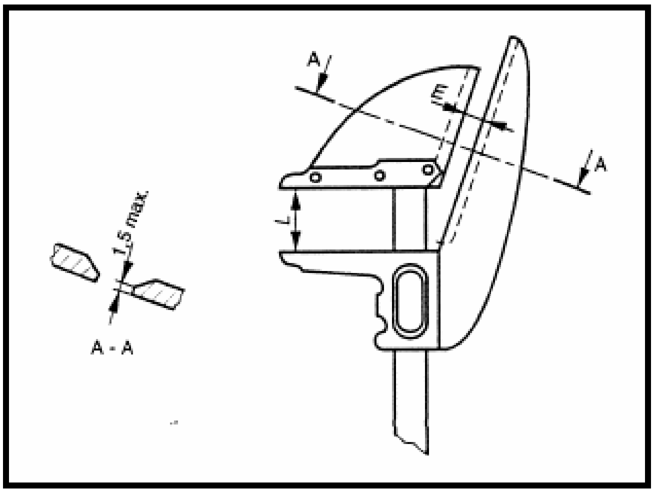



**Figure 3.1: Particle slide gauge – dimensions in millimeter (BS EN 933-4)**

### 3.3.2 Toughness and Abrasion Resistance

There are several test methods to determine toughness and abrasion resistance of aggregate. These include the micro-Deval abrasion test, Los Angeles test, gyratory compaction, aggregate impact value (AIV) test, aggregate crushing value (ACV) test and ten per cent fines value (TFV) test. However, only AIV, ACV and TFV are discussed here.

### 3.3.2.1 Aggregate Crushing Value (*ACV*) Test

The ACV gives a relative measure of resistance of an aggregate to crushing under a gradually applied compressive load. In principle the test specimen is compacted in a standardized manner into a steel cylinder fitted with a freely moving plunger. The specimen is then subjected to a standard loading regime applied through the plunger. This action crushes the aggregate to a degree which is dependent on the crushing resistance of the material. This degree is assessed by a sieving test on the crushed specimen and is taken as a measure of the aggregate crushing value (*ACV*). The test is normally performed on the aggregate fraction passing 14 mm and retained on 10 mm (BSI, 1990). However, the BS includes a set of tables that allows the ACV test to be carried out on smaller aggregate sizes. Details of the procedure of this test method can be found in BS 812-110:1990.

The *ACV* value is expressed as a percentage, to the first decimal place, of the mass of fines formed to the total mass of the test specimen from the following equation:

$$AVC(\%) = \frac{M_2 \times 100}{M_1} \qquad \ldots(3.2)$$

where:

$M_1$ is the mass of the test specimen (in g).

$M_2$ is the mass of the material passing the 2.36 mm test sieve (in g)

Work on the *ACV* test showed that the smaller aggregate size, the stronger the aggregate (Markwick and Shergold, 1945). They also found that the range of the test values varied with type of aggregate. The values 10 to 20% were for hard igneous rock, quartzite, flints and sandstone; 20 to 30% for limestone and greater than 25% for slag. Under the current standards, there is no requirement by the Highways Agency for aggregate to posses a particular minimum value of ACV. However, a value of less than 25% has typically been regarded as a suitable requirement for natural aggregates (Woodward, 2000).

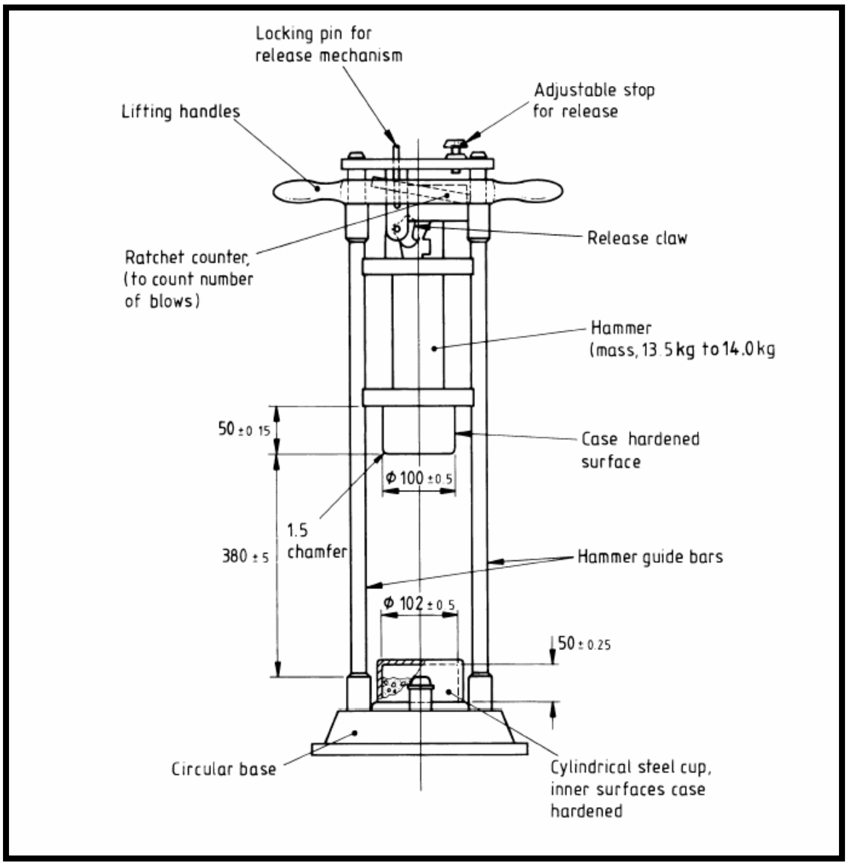



**Figure 3.2: AIV equipment -all dimensions are in millimeters (BS 812-112)**

### 3.3.2.2 Aggregate Impact Value (*AIV*) Test

The *AIV* test gives a relative measure of the resistance of an aggregate to sudden shock or impact (Figure 3.2). It provides a measure of resistance to granulation. The principle of this test is explained in BS 812-12 (1990). A test specimen is compacted, in a standardized manner, into an open steel cup. The specimen is then subjected to a number of standard impacts from a dropping weight. This action breaks the aggregate to a degree which is dependent on the impact resistance of the material. This degree is assessed by a sieving test on the impacted specimen and is taken as the aggregate impact value (AIV). A lower numerical value indicates a tougher, more wear-resistant aggregate. The *AIV* test has good reproducibility and is cheap to operate. However, the *AIV* does not separate suitable and unsuitable aggregate (Bullas and West, 1991). So a combination of physical tests can be used to assess aggregate durability (Fookes, Gourley and Ohikere, 1988). Under SHW (Specifications for Highway Works) specifications, the maximum acceptable value of *AIV* is 30 for aggregates in asphalt mixture.

### 3.3.2.3 Ten Per Cent Fines Value (*TFV*) Test

This test is frequently used as a main specification requirement for aggregate strength. Under the SHW specification, the aggregate for use in asphalt must have a *TFV* dry value greater than 140 kN whilst unbound sub-base aggregates must have a soaked value greater than 50 kN.

The *TFV* test uses the same apparatus as the *ACV* test. Also, there is little difference between both in terms of methodology. However, the TFV differs in that the load applied during testing is not constant but varies according to the expected strength of the aggregate.

In *TFV*, a test specimen is compacted in a standardized manner into a steel cylinder fitted with a freely moving plunger. The specimen is then subjected to a load applied through the plunger. This action crushes the aggregate to a degree which is dependent on the crushing resistance of the material. The degree of crushing is assessed by carrying out a sieving test on the crushed specimen. The procedure is repeated with various loads to determine the maximum force which generates 10% of material finer than the 2.36mm sieve size. This force is taken as the ten per cent fines value (*TFV*). There are two types of *TFV* test, namely TFV test in a dry condition and TFV test in a soaked condition. However, both tests are performed using a test portion passing on 14mm and retained on 10mm sieve size. Details of this method can be found in BS 812 – 111:1990 (BSI, 1990)

The force F (in kN) required to produce 10% of fines for a test specimen, with the percentage of material passing in the range 7.5% to 12.5%, can be calculated from the following expression:

$$F = \frac{14f}{(m+4)} \qquad \ldots(3.3)$$

where

$f$ is the maximum force (in kN)

$m$ is the percentage of material passing the 2.36mm test sieve at the maximum force

### 3.3.3 Moisture Content

Oven drying provides a measure of the total free water present in a test portion of aggregate. The water can be from the surface of the aggregate and from water accessible pores within the aggregate particles. The water content of aggregate can be determined according to the BS EN 1097-5 (1999). A test portion of the sample is weighed and then placed in a ventilated drying oven at a temperature of (110 ± 5) $^{o}$C until a constant mass is achieved. To ascertain that a constant mass has been achieved, the dry sample is weighed at several time intervals until there is no more reduction in mass with further drying time. The water content is determined as the difference in the mass between the wet and the dry mass. Water content ($\omega$) is expressed as a percentage of dry mass of the test portion. The calculation of water content, $\omega$, is in accordance with the following equation:

$$\omega = \frac{M_1 - M_3}{M_3} \times 100 \qquad \ldots(3.4)$$

Where:

$M_1$ is the mass of the test portion, in grams

$M_3$ is the constant mass of the dried test portion, in grams

### 3.3.4 Particle Density and Water Absorption

Particle density is determined from the ratio of mass of a material to its volume. The test portion is weighed in the saturated and surface dried condition and again in the oven dried condition. Depending on the aggregate size, volume is determined from the mass of the water displaced, either by measurement of the mass of a submerged specimen using a wire-basket method or by using a pyknometer. If the size of aggregate is between 31.5mm and 63mm, the wire-basket method is used and if the size of aggregate is between 0.063mm and 31.5mm, the pyknometer method is used.

Most of the natural aggregates have particle densities between 2.6 and 2.7 although values of 2.4 to 3.0 may be encountered. Alternative materials can be much higher (many slags) or lower (e.g. tyre crumb). Water absorption, calculated by comparing the saturated and dried weights, can be used as a crude indicator of aggregate durability as it indicates the amount of water available to freezing and thawing. High absorption is often an indicator of unsound aggregate.

### 3.3.5 Environmental Stability or Weathering Properties of Aggregates

#### 3.3.5.1 Magnesium Sulphate Soundness Test

The magnesium sulphate test is used to estimate the resistance of aggregate to weathering. Weathering is described as disintegration when aggregate is physically degraded into small particles due to the influence of the atmosphere and hydrosphere such as wetting and drying, freezing and thawing and thermal cycling at elevated temperature (Broadhead and Hills, 1990). In other words, the magnesium sulphate test is a measure of the durability of an aggregate in service.

Overall, researchers have a mixed view regarding the performance of the magnesium sulphate test, for example, Paul reported the test had good correlation with the performance of the aggregates, while Garrity and Kriege reported poor correlation (see

Kandhal and Parker, 1998). Later studies by Gandhi and Lytton (1984), Hasan, et al. (1991), Rogers et al. (1991) and Senior and Rogers (1991) also reported mixed reviews for performance predictions based on this test. However, reproducibility of this test is believed to be fair and poor for coarse and fine aggregates respectively (Kandhal and Parker, 1998).

In the BS 812-121 *Testing Aggregate – Method for determination of soundness* (BSI, 1989), a test portion of specimen in the size range 10 mm to 14 mm placed in a perforated container, is immersed in a saturated solution of magnesium sulphate for 17 hours, drained for 2 hours, followed by oven drying at (110 ± 5) $^{o}$C for 24 hours. The complete immersion and drying cycle is repeated five times. This subjects the laboratory aggregate sample to the disruptive effects of repeated crystallization and rehydration of magnesium sulphate within the pores of the aggregate. The degradation arising from the disruptive effects is measured by the extent to which material finer than 10 mm in particle size is produced.

The magnesium sulphate value (*MS*) as a percentage by mass can be calculated using the following equation:

$$MS(\%) = \frac{(M_1 - M_2)}{M_1} \times 100 \qquad \ldots(3.5)$$

Where:

$M_1$ is the initial mass of the test specimen

$M_2$ is the final mass of aggregate retained on the 10mm sieve.

The current SHW requires that more than 75% mass remains at the end of the test.

### 3.3.5.2 Loss on Ignition (*LI*)

The loss on ignition test is used to determine the presence of volatile elements in the aggregate particles. It is determined in an oxidizing atmosphere (air). By igniting the aggregate in air at 975 $^{o}$C for at least 60 minutes, the carbon dioxide and the water not evaporated during drying are driven-off as are any oxidizable volatile elements present.

The loss on ignition (*LI*) can be calculated from the following equation (BS EN 1744-1:1998):

$$LI(\%) = \frac{M_1 - M_2}{M_1} \times 100\% \qquad \ldots(3.6)$$

where

$M_1$ is the mass of the test portion in grams

$M_2$ is the mass of ignited test portion in grams

**3.3.5.3 The Expansion of Slag**

Some alternative aggregates, such as steel slag, tend to expand if not properly weathered. The late hydration of dead burned free lime and/or free magnesium oxide can cause significant swell and pop-outs and lead to the disintegration of the aggregate which will affect the performance of the pavement structure. The expansion of slag is determined in accordance with BS EN 1744-1 (BSI, 1998). In this test, a compacted slag specimen, combined from known grain sizes, is subjected to a flow of steam at 100$^{o}$C in a steam unit at ambient pressure. By this means, the necessary moisture for reaction with the free lime and free magnesium oxide is continuously conveyed to the test specimen. Any change in the volume caused by this reaction is read off from a dial test indicator directly at the top of the specimen. The increase in volume is given as the result, calculated in percentage volume in relation to the original volume of the compressed slag specimen (see Figure 3.3).

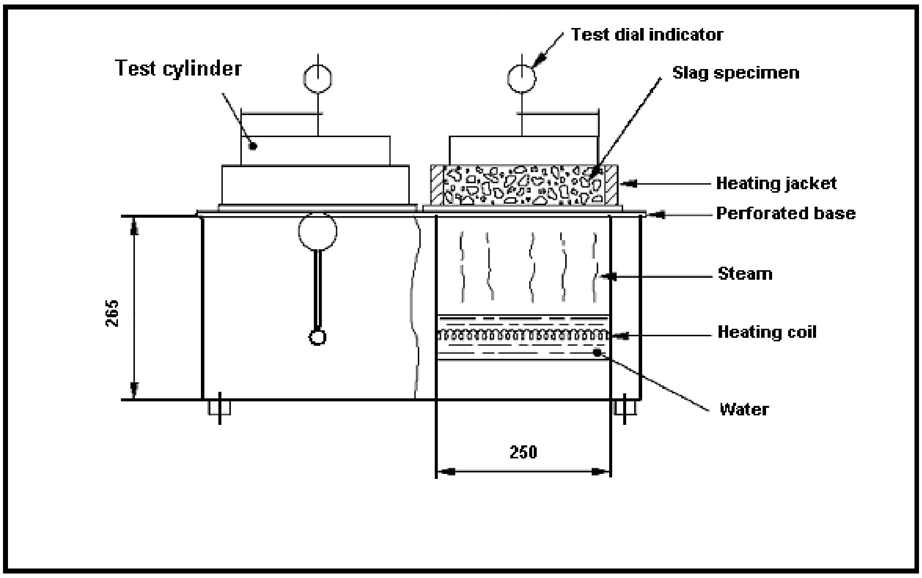



**Figure 3.3: Vertical section of typical steam equipment - all dimension in millimetres (BSI, 1998)**

## 3.4 LABORATORY TESTING PROGRAMME FOR TIN SLAG MATERIALS

The properties of recycled materials are very different compared to natural aggregates. There are not many established standards to be used on alternative materials. Some standard tests, used on natural aggregates, are not appropriate to be used on recycled materials (Reid et al., 2001). However, if recycled materials want to be used in road construction, they usually have to meet specification requirements and be classified in the same way as for road-making materials already in use.

ALT-MAT, a collaborative research project funded by the European Commission, has performed a study to define methods of assessment on alternative materials for use in road construction. The work of this project was co-ordinated by the UK and most of the Western European Countries were represented. The methods, covered under ALT-MAT project, were mechanical properties, functional requirements, leaching potential and long-term stability of the materials for unbound bound granular materials. For the purpose of this research, the following section describes the programme developed for evaluation of the physical and mechanical properties of tin slag. The programme of the tests selected drew on the findings and recommendations of earlier research.

### 3.4.1 Laboratory Testing Programme

#### 3.4.1.1 Physical and Mechanical Properties of Tin Slag

At this stage, the laboratory-testing programme was planned with the intention of studying the characteristics of tin slag. The physical and mechanical properties of slag were assessed to determine whether the material complies with all the tests required in the present specification for road construction by the UK Highway Agency in its Specifications for Highway Works (Highway Agency, 2001). A comprehensive range of tests was performed to ascertain the feasibility of using tin slag in road construction. Most of the testing was performed in accordance to the methods described in British Standards Institute and as outlined in Section 3.3. A list of test methods used in this study and the relevant BS reference is presented in Table 3.1.

When determining the aggregate crushing value, aggregate impact value and ten percent fines values it was not possible to test the slag samples using the standard aggregate size which is between 14mm and 10mm. The sizes of tin slag used for these tests were between 5.00mm and 6.63mm. These sizes were selected because of their abundance and being the nearest to the suggested standard aggregate size. Since the size of tin slag used for determination of ACV, AIV and TFV was not the suggested standard size, it was therefore decided to use granite with the same size as the tin slag as a control. This allowed direct comparison between test results of the tin slag and natural aggregate.

Some of the fundamental tests for natural aggregate were not conducted on the tin slag specimen in this investigation. For example, Atterberg limits test was not included in the proposed testing programme although required by the SHW (Highways Agency, 2001). The objective of the Atterberg limits testing on aggregate is to ensure that the fines fraction in an aggregate are non-plastic. However, the test was not required for tin slag because the slag had less than 5% passing the 0.075 test sieve.

**Table 3.1: Test methods used for selected materials**

| Test Method | Standard Reference |
|---|---|
| **Test for geometrical properties of aggregate** | |
| Determination of particle size distribution – Sieving method | BS EN 933-1:1997 |
| Image Analysis | - |
| **Test for mechanical and physical properties of aggregates** | |
| Determination of water content | BS EN 1097-5:1998 |
| Determination of particle density | BS EN 1097-6:2000 |
| Determination of water absorption | BS EN 1097-6:2000 |
| Determination of aggregate crushing value (ACV) | BS 812-110:1990 |
| Determination of aggregate impact value (AIV) | BS 812-112:1990 |
| Determination of ten percent fines value (TFV) | BS 812-111:1990 |

Elongation index and flakiness index were not determined in this research although they are essential tests for natural aggregate. The tests were not vital for tin slag because the shape of the tin slag particles was mostly rounded and angular. Furthermore, the tests are not applicable to materials passing a 6.30mm test sieve, therefore it was inappropriate to perform the tests on the tin slag which had a predominance of particles less than 6.30mm. Based on the same reasons, the shape index was also not determined. However, the above tests were replaced by studying the particle shapes of tin slag using computerized image analysis.

The frost susceptibility of the slag was not determined for the reason that if the research is successful, the tin slag is only likely to be used in the countries of origin which are predominantly tropical countries, such as Malaysia, Thailand, or Indonesia and probably China. In these countries, except for China, there is no freezing zone to take into account.

#### 3.4.1.2 Environmental and Chemical Properties of Tin Slag

In the early stage, there were four proposed tests to determine the environmental and chemical properties of tin slag. These were magnesium sulphate test, determination of the expansion of slag, determination of loss on ignition and determination of free lime and pozzolanic property of tin slag. However, after several further considerations none of the above tests were deemed applicable to tin slag except pozzolanic property. The determination of pozzolanic property is discussed in detail in Chapter 5.

As already discussed in paragraph 3.3.5.2, the loss on ignition test is determined by igniting the specimen at 975 $^{o}$C to detect the present of volatile elements on specimen. Since the temperature needed for the production of tin slag is more than 1300 $^{o}$C, therefore to determine the loss on ignition is unnecessary. For that reason, the test was not performed on the tin slag. The magnesium sulphate test is used to determine the durability of the aggregate as an estimation of aggregate resistance to weathering action. The test portion of specimen for this test is in the size range of 10 mm to 14 mm (BSI, 1998). Since most of the tin slag size is less than 10 mm, and as the reproducibility of the test is poor for fine aggregate (Kandhal and Parker, 1998), therefore the magnesium sulphate test was not performed. Furthermore, the different opinions of many researchers regarding this test as explained in paragraph 3.3.5.1 were also taken into consideration.

An expansion test was also not performed on tin slag. It was assumed that tin slag would not face any expansion problem, based on its chemical composition as shown in Table 2.13. The table shows that tin slag contains low percentages of calcium oxide (CaO) and magnesium oxide (MgO), which are within the range of 16-20% and 2-4% respectively.

## 3.5 RESULTS AND DISCUSSION

The laboratory test results obtained from testing the tin slag are presented in the following sections.

### 3.5.1 Gradation

Figure 3.4 shows the results obtained from two tin slag particle size distribution analyses performed on notionally identical specimens. Particle size analysis revealed the following results:

- None of the tin slag particles had a size greater than 16mm.
- Only 2 % to 3.4 % of the tin slag particles had a size between 10mm and 16mm.
- Most of the tin slag was retained on 0.6mm sieve and passing 3.35mm.
- The tin slag had a very small amount of filler i.e. only 0.5 % of the particles were less than 0.075mm.

The particle size distribution in Figure 3.4 shows a nearly vertical curve in the mid-size range and flat and near zero in the small-size range of tin slag. It can be concluded that the tin slag has an open graded gradation. For that reason, it was expected that such a mix will result in more air voids following compaction than continuously graded mixes because there are not enough fine particles to fill in the voids between the larger particles. Given the rounded shape of slag particles, it may be further concluded that stability under direct shear loading (e.g. as imposed by traffic) may be lower than other more cubical aggregates. Therefore, this should be taken into consideration when designing mixes for bound and unbound road layers.

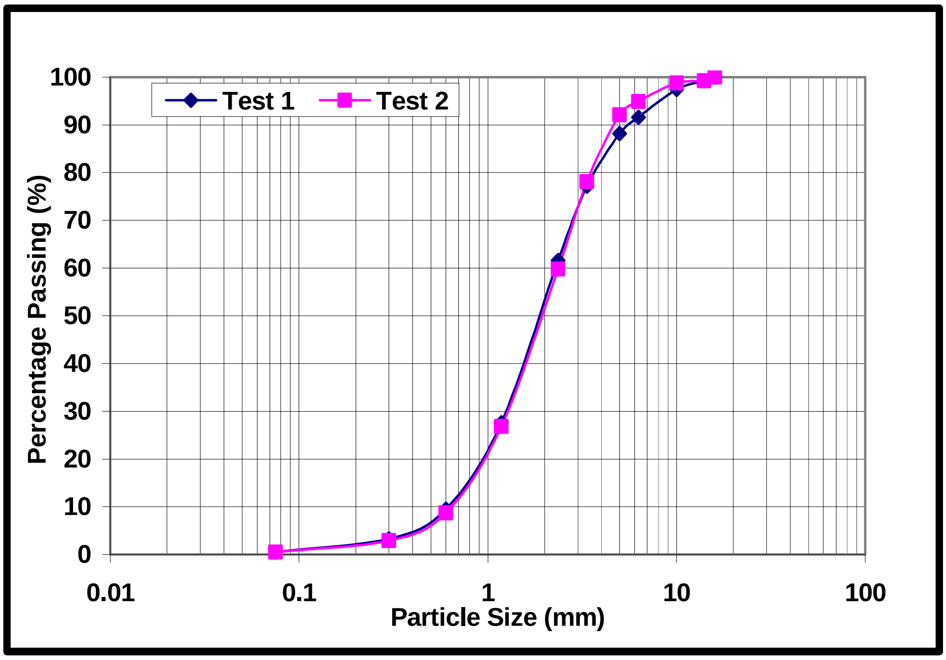



**Figure 3.4: Particle size distribution of tin slag**

### 3.5.2 Particle Shape and Surface Texture

Figure 3.5(a) shows the overall picture of tin slag. The tin slag is in granulated form, glassy and black in colour. Figures 3.5(b) and 3.5(c) show the particle shape and surface texture of the tin slag. Most of the tin slags with particle size greater than 10mm have rounded shape as shown in Figure 3.5(c). However, overall angular shapes are more dominant for tin slag as shown in the examples in Figures 3.5(b) and 3.5(c).

The proportion of elongated and flaky particles in any tin slag sample was insignificant. Hence, it is likely that particle fracture during the compaction process should not be a major consideration. Another interesting observation was that tin slag particles contained needle shaped particles, especially with aggregate sizes less than 6.3mm. Therefore, when handling the tin slag on site, care must be exercised, as some of the particles are needle shaped, very pointed and can cause injury.

**Figure 3.5 (a): Tin slag**

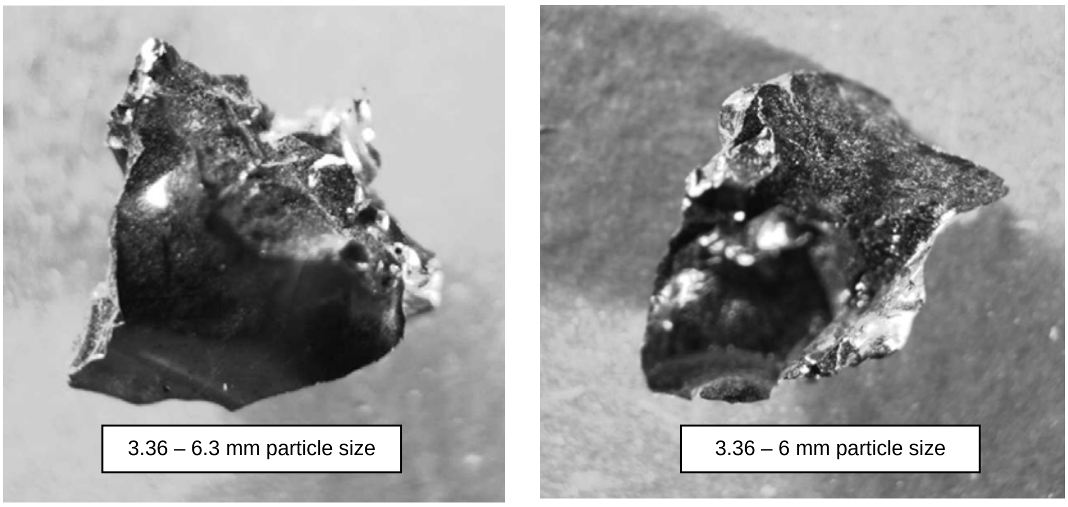



**Figure 3.5(b): Tin slag**

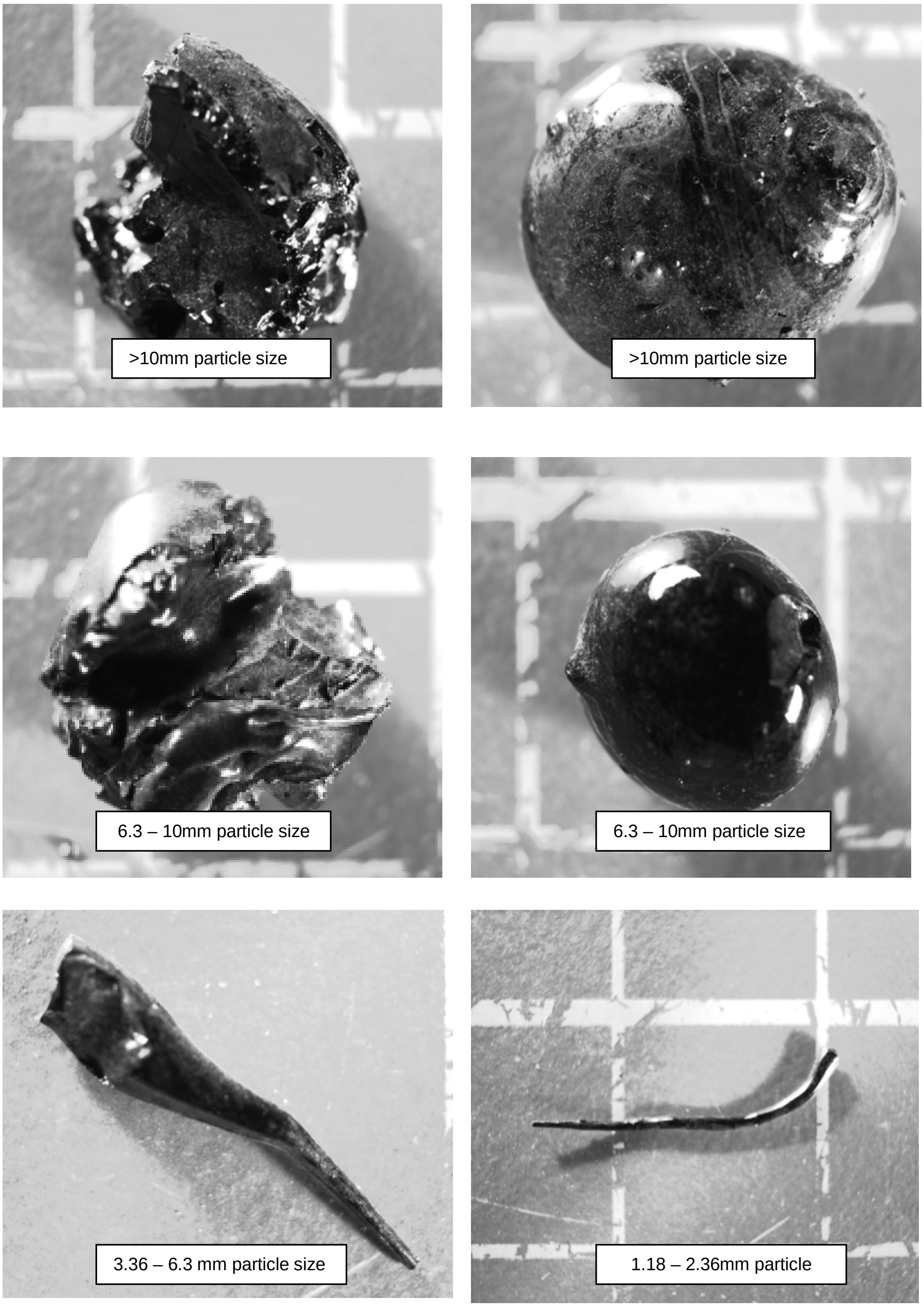



**Figure 3.5(c): Tin Slag**

### 3.5.3 Particle Density

Two methods, British Standard water pyknometer and a helium pyknometer, were used to determine particle density of tin slag. The results from both methods are shown in Table 3.2. The helium pyknometer test apparatus was based at the School of Civil Engineering, the University of Leeds.

**Table 3.2 Particle density and water absorption of tin slag**

| Property | Particle density |
|---|---|
| Apparent particle density ($\rho_a$) | 3.59 Mg/m$^3$ |
| Particle density on an oven-dried basis ($\rho_{rd}$) | 3.35 Mg/m$^3$ |
| Particle density on a saturated and surface-dried basis ($\rho_{rrd}$) | 3.42 Mg/m$^3$ |
| Particle density (using helium pyknometer) | 3.52 Mg/m$^3$ |

When comparison is made between tin slag, other slags and natural aggregates, the density value of tin slag seems to be relatively similar to that of steel slag and copper slag (see Table 3.3). However, the density of tin slag is higher than natural aggregates e.g. limestone, granite. Aggregate particle density is useful in making weight-volume conversions and in calculating the void content in compacted hot mix asphalt (Roberts et al., 1996). Particle density results suggested that a high-density mixture will be produced when tin slag is used as the aggregate. The dry density of hydraulically bound and the bulk density of asphalt mixtures will be affected by the increased particle density. Hence, provided the packing properties of slag mixes are similar to those of conventional aggregates, the slag mixes will be denser.

**Table 3.3 Comparison of particle densities of selected aggregates and slags**

| Type of Materials | Particle density ($Mg/m^3$) |
|---|---|
| Tin slag | 3.52 |
| Blast furnace slag | 2.38 - 2.76 [a] |
| Steel slag | 3.1 – 3.5 [a] |
| Copper slag | 3.4 – 3.6 [b] |
| Phosphorus slag | 2.7 [a] |
| Granite | 2.69[c] |
| Limestone | 2.66[c] |

a Lee (1974)

b Mineral research & Recovery Inc. (2001)

c Neville (1983)

**Table 3.4 Water absorption of tin slag compared with other slags**

| Type of Slags Materials | Water absorption % by mass |
|---|---|
| Tin slag | 1.97 |
| Blast furnace slag | 1.5 – 6.0 [a] |
| Steel slag | 0.2 – 2.0 [a] |
| Copper slag | 0.13 [b] |
| Phosphorus slag | 1.10 [a] |
| Granite | 0.07 – 0.3[c] |

a Lee (1974)

b Mineral Research & Recovery Inc. (2001)

c Marek (2001)

### 3.5.4 Water Absorption and Water Content

The water absorption of tin slag was measured at 1.97%. The value is moderate when compared with other slags. Table 3.4 presents the water absorption values of various slags. Other slags having similar water absorption value include blastfurnace and steel slags. Generally, this level of water absorption is not a major concern when using tin slag in asphalt mixtures because tin slag is thoroughly dried during the hot asphalt mix production process but higher absorption values could cause excessive demand for bitumen. However, care should be exercised when using tin slag for cement bound mixtures because the tin slag is generally not dried and therefore the moisture content in the slag will affect the overall water content (thus affecting the water-cement ratio). The water content will also affect tin slag proportioning because it contributes to aggregate weight.

### 3.5.5 Mechanical Properties

The results of aggregate crushing value (ACV), aggregate impact value (AIV) and ten percent fines value (TFV) for tin slag and granite are shown in Table 3.5. The results indicate that tin slag is approximately three times weaker than granite in terms of crushing strength. However, the test results might not be a true representation of their value and could be inaccurate since the sizes of the aggregates used for the test were not compliant with standard BS sizes. According to BACMI (1992), the aggregate is considered too weak to use in normal road construction if the ACV is over 35%, although such an aggregate might be useable in some circumstances. Another study by Hosking and Tubey (1969) reported that aggregates with ACVs ranging between 30% and 35% could be adopted for a low volume road. In this respect, tin slag, which has 31% ACV, might be considered suitable for use as aggregate for a low-medium volume road which have no requirement in the current Specification for Highway Works (SHW).

Table 3.5 also shows that the tin slag and granite have 24% and 10.4% AIV respectively. These values are far below the value suggested in the Specification for Highway Works (SHW, 2002) as shown in Table 3.6 for aggregates suitable for use in bituminous bound materials. Therefore, in terms of AIV, tin slag can be used in bituminous bound material without any doubt. However, aggregates having AIV between 30% and 35% could be considered for use in low volume and low cost road construction (Hosking and Tubey, 1969).

Table 3.5 shows the $TFV_{dry}$ for tin slag and granite to be 22.1kN and 100kN respectively. It shows that tin slag is approximately five times weaker than granite in terms of $TFV_{dry}$. However, the SHW specifies that aggregate for use in bituminous bound materials must have $TFV_{dry}$ values of more than 140kN and 85 kN for natural aggregates and blastfurnace slag respectively (Table 3.6). In this investigation, both tin slag and granite had $TFVs_{dry,}$ that were too low and this requires further investigation. Additional tests to determine mechanical properties by using performance tests such as repeated load triaxial tests may be more suitable for waste materials like tin slag.

In general, tin slag showed satisfactory performance in terms of toughness tests although the TFV seemed unduly low. Although small particles usually give higher TFV value (Dawson, 1989) this is not always true. For example, in the case of slate waste, the use of small sized particles resulted in a lower TFV value, probably due to the flakiness of the particles (Nunes, 1997). It must be emphasised that in this investigation all the mechanical tests performed were not in accordance with standard size aggregates i.e. not between 10 and 14 mm. As a result of the natural particle size distribution of the tin slag, it was not possible to obtain adequate quantities of slag particles between 10 – 14mm. As discussed earlier, some of the tin slag has needle shaped particles and this would have effected the TFV values.

**Table 3.5 Mechanical properties of tin slag compared with granite**

| | **Material** | |
|---|---|---|
| **Property** | **Tin slag** | **Granite** |
| Aggregate Crushing Value, ACV (%) | 31.4 | 11.6 |
| Aggregate Impact Value, AIV (%) | 24.9 | 10.4 |
| Ten Percent Fine Value, $TFV_{dry}$ (kN) | 22.1 | 100.0 |

**Table 3.6 Mechanical properties of aggregates for bituminous bound materials according to the Specification for Highway Works (2002)**

| | Material | |
|---|---|---|
| **Property** | **Blastfurnace slag** | **Natural aggregate** |
| Aggregate Impact Value, AIV (%) | < 35% | < 30% |
| Ten Percent Fine Value, TFV (kN) | > 85 kN | > 140 kN |

## 3.6 SUMMARY

- The testing programme set up for evaluating the tin slag was described and the results obtained were presented. The results were compared against the Specification for Highway Works requirements.

- The particle size distribution performed on tin slag showed that tin slag has an open graded gradation.

- An assessment on the shapes showed that most of the tin slag has angular shape. However, round and needle shapes can also be found in tin slag.

- In terms of physical and mechanical properties, tin slag seems to meet most of the requirements stated in the Specification for Highway Works. Therefore, tin slag possesses properties demonstrating some potential for use in bituminous bound materials and hydraulically bound materials, although care must be exercised.

- For unbound applications of tin slag, performance capacity should be verified through performance tests such as the repeated load triaxial test as its TVF value seems unduly low.

Chapter 4

# INVESTIGATING THE POTENTIAL USE OF TIN SLAG AS UNBOUND GRANULAR MATERIAL

## 4.1 INTRODUCTION

Unbound granular materials (UGM) are generally used as base and sub-base layers in road pavements. As a base course, they play a structurally important role, especially in medium and low volume roads where the surfacing course may be quite thin. As a sub-base, they protect the sub-grade from surface load stresses and act as an insulating layer against frost action (European Commission, 2001). The UGM may be a natural material like gravel, sand or crushed rock, or it may be made from by-products or wastes from an industrial process such as crushed slag or crushed concrete mixed with crushed masonry and asphalt planings.

Although they form intermediary elements of the pavement structure, the correct functioning of the UGM layers is vitally important. UGM does give rise to pavement failures due to either inadequate support provided to upper layers or being insufficiently stiff to distribute the load to the sub-grade. These pavement failures usually necessitate complete pavement reconstruction, and not just remedial treatment of the pavement surface where the problem is visible. Therefore, it is very important to evaluate UGM before it can be incorporated into road pavements.

The performance of UGM can be assessed by its shear strength and stiffness modulus. There are several methods that can be used to measure shear strength and stiffness modulus of UGM. However, the triaxial test method is preferred by most researchers. The advantages of using this method are described below (Saeed et al., 2001):

- The test is accepted universally for measuring soil strength;
- The test method can accommodate different stress states;
- The test method can consider repetitions of stress;
- The test method provides a measure of permanent deformation;
- The test method can accommodate changes in moisture content;
- The degree of test complexities can be varied to suit the test objectives;

Although there is a little doubt about the capabilities of the triaxial test method, yet there are some disadvantages in using this test method (Saeed et al., 2001) including:

- The method can be very complex and require special equipment;
- Test specimens are difficult to prepare;
- The test is time-consuming and difficult to set-up.

## 4.2 SHEAR STRENGTH

Shear strength is the single most important property that governs the performance of an unbound pavement layer in a pavement structure. Shear strength of a material is defined as the ultimate mobilized shear stress before shear failure take places (Xie and Lekarp, 2001). According to these authors, the strength of granular material is important in two respects. Firstly, the strength may be related to stiffness and to permanent deformation behaviour. The strength may provide an important substitute measure of stiffness or permanent deformation behaviour since the strength of a granular material may be easier to determine. Secondly, the strength of individual particles may have major impact on the behaviour of the material as a whole. For example, a crushed rock with weak particles that are damaged during compaction will exist in the road construction with a smaller mean particle size than when initially placed. Such a change in grading is likely to have a major effect on both stiffness and permanent deformation response.

One of the techniques used to determine shear strength is repeated load vacuum triaxial test which was used by Sweere (see NCHRP, 1997) when assessing shear strength of granular materials. In this test, a cylindrical specimen having a porous disc on the top and bottom surface is enclosed by rubber membrane, and rests on a base plate of a loading frame. The compressive stress, $\sigma_3$, (confining pressure) is applied by applying a partial vacuum of known magnitude on the inside of the specimen instead of using a conventional triaxial chamber (see Figures 4.1 and 4.2). The axial stress, $\sigma_1$, is increased at a certain rate by $\Delta\sigma_1$, i.e. $\sigma_1 = \sigma_3 + \Delta\sigma_1$, under constant confining pressure. Shear failure occurs when there is a clear and marked fall-off in the value of the applied deviator stress, $\sigma_d$ which is the difference of axial stress and compressive stress (i.e. $\sigma_d = \sigma_1 - \sigma_3$). This procedure is then repeated at least for three different confining pressures. The values of the deviator stress are then used in drawing the Mohr's circles (see Figure 4.3). The slope of the tangent (as the angle of internal friction, ø, of aggregate) is then measured and subsequently the shear strength is evaluated. The shear strength can be determined using the Mohr-Coulomb equation which is expressed in Equation 4.1.

$$\tau_f = c + \sigma \tan \phi \qquad \ldots(4.1)$$

where

| | |
|---|---|
| $\tau_f$ | is the shear strength |
| $c$ | is the apparent cohesion |
| $\sigma$ | is the normal stress |
| $\phi$ | is the angle of internal frictional resistance |

Note that the equation and its constituent terms are written in terms of total, not effective, stresses because, in moist aggregate, pore suction cannot be measured and hence, the effective stress cannot be determined.

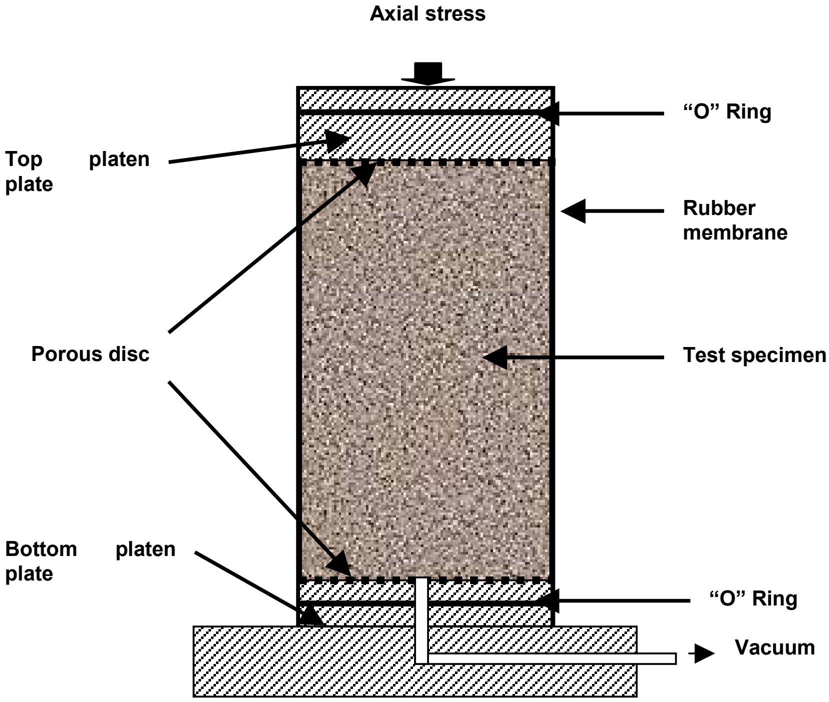



**Figure 4.1: A vacuum triaxial test equipment**

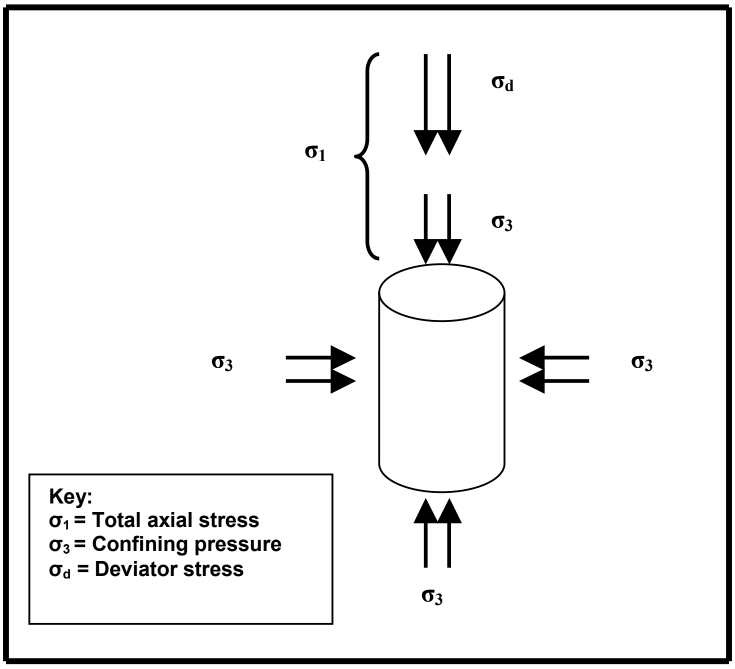



**Figure 4.2: Stress applied to a triaxial specimen**

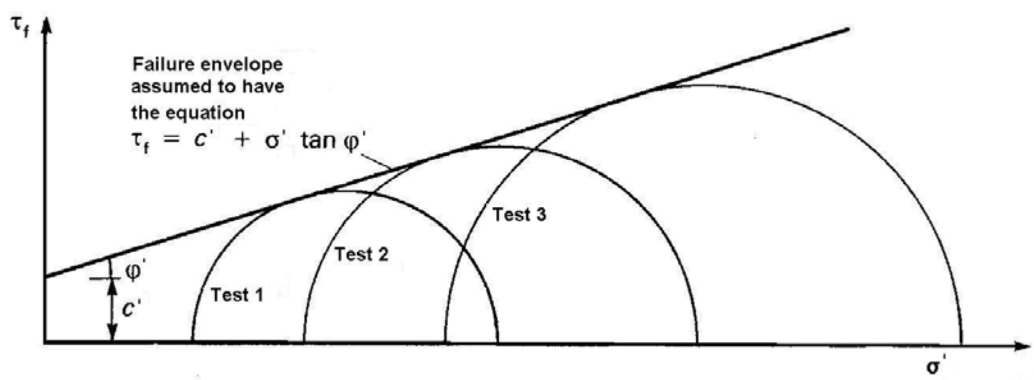



**Figure 4.3: Obtaining the Mohr-Coulomb envelope (Whitlow, 1990)**

## 4.3 RESILIENT MODULUS

UGMs in pavement layers must exhibit high resilient modulus in order to spread load adequately and to reduce the resilient deformations of upper bituminous layers. Resilient modulus is a measurement of the elastic property of materials and can be determined from recoverable strains during unloading. Therefore, resilient modulus ($M_r$) is defined as the ratio of the peak applied axial repeated stress to the recoverable axial strain occurring within the specimen (Barksdale et. al, 1997). The resilient modulus is presented in the following equation:

$$M_r = {(\sigma_1 - \sigma_3)}\big/{\varepsilon_{1r}} \qquad \ldots(4.2)$$

where:

$M_r$ is a resilient modulus

$\sigma_1$-$\sigma_3$ is the maximum repeated axial stress (deviator stress)

$\varepsilon_{1r}$ is the maximum resilient axial strain

It was reported that the stiffness of granular material increased non-linearly with applied stress (Brown, 1967). Hicks and Monismith (1971) reported that the resilient stiffness of aggregate was heavily influenced by the summation of the three principal stresses. They found that materials became stiffer when confining stress was increased. Also, the stiffness decreased initially with increasing deviator stress and then increased when higher deviator stresses were applied.

Boyce (1976), in his literature review on factors affecting the resilient behaviour of granular materials, concluded that the density could affect resilient properties up to 50%. However, Thom and Brown (1988) found that the degree of compaction (i.e. density) had little effect on resilient modulus. This finding was supported by Thomson (2001) who mentioned that for a given material, specimen density has only a limited effect on the resilient modulus. He also stressed that, at moisture contents above optimum, resilient modulus tends to decrease. However, for materials with limited fines, the effect of moisture change is minor and materials that do not contain fines are practically moisture insensitive.

Barksdale and Itani (1989) found that the stiffness was affected by the surface characteristic of granular material such as particle shape, angularity, surface texture and roundness. They found that angular materials with the same grading had a higher resilient modulus than rounded gravel. The work of Nataatmadja (see Cheung, 1997) showed that at the same comparative effort and with the same shape of particle grading curve, materials with larger maximum particle size produced smaller resilient strains.

The resilient modulus of UGM can be determined using the repeated load triaxial test as described in literature such as BS EN 13286-7:2004 (BSI, 2004) and Soil Manual No. 10 (Asphalt Institute, 1993). In this study, the tests were performed in accordance with Manual No. 10 with some modifications to the methodology. In this study, a vacuum triaxial method was used as described previously in Section 4.2, rather than using a conventional triaxial chamber.

## 4.4 LABORATORY ASSESSMENT OF MATERIAL POTENTIAL

### 4.4.1 Gradation

For the purposes of this investigation, an unaltered gradation of tin slag (as received) was used to study its shear strength and resilient modulus. Conventional aggregate was used as a reference material for comparison purposes. A Modified Fuller packing equation (Cooper et al, 1985) with exponent n = 0.5 was used to obtain a maximum density of the reference specimen (see Equation 4.3). Figure 4.4 shows the gradation of tin slag and conventional aggregate used in this investigation.

$$P = \frac{(100 - F)(d^n - 0.075^n)}{(D^n - 0.075^n)} \times F \qquad \ldots(4.3)$$

where:

$P$ = total percentage by mass passing sieve size $d$ (mm)

$D$ = maximum aggregate size in the sample (mm)

$F$ = filler content (%)

$n$ = a parameter established by Fuller to determine shape of the grading curve.

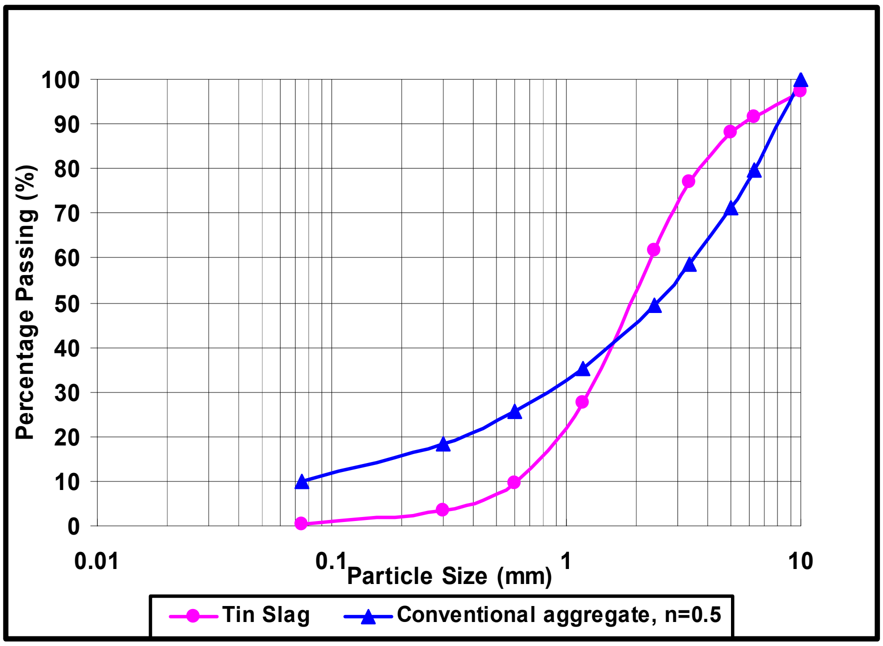



**Figure 4.4: Tin slag and conventional aggregate gradation used in investigation of shear strength and stiffness modulus**

### 4.4.2 Sample Preparation

A three piece split aluminium compaction mould of 100mm internal diameter and 200mm height was used in preparing the aggregate specimen. First, the porous stone was placed on the bottom platen. A rubber membrane was attached to the bottom platen with a rubber "O" ring. The mould was assembled directly on top of the base plate around the rubber membrane which then folds down over the mould. A vacuum was applied to hold the membrane to the sidewall of the mould. Care was taken to ensure the rubber membrane was not twisted or pinched. The specimens were placed in the mould and then compacted by tamping. The materials were compacted in three layers and each layer was compacted with 25 blows. To reduce stratification, the top of the layer was scarified before placing the next layer.

### 4.4.3 Compactibility test

Separate compactibility tests were performed to determine the optimum moisture content and dry density for all mixtures. During the compaction process the solid particles are packed more closely together, thereby increasing the dry density of the material tested. The dry density which can be achieved depends on the degree of compaction applied and on the amount of water present in the material. For a given degree of compaction, there is an optimum moisture content at which the dry density obtained reaches a maximum value

The determination of optimum moisture content was carried out in accordance with BS 1924-2 (1990). Specimen preparation for the compactibility test was similar to that explained previously (see Section 4.4.2). However, in this case, after removal of the specimen mould, weighing and measuring the height, the specimens were placed on trays and dried in an oven at 105$^{o}$C until a constant weight was observed. The bulk density, dry density and water content were then calculated. A graph of moisture content against dry density was then plotted to determine the optimum moisture content.

### 4.4.4 Shear Strength and Stiffness Modulus Determination

The specimens used to determine shear strength and stiffness modulus were prepared in accordance with Sections 4.4.2 and 4.4.3 at their optimum moisture contents. The vacuum line was then released from the mould and attached to the base plate. With the vacuum then applied to the specimen, the mould was removed. The rubber membrane was observed for any holes or leaks. If any hole or leak was found the specimens were then rebuilt with a new membrane. The confining pressure had to change in line with the maximum capacity that can be achieved by the vacuum pump. Therefore, for the purpose of this investigation, the confining pressures were set to 40, 60 and 80 kPa. Four height measurements and three diameter measurements (i.e. at the top, at mid-height and at the base) were determined. The measurements were averaged and the volume of the compacted specimen computed. The compacted specimens were then transferred to a loading machine (in this study an Instron hydraulic machine was used), ready for testing.

## 4.5 RESULTS AND DISCUSSION

### 4.5.1 Compactibility Test

The results obtained from the compactibility tests on tin slag and conventional aggregate are shown in Figure 4.5 and summarised in Table 4.1. The trend lines clearly show that each compaction curve maximises at a peak defining the optimum moisture content and maximum dry density values.

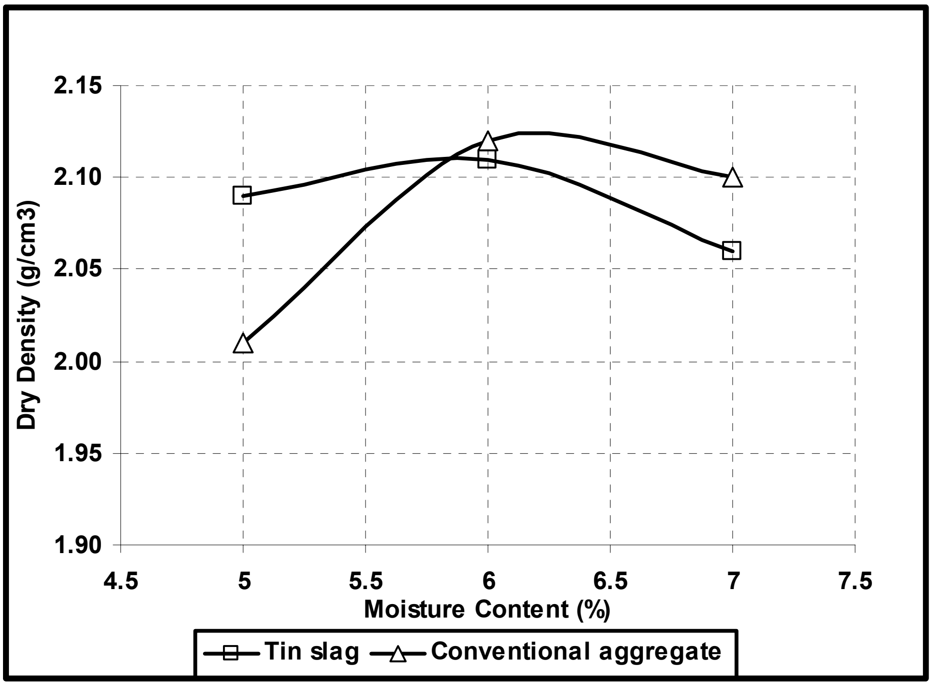



**Figure 4.5: Compactibility test results for tin slag and conventional materials**

Although the peak dry density values were very similar, the much higher $SG_{agg}$ value of the tin slag caused the dry compacted porosity to be much higher as shown in Table 4.1.

**Table 4.1: Compactibility tests for tin slag and conventional aggregate**

| | Tin Slag | Conventional Aggregate |
|---|---|---|
| OMC (%) | 5.9 | 6.2 |
| Dry Density ($Mg/m^3$) | 2.11 | 2.12 |
| Dry Porosity (%) | 40.06 | 23.47 |

## 4.5.2 Shear Strength

Shear strength envelopes for tin slag and conventional aggregate obtained from the tests are shown in Figures 4.6 and 4.7 respectively. By evaluating the line in Figure 4.6, it was found that the value of angle of friction (ǿ) for tin slag was 40.24$^o$ and the value of apparent cohesion (c') was 15kPa. With regard to a conventional aggregate (see Figure 4.7), it was found that the angle of friction was 45$^o$ and the apparent cohesion was

10kPa. It seems that the angle of friction value for conventional aggregate (granite) in this study exhibits a medium value compared with Cheung (1994), who quoted 32$^{o}$ for poorly compacted granite and 56$^{o}$ for denser granite. The tests indicate that the shear strength of the tin slag is marginally lower than the shear strength of the conventional aggregate (reference material). This can be explained by looking at the particle shapes and the gradation of tin slag (see Figures 3.2(b) and 4.4). From observation, it was found that some of the tin slag had particle sizes between 1.15 and 6.3mm and an angular shape.

With regard to the apparent cohesion value, it was found that tin slag had an apparent cohesion value higher than that of the control aggregate. In reality, this value is largely due to suction and exists as long as tin slag or aggregate retains some moisture. It would be lost if the aggregate were to be dried out or to become saturated or submerged. Therefore, the extra strength attributed to apparent cohesion is generally neglected in foundation design (McCarthy, 1988). It can be concluded that the tin slag had a shear strength which is very similar to that of a typical, conventional, aggregate.

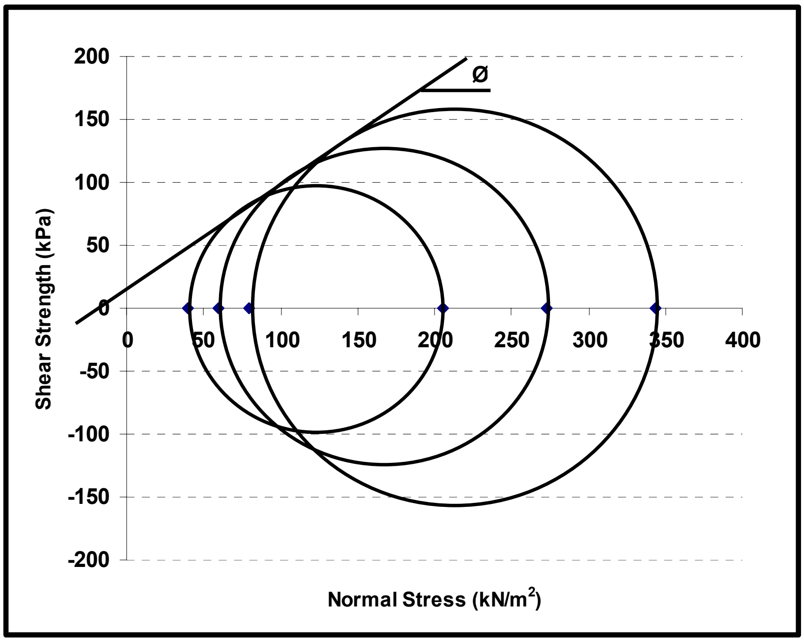



**Figure 4.6: Shear strength test for tin slag material**

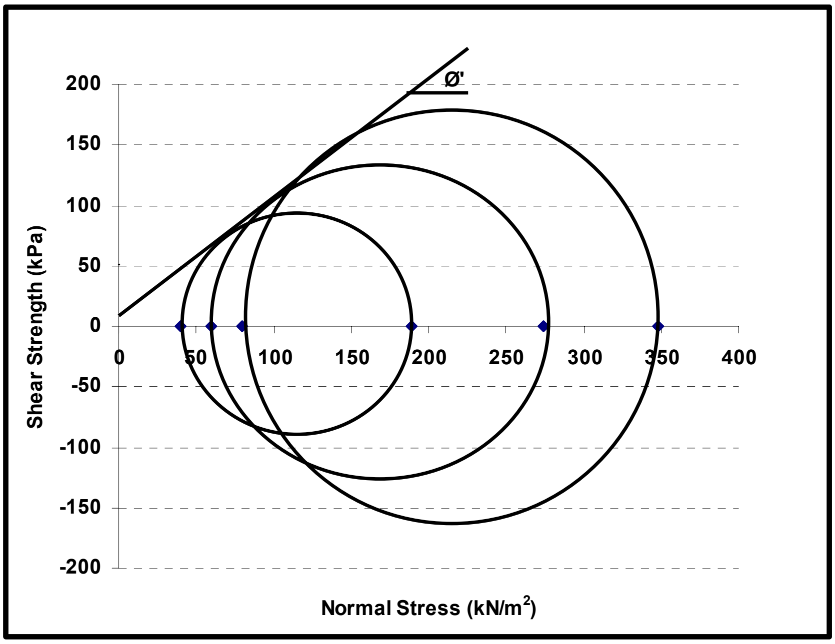



**Figure 4.7: Shear strength test for conventional material**

### 4.5.3 Resilient Modulus

A single specimen of both tin slag and conventional aggregate was tested for resilient modulus. Figures 4.8 to 4.13 show typical resilient behaviour obtained from repeated load triaxial tests performed in this investigation. In all these figures a single load cycle (the $4^{th}$ cycle) is represented, with a degree of scatter in individual data points being evident. A value of resilient modulus has been determined by dividing maximum deviator stress by the resilient strain. It was found that the resilient strain response was dependent upon the confining stress applied, i.e. the higher confining stress, the lower resilient strain and therefore the higher the resilient modulus.

The test results are summarised in Table 4.2. The results show that the stiffness modulus for tin slag at 40kPa and 80kPa confining pressures was slightly lower than the stiffness modulus values obtained from conventional aggregate. However, at 60kPa confining pressure, tin slag had a slightly higher stiffness modulus value compared with conventional aggregate. In general, when comparing the unbound tin slag and conventional aggregate, it seems that there is only a very slight difference in stiffness modulus between unbound tin slag and conventional aggregate under repeated loading. The results show that unbound tin slag material performs reasonably well under repeated loading and has comparable performance to conventional aggregate.

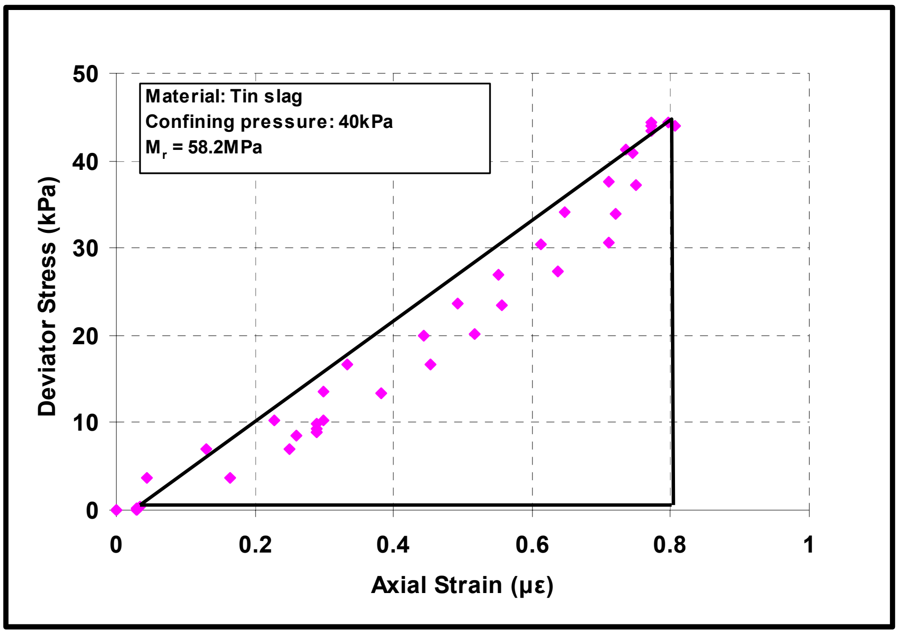



**Figure 4.8: Typical resilient behaviour of unbound tin slag under repeated loading (Confining pressure = 40kPa)**

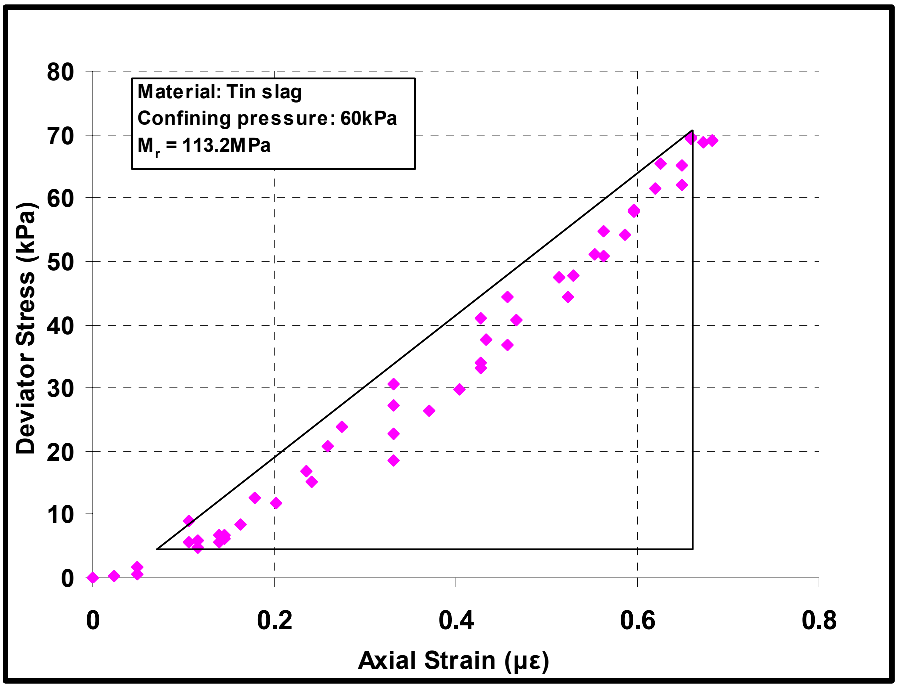



**Figure 4.9: Typical resilient behaviour of unbound tin slag under repeated loading (Confining pressure = 60kPa)**

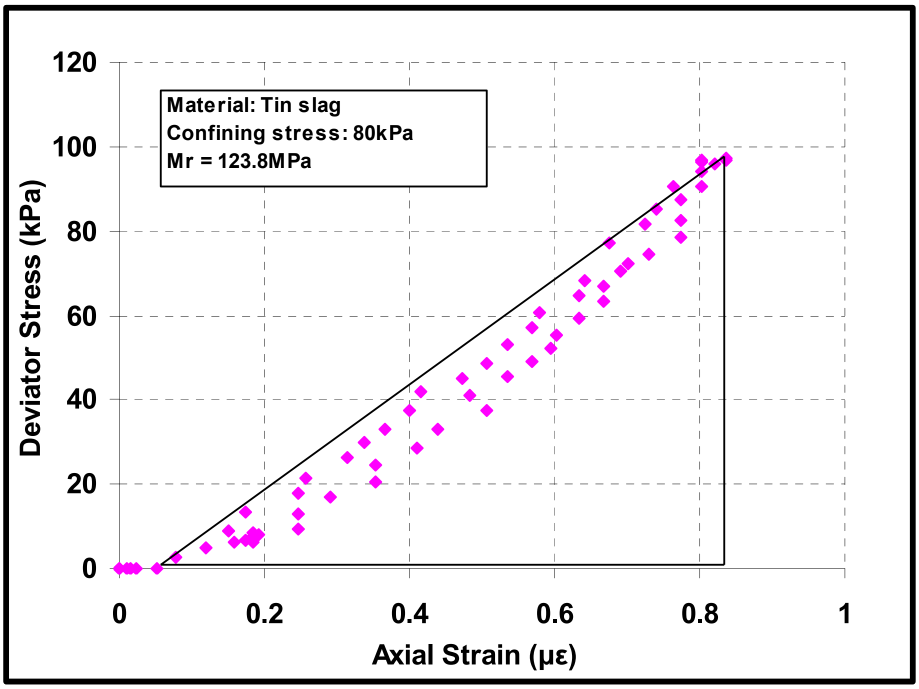



**Figure 4.10: Typical resilient behaviour of unbound tin slag under repeated loading (Confining pressure = 80kPa)**

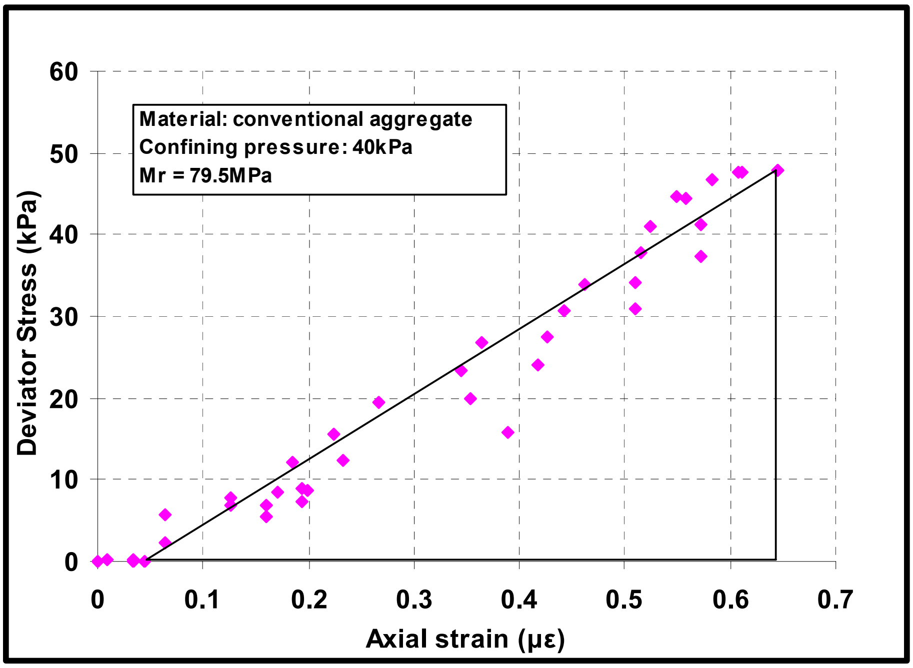



**Figure 4.11: Typical resilient behaviour of unbound conventional aggregate under repeated loading (Confining pressure = 40kPa)**

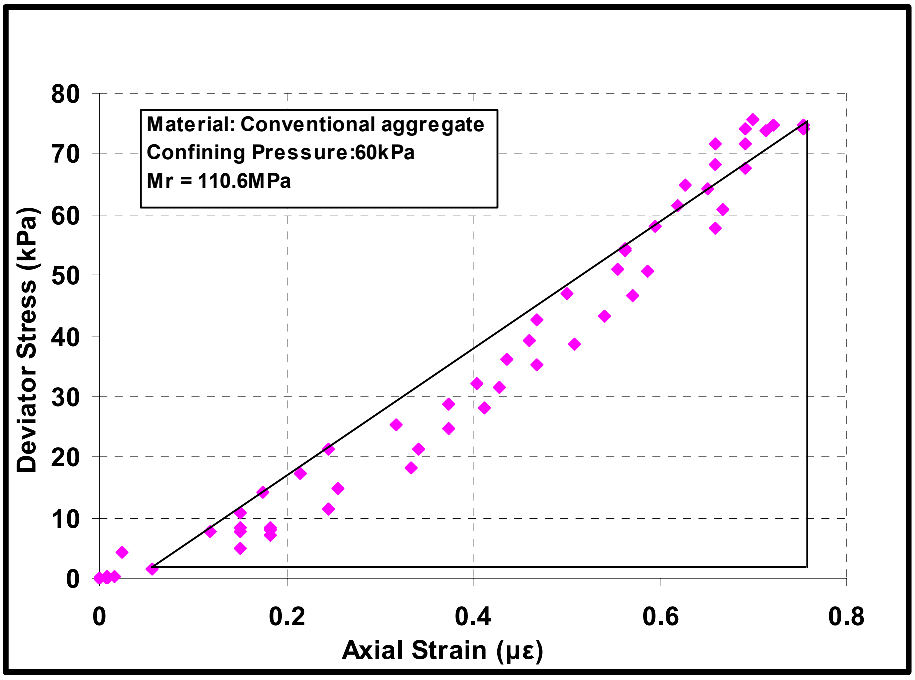



**Figure 4.12: Typical resilient behaviour of unbound conventional aggregate under repeated loading (Confining pressure = 60kPa)**

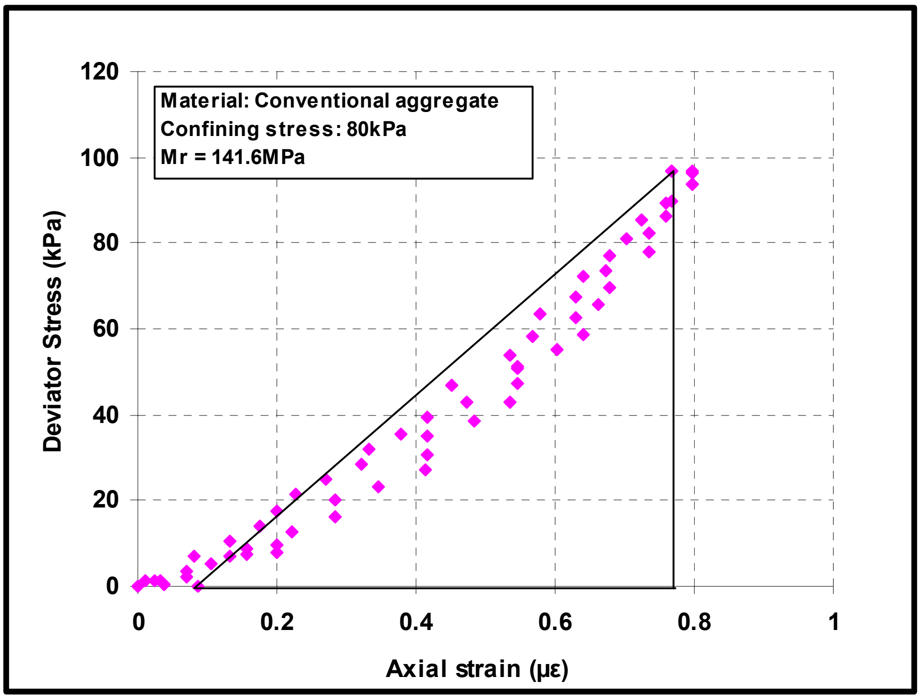



**Figure 4.13: Typical resilient behaviour of unbound conventional aggregate under repeated loading (Confining pressure = 80kPa)**

**Table 4.2: Summary of resilient modulus test results**

| Test | Confining pressure $\sigma_3$ (kPa) | Resilient Modulus MPa |
|---|---|---|
| Tin slag | 40 | 58.2 |
| | 60 | 113.2 |
| | 80 | 123.8 |
| Conventional aggregate | 40 | 79.5 |
| | 60 | 110.4 |
| | 80 | 141.6 |

## 4.6 SUMMARY

- Shear strength and stiffness moduli of unbound tin slag and conventional aggregate were evaluated using triaxial testing equipment in accordance with the procedure set in Soil Manual No. 10 (Asphalt Institute, 1993) with some modification to allow for the limitations of the equipment used.

- The results from shear strength tests indicate that the unbound tin slag had shear strength slightly, but not significantly, lower than that of the conventional aggregate.

- In terms of stiffness modulus, the unbound tin slag material performed reasonably well under repeated load although slightly less competently than conventional aggregate.

- Shear strength and stiffness modulus tests indicate that unbound tin slag could be potentially used as an unbound granular material for road making.

# Chapter 5 ASSESSMENT OF TIN SLAG IN HYDRAULICALLY BOUND MIXTURES

## 5.1 INTRODUCTION

A hydraulically bound mixture is defined as a mixture that hardens by hydraulic, pozzolanic, sulphatic and/or carbonatic reaction, which usually has a workability to suit compaction by rolling and which is generally used in bases, sub-bases and capping layers Based on the above definition, hydraulically bound mixtures can be, therefore, divided into cement bound materials and hydraulically bound mixtures other than cement bound materials.

Cement bound materials have been used in road pavements for many years. The cement-stabilised concept is attributed to a trial by H.E. Brooke-Bradley on Salisbury Plain in 1917 (Williams, 1978). However, not until 1935 onwards were the materials extensively used in many countries. Generally, cement bound materials are described as materials in which cement is added to make them suitable for use in road construction or to enhance their suitability.

Hydraulically bound materials include slag bound mixtures, pulverised fuel ash bound mixtures, lime treated mixtures and natural pozzolana bound mixtures. Some hydraulically bound materials such as natural pozzolana have been used more than 2000 years ago by the Greeks and Romans, where they used a mixture of lime and fine volcanic ash (pozzolana) to build structures such as the Coliseum (Western Pozzolan, 2003). At the present day, natural pozzolana is not the only material used in hydraulically bound materials but slag and pulverised fuel ash are also widely used especially in road construction. Section 5.4 explains in more detail the properties of slag bound material.

With regard to Chapter 5, the principle objectives of this part of the study were:

- To investigate the possibility of using tin slag as a binder.
- To assess the possibility for tin slag to be used in slag bound materials and/or cement bound materials.
- To establish an appropriate laboratory methodology and test to identify the pozzolanic properties of the materials tested.

Thus, to achieve the above objectives, the first step was to determine the pozzolanic properties of tin slag. The preferred use of tin slag may be dictated by whether it can be shown that tin slag has a significant pozzolanic property or not.

## 5.2 HYDRAULIC BINDERS

There were two types of hydraulic binders considered in this part of the investigation, namely:

- Primary binder (conventional binder) – a Portland cement and lime were used in this research.
- Secondary binder – ground tin slag was investigated as an option.

### 5.2.1 Primary Binder (Conventional Binder)

Generally, the raw materials used to produce Portland cement are calcium carbonate, found in the form of limestone or chalk and alumina silica and iron oxide normally found combined in clay or shale. The mixture, with proportioned amount of raw materials, is calcined in a kiln at about 1450$^{o}$C, where the components of lime and clay combine to form clinkers composed of tricalcium silicate ($3CaO.SiO_2$), dicalcium silicate ($2CaO.SiO_2$), tricalcium aluminate ($3CaO.Al_2O_3$) and calcium aluminoferrite ($4CaO.Al_2O_3Fe_2O_3$). The behaviour of each type of cement depends on the proportion of these materials. The burnt clinkers are then allowed to cool and taken to a ball mill where they are ground to a fine powder. The fineness of the cement has an influence on the rate of hydration. Greater fineness increases the surface availability for hydration, causing early strength and more rapid heat generation. Gypsum is also added during the grinding process. Gypsum acts as a regulator of the initial rate of hydration of Portland cement. Portland cement possesses hydraulic properties and so it can react with water, hydrating to form cementitious compounds being a complex combination of silicate, aluminate and aluminosilicate hydrates. A cement bound mixture was tested in this study and is described in Section 5.7.

Lime or quicklime (CaO) is produced when limestone, which is primarily composed of calcium carbonate ($CaCO_3$), is heated at temperature more than 900$^o$C. This calcining process will convert the calcium carbonate into calcium oxide. The product of this process is quicklime. On the other hand, hydrated (or slaked) lime is produced by reacting quicklime with water in continuous hydrators where all calcium oxide will be converted to calcium hydroxide [$Ca(OH)_2$]. Lime is used to aid in the modification and stabilisation of soil in road construction. Using lime can substantially increase the stability, impermeability and load bearing capacity of the subgrade. Modification of soils occurs when calcium cations supplied by the hydrated lime replace the cations normally present on the clay surface promoted by the high pH environment of the lime-water system. Thus, the clay surface mineralogy is altered producing the following benefits: reduction of plasticity, reduction in moisture-holding capacity (drying), swelling reduction, improved stability and the ability to construct a solid working platform (NLA, 2001).

### 5.2.2 Secondary binder

A secondary binder is a hydraulic binder which contains material with pozzolanic properties. Hydrated calcium silicate, a source of calcium oxide is required as an activator for secondary binders to form high strength. Lime is commonly used as an activator for pozzolonic activity. Some examples of secondary binders include pulverised fuel ash, granulated blast furnace slag and cement kiln dust. Pulverised fuel ash is a pozzolanic material and when in combination with lime acts as a hydraulic binder. Section 5.4

discusses slag bound materials in more detail.

## 5.3 CEMENT BOUND MATERIALS

Cement has been used for over a century as a cost-effective construction material. The United Kingdom's Highways Agency (2002) divides cement bound materials into seven categories; CBM1, CBM1A, CBM2, CBM2A, CBM3, CBM4 and CBM5. The only differences between these categories are the range of aggregate sizes used and the minimum seven days cube compressive strength. In this chapter, only CBM1 and CBM2 (more lightly cemented materials) were thoroughly investigated. According to the specification for Highway Works, the CBM1 and CBM1A categories should have gradings as shown in Figure 5.1. However, the Highway Agency does not specify what type of material should be used for these categories. For cement bound material category CBM2 and CBM2A the material shall be made from gravel-sand, washed or processed granular material, crushed concrete, recycled aggregate, crushed rock, all-in aggregate, blastfurnace slag or any combination of these. Nevertheless, these materials shall fall within the grading limits as shown in Figure 5.1.

In the United Kingdom, cement bound materials shall satisfy the strength requirements that are specified in the manual of Contract Documents for Highway Works (Highways Agency, 2002) before they can be laid. BS 1924-2 and BS EN 13286-41 detail the procedures for determining compressive strength of stabilised materials or hydraulically bound materials. The cement bound material categories CBM1 and CBM2 shall have compressive strength values not less than those indicated in Table 5.1.

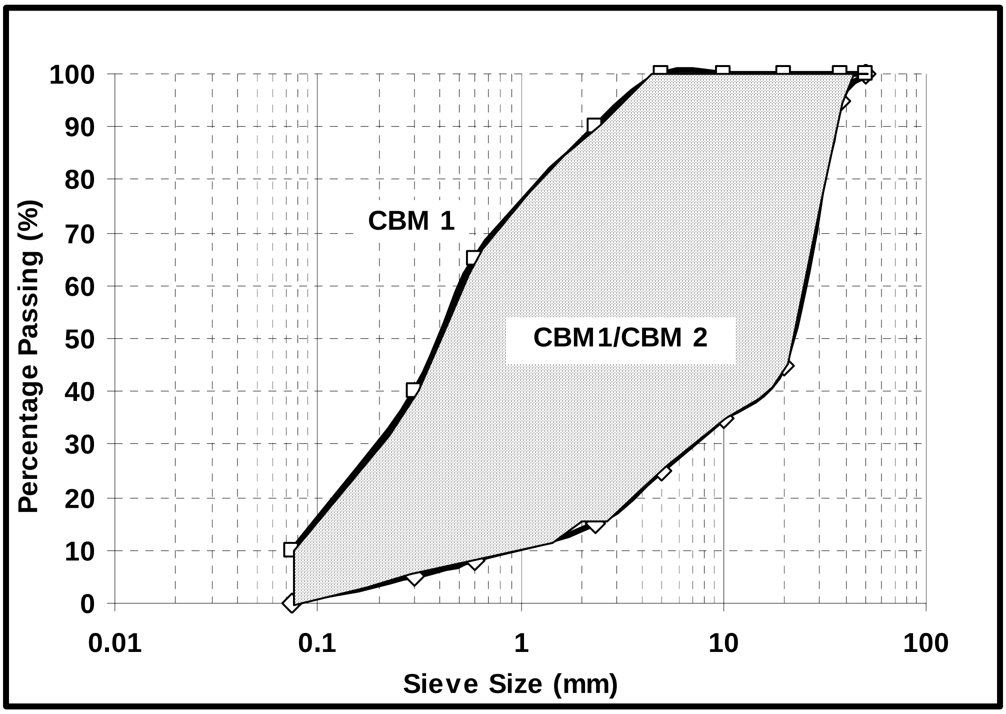



**Figure 5.1: Particle size distribution for CBM1 and CBM 2 (after DETR, 1999)**

**Table 5.1: Compressive strength requirement for Cement Bound Materials (Highways Agency, 2002)**

| Category | Compressive Strength at 7 days ($N/mm^2$) | |
|---|---|---|
| | Average | Individual |
| CBM1 | 4.5 | 2.5 |
| CBM1A | 10.0 | 6.5 |
| CBM2 | 7.0 | 4.5 |
| CBM2A | 10.0 | 6.5 |

In addition to the above requirements, an immersion test should be carried out on cement bound materials to assess their durability. The immersion test is used to detect whether the aggregate used in the cement bound material is suspected to contain sulfate/sulfides, expansive clay minerals or other deleterious constituents. These materials may have a disruptive effect when water comes into contact with the cement-bound layer. The procedure described in BS 1924-2 can be used for determining the effect of immersion in water on the compressive strength of cement bound materials. The cement bound materials shall be regarded as sound provided that the specimens do not lose more than 20% of their unconfined average compressive strength. The specimens shall also not show any signs of cracking or swelling after an immersion period of seven days.

Although cement bound materials have been used for many years, reflective cracking is still one of the problems facing cement bound materials (Walsh and Williams, 2002). This problem is due to overnight temperature changes in the first day or so after laying, causing tensile stresses in the outer layer. It is not possible to reduce the strength of cement bound materials sufficiently to prevent this from occurring.

## 5.4 SLAG BOUND MIXTURES

The uses of slag bound materials as alternatives to cement bound materials and/or bitumen bound materials are not new. Slag bound material (or 'Grave-Laitier' in French) was developed in France by LCPC and Laitier Nord (Dunkirk) in the early 1970's (Robinson, 1999). Between 1972 and 1992, approximately 10,000 km of the French road network was strengthened using hydraulically bound materials of which 65% was composed of grave-laitier (Sherwood, 2001).

In the UK, the use of slag bound mixtures was introduced in Kent in 1987 where the first full scale trial was laid near Pembury. In this case, instead of using air-cooled blast furnace slag as the coarse component, phosphorus slag was used. A blend of 85% phosphoric slag mixed with 15% granulated blast furnace slag was used as a road base material (Walsh, 1999). The trial in Kent has shown that hydraulically bound mixture can be used in sub-base and road base layers.

Studies of slag-bound mixture-design have been carried out by several researchers.

Researchers at SETRA (1980) in France and further work performed by Elliot (1995), Nunes (1997) and Atkinson et al (1999) have shown the successful possibilities for slag-bound mixture design. At present there are no guidelines or standards for the constituent materials and no specific mixture formulae to be followed, so the potential number of mixtures to be considered is immense.

The methodologies proposed by the above researchers for the study of treated secondary material were based on short and long-term structural capacity, immediate stability to enable initial trafficking, and slow setting times to increase the flexibility during laying and compacting the layers. Also, practical proportions of the constituents for successful mixing were taken into account. The formulations proposed by SETRA (1980) and Atkinson et al (1999) are shown in Tables 5.2 and 5.3 respectively. Atkinson et al (1999) studied both laboratory and field performance. They found that all the mixtures listed in Table 5.3 can be considered to be equally adequate for use in pavement construction except mixture number 5 as there was some concern over its durability.

**Table 5.2: Formulation of mixtures proposed by SETRA [1980]**

| Components (%) | Stabilised Materials | | | |
|---|---|---|---|---|
| | Cement-bound courses | Slag-bound courses | Fly ash-lime-bound courses | Pozzolana-lime-bound courses |
| Aggregate | 95.5 to 96.5 | 80 to 85 | 85 to 90 | 85 |
| Cement or hydraulic fly ash | 3.5 to 4.5 | | | |
| Granulated or pelletised slag | | 15 to 20 | | |
| Fly ash | | | 8 to 12 | |
| Pozzolans | | | | 12 |
| Lime | | 1 | 2 to 3 | 3 |
| Water | 4 to 7 | 6 to 9 | 4 to 9 | 6 to 9 |

**Table 5.3: Selected mixtures proposed by Atkinson et al (1999)**

| Mixture | Aggregate/binders | Proportions (%) |
|---|---|---|
| 1 | Fa/C | Fa 93%, C 7% |
| 2 | Fa/CdC | Fa 90%, Cd 7%, C 3% |
| 3 | Fa/GyL | Fa 91%, Gy 5%, L 4% |
| 4 | Fa/CdGs | Fa 70%, Cd 20%, Gs 10% |
| 5 | Gy/FaC | Gy 82%, Fa 10%, C 8% |
| 6 | Cc/C | Cc 93%, C 7% |
| 7 | Cc/CdC | Cc 90%, Cd 7%, C 3% |
| 8 | Cc/FaL+ | Cc 80%, Fa 15.5%, L 4%, + 0.5% |
| 9 | Cc/GgsLGy | Cc 92.5%, Ggs 6%, L 1%, Gy 0.5% |
| 10 | T1/C | T1 96%, C 4% |
| 11 | T1/FaLGy | T1 92%, Fa 6%, L 1.5%, Gy 0.5% |
| 12 | Bs/GsL | Bs 84%, Gs 15%, L 1% |
| 13 | Bs/GsSs | Bs 70%, Gs 20%, Ss 10% |

Fa = Pulverised fuel ash, C = cement, Cd = Cement kiln depth, L = lime, Cc = China clay sand, Gs = Granulated blast furnace slag, Ggs = Ground granulated blast furnace slag, Bs = Blast furnace slag, Ss = steel slag, T1 = conventional crushed aggregate.

The use of slag bound mixtures offers considerable advantages over cement bound materials as construction materials as described below (Robinson, 1999; Sherwood, 2001):

- Improved resistance to reflective cracking compared to cement bound materials which also allows a comparative reduction in asphalt thickness.
- Improved resistance to frost heave compared to granular sub-base.
- Less segregation compared to unbound materials.
- Excellent for working under wet/wintry conditions. In the case of heavy rain, excess water is simply allowed to drain off before proceeding with compaction. If necessary, materials may be spread, allowed to dry out and than compacted.
- Particularly suited to a weak sub-grade forming a strong bond and adapting to the shape of the sub-grade as it deforms under traffic.
- Remains useable for up to four days after manufacture compared to cement bound materials which must be placed within 2 hours of production.

- Take a relatively long time to set allowing for several days of storage without difficulty. It also allows flexible organization of the road works, each machine operating individually and at its maximum output.
- A relatively large quantity of granulated slag facilitates a homogeneous distribution of the binder in the mass. Part of the slag remains available, enabling a renewed setting (self-healing) should cracking occur.
- Road-works equipment can be allowed to circulate over the grave-laitier as soon as it is laid. Post-compaction due to traffic is good. The material is suitable for strengthening purposes while traffic is maintained.
- Although the hardening process is halted under frost, the hardening recommences once normal temperatures are reached.
- The strength is not affected by an initial delay in hardening because it takes a long time to build up fully (one year or longer).
- The slow rate of hardening allows the moduli of the grave-laitier layers to increase progressively with the consolidation of the subgrade and with increasing traffic.
- Requires less energy to manufacture compared to asphalt and concrete.

The strengths obtained from grave-laitier are only half those that would be obtained if Portland cement were used (Sherwood, 2001). However, when used as sub-base slag bound mixtures reduce the whole life pavement cost by extending the period to reconstruction or they allow the asphalt overlay thickness to be reduced hence reducing the initial construction cost.

## 5.5 THE EFFECTS OF BOUND MIXTURES

### 5.5.1 Improvement in Mechanical Performance

Nunes (1997) and Atkinson et al (1999) have studied the effect of slag bound mixtures on mechanical performance. They tested slag bound mixtures for a range of properties at different curing times. The compressive strength was measured at 7 days for short-term trafficability. To assess long-term performance, the tensile strength, stiffness modulus, resistance to fatigue and durability properties were measured. They found that the slag bound materials performed well enough to make them conceivable road construction materials.

Richardson and Haynes (2000) reported that the typical properties of slag bound materials in the hardened state fall within the following ranges:

- Compressive strength: 6-10MPa
- Tensile strength: 0.6-1.5MPa
- Elastic modulus: 15-20GPa.

They also reported that a study using a falling Weight Deflector indicated that the performance of the slag bound materials after about one year in service were comparable with those of heavy duty macadam asphalt bases of similar thickness and position in the pavement construction.

### 5.5.2 Containment of Contaminants

In addition to improvements in mechanical properties, hydraulically bound materials are also important as they have the ability to contain contaminants and minimise their release from wastes or alternative materials when used in the mixtures. Leaching can be minimized through both physical and mechanical mechanisms. The binder in the hydraulically bound materials can coat the surface of the contaminated materials resulting in a reduction of direct contact between the materials and leachant (e.g. water) and can also reduce the overall permeability, thus reducing the rate of leaching of contaminants.

The pH and redox potential are two of the parameters that control the leaching process. Cement and pozzolan based binders work at high pH. Generally, the leaching processes slow at high pH values. Therefore, leaching of contaminants from hydraulically bound materials can be minimized by using a slag binder.

## 5.6 POZZOLANIC PROPERTIES OF TIN SLAG

Pozzolans are materials containing reactive silica and/or alumina which on their own have little or no binding property but when mixed with lime in the presence of water will set and harden like cement. Nowadays, a wide variety of siliceous or aluminous materials are used for producing pozzolanas, the common materials being pulverised fuel ash, volcanic ash and ash from agricultural residues such as rice husks. The chemical composition of pozzolans varies considerably, depending on the source and the preparation technique. Generally, a pozzolana will contain silica, alumina, iron oxide and a variety of oxides and alkalis, each in varying degrees. It is also generally agreed that although the chemical content of a raw material will determine whether or not it is pozzolanic and will react when mixed with lime or ordinary Portland cement, the degree of reaction and subsequent strength of the hydrated mixture cannot be accurately deduced from just the chemical composition (except for a small number of known pozzolanas (ITDG, 2003)). In most cases no direct correlation can be found between chemical content and reactivity.

Other characteristics of the pozzolan also affect its reactivity, such as fineness and crystalline structure. It is also argued that because pozzolanas are used for a variety of different applications, such as in mortars, concretes, block manufacture, etc., and mixed with a variety of other materials such as lime, ordinary Portland cement, sand, etc., (which can also radically affect the reaction of the pozzolan), then perhaps it is better to develop a test to determine the desired properties of the mixture in the context for which it is intended. This provides valuable information for specific project applications and can also help determine the general characteristics of a pozzolan for cases where the application of the pozzolan is not specified.

Tests for pozzolanic materials are required for a number of reasons;

- To assess the viability of a new potential pozzolanic deposit
- To provide quality control on a day-to-day basis as part of a production process
- To provide long term quality control of the pozzolanic resource

From the above explanation, thus the principle objectives of this part of the investigation can be derived as follows:

- To determine the presence of pozzolan in the tin slag.
- To propose suitable binder treatment methods that can make tin slag work.

## 5.6.1 Testing Programme

### 5.6.1.1 Mixture design

To obtain an understanding of tin slag's pozzolanic properties, it is necessary to investigate a range of mixtures. There were four mixture compositions used in this study (Table 5.4). The first mixture was a combination of 80% sand and 20% ground tin slag. The other three mixtures were combination of 80% sand, 16% ground tin slag and 4% cement; 80% sand, 16% ground tin slag and 4% lime; and 80% sand, 16% limestone filler and 4% cement. The component's proportions in the mixtures were selected based mainly on known successful mixtures of other slags as studied by Elliot (1995), Nunes (1997) and Atkinson et al (1999).

**Table 5.4: Mixtures composition for determination of pozzolanic properties of tin slag**

| Mixture | Aggregate (Sand) | Binder content (%) | | | |
|---|---|---|---|---|---|
| | | Ground Tin slag | Cement | Lime | Limestone Filler |
| S/T | 80 | 20 | | | |
| S/T/C | 80 | 16 | 4 | | |
| S/T/L | 80 | 16 | | 4 | |
| S/LF/C | 80 | | 4 | | 16 |

The first mixture, comprising 20% ground tin slag, was selected on the basis that ground tin slag was believed to have pozzolanic properties. Lime was used in combination with ground tin slag in the third mixture, to determine whether tin slag does possess hydraulic properties that can be activated by lime. In this case lime was as a potential activator of the ground tin slag. If the mix was found to be pozzolanic, it could have been used as secondary binder in a further study. Limestone filler is known as a non-reactive material in terms of hydraulic properties. For this study's purposes the limestone filler was used as a control material so that the effect of cement with ground tin slag (second mixture) may be distinguished from the effect of cement alone. So, at the end of this study a comparison can be made between ground tin slag, cement and hydrated lime in terms of their hydraulic properties.

#### 5.6.1.2 Sample Preparation

Fineness is one of the most important properties that controls hydraulic materials. Therefore, before raw tin slag may be used in a hydraulically bound material, its particle size needs to be reduced. In this study, the size of the tin slag was reduced to the particle size of ordinary Portland cement using a ball mill grinder. The literature indicates that cement particles with the size range between 3 and 32µm are optimum for the cement performance and that cement particles larger than 32µm were found to be too large to hydrate completely during the hydration reaction (Cement Australia, 2004). According to Cement Australia, the most common cement particle size was approximately 24µm. Therefore, in this investigation, 24µm was selected as the particle size to which tin slag was ground using a ball mill grinder. The ground tin slag was thus sieved and the material passing 24µm collected.

The specimens were prepared largely in accordance with Draft BS EN 13286-53, "Unbound and hydraulically bound mixtures - Methods for making test specimens Part 53: Making cylindrical specimens by axial compression" (BSI, 2002). However, some modifications were carried out on the mould dimensions. In this study PVC moulds with a 65mm internal diameter and 150mm height were used. To make a mixture, 350g of combined materials was placed in a mixing pan and thoroughly dry blended by hand. The required volume of water was then added and manual mixing was carried out until the materials achieved a uniform consistency. The specimen was then compacted under a static (press) load (see Figure 5.2). This method of compaction was selected because all the materials used in this study of pozzolanic properties were very fine (i.e. less than 24μm) except sand which has size between 0.075mm and 1.18mm. Also, the fine ground tin slag stock was very limited. It should be noted that the ground tin slag with size 24μm was produced manually by hand, therefore the process was very time consuming.

Prior to mix compaction, the maximum load that the PVC mould could tolerate during compaction of the specimen had to be established. First, a specimen with about 10% water was placed into the mould. A maximum load of 16kN was then introduced in 4 increments where each step was 4kN using an Instron hydraulic loading machine. The loading rate was 200kN/s. The load was held for 10 seconds for each step except for the peak load (i.e. last step) which was held for 1 minute. It was found that the PVC moulds could easily tolerate the 16kN load. The procedure was repeated using a maximum load value of 24kN with 4 steps where each step was 6kN. It was found that the mould could still tolerate the given load. Finally, a 32kN load was applied in four 8kN increments. At this load level, the mould was found to deform or bulge in the middle. Thus, it was decided that the most suitable and safe maximum load for compaction was 24kN.

The following were some of the problems faced whilst producing the specimens;

- The time required to grind and sieve the tin slag was very long. Therefore, the amount of ground tin slag produced was very limited.
- Long specimens could not be practically produced with the compaction moulds used in this investigation. The plunger used for compaction tended to move slightly off centre when the load was applied.

Due to the problems listed above, the resultant compacted specimens being produced were relatively small and short (see Figure 5.3).

#### 5.6.1.3 Compactibility Test

It was necessary to conduct compactibility tests on the specimens to evaluate the potential availability of the pozzolanic property in tin slag. The compactibility test is used to determine the optimum moisture content and dry density for each of the mixtures. The determination of the optimum moisture content was performed in accordance with BS 1924-2 (BSI, 1990). However, some changes were made where an axial press was used for compaction instead of using a vibrating hammer. The load of 24kN was used for compaction of the specimens as discussed above. Compactibility test were performed on all four mixtures. After compaction, the specimens were extruded from the mould, weighed and then the height and diameter were measured. The specimens were then placed on a tray and dried in an oven at a temperature of 105$^{o}$C to determine moisture content. The bulk density and dry density values for each mix at each moisture content were calculated.

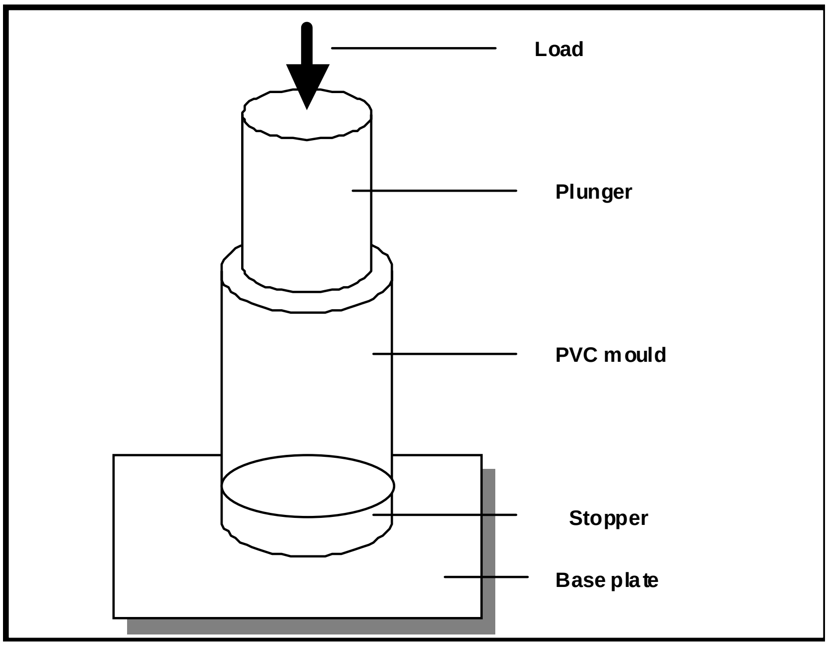



**Figure 5.2: Schematic diagram of developed compaction mould (not to scale)**

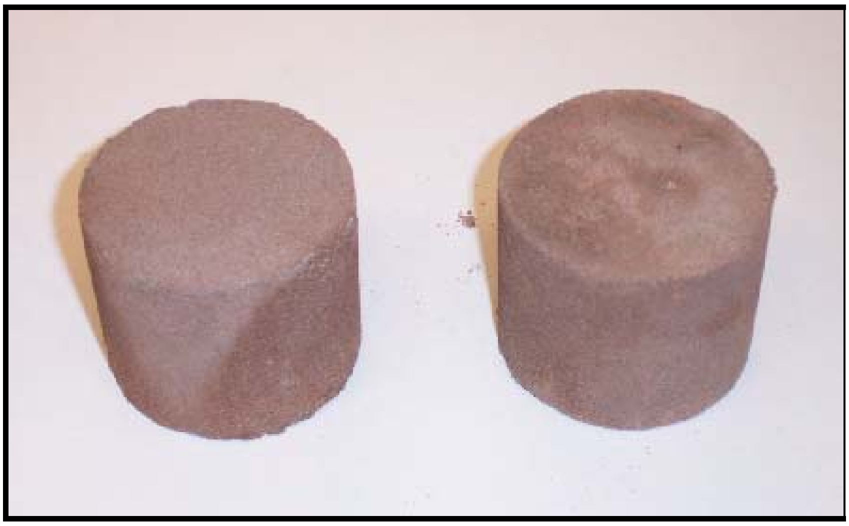


**Figure 5.3: Specimens produced for the pozzolanic property determination of tin slag**

### 5.6.1.4 Compressive Strength Test

Determination of the compressive strengths of the cylindrical specimens was performed in accordance with BS EN 13286-41 - test method for determination of the compressive strength of hydraulically bound mixtures (BSI, 2003). However, the height of a typical specimen produced following compaction was around 58mm, which was less than the specimen diameter (65mm). It was not possible to produce specimens with the ideal heights, i.e. two times the diameter, because of the limited amounts of ground tin slag and the problems with the plunger during compaction. It may be argued that the specimen height to diameter ratio was inadequate for the compressive test to be conducted. However, in this investigation the compressive strengths of tin slag specimens were directly compared with control specimens which were produced in the same way and having the same dimensions.

After compaction the specimens were stored for curing according to the following procedure:

- Immediately after compaction the specimens were extruded from the moulds.
- The specimens were then weighed and the heights and diameters measured.
- The specimens were then carefully sealed using plastic membranes to avoid the loss

of moisture. This is essential for strength development.

- Sealed specimens were then stored in a temperature-controlled room at a constant temperature of 20$^{o}$C.

At the end of the curing period the specimens were placed centrally on the lower platen of the compression-testing machine, i.e. Instron machine. The force was applied in a continuous and uniform manner without shock at a deformation rate of 1mm/min (see Figure 5.4). The compressive strengths ($R_c$, in MPa) were calculated from the crushing force and cross-sectional area of the cylinder using the following equation:

$$R_c = \frac{F}{A_c} \qquad \ldots(5.2)$$

where

$R_c$ is the compressive strength of the specimen in N/mm$^2$ (MPa)

$F$ is the maximum-recorded load in N

$A_c$ is the cross-sectional area of the specimen in mm$^2$

**Figure 5.4: Performance test for determination of pozzolanic properties of tin slag**

### 5.6.2 Results and Discussion

#### 5.6.2.1 Compactibility Test

The Compactibility test is used to determine the optimum moisture content (OMC) and maximum dry density. During the compaction process the solid particles are packed more closely together, thereby increasing the dry density of the material tested. The dry density which can be achieved depends on the degree of compaction applied and on the amount of water present in the material. For a given degree of compaction, there is an optimum moisture content at which the dry density obtained reaches a maximum value (see Figure 5.5). Ideally, the range of moisture contents chosen for each mixture should include the estimated OMC and two values on either side of this.

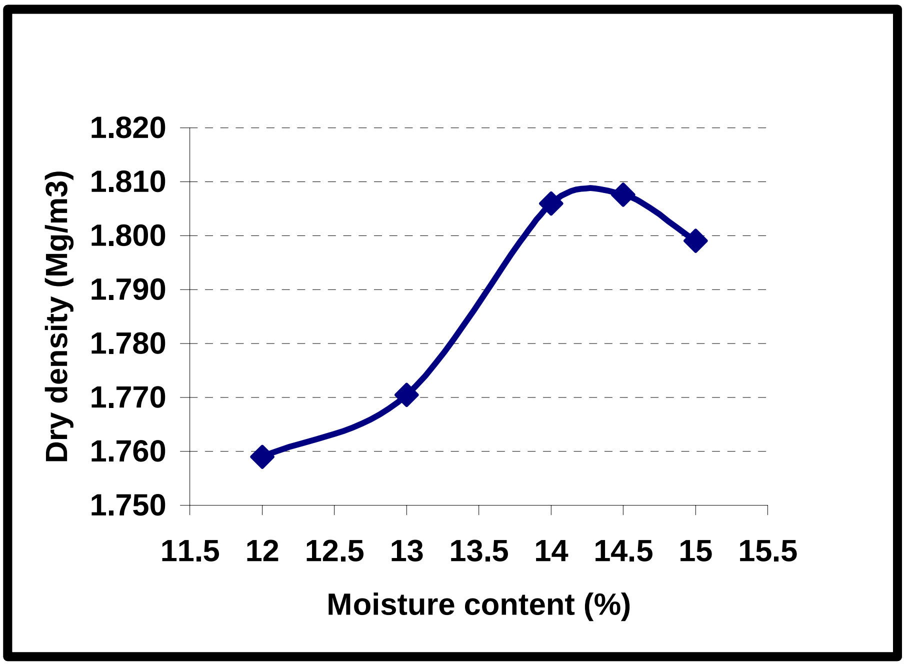



**Figure 5.5: Typical compaction profile**

The results obtained from the compactibility tests on the ground tin slag mixtures are shown in Figure 5.5, Figure 5.6 and Table 5.5. Figure 5.5 shows an example of a typical curve defining optimum moisture content and dry density of a mixture. It is very clearly shown that the compaction curve reaches a peak defining the optimum moisture content and maximum dry density values. The compaction curves for all the mixtures are shown in Figure 5.6. Most of the mixtures have a dry density value less than 1.811Mg/m$^3$, except S/LF/C (sand/limestone filler/cement) mixture which has 1.830Mg/m$^3$. The optimum moisture content and maximum dry density values for all mixtures are presented in Table 5.5. There were no difficulties encountered during compaction of the mixtures. However, extra precaution had to be taken when extruding the specimens from the mould because the specimens were still in a weak fragile condition.

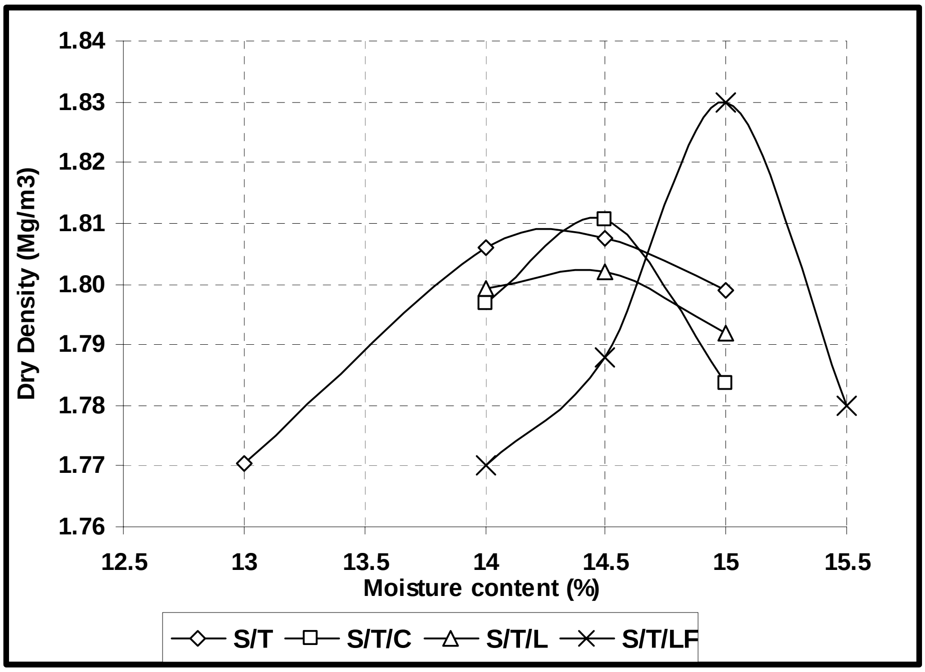



**Figure 5.6: Compaction curves for all mixtures**

**Table 5.5: Results of compactibility test**

| Mixture | Aggregate (Sand) | Binder content (%) | | | | Compactibility Test | |
|---|---|---|---|---|---|---|---|
| | | Tin slag | Cement | Lime | Limestone Filler | OMC (%) | MDD (Mg/m$^3$) |
| S/T | 80 | 20 | | | | 14.5 | 1.808 |
| S/T/C | 80 | 16 | 4 | | | 14.5 | 1.811 |
| S/T/L | 80 | 16 | | 4 | | 14.5 | 1.802 |
| S/LF/C | 80 | | 4 | | 16 | 15 | 1.830 |

Note: S/T - mixture sand and ground tin slag
S/T/C - mixture sand, ground tin slag and cement
S/T/L - mixture sand, ground tin slag and hydrated lime
S/LF/C - mixture sand, ground tin slag and limestone filler

### 5.6.2.2 Compressive Strength

Three specimens were used to determine compressive strength for each mixture. The results obtained from the compressive strength tests are presented in Table 5.6, Figures 5.7 and 5.8 as a function of time for each mixture. Figure 5.7 shows the compressive strength development process of the mixtures. The investigation shows that there is little difference in terms of the strength development in mixtures S/T and S/T/L between 28 days, 90 days and 365 days. This indicates that mixtures S/T and S/T/L do not show any sign of either slow early strength development (typical of other slag bound materials, even after one year) or fast strength development as measured for mixture S/T/C.

In order to fully understand the pozzolanic properties of tin slag, the graph in Figure 5.8 was drawn. In the graph, the comparative analysis of the compressive strength can be made between the mixtures. It shows that the sand/slag mixture had the lowest strength at all curing stages up to one year, while the sand/slag/cement mixture had the highest strength. The sand/limestone filler/cement mixture yielded a similar result to the sand/slag/cement mixture while the other two mixtures, without cement, were weaker. Therefore, the compressive strength results indicated that cement is responsible for the majority of the strength gain. Slag may be responsible for continued strength gain between 90 and 365 days (see the 365 days result for S/T/C) but, otherwise the slag does not appear effective. The addition of lime helps develop a little early strength, but not in the long-term. Thus the effect of lime may be due to better mechanical interlock when lime fines are present. The laboratory tests clearly indicate that the compressive strength in the sand/slag/cement and sand/slag/lime mixtures do not originate from the reaction between ground tin slag and cement or hydrated lime since there is minimum pozzolanic reaction in sand/slag mixture.

From the results obtained, it was possible to draw the following conclusions:

- Tin slag can be considered to have, at best a modest pozzolanic property.
- Tin slag has little potential to be applied in slag bound material.
- Tin slag may be utilised in cement bound materials.

**Table 5.6: Compressive strength test results with 95% confidence limits.**

| Mixture | Compressive Strength (N/mm2) | | |
|---|---|---|---|
| | 28 days | 90 days | 365 days |
| S/T | 0.028±0.004 | 0.09±0.03 | 0.04±0.004 |
| S/T/C | 1.115±0.24 | 1.10±0.15 | 1.94±0.65 |
| S/T/L | 0.193±0.056 | 0.24±0.05 | 0.18±0.03 |
| S/LF/C | 1.361±0.26 | 1.47±0.36 | - |

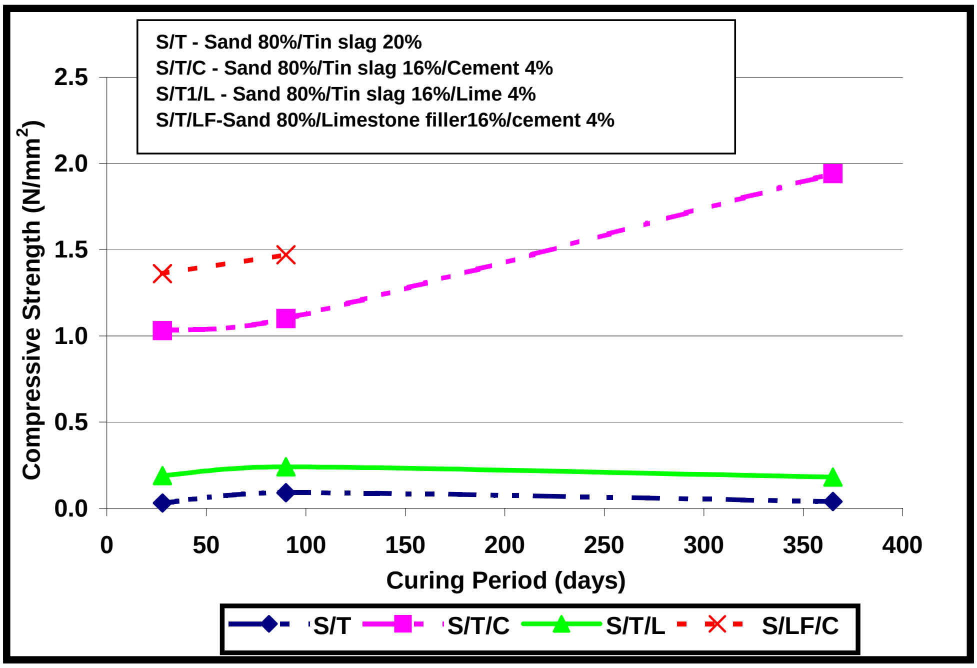



**Figure 5.7: Variation in compressive strength values of the mixtures with times**

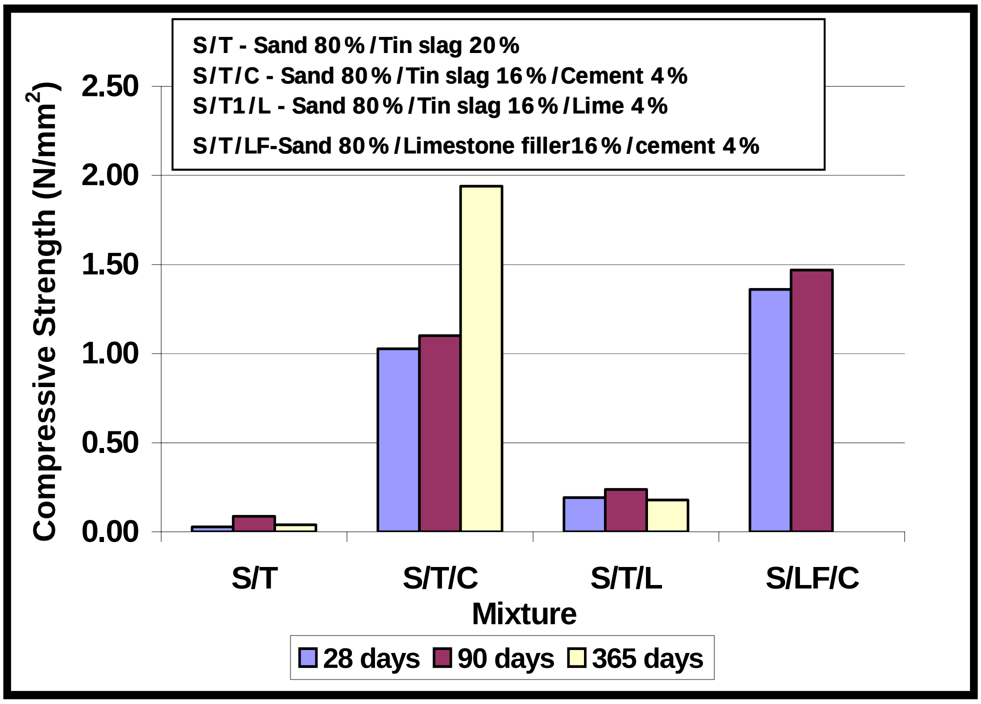



**Figure 5.8: Compressive strength test results**

## 5.7 MIX DESIGN FOR CEMENT BOUND MATERIALS

The previous study in Section 5.6 has shown that the tin slag has no potential to be used in slag bound materials. It was suggested that tin slag could be more effectively used in cement bound materials. Therefore, the primary objective of this part of study was to assess the possibility of using tin slag as part of a cement bound material. The secondary objective includes assessment of the durability of tin slag in cement bound materials.

The bearing capacity or the strength of fully cured cement bound materials is highly dependent upon cement content and dry density. For constant cement and water contents, the bearing capacity or strength increases as the dry density increases and for constant dry density and water content, the bearing capacity or strength increases as the cement content increases (Kennedy, J. and Hopkins, M., 2000). This strength is dependent upon dry density which is in turn dependent upon moisture content. Hence, the main objective of the first stage of a mix design procedure for cement bound materials is to determine the correct water content for compaction of the material and the cement content that is required at that moisture content to satisfy the required mechanical performance. When designing cement-bound materials, 2% extra moisture is routinely added on top of the optimum moisture content defined by the compaction to ensure complete hydration (Kennedy, J. and Hopkins, M., 2000).

### 5.7.1 Testing Programmes

#### 5.7.1.1 Gradation

Aggregate gradation is a very important characteristic for stabilisation and affects the type and amount of binder required. Particle size and distribution also affects the main properties of mixture design such as mechanical strength and durability.

For the purpose of this assessment two gradations were used. For the first mixture, a tin slag cement bound mixture, there were no alterations carried out on the tin slag gradation, which meant the tin slag was used as received. Figure 5.9 shows the gradation of the tin slag compared to the gradation limits proposed in the Specification for Highway Works (1998) for CBM2. The gradation shows that tin slag has almost fully complied with the CBM2 gradation specification except for the proportion of material falling slightly below the limits at 0.5mm.

The second mixture investigated was a cement-bound tin slag/sand mixture. For this mixture, the original tin slag gradation was altered and sand with particle size between 1.18mm and 0.075mm was added at a ratio of 20% by weight (see Figure 5.9).

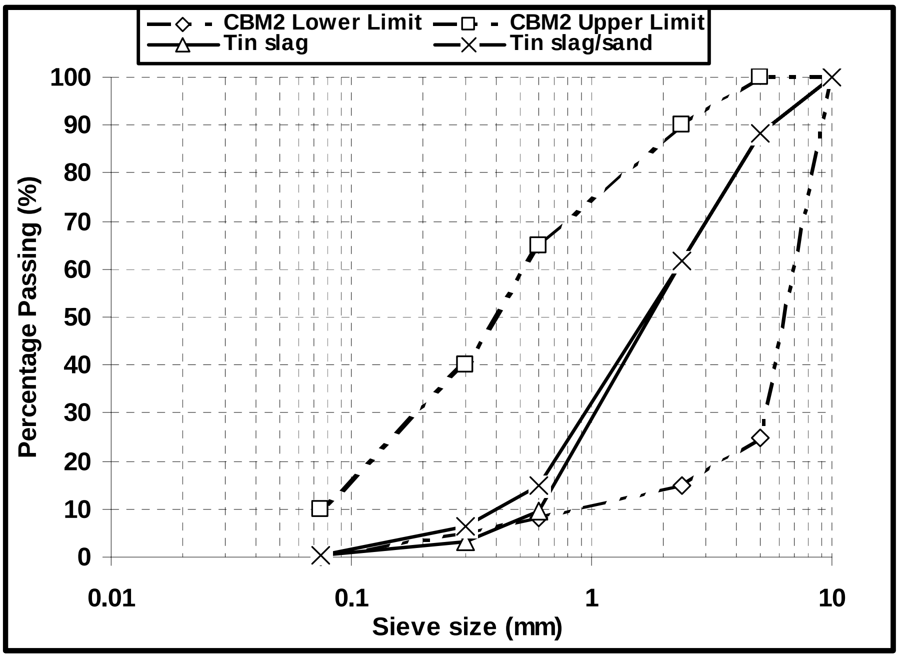



**Figure 5.9: Gradation of cement-bound tin slag and tin slag/sand mixtures**

### 5.7.1.2 Mix Design

The decision to add sand in the second mixture was based on a preliminary investigation which showed that the strength of the cement bound material increased when sand was added. The amount of cement was chosen (see Table 5.7) to bracket the estimated optimal treatment (high strength but modest cement addition). Less cement was added to the 20% sand mixtures due to the strength gain likely to result from using sand.

**Table 5.7: Combination of material for cement bound material mix design**

| Mixture Code | Percentage of Material by weight | | |
|---|---|---|---|
| | Tin slag | Sand | Cement |
| T4C | 96 | | 4 |
| T7C | 93 | | 7 |
| T10C | 90 | | 10 |
| T/S3C | 77 | 20 | 3 |
| T/S6C | 74 | 20 | 6 |
| T/S9C | 71 | 20 | 9 |

### 5.7.1.3 Sample Preparation

Dry tin slag specimens were prepared according to the methodology proposed by Nunes et. al. (1996). The standard compaction rig (BS 5853) was used for compaction and a specially built mould was used for this purpose (see Figures 5.10, 5.11 and 5.12). The outer mould was made from steel while the specimen mould was made from plastic (PVC) with size 150mm internal diameter and 70mm height. There were several benefits of using plastic moulds. The moulds enabled the specimens to cure for a sufficient time for strength to develop without removal from the moulds. The plastic moulds were also relatively inexpensive and were easy to manufacture in large numbers. It was also found that the plastic moulds had relatively low adherence to the specimen because of the smoothness of the internal mould surfaces and the absence of corners where the material tends to get trapped. Plastic moulds also do not corrode thus making it easy to extrude the cured specimens from the moulds.

The following methodology was adopted for curing the specimens in order to make the specimens suitable for testing as stipulated in BS 1924-2 (BSI, 1990).

- Immediately after compaction, the specimens, which were kept inside the moulds, were carefully sealed with a plastic membrane to avoid loss of moisture. This was essential for strength development.

- Sealed specimens were stored at a constant temperature of 20±2$^{o}$C for the desired curing period.
- The specimens were extruded from their moulds after seven days curing and were ready for compressive strength testing.

**Figure 5.10: Compaction mould**

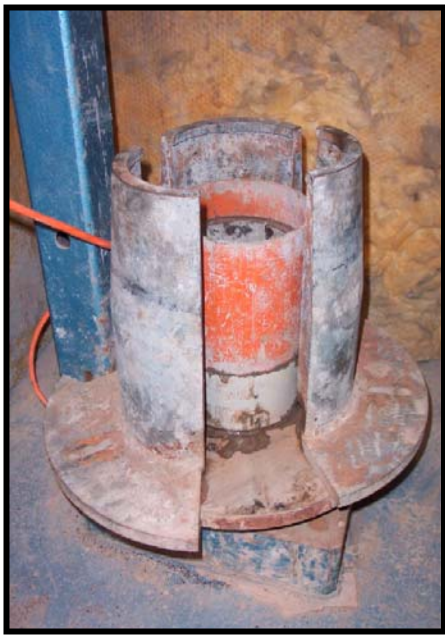


**Figure 5.11: View of inner specimen mould**

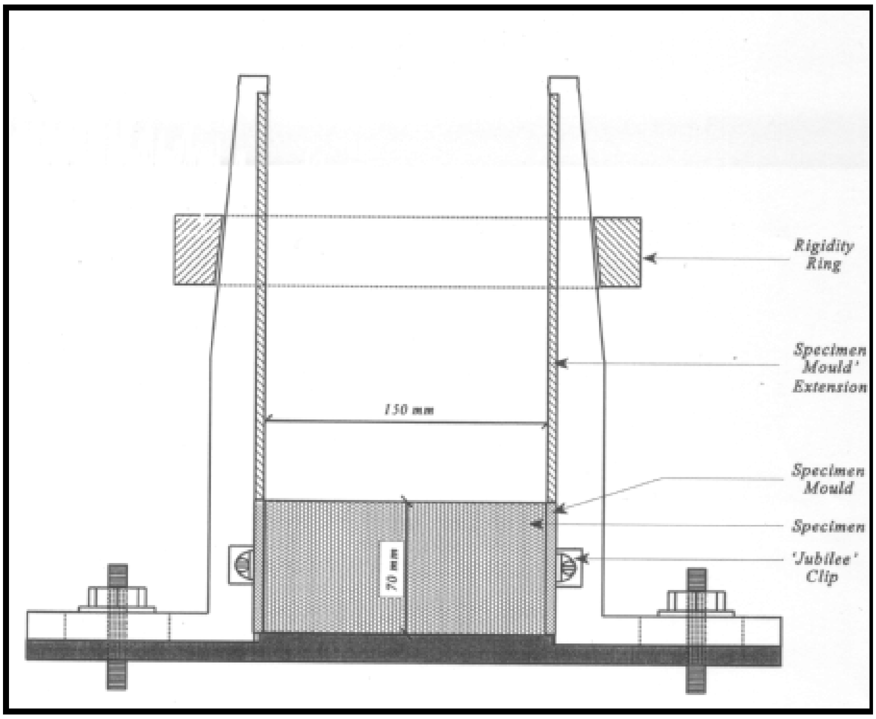



**Figure 5.12: Schematic view of compaction mould (Nunes, 1997)**

### 5.7.1.4 Determination of Optimum Moisture Content (OMC)

In this study, the results for the determination of OMC which were obtained from an earlier part of this investigation on pozzolanic properties as discussed in Section 5.6 were used as a guideline to estimate the OMC of the CBM mixture. Kennedy and Hopkins (2000) also proposed as estimate for the OMC for granular CBM mixtures to lie between 5% and 7% by weight.

It is very important to include cement in the determination of OMC because it constitutes part of the aggregate grading. It also affects the moisture demand and hence the density achieved in the tests. The aggregate, cement and water were mixed until a uniform mixture was produced. The specimens were then transferred into plastic moulds ready for compaction. The materials were compacted in one layer, using a standard vibrating hammer method for a period of three minutes as suggested in BS 1924-2 (1990) but in the equipment as described above.

### 5.7.1.5 Determination of Optimum Cement Content

For the determination of optimum cement content, a range of cement contents was used for each mixture as proposed by Kennedy and Hopkins (2000). The procedure and specimen manufacture depends on whether performance is specified by compressive strength or California Bearing Ratio (CBR). Results from early performance tests on CBM conducted in this study showed that to achieve 4.5MPa in 7 days, the cement content of tin slag mixture was likely to be in the range of 3% to 6% by weight. For the tin slag and sand mixture, the cement content was between 2.5% and 5% by weight. Therefore, three cement contents for each mixture were tested to assist in the determination of optimum cement content.

Water contents corresponding to the OMC of each mixture listed in Table 5.7 were used for compactibility. The specimens, which consisted of cement, aggregate (tin slag or tin slag with sand) and water, were mixed together until they were homogeneous. The specimens were then compacted to refusal using vibratory compactor in accordance with BS 1924-2 as before. The specimens were then cured for seven days as described in Section 5.6.1.4. After seven days of curing the specimens were crushed to determine the compressive strength as described in Section 5.6.1.4. Using the average of each set of results, a graph of compressive strength against cement content was plotted. The cement content of the specimen, which achieved 4.5N/mm$^2$ compressive strength, was then selected.

### 5.7.1.6 Durability Test

The durability tests were performed in accordance with BS 1924-2. For the purpose of durability assessment, a selection from the above mixtures was made based on the following criteria:

- Strength of the mixture
- Cost of constituents

With regard to the durability assessment, two sets of identical specimens from the selected mixtures were prepared. Both sets were cured in the normal manner at constant moisture wrapped in cling film for 7 days as described in Section 5.6.1.4. At the end of 7 days curing, one set (after the cling film was removed) was totally immersed in water at 20$^o$C to a depth of 25mm below the surface of the water for 7 days whilst the other set was left to continue curing at constant humidity wrapped in cling film at 20 ± 2$^o$C for another 7 days. After curing for 14 days, both sets of specimens were tested for compressive strength according to Section 5.6.1.4 and the average of the compressive strength values was calculated. The durability index, $R_i$, which is the strength of the set immersed in water as a percentage of the strength of the set cured at constant moisture content, was then calculated from the Equation 5.3.

$$R_i = \frac{P_i}{P_c} \times 100 \qquad \ldots(5.3)$$

where

$P_i$ is the mean compressive strength of immersed specimens, in N/mm$^2$

$P_c$ is the mean compressive strength of control specimens, in N/mm$^2$

## 5.7.2 Results and Discussion

### 5.7.2.1 Compactibility Test

The results obtained from the compactibility tests are presented in Figures 5.13 and 5.14. The values of optimum moisture content and maximum dry density are well defined. It was observed that the maximum dry density values for all mixtures (tin slag/cement mixture and tin slag/sand/cement mixture) were relatively high compared with the test results with other materials tested by Nunes (1997). It was also found that the higher cement content in the mixture, the higher the maximum dry density value of the mixture. This is likely to be the result of increased particle packing and hence reduced porosity as more cement powder was added. However, there was no significant difference in optimum moisture content values between the mixtures. The high dry density values of the mixtures were most probably due to the high specific gravity of the tin slag (S.G. = 3.52). Table 5.8 shows the compactibility test results for dry density and optimum moisture content values obtained from each mixture.

**Table 5.8: Results of the compactibility tests**

| Mixture Code | Percentage of Material by weight | | | Optimum Moisture Content (%) | Maximum dry density (Mg/m$^3$) |
|---|---|---|---|---|---|
| | Tin slag | Sand | Cement | | |
| T4C | 96 | | 4 | 4.8 | 2.56 |
| T7C | 93 | | 7 | 5.0 | 2.68 |
| T10C | 90 | | 10 | 5.0 | 2.75 |
| T/S3C | 77 | 20 | 3 | 5.0 | 2.63 |
| T/S6C | 74 | 20 | 6 | 5.0 | 2.71 |
| T/S9C | 71 | 20 | 9 | 5.0 | 2.75 |

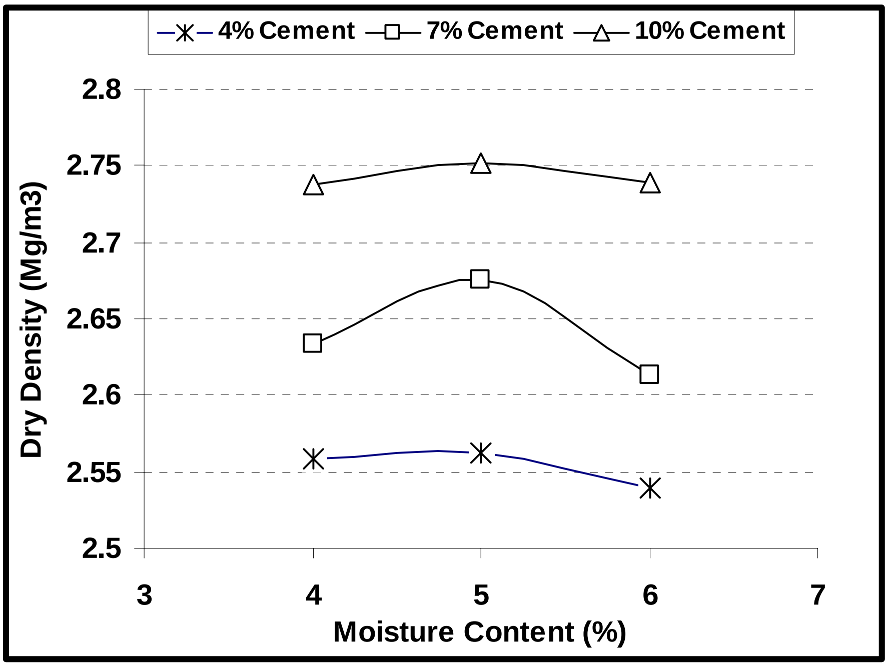



**Figure 5.13: Compaction curve for tin slag cement bound materials**

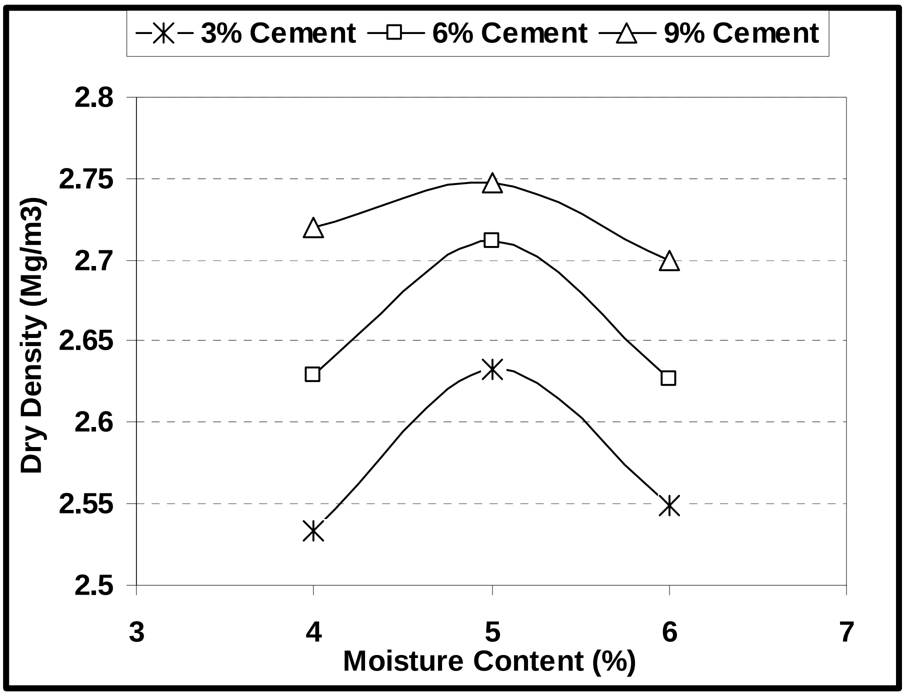



**Figure 5.14: Compaction curve for tin slag/sand cement bound materials**

### 5.7.2.2 Compressive Strength Test

Figures 5.15 and 5.16 show the appearance of the cement bound material with slag only and cement bound material with slag/sand respectively after seven days curing. Physically, there was not much difference between the mixtures except that the cement bound material made with tin slag/sand had a brownish colour due to the sand content.

The compressive strength test results for cement bound tin slag mixtures and cement bound tin slag/sand mixtures are shown in Table 5.9. Three specimens from each mixture were tested and the average compressive strength calculated. It can be clearly seen that the strength of the mixtures increases when cement content increases, as expected. Thus, there is no doubt that cement plays a very important role in the mixture strength.


**Figure 5.15: Cement Bound Material with tin slag and cement only**


**Figure 5.16: Cement Bound Material with tin slag/sand and cement**

The results also show that the cement-bound tin slag/sand mixtures have higher compressive strength values than cement-bound tin slag mixture at the same quantity of cement. The comparison between these two mixtures is made clearer in Figure 5.17. Explanations can be made noting that strength is achieved not only by binding action but also by improved packing, as the fabric of a well-packed specimen is more difficult to disrupt. In particular:

- Tin slag gradation, (Chapter 3) showed that tin slag consists of a very small amount or negligible amount of filler. Therefore, during compaction of the specimens some of the cement will take over as a filler to deliver a better packing. Only the excess cement not used up in packing will act as a binder. Therefore, such an open graded mixture demands more cement for optimum performance.
- In the cement-bound tin slag/sand mixture, the sand used was finely graded, mostly below 1.18mm. The added sand will partially fill the gaps between the tin slag particles. Therefore, most of the cement added in the mixture will act as a binder and not as filler to improve packing.

With regard to Figure 5.17, the main objective in presenting this figure was to determine the cement content requirements for the mixture to be classified as either CBM1 or CBM2 as defined in the Specification for Highway Works (2000). From the curves in Figure 5.17, it was found that a cement-bound tin slag mixture needs 4.6% cement to achieve 4.5MPa compressive strength to be classified as CBM1 and 5% cement to achieve 7MPa compressive strength to be classified as CBM2. On the other hand, the cement-bound tin slag/sand mixture only required 3% cement and 3.4% cement for the same strengths. From the results obtained in this part of the investigation the following conclusions may be drawn:

- Tin slag can be successfully used in cement bound materials either for CBM1 or CBM2.
- The aggregate packing and compressive strength of the mixtures can be increased by adding a certain amount of sand. By this means the cement content of the mixtures can be reduced without any compromise in strength.
- There is a significant advantage for a mixture to include added sand as this can reduce the overall cost of the CBM.

**Table 5.9: Compressive strength test results for tin slag mixture and tin slag/sand mixture with standard deviations**

| Tin slag Mixture | | Compressive Strength (MPa) | Tin slag/sand Mixture | | Compressive strength (MPa) |
|---|---|---|---|---|---|
| T4C | 1 | 2.33 | T/S3C | 1 | 4.97 |
| | 2 | 2.17 | | 2 | 4.42 |
| | 3 | 3.33 | | 3 | 4.55 |
| | **Mean** | **2.61±0.6** | | **Mean** | **4.64±0.3** |
| T7C | 1 | 14.94 | T/S6C | 1 | 20.26 |
| | 2 | 20.14 | | 2 | 21.61 |
| | 3 | 14.37 | | 3 | 21.84 |
| | **Mean** | **16.48±3.2** | | **Mean** | **21.24±0.9** |
| T10C | 1 | 29.76 | T/S9C | 1 | 37.34 |
| | 2 | 38.25 | | 2 | 41.30 |
| | 3 | 30.67 | | 3 | 37.51 |
| | **Mean** | **32.89±4.7** | | **Mean** | **38.72±2.2** |

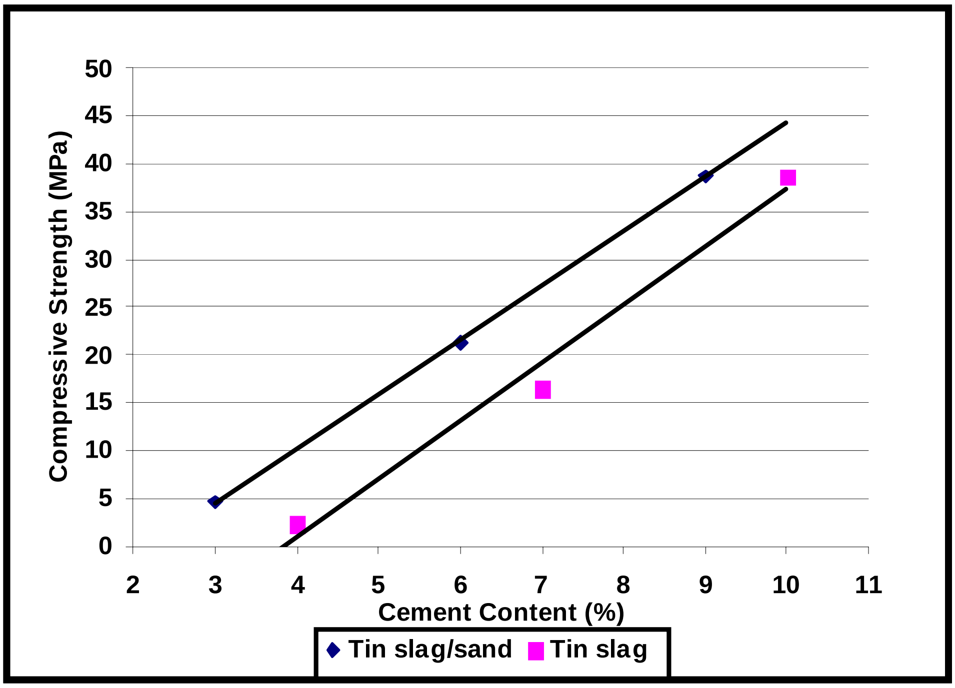



**Figure 5.17 Compaction curve of Cement Bound Materials**

It should be noted that in this investigation the compressive strength tests were performed on specimens that were not the standard cylindrical dimensions. Also, under the SHW requirement, compressive strength should be tested on cube specimens. Therefore, the results obtained from the compressive strength tests were likely to be lower than the results of cube compressive tests. Based on British Standard BS 1881-120 (BSI, 1983) and Johnson, et al (1960) it was estimated that the compressive strength of a standard cube of cement stabilised material was between 15% and 25% higher than the compressive strength of cylindrical specimens as tested in this investigation.

### 5.7.2.3 Durability

Compressive strength test results (Table 5.9 and Figure 5.17) show that the tin slag/sand cement bound mixtures had achieved higher compressive strength values than tin slag only cement bound mixtures at the same quantity of cement. It was, therefore, decided that only tin slag/sand mixtures would be used for the durability study. The tin slag/sand cement bound mixture with a cement content of 3% was used for the immersion test. At the end of the immersion test, the specimens were visually assessed for any signs of swelling and cracking, and none were observed.

Immersion in water resulted in higher compressive strength values which resulted in a durability index value greater than 100% (see Table 5.10). This is most probably due to an amount of binder that did not fully react with water during the conventional hydration process. This suggests that the optimum moisture content may not be adequate during compaction and curing stages of the mixture production. Therefore, some further work needs to be carried out on moisture content optimisation to achieve the best end-product in cement bound materials. From the results in Table 5.10, it can be seen that tin slag/sand cement bound material has an average durability index exceeding the acceptable level imposed by the Specification for Highway Works of 80%.

**Table 5.10: Result from durability assessment**

| Average Compressive Strength (MPa) | | Durability Index (%) |
|---|---|---|
| Control | Immersion in Water | |
| 6.37 | 7.05 | 112 |

## 5.8 SUMMARY

- Laboratory specimens for compressive strength tests were produced using axial compression with minor modifications to the methodology as stated in Draft EN 13286-53, “Unbound and hydraulically bound mixtures - Methods for making test specimens Part 53: Making cylindrical specimens by axial compression”.
- The pozzolanic properties of ground tin slag were determined. Cement, hydrated lime and limestone filler as a control were also tried as partial replacements of the ground tin slag. The tests included optimum moisture content, dry density and compressive strength evaluations.

- The compressive strength tests showed that ground tin slag has, at best, only a modest pozzolanic property. Therefore, it can be concluded that ground tin slag is not suitable for use in slag bound materials.
- Tin slag gradation almost complies with the requirement of the SHW (Highways Agency, 2002) for use in cement bound materials. The laboratory tests showed that both cement-bound tin slag mixture and cement-bound tin slag/sand mixture could be used as cement-bound materials in road-base.
- Laboratory compressive strength tests indicated that cement-bound tin slag/sand mixtures were stronger than cement-bound tin slag mixtures (without sand) at the same cement content. Adding sand to the mixture improved the packing, allowing more of the cement to be used for hydration. In the cement-bound tin slag mixture, it is probable that the cement acts as a filler since the unmodified tin slag contains negligible filler content.
- It can be concluded that there is the possibility of producing cement-bound materials of higher strength by adding sand. Using sand can reduce the need for cement, thus reducing the cost of producing cement bound mixture without any compromise to the strength of the mixture. Cement is the most expensive material in the mixture.
- Durability tests showed that cement-bound tin slag/sand mixture does not lose more than 20% of its compressive strength. Indeed, some strength gain was observed suggesting that some cement remained unhydrated following compaction and conventional curing.
- In durability terms, the tin slag complies with the requirements set in the UK's Specification for Highway Works.
- On the basis of satisfactory performance in the laboratory, further laboratory testing and small-scale trials are recommended to develop a more detailed understanding of these mixtures. Limited laboratory testing has proved that there is a potential to design cement-bound tin slag/sand mixtures for use as sub-bases.
- Further investigation into the optimum amount of sand could be undertaken. More work in optimising moisture content for hydration as well as compaction is warranted.

# Chapter 6 INVESTIGATING THE POTENTIAL FOR INCORPORATING TIN SLAG IN BITUMINOUS BOUND MATERIALS

## 6.1 INTRODUCTION

A review of the literature shows that both ferrous and non-ferrous slags have been used in road pavements in many countries for many years. Steel slag is normally utilised as part of the coarse aggregate fraction of the mixture at percentages ranging from 20% to 100% depending on the specific application of the mixture. In the midwest and eastern states of the United States of America, the incorporation of steel slag into hot mix asphalt concrete has been reported to improve performance characteristics of the pavement surfacing (Ramirez 1992). The use of steel slag can also improve the skid resistance properties of the pavement. Steel slag aggregates also provide high stability and resistance to rutting because of the aggregates' rough surfaces, high specific gravity and angular interlocking features (Ramirez 1992; Noureldin & McDaniel, 1990; Lemass, 1992). However, the stability of the mixture may be a function of the steel slag type used.

In the United Kingdom, there is no restriction in the Specification for Highway Works (SHW) on the use of steel slag in bituminous bound layers. Under the latest amendments of the SHW, steel slag is permitted for use in bitumen bound layers. Clause 938 of SHW covers the use of crushed steel slag in porous asphalt while Clause 942 covers the use of crushed steel slag in thin surface course systems (Highways Agency, 2001). The latest work carried out to date confirms that properly weathered steel (BOS) slag is a competent roadstone coarse aggregate that can be used without compromising the integrity of asphalt mixtures (Rockliff, et al., 2002).

However, there are very few studies that have been conducted on the use of non-ferrous slags such as zinc slag, copper slag, tin slag and phosphoric slag in bituminous bound layers. In 1993, copper slag was used as paving materials in asphaltic concrete at El Paso, in the United State of America. However, the project was discontinued as a result of environmental concerns and none of the reasons given had anything to do with the materials' workability or strength (Texas Department of Transport, 2003). Fine copper slag has reportedly been used in hot mix asphalt pavements in California and granulated copper slag has been incorporated into asphalt mixes in Georgia to improve stability. Although it is rarely used, Michigan Department of Transportation Specifications consider copper slag to be a conventional coarse and fine aggregate for hot mix asphalt pavements (Collins and Cieleski, 1994).

## 6.2 BITUMINOUS BINDERS

There are three types of bituminous binders:

- Petroleum bitumen

  Petroleum bitumen is derived from the refining of certain types of crude oils and is the most common material used in road construction today. Section 6.2.1 discusses in some detail this type of bitumen and for the purpose of this research the term "bitumen" refers to petroleum derived bitumens.

- Tar

  Tar is produced from the carbonisation of coal in the production of coke for metallurgical purposes or smokeless fuels. Tar offers increased adhesion to aggregate and imparts a high resistance to attack by fuel spillage compared to petroleum bitumens. However, tar is now classified as a carcinogen and is no longer used in road construction.

- Natural bitumen

  Natural bitumen is bitumen found in natural asphalt. There are three main sources of natural bitumen in the world, Trinidad Lake which is located in Trinidad, Gilsonite which is found in the USA and rock asphalt which is found mainly in France, Italy and Switzerland. Lake asphalt is the most extensively used and best-known form of natural asphalt. The refined product of Trinidad Lake asphalt is called Trinidad Epuré and typically has, by mass, 54% binder, 36% mineral matter and 10% organic matter (Shell Bitumen, 1990). However, the Epuré is too hard to be used in asphalt mixes and is generally used in a 50/50 blend with 200 pen bitumen so that the penetration of the product can be reduced to 50 pen.

  The rock asphalt is formed by the impregnation of calcareous rock such as limestone or sandstone with seepages of natural bitumen. The asphalt rock contains up to 12 % by mass of bitumen. However, currently the use of rock asphalt has almost ceased.

  Gilsonite is a very hard material having a penetration of zero with softening point between 115$^{o}$C and 190$^{o}$C. The penetration of gilsonite is increased by adding bitumen, thus increasing the softening (Read and Whiteoak, 2003). Currently the use of gilsonite is very limited due to the high production costs.

### 6.2.1 Bitumen

The Oxford English Dictionary Online gives the following definition to bitumen.

> " The generic name of certain mineral inflammable substances, native hydrocarbons more or less oxygenated, liquid, semi-solid, and solid, including naphtha, petroleum, asphalt, etc."

However, the term "bitumen" is believed to have originated in Sanskrit where the words *'jatu'* means pitch and *'jatu-krit'* means pitch creating, referred to the pitch-producing properties of certain resinous trees. Then the Latin used the word *'gwitu-men'* that means pertaining to pitch and *'pixtu-men'* that means bubbling pitch, which became shortened into "bitumen" when passing via French to English (Read and Whiteoak, 2003).

Bitumen is one of the most important pavement materials. It is manufactured from crude oil and is a mixture of high molecular weight hydrocarbons with very complex chemical structures. In addition, there are a number of other structures containing hetero-atoms such as oxygen, sulphur and nitrogen. The mixture of hydrocarbon and heteroatoms gives bitumen its unique balance of properties.

In general, the physical properties of bitumen are susceptible to temperature. Therefore, the classification criterion for bitumen can be based on its susceptibility to temperature. Bitumen has the following physical properties (Lancaster, 2000):

- High viscosity at low temperatures.
  At low temperature, the molecules in bitumen are unable to move over each other. The movement of molecules is very small or there is no movement at all.

- Low viscosity at high temperatures.
  When the temperature increases some of the molecules begin to move and some of the intermolecular bonds may be broken down. As a result, the rate of molecular movement increases and the viscosity decreases. Hence the bitumen behaves as a liquid at high temperature.

As a binder, viscosity of bitumen is a very important factor in the manufacture of asphalt mixtures. If the viscosity of the binder is too high during the mixing stage then it is unlikely that all of the aggregate will be evenly coated and a poorly bonded mixture will result. Conversely, if the viscosity is too low then there is a chance that the binder will drain from the aggregate during storage and transportation. The viscosity of the binder has a significant contribution to the compactibility of an asphalt mixture. A high viscosity binder can result in a reduction in workability and poor compaction while a low viscosity binder will produce mixtures which are excessively mobile and which tend to push out in front of the roller. It is important to control the viscosity of the binder to within a practical temperature range to allow effective compaction whilst maintaining stability.

### 6.2.2 Bitumen Specifications

Four types of bitumen are normally manufactured namely penetration grades, oxidised and hard grades and cutback grades (also emulsions). However, penetration grade bitumens are most commonly used in road pavements. The rest of the types are more suitable for industrial and domestic applications.

Penetration grade bitumen is specified by the penetration and softening point tests. The penetration is defined as the distance travelled by a needle into a pot of bitumen in 5 seconds (Read and Whiteoak, 2003). Penetration is measured in tenths of a millimetre (decimillimetre, dmm). However, these bitumens are normally designated by penetration only, for example 100 pen bitumen has a penetration value of 100 ± 20 decimillimetre (dmm) and a softening point of 46 ± 5$^{o}$C. The pen number of bitumen represents the hardness of a bitumen. The lower the pen number, the harder the bitumen and the higher pen number the softer the bitumen. Details of the bitumen standards and testing methodologies can be found in BS EN 12591:2000 and The Shell Bitumen Handbook (1990).

## 6.3 ASPHALT MIXTURE DESIGN

The overall objective of mix design is to determine an economical blend and gradation of aggregates (within the specification limits) and the proportions of the materials comprising the mixture such that optimum in-service performance is achieved. Mixture design involves selection of an appropriate aggregate gradation and selection of grade and amount of bitumen. The final mixture should have the following properties (Asphalt Institute, 1989):

- Sufficient bitumen to ensure adequate fatigue cracking resistance and durability.
- Sufficient mixture stability and stiffness to resist deformation due to traffic loading.
- Sufficient percentage of air voids to allow for thermal expansion and slight compaction under traffic loading without flushing, bleeding, or loss of stability. The voids content also should be low enough to prevent intrusion of water and to reduce the amount of oxidation.
- Sufficient workability to permit efficient placement of the mixture without segregation.
- Sufficient skid resistance if the mixture is to be used as wearing course.
- Sufficient performance characteristics over the service life of the pavement.

Table 6.1 shows the general requirements of asphalt mixture design. It gives a general indication of the levels of binder content, aggregate gradation and air voids needed to achieve each requirement.

**Table 6.1 Summary of mixture design requirements (The Asphalt Institute, 1989)**

| **Mixture Property** | **Bitumen content** | | **Aggregate Gradation** | | **Air Voids** | |
|---|---|---|---|---|---|---|
| | **Low** | **High** | **Dense** | **Open** | **Low** | **High** |
| Stability | X | | X | | x | |
| Durability | | x | x | | x | x |
| Flexibility (resistance to non-load related cracking) | | x | | x | | x |
| Fatigue cracking resistance (resistance to load related cracking) | | x | x | | x | |
| Skid resistance | X | | | x | | x |
| Imperviousness | | x | x | | x | |
| Fracture strength | | x | x | | x | |

The objective of mixture design can be achieved by ensuring the mixture contains an appropriate quantity of bitumen to adequately coat all of the aggregate particles, provide good workability, and such that when it is compacted, it possesses adequate stiffness, deformation resistance and air voids. This can be accomplished through preparation and testing of representative mixture design specimens as shown by the flow chart in Figure 6.1.

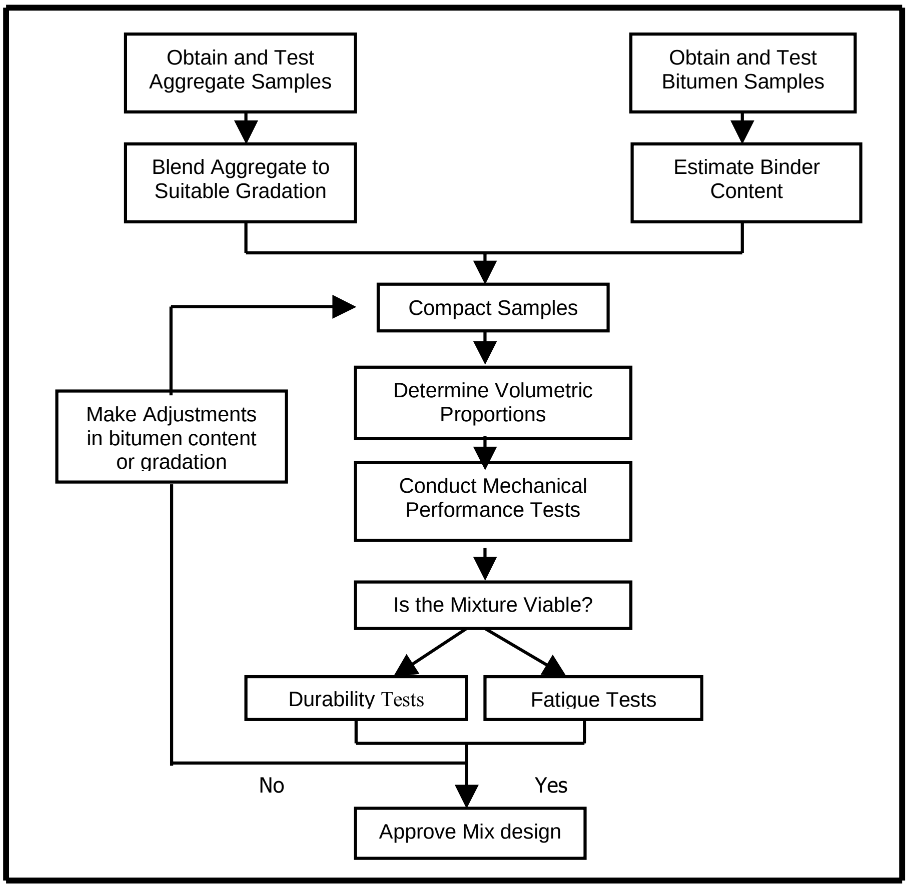



**Figure 6.1: Asphalt mixture design process**

### 6.3.1 Present Methodology on Designing Bituminous Bound Materials

Mix design is an essential part of pavement construction. At present there are several design methods that can be used when designing bituminous bound materials such as Marshall design method, Hveem design method, University of Nottingham design method and SHRP Superpave design method (Airey, G.D., 2002). However, in the

United Kingdom, the design method which is commonly used is a recipe method which is based on practical experience that has been gained over long periods of time. The Marshall design method is only used when designing wearing course layers and thin base courses. The following section will discuss in some detail the Marshall mix design method and the Nottingham University mix design method.

#### 6.3.1.1 Marshall Mixture Design Method

The basic concepts of the Marshall mix design method were originally developed by Bruce Marshall, formerly the Bituminous Engineer for the Mississippi Highway Department around 1939 and then subsequently refined by the U.S. Army Corps of Engineers. There are two major features of the Marshall method of mix design namely density voids analysis and stability-flow tests (i.e. Marshall stability test).

The Marshall stability and flow test provides the performance prediction measure for the Marshall mix design method. They are determined in accordance of Draft BS EN 12697-34 (BSI, 1999). Prior to stability and flow testing, the specimens are immersed in a water bath at 60±1$^{o}$C for at least 40 minutes and not longer than 60 minutes. The Marshall testing machine, compression-testing device (in this study Instron machine as shown in Figure 6.2), is designed to apply loads to test specimens through cylindrical segment testing heads at a constant rate of vertical strain of 50mm/min. The stability value is calculated by multiplying the maximum load applied by a correction factor obtained from the table provided in the standard. The flow is obtained by monitoring the displacement between the starting point of load and maximum point of load. Figures 6.3 and 6.4 show the principle of determining of Marshall Stability and Marshall Flow values in Marshall mix design method.

Another Marshall parameter of interest is the Marshall Quotient which is the ratio of Marshall stability to Marshall flow. This parameter is thought to be an indicator of the material's resistance to permanent deformation when designing asphalt mixtures. According to the Asphalt Institute (1989) the Marshall stability shall be in range 2kN to

8kN and Marshall flow shall be in the range 2.5mm to 5.0mm. The minimum Marshall Quotient shall be 1.4kN/mm. Generally, the stability is found to increase with increasing binder content up to a maximum value after which it declines rapidly. On the other hand, the flow generally continues to increase with increasing binder content.

In order to investigate the density-void relationship, the volumetric properties of the mixture need to be studied. (Density-Voids analysis is discussed further in Section 6.3.2). In these analyses, the bulk density, the porosity (air voids), the voids in mineral aggregate are determined and plotted against a range of binder contents. The results are then compared with the recommended values. The percentage of porosity in the Marshall mix design method should be in between 3% - 5% (Asphalt Institute, 1989). Figure 6.5 shows typical plots from the Marshall mix design method which are usually used to determine optimum binder content of the mixture.


**Figure 6.2: Instron machine arrangement for Marshall stability test**

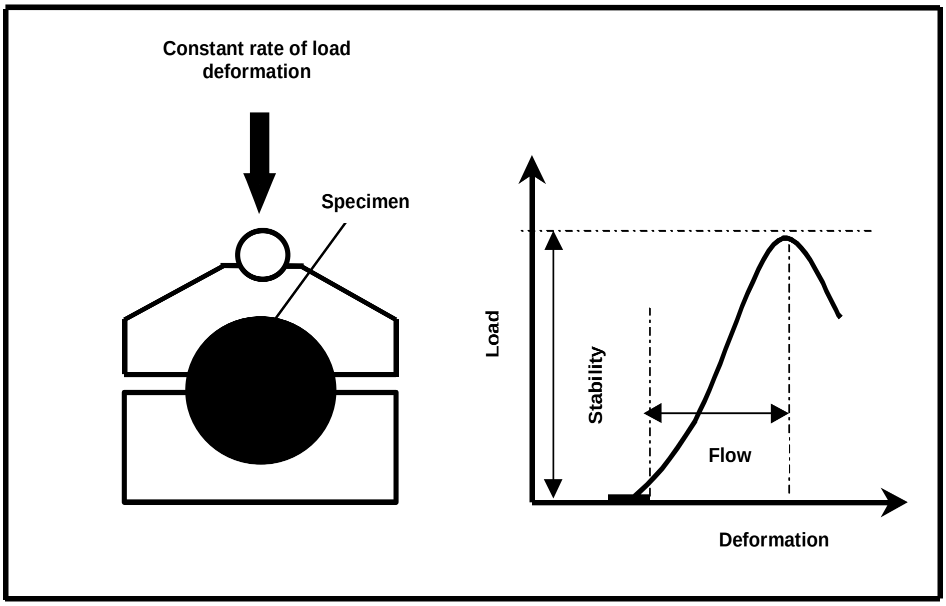



**Figure 6.3: Determination of Marshall Stability and Flow values**

.

**Figure 6.4: Marshall test head with specimen arrangement**

Although the Marshall mixture design methodology is widely used, it has several shortcomings. The method is based on empirical tests only and does not test for fundamental engineering properties. Therefore, it cannot relate to the in-service mixture performance which is based on engineering properties. Thus, the method can produce mixtures that are susceptible to deformation.

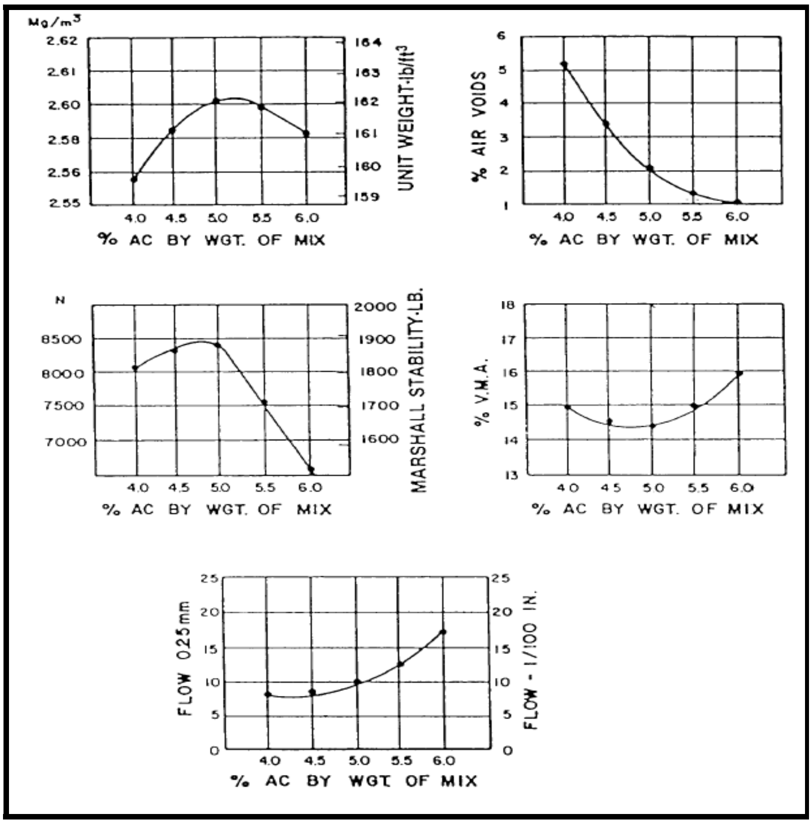



**Figure 6.5: Typical data from the Marshall Method of mix design (Asphalt Institute, 1989)**

### 6.3.1.2 University of Nottingham Method

The University of Nottingham mix design method was proposed by Cooper et al. (1992) for the design of continuously-graded mixtures i.e. basecourses and roadbases. This method enables the determination of the best gradation and binder content for optimum mechanical properties at a given level of compaction. It is aimed at maximizing resistance to deformation using values of exponent number, n, greater than 0.45 in a modified Fuller equation (Equation 4.3). For optimum packing, a value of 0.5 is normally used. The method is similar to the Marshall method in terms of:

- Preparation of test specimens at a range of binder contents
- Volumetric analysis
- Assessment of mechanical properties
- Selection of the optimum mixture

However, the two methods differ in the details for each of the above points, such as compactive effort and selection of binder content. With respect to assessment of mechanical properties, the Nottingham University method has more emphasis on mixture stiffness and permanent deformation resistance. Compaction efforts are classified at three levels - compaction to refusal, intermediate level and low level using vibratory kneading action. Selection of binder content is based on UK practice with dense bitumen macadam (DBM) and covers the range 3.5% to 4.7% by total weight of mixture. The air voids content and voids in mineral aggregate (VMA) are then checked against volumetric criteria and if the values are acceptable, the mixtures are subsequently tested for stiffness modulus and resistance to permanent deformation and the results checked against the acceptable criteria for these tests. The optimum binder content of the mixtures is then determined. The final mixtures at optimum binder content are then tested for fatigue cracking and durability.

### 6.3.2 Volumetric Analysis

Asphalt mixtures are designed to meet certain minimum volumetric requirements so that the mix will have a reasonable assurance of performance. Volumetric analysis of asphalt mixtures is universally recognised as being very important for the design and evaluation of performance of the mixtures. The volumetric properties of asphalt mixtures include the voids in the mineral aggregate, the voids filled with binder and the air voids content. In order to assess the volumetric properties of an asphalt mixture, the mineral aggregate particle density should first be determined.

Efforts should be made to limit the value of compacted air voids in a compacted dense asphalt mixture to between 3% and 7%. Once the voids content exceeds 8%, the voids will become interconnected thus allowing air and moisture to permeate the pavement and this is known to reduce the durability of the pavement. On the other hand, if the air voids fall below 3%, there will be inadequate room for expansion of the asphalt binder in hot weather and when the void content drops to 2% or less, the mix can become plastic and unstable.

The voids in mineral aggregate (VMA) are defined as the volume of inter-granular void space between the aggregate particles of a compacted asphalt mixture that includes the air voids and the volume of bitumen not absorbed into the aggregates. The VMA property is intended to ensure that an asphalt mixture will have sufficient inter-granular space for both the bitumen binder and air voids. VMA is a durability requirement that is strongly affected by laboratory compaction procedures. There are two approaches that can be followed in attempting to achieve a minimum VMA. The first is to alter the gradation of the mixture. Dense-graded mixtures (those that follow the maximum density line) often have difficulty in achieving adequate VMA values. Unfortunately, there is no consistent method of adjusting gradation to achieve the desired VMA that holds true for all combinations of aggregates. The second approach that a mix designer can use to achieve VMA is to change the aggregates used in the mix. Increasing the use of clean, angular, cubical aggregates will typically help to increase VMA.

### 6.3.3 Compaction

Compaction is one of the very important factors in relation to void analysis. Compaction, a mechanical process, compresses the asphalt mixture into a smaller, denser volume. Fordyce (1997) described the main aim of compaction as to optimise the packing of the aggregates, to uniformly distribute the bitumen and air voids, and minimize the residual air voids. Compaction increases the mixture stability by forcing the bitumen-coated aggregate particles closer together thus achieving greater particle-to-particle contact. Compaction will increase the density of mixture and will reduce the volume but will not change its mass. The volume of voids in bituminous mixtures is directly related to density.

Primary factors that affect asphalt mixture compaction include the material properties, equipment operation and confinement of the mix. Confinement forces result from the compressive forces applied by the compactors and from the resistance of the mix itself to compaction. Improper compaction will result in an asphalt mixture with air voids that are either too high or too low. High air voids allow moisture to penetrate the mixture and can cause stripping or surface ravelling. High air voids can also lead to rutting due to additional densification of the mixture under traffic. Low air voids in a compacted mix will not allow for thermal expansion of the bitumen and can cause flushing. Furthermore, if too few voids are present, rutting can occur due to plastic flow of the mixture where an asphalt mixture deforms when loaded but does not rebound to its original state.

There are several compaction methods available for producing specimens in the laboratory such as Marshall hammer, vibratory, kneading, gyratory compactor and roller (wheel or slab) compaction. The impact of compaction methods on mechanical properties of bituminous mixtures has been widely studied by many researches (Hartman et al. 2001, Harvey et al. 1994, Button et al. 1994). Harvey et al. (1994) found that the mechanical performance properties influenced by compaction method include fatigue, stiffness and permanent deformation. However, Button et al. (1994) concluded that the mechanical properties of asphalt mixtures with stiff binders were relatively unaffected by compaction method. Hartman et al (2001) found that the influence of the compaction method on fatigue strength of bituminous mixture would appear to be mixture dependent. Mixes with grading profiles that are designed for aggregate interlock were found to have higher fatigue strengths, provided the material was compacted using a method that would facilitate reorientation of the aggregate such as gyratory compaction.

The Marshall hammer method was developed by Bruce Marshall and has been used for many decades as a compaction method. The Marshall hammer produces specimens that are considerably different from actual field cores (Button et al. 1994). The mechanism of compaction lacks the kneading action required to re-orientate the aggregate and additionally the impact forces can degrade the aggregates. On the other hand using a static compression method, very high pressures are required to achieve the required densities, resulting in aggregate crushing and squeezing of the binder film. This affects the microstructure of the compacted mixture and hence the end product is different from that of the material compacted in-situ.

In general, kneading compaction produces specimens having greater resistance to permanent deformation (Sousa et al., 1991; Harvey et al., 1993) but this method of compaction tends to trap voids in the lower part of the compacted specimens (Harvey et al., 1994). Gyratory compaction produces specimens that have great resistance to fatigue cracking (Sousa et al., 1991). It also produces specimens closer to field compacted materials than either impact or static compression specimens, but they are not always homogeneous. During gyratory compaction, larger aggregates tend to segregate towards the sides of the cylindrical moulds, which results in higher void contents towards the exterior of the specimens (Harvey et al., 1994). On the other hand, for both permanent deformation and fatigue resistance, rolling-wheel specimens are ranked in between gyratory and kneading specimens (Sousa et al., 1991). It appears that rolling-wheel compaction has similarities with field compaction in terms of compaction action and void distribution.

## 6.4 MECHANICAL PERFORMANCE OF BITUMINOUS BOUND MIXTURES

In flexible pavement structures, bituminous paving mixtures are used as surface or base layers. They distribute loading stresses and also protect underlying unbound layers from the effects of water. The mixtures, therefore, must withstand the effects of air and water, resist permanent deformation and resist cracking caused by traffic loading and thermal stress. There are many factors affecting the integrity or stability of bituminous mixtures such as mixture design, construction practices, properties of the mixtures' component materials and additives.

There are three fundamental properties of bituminous materials which govern the life of the structural layers in bituminous pavements (Curtis et al., 1993). These properties are:

- Stiffness modulus which relates to the load spreading ability of the layer.
- Fatigue strength which relates to resistance to crack development caused by repeated traffic loading.

- Resistance to deformation characteristics which relates to creep under repeated loading and which can occur both within the pavement structure and/or in the surface layer.

However, there are two further properties that are perhaps considered of lesser importance:

- Water sensitivity
- Long term ageing, hardening and oxidation of the binder which occur with time.

### 6.4.1 Stiffness Modulus

Stiffness, in general terms, is the ratio between a stress and a corresponding strain. In pavement engineering, the stiffness modulus is a ratio of stress to strain under axial loading conditions. It is analogous to Young's Modulus of elasticity. Stiffness modulus is a function of load, deformation, specimen dimensions and Poisson's ratio and is therefore not a constant for a particular material. It is also a function of temperature and loading time because of the visco-elastic behaviour of bituminous materials. Therefore, different stiffness values are obtained at different loading times and/or temperatures.

There are two main types of stiffness i.e. elastic stiffness and viscous stiffness (Zoorob, 2000).

- Elastic stiffness

  Elastic stiffness is relevant only to low temperatures or short loading times. Under these conditions, the stiffness of a mixture depends only on that of the bitumen and voids in mineral aggregate (VMA) of the mixture. Elastic stiffness is used to calculate critical strains in the pavement structure for use in analytical design
- Viscous stiffness

  Viscous stiffness is relevant at high temperatures or longer loading times. Under these conditions, the stiffness of a mixture depends on the type, grading, shape and texture of the aggregate, the confining conditions and the method of compaction in addition to the bitumen stiffness and VMA. Viscous stiffness is used to evaluate the resistance of the material to deformation.

The stiffness modulus is considered to be a very important performance property of the roadbase or basecourse and it dictates the load-spreading ability of the bituminous layers. It controls the levels of the traffic-induced tensile strains at the underside of the lowest bituminous bound layer, which are responsible for fatigue cracking, as well as the stresses and strains induced in the sub-base and subgrade that can lead to plastic deformation (Zoorob, 2000). Measurement of bituminous mixtures' stiffness values can be used as follows:

- To provide an indication of binder grade and temperature susceptibility by carrying out the test at a range of temperatures;
- To provide an indication of resistance to moisture related durability problems by carrying out the test before and after a water conditioning regime.
- To evaluate both the stress and strain distribution in asphalt pavements.
- As an indicator of mixture quality for pavement and mixture design.
- To evaluate damage and age-hardening trends of bituminous mixtures in the laboratory and the field.

Higher stiffness mixtures reduce load-induced stresses to underlying layers but increase both load-induced and thermal stresses in the bituminous mixture. Higher stiffness mixtures can be achieved by changing any of the mixture gradation, bitumen content, compaction level, filler content, and binder types including additives.

The stiffness modulus of bituminous mixtures can be determined in the laboratory by several test methods such as repeated load beam test, uniaxial repeated load test and indirect tensile test. In the U.K., the Indirect Tensile Stiffness Modulus (ITSM) test method using the Nottingham Asphalt Tester (NAT), which is a non-destructive method, has been widely adopted for determination of stiffness modulus values (Cooper and Brown, 1989).

Draft of British Standards, *BS DD 213 Method for determination of the indirect tensile stiffness modulus of bituminous mixtures* (BSI, 1993) specifies the method to determine the indirect tensile stiffness modulus (ITSM) of bituminous mixtures using cylindrical specimens that may be prepared in the laboratory or sampled from the field. The standard target parameters pertaining throughout testing are as follows:

| | |
|---|---|
| Test temperature | $20^oC$ |
| Rise time | $(124 \pm 4)$ ms |
| Horizontal deformation | $(5 \pm 2)$ µm |
| Poisson ratio | 0.35 |

Prior to testing, all specimens are conditioned in an environmental cabinet at the test temperature of $20^oC$ for at least two hours. During testing, it is important to adjust the magnitude of peak load so it results in a transient horizontal deformation value of $(5 \pm 2)$ µm for a specimen with nominal diameter size 100 mm. Figure 6.6 shows the typical test configuration of ITSM. Based on the ITSM test, the stiffness modulus of bituminous mixtures, $S_m$, can be determined using Equation 6.1 below:

$$S_m = \frac{L(\nu + 0.27)}{Dt} \qquad \text{…(6.1)}$$

where:

| | |
|---|---|
| $S_m$ | indirect tensile stiffness modulus (MPa) |
| $L$ | peak value of the applied vertical load in Newton (N); |
| $D$ | mean amplitude of the horizontal deformation obtained from two or more applications of the load pulse in millimetre (mm) |
| $t$ | mean thickness of the test specimen in millimetre (mm) |
| $\nu$ | Poisson's ratio which for bituminous mixtures is normally assumed to be 0.35 |

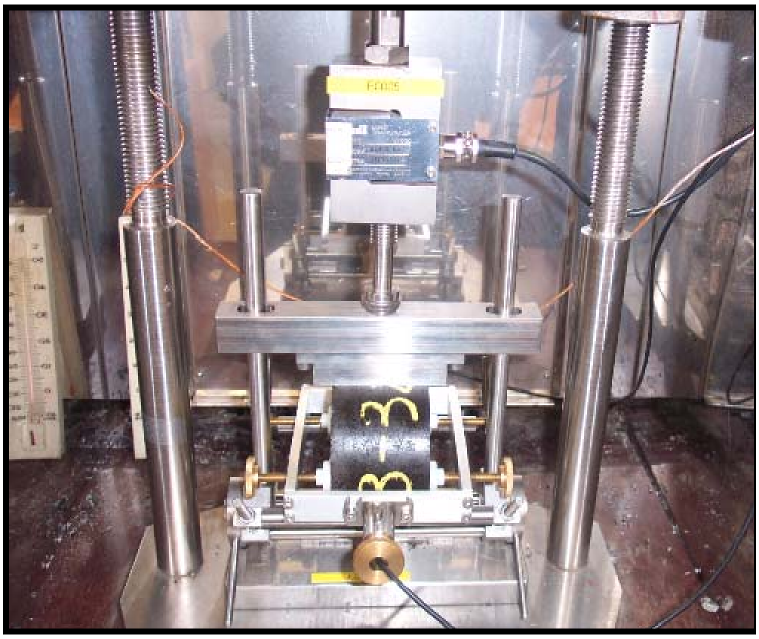


**Figure 6.6: Indirect tensile stiffness modulus test configuration**

According to Cooper et al. (1989), trials have demonstrated that NAT test results give an excellent correlation with other test methods such as bending beam and wheel tracking. The NAT provides the most inexpensive and user friendly means of measuring and assessing the mechanical properties of asphaltic paving materials on a routine basis. It is used to derive the maximum information about a material using either cores cut from the pavement or laboratory moulded specimens. However, although the NAT test results give an excellent correlation with other test methods, there are several other factors which affect the test results including:

- The number conditioning of pulses
- Selection of the rise time
- Specimen dimensions
- Actuator capacity
- Pulse shape

Table 6.2 lists some additional important factors which significantly affect the stiffness modulus of asphalt mixture.

**Table 6.2: General effects of the main factors affecting the ITSM (Read, 1996)**

| Factor | General Effects on ITSM |
|---|---|
| Specimen temperature during test | High temperature → low stiffness modulus<br>Low temperature → high stiffness modulus |
| Loading frequency | Low frequency → low stiffness modulus<br>High frequency → high stiffness modulus |
| Stress amplitude | High stress → low stiffness modulus<br>Low stress → high stiffness modulus |
| Poisson's ratio (assumed) | Low Poisson's ratio → low stiffness modulus<br>High Poisson's ratio → high stiffness modulus |
| Bitumen grade (for particular mixture type) | High penetration → low stiffness modulus<br>Low penetration → high stiffness modulus |
| Bitumen content (for particular mixture type) | Highest stiffness modulus is generally achieved at or very near the optimum binder content for compacted aggregate density (BS 598) |
| Bitumen modifiers | Use of modified bitumen in mixtures can significantly increase or reduce the stiffness modulus of the mixture and the magnitude of the effect greatly depends on the type of modifier. It should be noted, however, that modifiers are generally used to improve characteristics of the mixture other than its stiffness modulus. |
| Void content (for particular mixture type) | High air voids → low stiffness modulus<br>Low air voids → high stiffness modulus<br>(for some mixture very low air voids can result in a reduction in stiffness modulus) |
| Aggregate type and gradation | Materials made with crushed rock tend to have higher stiffness than those with gravels. For continuously graded materials the larger the aggregate the higher the stiffness. For all mixtures, the higher the quantity of coarse aggregates the higher stiffness. |

With regard to the precision and practicability of the indirect tensile stiffness modulus test Nunn et al. (1997) concluded that:

- The precision of the ITSM test can be improved by using a correction factor that takes account of the shape of the load waveform.
- An option to relax the temperature tolerances and correct to the target temperature

will improve the practicality of the test.

- Testing in two orientations should be retained but retesting should not be allowed. If the differences of measurement in the second orientation is more than 20% lower or 10% greater than that in the first orientation the results should be rejected.

### 6.4.2 Fatigue Strength

The Oxford English Dictionary Online (Oxford, 2002) gives the definition of fatigue as:

> "The condition of weakness in metals or other solid substances caused by cyclic variations in stress"

In terms of pavement engineering, fatigue resistance of an asphalt mix is related to its ability to withstand repeated bending without fracture. Flexible pavement structural layers are subjected to continuous flexing under traffic loading. Read (1996) proposed a new definition for fatigue as follows:

> "Fatigue in bituminous pavements is the phenomenon of cracking. It consists of two main phases, crack initiation and crack propagation, and is caused by tensile strains generated in the pavement by not only traffic loading but also temperature variations and construction practices."

It is important to have a measure of fatigue characteristics of specific mixtures over a range of traffic and environmental conditions so that fatigue considerations can be incorporated into the process of designing asphalt pavements. The fatigue characteristics of asphalt mixes are usually expressed as the relationship between the initial stress or strain and the number of load repetitions to failure. The fatigue failure of a specific asphalt mix can be characterized by the slope and relative level of the stress or strain versus the number of load repetitions to failure on a log-log plot. It is normal to test several specimens at each stress or strain level because of the scatter in results. Typical profiles of constant stress fatigue life characteristics are shown in Figure 6.7.

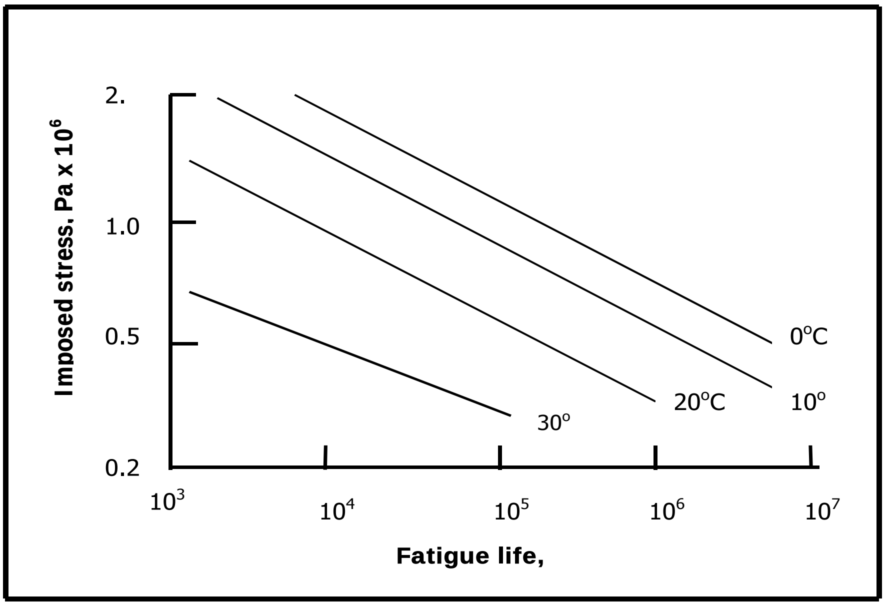



**Figure 6.7: Typical constant stress fatigue life characteristic**

The general relationship defining the fatigue life based on crack initiation is as follows:

$$N_f = c \times \left( \frac{1}{\varepsilon_t} \right)^m \quad \ldots(6.2)$$

where:

- $N_f$ number of load applications to initiate a fatigue crack
- $\varepsilon_t$ maximum value of applied tensile strain
- *c, m* factors depending on the composition and properties of the mixture

Factor *m* defines the slope of the strain-fatigue life line and for many mixtures has a value of approximately 5 or 6. Softer grades of bitumen give steeper lines than hard grades. Monismith et al. (1985) developed the relationship further by taking into account the initial mix stiffness when defining the fatigue life. They defined fatigue life as follows:

$$N_f = a(1/\varepsilon_o)^b \times (1/S_o)^c \quad \ldots(6.3)$$

where:

$N_f$ fatigue life

$\varepsilon_o$ tensile strain

$S_o$ initial mix stiffness

*a, b, c* experimentally determined coefficients

In general, the main factors affecting the fatigue property of continuously graded bituminous mixtures are aggregate content, filler content, mixture stiffness, binder content and voids content. Harvey and Monismith (1993) found that a 3% reduction in fines content significantly affects fatigue performance. The gradation of the aggregate also contributes to fatigue resistance of bituminous mixtures. Studies by Sousa et al. (1998) showed that fine graded mixtures seem to have better fatigue performance than mixtures containing coarser gradations. However, the maximum nominal aggregate size was not found to significantly affect fatigue resistance (Moutier et al., 1988). On the other hand, Read (1996) reported aggregate shape plays a major role in the crack propagation phase with flaky aggregate oriented normally to the applied load giving slower rates of crack propagation than nominally spherical aggregate.

Read (1996) argued that if the binder content of the asphalt mixture increases, within a practical range, the fatigue life increases. According to Gibb (1996), the volume of bitumen should be as high as possible for the longest fatigue life, but has to be limited to satisfy the permanent deformation requirements. Another factor that affects fatigue strength is binder stiffness, where longer fatigue lives can be achieved by incorporating higher stiffness binders. Hard binders have better fatigue performance especially when used in thicker pavements (Sousa et al., 1998). However, temperature is also a very important factor that affects the fatigue strength of asphalt mixtures in the way that the temperature affects the stiffness.

With regard to the air voids content, generally, mixtures with lower air voids contents are more likely to achieve higher resultant fatigue lives. However, if the air voids are reduced too much, the mixture will rut faster because the voids become over filled with bitumen which causes the aggregates to be pushed apart (Gibb, 1996).

Under the present standards, the indirect tensile fatigue test (ITFT) protocol is covered by draft DD ABF – 2000 – "*Method for the determination of the fatigue characteristics of bituminous mixtures using indirect tensile fatigue"* (BSI, 2000). This test was carried out at a standard temperature of $20 \pm 1^{o}C$ using 100mm diameter specimens. The test was performed at various stress levels at a rate of 40 pulses/minute. Each specimen was repeatedly loaded until it failed by cracking. The testing procedure is briefly described as follows:

- The 100mm diameter specimens were conditioned at the test temperature at least two hours prior to positioning in between the loading strips of the test frame as shown in Figure 6.8.
- A number of specimens from each mixture were tested at a range of applied loads resulting in different target stress levels at the centre of each specimen.
- The maximum horizontal tensile strain at the centre of each specimen was then calculated using Equation 6.4.
- The results were plotted as the maximum tensile strains ($\varepsilon_{x,max}$) against the number of cycles to failure ($N_f$) using logarithmic scales. Linear regression analysis was used to describe the resultant fatigue relationship.

The results obtained from ITFT tests can be expressed in the form of a relationship between the maximum tensile horizontal strain and the number of cycles to failure. The maximum tensile strain $\varepsilon_{x\,max}$ is defined as:

$$\varepsilon_{X\max} = \frac{\sigma_{X\max} \times (1+3v)}{Sm} \times 1000 \qquad \ldots(6.4)$$

where:

$\sigma_{X\,max}$ = maximum tensile stress at the centre of the specimen (kPa)

$$\sigma_{X\max} = \frac{2 \times P}{\pi \times d \times t}$$

*P* = Maximum applied load (N)

$d$ = diameter of specimen (mm)

$t$ = average height of the specimen (mm)

$\nu$ = Poisson's ratio (typically assumed as 0.35 for 100 mm diameter specimen)

$S_m$ = indirect tensile stiffness modulus at $\sigma_{X\,max}$ (MPa)

The results of the ITFT can be used to compare against the performance of well known materials, or, by application of an appropriate shift factor, to represent actual performance on site, and thus to validate the assumptions made in pavement evaluation in the calculation of residual life and appropriate remedial treatments.

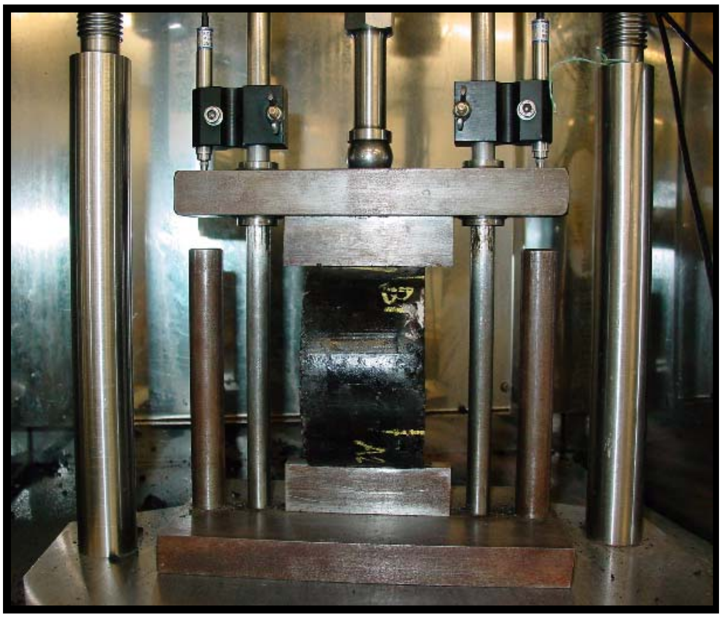


**Figure 6.8: Indirect tensile fatigue test (ITFT) arrangement using NAT apparatus**

### 6.4.3 Deformation Resistance

Deformation resistance of bituminous mixtures is influenced by the properties and composition of the mixture such as texture, grading, size and shape of the aggregate as well as the stiffness of the binder used and the compaction methods. A study carried out by Harvey et al. (1993) showed that compaction method selected in the lab (gyratory, rolling wheel and kneading compaction) is a significant factor in permanent deformation performance. They also found that a reduction in the fines content by 3% significantly affects both permanent deformation and fatigue performance. While Brown (1967) indicated that resistance to permanent deformation depends mainly on the aggregate grading and the particle characteristics. Angular and rough crushed aggregates show better resistance to permanent deformation than smooth, rounded aggregates however; a low but adequate binder content is needed for the angular and rough crushed aggregate mixture.

A variety of tests are available for assessing the resistance of bituminous mixtures to permanent deformation. The most widely used fundamental tests for assessing permanent deformation characteristics are the repeated load axial test (RLAT) and the repeated load triaxial test. The RLAT is increasingly used, as the pulsed load is more simulative of the traffic load compared to other tests. During the RLAT, a pulsed load is applied to the test specimen and the resultant deformations are measured. The RLAT is also described in a draft British Standard (BS DD226). Figure 6.9 shows the configuration of RLAT. The test is carried out as follows:

- All specimens are treated with graphite powder to ensure smooth, friction free surfaces for uniform load application.
- The specimens are conditioned at a standard testing temperature of $40^{o}C$ for at least two hours prior to positioning on the loading plate.
- The specimen is centred on the bottom plate and a top-loading plate is then placed on top of the specimen.
- The specimen is then subjected to a pre-test conditioning regime consisting of the application of a 100kPa static stress for a duration of 10 minutes. The main purpose of this conditioning period is to ensure that the loading platens are firmly seated on the specimen prior to the commencement of deformation measurement.
- In total 3,600 load pulses are applied to each specimen, each cycle consists of the application of 100kPa vertical axial stress for one second.
- The axial cumulative deformations are continuously measured during the test. The test results are then expressed as a relationship between the accumulated axial strain and test duration (a number of load cycles).

**Figure 6.9: Repeated load axial test (RLAT) arrangement using NAT apparatus**

### 6.4.4 Indirect Tensile Strength (ITS)

The Indirect Tensile Strength (ITS) Test is conducted on asphalt specimens to measure the tensile strength characteristics of the mix. The results from the ITS can be used to determine fatigue and low temperature cracking. The test method can also be used to determine the water sensitivity of bituminous specimens once the volumetric design has been completed. British Standard BS EN 12697-23 *Determination of the indirect tensile strength of bituminous specimens* (BSI, 2003) contains the procedure for determination of the ITS of bituminous specimens. In accordance to the standard, the ITS test consists of applying diametrical load continuously at a constant rate of deformation of 50mm/minute at the test temperature of 25$^o$C. Testing for fatigue cracking is performed at a rate of 50mm/minute at test temperatures of -10, 4, and 20$^o$C. Testing for low temperature cracking is conducted at a rate of 12.5mm/minute at test temperatures of -20, -10, and 0$^o$C.

In this test, a cylindrical sample is subjected to compressive loads between two loading strips, which creates tensile stress, perpendicular to and along the diametral plane causing a splitting failure. The diametrical load with constant rate of deformation of (50 ± 2) mm/min is applied continuously and without shock until the peak load is reached at which point the specimen fractures. Figure 6.10 shows the test configuration.

The ITS value is calculated using the following equation:

$$ITS_i = \frac{2P_i}{\pi D_i L_i} \qquad \text{...(6.5)}$$

and the average value follows:

$$ITS = \sum_{1}^{i} (ITS_i) / i \qquad \text{...(6.6)}$$

where

| | |
|---|---|
| *ITS* | is the indirect tensile strength, expressed in kiloPascals (kPa) |
| *P* | is the peak load, expressed in kiloNewtons (kN) |
| *D* | is the diameter of the specimen in mm |
| *L* | is the length of the specimen in mm |
| *i* | is the number of the determination. |

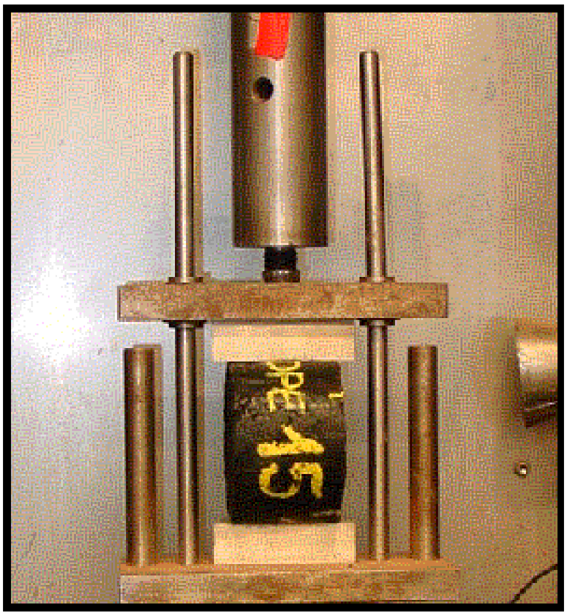


**Figure 6.10: Indirect tensile strength (ITS) test configuration**

## 6.5 DURABILITY OF BITUMINOUS PAVING MIXTURES

Durability means a capability of withstanding change, decay or wear for a long period of time without significant deterioration. For the highway industry, durability generally refers to the ability of bituminous mixtures to withstand effects related to the environment, namely moisture and ageing. Durability can also be defined as the ability to maintain satisfactory rheology, cohesion and adhesion in long-term service (Whiteoak, 1990). However, Scholtz (1995) suggested the following definition for durability.

> *"Durability as it applies to bituminous paving mixtures is defined as the ability of materials comprising the mixture to resist the effects of water, ageing and temperature variations, in the context of a given amount of traffic loading without significant deterioration for an extended period".*

The durability of bituminous mixtures is a very important criteria that must be considered when designing the lifetime of mixtures, as the costs of maintenance and rehabilitation of pavements structures are very much dependent on it. From the above definition, generally, there are two environmental factors which have a profound effect on the durability of bituminous mixtures namely moisture susceptibility and age hardening of the binder.

### 6.5.1 Water Susceptibility

There are three main mechanisms by which water can degrade the integrity or strength of asphalt mixtures (Terrel and Al-Swailmi, 1994; Scholtz, 1995) as follows:

- Loss of cohesion (strength) of the bitumen film that may be caused by several mechanisms.
- Failure of the adhesion (bond) between the aggregate and bitumen.

- Degradation or fracture of individual aggregate particle when subjected to freezing.

Loss of cohesion in the bitumen and aggregate mixture results in a reduction of mixture strength and stiffness and thus a reduction of the pavement's ability to support traffic-induced stresses and strains (Scholtz, 1995). A similar phenomenon occurs when the bond between the bitumen and aggregate fails. Therefore, when the mixtures are permeable to water, the bitumen is often stripped away and the mixtures tend toward stripping. Stripping leads to loss in integrity of asphalt mixtures and ultimately leads to failure of the pavement as a result of ravelling, rutting or cracking. The risk of stripping can be reduced by designing asphalt mixtures that have low void contents.

Curtis et al. (1993) identified that stripping of bitumen from the aggregate stems from the intrusion of water into the asphalt-aggregate system. They report that the modes of failure are many and dependent on the character of the system. The most important modes of failure are:

- Separation of the bond at the interface
- Failure within the bitumen where soluble components are removed
- Cohesive failure within the coated aggregates
- Phase separation of components when the presence of water increases the solubility of polar compounds through hydrogen bonding.

Water penetration into the system can take place by diffusing through the bitumen film removing, along the way, those asphaltic components that are solubilized. If cracks occur in the film, then water can intrude to the bitumen-aggregate interface, causing failure at or near the interface. The failure can be interfacial or cohesive, either in the bitumen or in the aggregate.

There are several methods that have been developed and used to predict the moisture susceptibility of asphalt mixture such as Boiling Water Test, Texas Boiling Water Test, Static Immersion Test, Lottman Test, Tunnicliff and Root Conditioning Test, Modified

Lottman Test, Immersion Compression Test, Hamburg Wheel Tracking Device, and Texas Freeze-Thaw Pedestal Test. But none as yet has been universally accepted as a suitable method. However, in the UK and other European countries draft standard prEN 12697-12, "*Determination of the water sensitivity of bituminous specimens*" (BSI, 2002) is in use. According to the standard, at least six specimens are needed for each mixture to be tested, three for a "dry" subset and three for a "wet" subset. Box 6.1 and Figure 6.11 provide a summary of the test methodology.

Water sensitivity, can be expressed by the ITS ratio, i.e. the indirect tensile strength of wet (water conditioned) specimens to that of dry specimens, expressed as a percentage (Equation 6.7). However, in this study, indirect tensile stiffness modulus was also used to evaluate the water sensitivity of bituminous mixture. The ITSM ratio is expressed in Equation 6.8.

$$ITSR = \frac{100 \times ITS_w}{ITS_d} \qquad \ldots(6.7)$$

and

$$ITSMR = \frac{100 \times ITSM_w}{ITSM_d} \qquad \ldots(6.8)$$

where:

ITSR is the indirect tensile strength ratio, in percent (%)

$ITS_w$ is the average indirect tensile strength of the wet (conditioned) group, kPa

$ITS_d$ is the average indirect tensile strength of dry (unconditioned) group, kPa

ITSMR is the indirect tensile stiffness modulus ratio, in percent (%)

$ITSM_w$ is the indirect tensile stiffness modulus of the wet group, MPa

$ITSM_d$ is the indirect tensile stiffness modulus of the dry group, MPa

**Water Sensitivity (Draft BS EN 12697-12)**

- The cylindrical test specimens with (100 ± 3) mm are prepared for each sample tested.
- Determine the dimensions and bulk density of each of the test specimens.
- Divide the specimens into two subsets having the same average length and average bulk density.
- Store the "dry" subset of specimens on a flat surface at (20 ± 2) $^{o}$C.
- Saturate the "wet" subset of specimens on the perforated shelf in the desiccator filled with distilled water at (20 ± 2) $^{o}$C.
- Apply a vacuum to obtain (6.7 ± 0.3) kPa and maintain for (30 ± 5) minutes.
- Then, let the atmospheric pressure slowly into the desiccator.
- Leave the specimens submerged in water for another (30 ± 5) minutes.
- Calculate the volume and reject the specimens, which have increase more than 1% in volume.
- Place the "wet" subset of specimen in a water bath at (40 ± 1) $^{o}$C for (68 ± 2) hours.
- Bring the specimens to the test temperature, which is (25 ± 1) $^{o}$C. [Note: the specimen should be stored at test temperature at least for 2 hours prior to the test]
- Determine the indirect tensile strength (ITS) of the specimen

**Box 6.1: Summary of water sensitivity test method for hot mix asphalt**

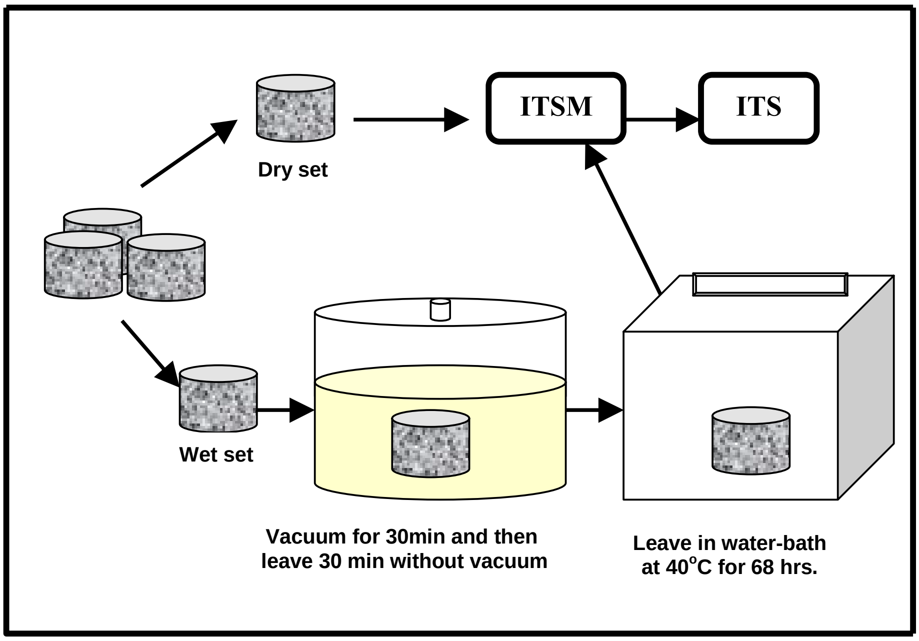



**Figure 6.11: Illustration of procedure for determination of water sensitivity**

### 6.5.2 Ageing

The phenomenon where the bitumen hardens (increases in viscosity) due to oxidation during construction and in service is called ageing (Scholtz, 1995). This hardening is manifested in the stiffness of bituminous mixture, which to some extent improves the load spreading capabilities of the bituminous mixture and gives better resistance to permanent deformation. However, if the hardening is excessive the mixture can become brittle and crack, thus resulting in a reduction of the pavement's ability to support traffic-induced stresses and strains (Scholtz, 1995).

Brown and Scholtz (2000) have investigated various methodologies for long term ageing of compacted bituminous mixtures and developed a test protocol for it. In their investigation, asphalt specimens were initially exposed to 85$^{o}$C for 120 hours in a force-draft oven in the absence of light and then the stiffness modulus test was carried out in accordance with BS DD 213 (BSI, 1993). They concluded that such a test protocol produces useful practical results.

Long-term ageing results in increasing of the elastic modulus and brittleness of bituminous mixtures due to volatilisation, oxidation and hardening of the binders. Increasing the elastic modulus will improve the ability of the mixture to distribute the load within the pavements structure, but on the other hand increasing the brittleness as a result of excessive hardening will lead to pavements cracking and loss durability in terms of water resistance and moisture susceptibility (Li et al., 1995).

At present, there is no standard test protocol available for ageing tests of bituminous mixtures in the United Kingdom. The protocol, which was used by Scholz (1995), which was based on the Strategic Highway Research Program (SHRP) methodology, was adopted to assess the durability of asphalt mixtures in this investigation. Two methods were established in SHRP, i.e. short-term oven ageing (STOA) and long-term oven aging (LTOA). However, in this investigation only LTOA was utilised as this test is conducted to simulate the ageing of asphalt mixtures in service.

Two mechanical properties of the mixtures were evaluated, indirect tensile stiffness modulus and indirect tensile strength. The protocol of the LTOA methodology is shown in Box 6.2.

**Long Term Aging Test Protocol**

- Compacted specimen is placed in a forced-draft oven at a temperature of 85±1°C for 120±0.25 hours.
- The oven is switched off at the end of the ageing period and left to cool to room temperature. (24 hours should be sufficient cooling period).
- The specimen is removed after it has cooled to room temperature.
- Indirect tensile stiffness modulus test is performed followed by the Indirect Tensile Strength (ITS) test.

(NOTE: The specimen should not be touched or removed from the oven until it has cooled to room temperature)

**Box 6.2: Summary of Long Term Aging Protocol (Scholz, 1995)**

In this investigation ITSM determinations were carried out in accordance with BS DD 213 (BSI, 1993) while the determination of ITS was carried out in accordance with BS EN 12697-23 (BSI, 2003). The LTOA test results were evaluated based on the magnitude of the increase in stiffness modulus of the mixture. The ratio of stiffness modulus after ageing to the stiffness modulus before ageing was referred to as the stiffness modulus ratio or ageing index. A similar evaluation was also carried out using the test results of the ITS to obtain an ageing index.

## 6.6 LABORATORY ASPHALT MIX DESIGN PROGRAMME

In general the overall work plan presented in this Chapter consisted of two tasks. The first task was the determination of optimum binder content of the target mixtures and the second was the laboratory tests carried out to determine the mechanical properties of the target mixtures. For the first task, the laboratory work involved determination of optimum aggregate gradation for the mixtures, mixing the aggregate with a range of bitumen contents, compaction, Marshall Stability and flow test, indirect tensile stiffness modulus test and indirect tensile strength (ITS) tests on all specimens. The second task was to further investigate the mechanical properties and durability of asphalt mixtures produced at the optimum binder content.

### 6.6.1 Selection of the Aggregate Gradation

The approach adopted in this study was based on earlier research carried out by Cooper et al. (1992) where a modification of Fuller's curve was used to define the maximum target aggregate gradation. The equation is based on the maximum aggregate size, the amount of added filler and the constant *n*. The equation was presented earlier as Equation 4.3.

Based on gradation (Equation 4.3) two exponent *n* values, 0.5 and 0.7 were selected for comparison purposes. The gradation of tin slag aggregate as received is shown in Figure 6.12 together with best packing gradation lines with exponent n values of 0.5 and 0.7. In Chapter 3, it was found that the tin slag has a very small amount of aggregate with size greater than 10mm. Therefore in this study it was assumed that all the tin slag has gradation passing 10mm sieve size and any tin slag particles with sizes greater than 10mm were omitted from the gradation.

Under the British Standard BS 4987-1:2001, the specification for 6mm dense graded surface course should follow the gradation limits as shown in Table 6.3. However when plotted and compared with the tin slag gradation, it was found that the slag gradation fell outside the British Standards. Figure 6.13 shows that in particular, tin slag particles with sizes above 1.18mm fell outside or above the upper limit specified by the standard. Based on the values in Table 6.3, Figures 6.12 and 6.13, and also for practical purposes it was decided not to modify the tin slag gradation to fit the BS limits.

For the purpose of this research, the unaltered gradation tin slag (as received) was used for manufacturing asphalt mixtures. For the first mixture, which was referred to as TS-tin slag mixture, it was proposed that the tin slag be used to manufacture asphalt mixtures without adding any conventional mineral aggregates. However, from gradation analysis of the tin slag, it was found that the tin slag had very small or negligible amounts of filler i.e. 0.5%. On this basis, 10 % by mass of limestone filler was added to the mixture.

**Table 6.3: Specification of 6mm size dense surface course (BS 4987-1:2001)**

| Test sieve aperture size | Aggregate % by mass passing |
|---|---|
| 10 mm | 100 |
| 6.3 mm | 90 to 100 |
| 3.35 mm | 55 to 75 |
| 1.18 mm | 20 to 50 |
| 300 μm | 8 to 25 |
| 75 μm | 2 to 10 |

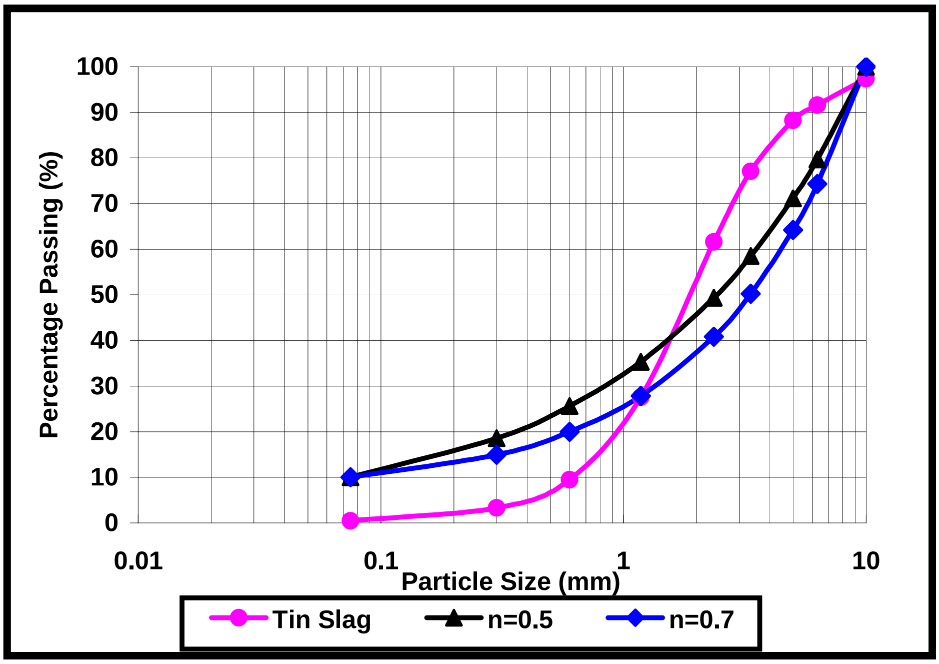



**Figure 6.12: Comparison between tin slag gradation and Fuller's gradation**

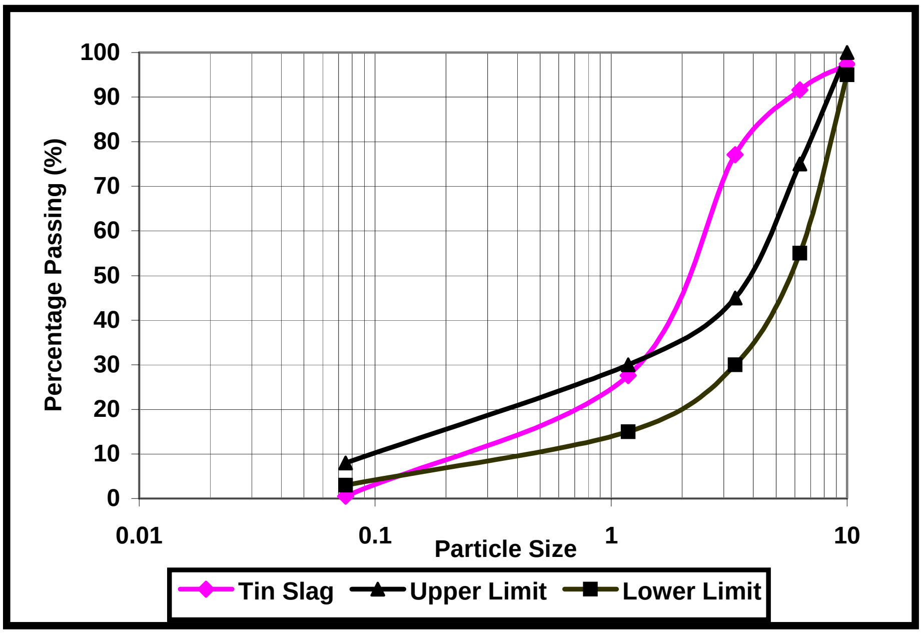



**Figure 6.13: Comparison between tin slag gradation and the upper and lower limits of 6mm dense graded surface course**

For the second tin slag mixture, which was referred to as TB-tin slag mixture, the gradation of the tin slag was modified by adding conventional coarse aggregates (i.e. granite) with size between 10 mm and 20 mm. The purpose of adding the conventional coarse aggregate was to evaluate any changes in the mechanical properties of the mixtures. The amount of conventional aggregate added was based on the modified Fuller's gradation with n=0.5. The gradations of the TS-tin slag and the TB-tin slag mixtures are presented in Tables 6.4 and 6.5 respectively.

The design of the control mixtures grading was also based on the modified Fuller's Equation 4.3 proposed by Cooper et *al*. (1992). Two control mixtures were designed in this study. The first control mixture, the CS-control mixture, was composed of conventional aggregate with 10mm maximum nominal aggregate size. The properties of this mixture were compared with the properties of TS-tin slag mixture. Table 6.6 shows the gradation of CS-control mixture. The second control mixture, CB-control mixture, was composed of conventional aggregate with 20mm maximum nominal aggregate size. This mixture was used to make a direct comparison with the TB mixture. Table 6.7

shows the gradation of CB mixture. Granite was used as the conventional aggregates for both control mixtures.

A summary of the percentages of each component in the slag and control mixtures is presented in Figure 6.14. The figure clearly shows that the CB-control mixture contains 90% conventional aggregate with 10% limestone filler and that the TB-tin slag mixture contains 27.1% granite coarse aggregate added to 61.9% unaltered tin slag with 10% limestone filler. On the other hand the CS-control mixture contains the same percentages of conventional aggregates, though of a smaller maximum size, as CB, whilst the TS mixture contains 90% unaltered tin slag with 10% limestone filler content. It should be noted that the grading of aggregate used in designing the mixtures was based on mass rather than volumetrics. However, when volumetric grading is used, the gradation of the control aggregate shows a small difference (see Figure 6.14) and for tin slag the grading by mass shows a difference between 2% and 4% compared to the volumetric grading. Nevertheless, these differences would not affect the determination of optimum binder content for mixtures since a range of bitumen content was used.

**Table 6.4: Aggregate grading for TS-tin slag mixture**

| Size (mm) | % passing by mass for n=0.5 | Aggregate mass (%) |
|---|---|---|
| 10 | 100.00 | 90 (tin slag) |
| 6.3 | 79.68 | |
| 3.35 | 58.50 | |
| 1.18 | 35.31 | |
| 0.075 | 10.00 | |
| Filler | | 10 |
| | | 100 |

**Table 6.5: Aggregate grading for TB-tin slag mixture**

| Size (mm) | % passing by mass for n=0.5 | Aggregate mass (%) |
|---|---|---|
| 20 | 100.00 | 0.00 |
| 14 | 84.34 | 15.66 |
| 10 | 71.92 | 12.42 |
| 6.3 | 57.94 | |
| 3.35 | 43.37 | 61.92 (tin slag) |
| 1.18 | 27.42 | |
| 0.075 | 10.00 | |
| Filler | | 10.00 |
| | | 100.00 |

**Table 6.6: Aggregate grading for CS-control mixture**

| Size (mm) | % passing by mass for n=0.5 | Aggregate mass (%) |
|---|---|---|
| 10 | 100.00 | 0 |
| 6.3 | 79.68 | 20.32 |
| 3.35 | 58.50 | 21.18 |
| 1.18 | 35.31 | 23.18 |
| 0.075 | 10.00 | 25.31 |
| Filler | | 10.00 |
| | | 100.00 |

**Table 6.7: Aggregate grading for CB-control mixture**

| Size (mm) | % passing by mass for n = 0.5 | Aggregate mass (%) |
|---|---|---|
| 20 | 100.00 | 0.00 |
| 14 | 84.34 | 15.66 |
| 10 | 71.92 | 12.42 |
| 6.3 | 57.94 | 13.98 |
| 3.35 | 43.37 | 14.57 |
| 1.18 | 27.42 | 15.95 |
| 0.075 | 10.00 | 17.42 |
| Filler | | 10.00 |
| | | 100.00 |

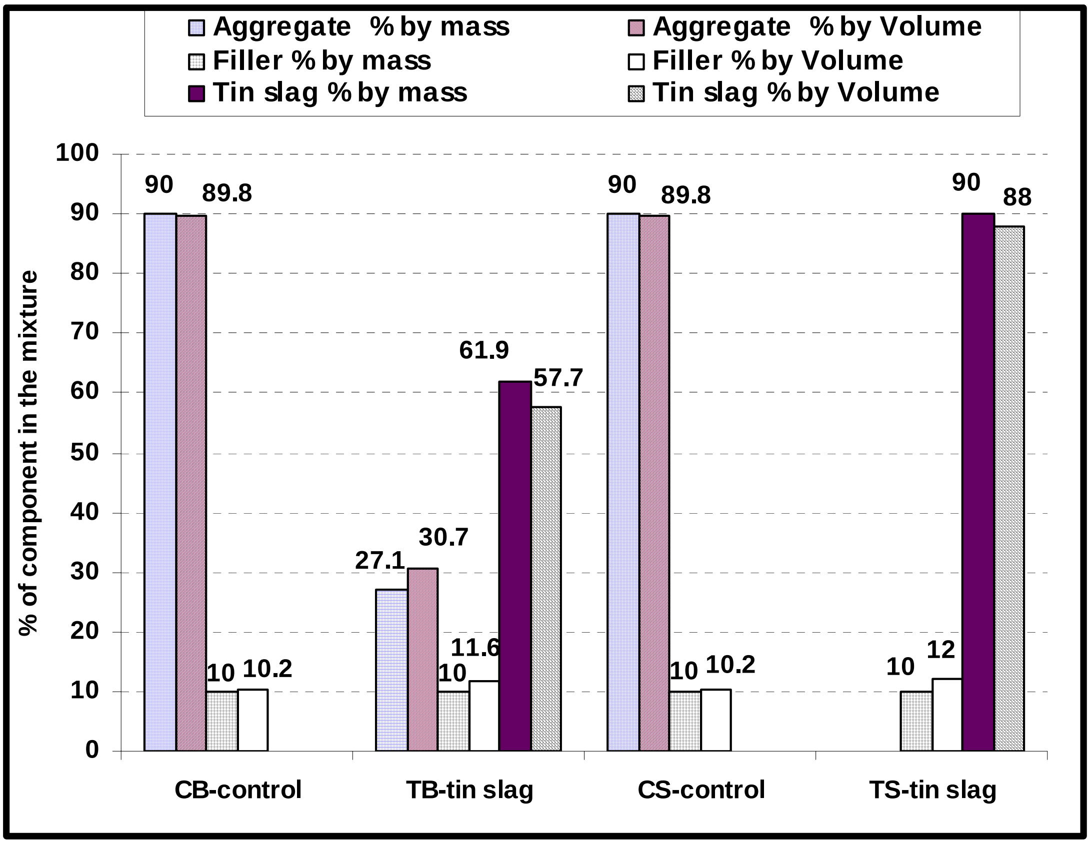



**Figure 6.14: Percentage of each component in the mixtures**

### 6.6.2 Selection of Binder Grade and Content

Selection of the bitumen grade to be used in a particular bituminous mixture is largely dependent on the conditions to which the mixture will be subjected such as traffic and climate. Hard bitumens would be normally selected for wearing course mixtures on roads with heavy traffic volumes in hot climates, whereas softer bitumens would be selected for the same layer in a cold climate. In this study, a 100 penetration grade Middle Eastern bitumen was used to manufacture all the bituminous mixtures. The selection of the grade of bitumen used in this research was on one hand based on the availability of bitumen types at the Nottingham Centre for Pavement Engineering (NCPE) at the time of study and on the other hand based on the most likely types of bitumens that would be used with tin slag in Malaysia. The binder was provided in kind by Shell Bitumen UK.

When selecting suitable binder content for each gradation, a procedure of mixing and compacting the graded aggregate specimens at different levels of binder contents was required. Mixture properties were determined at each level of binder content and graphs were generated showing the change in mixture properties with changes in the binder content. The optimum binder content for each mixture type was carefully selected after analysing this data.

The binder content requirement for a dense graded surface course asphalt mixture with a 6 mm top aggregate size is 5.7 % by mass of the total mixture [in accordance with BS 4987-1:2001 (BSI, 2001)]. This bitumen content is applicable for a mixture containing steel slag aggregates. Since there are no standards covering tin slag in asphalt mixtures, the initial proposed binder content ranges for the asphalt mixtures produced in this investigation were as follows:

CB-control mixture – 4.4%, 4.7%, 5.0%, 5.5% and 6.0% by weight of mixture
TB-tin slag mixture – 4.0%, 4.5% and 5.0% by weight
CS-control mixture – 4.4%, 4.7%, 5.0% and 5.1% by weight
TS-tin slag mixture – 4.0%, 4.5% and 5.0% by weight

The bitumen was preheated in 2 litre containers at the optimum mixing temperature in an oven for two hours prior to mixing. The heating duration was very important to ensure that the bitumen was at the correct mixing temperature and to prevent over-heating the bitumens which can cause bitumen hardening.

### 6.6.3 Filler Content

The filler content can influence mixtures by modifying the grading of the fine aggregates thus producing a denser mixture with greater aggregate contact. This will also influence the bitumen demand. Fillers also combine with bitumen to form the 'binder' which lubricates and binds the fine aggregate to form the 'mortar'. The filler stiffens and strengthens the bitumen so that the voids in the fine aggregate are filled with a filler/bitumen mixture of higher strength and lower temperature susceptibility (Suparma, 2001).

With regard to tin slag, a proportion of the filler was already present in the tin slag aggregates, but was less than 0.5% by mass of aggregate. This amount of tin slag filler was too low and more filler had to be added when producing asphalt mixtures. According to British Standards BS 4987-1:2001, the filler content requirement for a dense graded surface course with 6 mm top aggregate size is between 2% and 10%. Therefore, 10% limestone filler by weight of aggregate was added to all mixtures.

### 6.6.4 Specimen Preparation

#### 6.6.4.1 Preparation of Aggregate

The metallic pans containing the graded aggregates were placed in an oven set approximately 15$^{o}$C higher than the mixing temperature, which was 150$^{o}$C. At least two hours was required for the aggregate to reach the mixing temperature. All mixing equipment such as the mixing bowl, compaction moulds, spatulas and other metallic tools were also pre-heated to the same temperature.

#### 6.6.4.2 Mould Preparation

Standard steel gyratory moulds with 100mm internal diameter and 150mm height together with steel base and top plate were used for compacting the specimens. All components were cleaned and placed in an oven at mixing temperature for two hours prior to use. Figure 6.15 shows a steel mould together with top and bottom plates used for this research.

### 6.6.4.3 Preparation of Mixture

Mixing was conducted using a thermostatically controlled preheated twin paddle mixer bowl as shown in Figure 6.16, pre-set at the required mixing temperature. The heated aggregates were then charged into the mixer bowl and the required amount of bitumen was poured into the mixing bowl. The aggregates and bitumen were then mixed until the aggregate particles were thoroughly coated with bitumen.

Heated moulds were removed from the oven one at a time and a base plate was securely placed inside the mould. A plastic disk was also placed on top of the base plate. The plastic disks were used to assist in demoulding the specimens. The mould plus base plate was then placed on a balance, the internal surfaces of the mould were sprayed with silicone oil and the balance zeroed. With the aid of a scoop, the mix was transferred into the mould until the required specimen weight was achieved. The top surface of the loose hot mix was labelled and the specimen's weight and temperature noted. The weight of the sample was input into the computer programme that operates the gyratory compactor. A plastic disk was then placed on top of the levelled specimen, and finally the top plate was positioned on top of the mix. The specimen was thus ready for compaction.

**Figure 6.15: Gyratory Steel mould and end-plates**

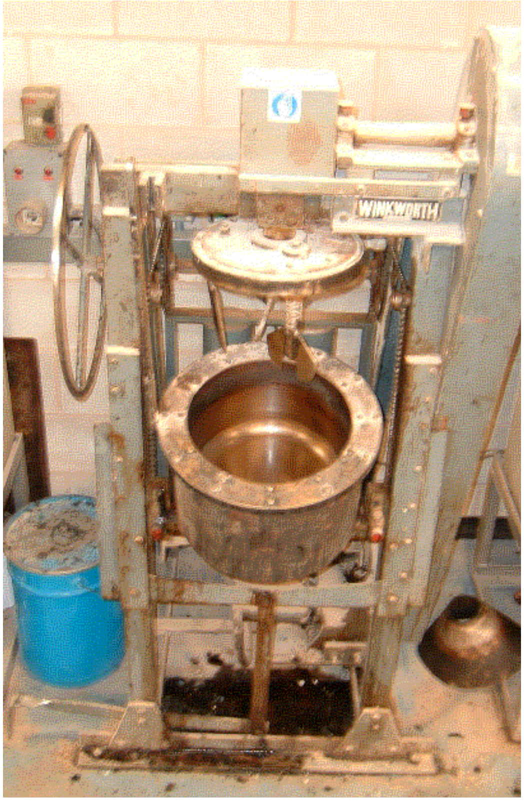



**Figure 6.16: The mixer used for asphalt specimen production**


**Figure 6.17: Gyratory compactor**

#### 6.6.4.4 Compaction of specimen

A gyratory compactor was used for compaction of all specimens (see Figure 6.17). The compactor was used rather than using the Marshall hammer, to prevent the tin slag from break during compaction since the impact value for tin slag was not very high. On the other hand, the roller compactor was not used because of the limited amounts of tin slag available.

The mould containing a hot asphalt specimen was placed into the gyratory compactor, centred under the loading ram and clamped into position. The compactor parameters were controlled by a computer programme. In this study, the gyratory compaction conditions were set to $1.25^{o}$ angle of gyration and the maximum number of gyrations was used to compact the specimens which were 300 gyrations or maximum density. During compaction, the ram loading system was maintained at a constant axial pressure of 600kPa. The gyratory compactor ceases compacting after reaching the maximum number of gyrations or the required density. The angle of gyration is released and the loading ram is raised automatically. The mould was then removed from the compactor and allowed to cool to room temperature before extruding the specimens. The plastic disks at the bottom and top of the specimen were then removed and the specimen was identified with a number.

### 6.6.5 Determination of Optimum Binder Content

The optimum binder content of each mixture was determined using the Marshall method. The Marshall method recommends that to determine the optimum binder content, each compacted mixture is subjected to volumetric analysis and Marshall Stability and Flow tests. However, in this study, along with the above approach, the mixtures were also tested to determine the indirect tensile stiffness modulus and indirect tensile strength values.
Volumetric analysis involved determination of bulk density of compacted specimens,

specific gravity of aggregate, specific gravity of mixture, maximum density of the mixture - Theoretical and measured (rice density), VMA and the porosity of the compacted mixture (voids content). The determination of the above properties has been discussed earlier in Section 6.3.2.

In this study, Marshall stability and Marshall flow values were determined in accordance with British Standard Draft BS EN 12697-34 as discussed in Section 6.3.1.1. Indirect Tensile Stiffness Modulus (ITSM) tests were carried out on the specimens using the Nottingham Asphalt Tester (NAT) apparatus according to the procedure mentioned in Section 6.4.1. Indirect tensile strength (ITS) test was carried out in accordance with BS EN 12697-23, *Determination of the indirect tensile strength of bituminous specimens* (BSI, 2003) as described in Section 6.4.4.

### 6.6.5.1 Interpretation of the Test Data

Before the optimum binder content can be determined the data obtained from the aforementioned tests must be analysed and interpreted. The following graphical plots were prepared to analyse the data.

- Stability versus bitumen content
- Flow vs. bitumen content
- Bulk density vs. bitumen content
- Percent air voids vs. bitumen content
- Percent voids in mineral aggregate (VMA) vs. bitumen content
- Stiffness modulus vs. bitumen content
- Indirect tensile strength vs. bitumen content

The test curves generally have the following properties:

- The stability value increases with increasing bitumen content up to a maximum value beyond which the stability decreases.
- The flow value increases with increasing bitumen content

- The curve for bulk density is similar to the stability curve except that the maximum bulk density occurs at a slightly higher bitumen content than the maximum stability.
- The percent of air voids decreases with increasing bitumen content, ultimately approaching a minimum air void content.
- The percent voids in the mineral aggregate (VMA) generally decreases to a minimum value then increases with further increase in bitumen contents.

Considering the above curves, the optimum binder content of asphalt mixture is then determined.

### 6.6.6 Results and Discussion

#### 6.6.6.1 CS-control Mixture Design

Figure 6.18 shows the variation in bulk density versus bitumen content for CS-control mixture. Based on the curve it is clearly evident that the maximum bulk density value was achieved at 4.9% bitumen content with a value of 2.52Mg/m$^3$. As recommended by the Asphalt Institute, the optimum voids content (porosity) for asphalt mixtures should be in between 3% and 5%. Here the 4% voids content was selected to determine optimum binder content. Therefore, at 4% void content, the mixture was found to have 4.7% bitumen content as shown in Figure 6.19. In terms of Marshall Stability, the CS-control mixture had maximum stability, which was 19.3kN, when the content of bitumen was 4.7% as shown in Figure 6.20. Also, at bitumen content 4.7% it was found that the Marshall flow value was 3.6mm which is also within the limits allowed by the Asphalt Institute (See Figure 6.21).

The indirect tensile strength (ITS) tests indicated that the CS-control mixture was strongest when the bitumen content was 4.7% as shown in Figure 6.22. The results show how the test can be used as an indicator to determine the optimum binder content of bituminous mixtures. Figure 6.23 shows the results of indirect tensile stiffness modulus tests on the CS-control specimens containing a range of bitumen contents. It was found that the relationship between ITSM and the binder contents of the CS-control specimens was reasonable to aid in determination of optimum binder content of the mixture. Based on this relationship, the stiffness modulus was found to be slightly higher at 4.7% bitumen content compared with the other bitumen contents. Hence, based on the results obtained from laboratory tests and the parameters calculated above, it can be concluded that the optimum binder content for the CS-control mixture was 4.7%.

Figure 6.24 shows the voids in mineral aggregate values of CS-control mixture investigated at a range of bitumen contents. Assuming the optimum binder content of the CS mixture was 4.7%, Figure 6.24 shows that the VMA value of the mixture at that bitumen content was 16%. In accordance with the Asphalt Institute recommendations, this value of VMA would be well within the acceptable range. A summary of the test results is shown in Table 6.8. After taking into consideration all the parameters mentioned previously it was decided that the optimum binder content for CS-control mixture was 4.7% by weight of mixture.

**Table 6.8: Summary of the test results used for determination of optimum binder content of CS-control mixture**

| Characteristic | Optimum Binder Content |
|---|---|
| Maximum bulk density | 4.9% |
| Maximum Marshall stability | 4.7% |
| 4% voids content | 4.7% |
| Maximum indirect tensile strength | 4.7% |

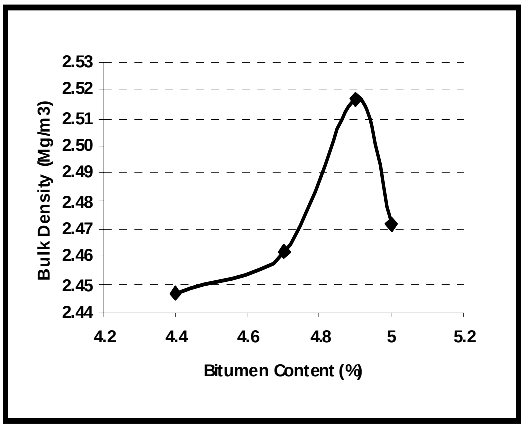
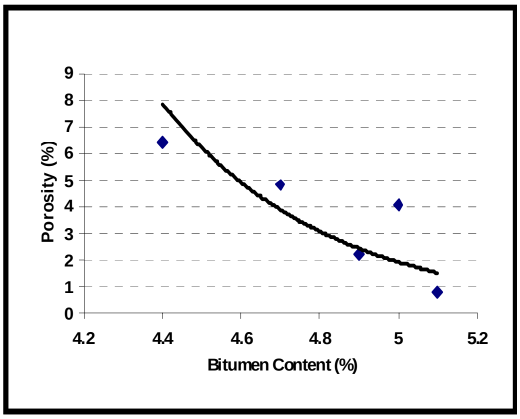



Figure 6.18: Bulk Density of CS-control mixture with a range of bitumen content




6.19: Porosity of CS-control mixtures measured using rice density method.

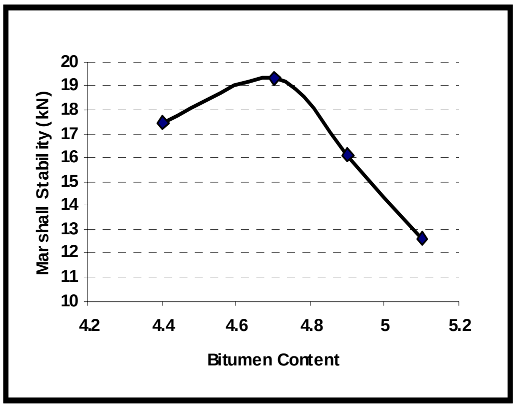



Figure 6.20: Marshall stability for CS-control mixture

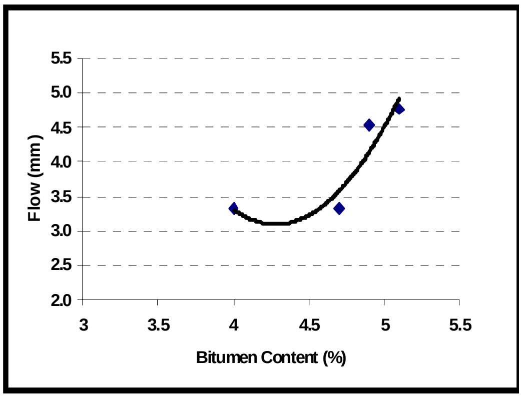



Figure 6.21: Marshall flow for CS-control mixture

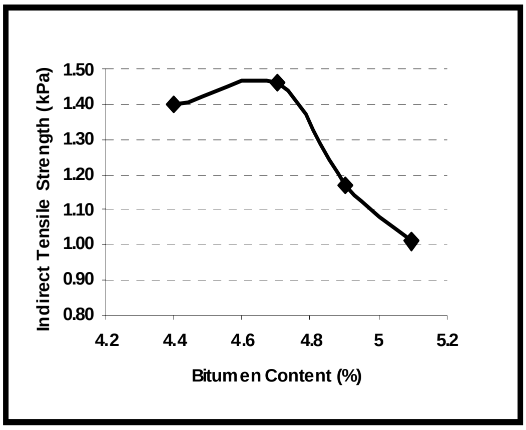



Figure 6.22: Indirect tensile strength test result for CS-control mixture

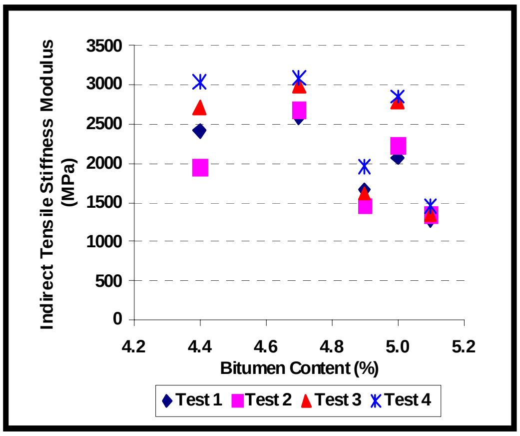



Figure 6.23: Indirect Tensile Stiffness Modulus (ITSM) test result for CS-control mixture

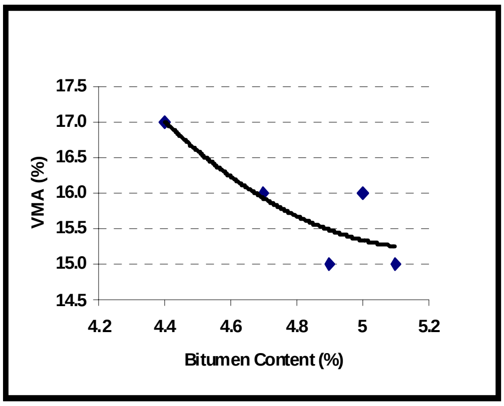



Figure 6.24: Voids in mineral aggregate for CS-control mixture at a range of bitumen content

### 6.6.6.2 TS-tin slag Mixture Design

The results obtained from the laboratory tests together with all the calculated parameters required for the determination of the optimum binder content for the TS-tin slag mixture are presented in Figures 6.25 to 6.30. Figure 6.25 shows that the relationship between bulk density and binder content was very poor and mainly for that reason, it was impossible to determine the value of optimum binder content. Results shown in Figure 6.26 for the Marshall Stability tests also clearly show a poor relationship between binder contents and stability of the TS-tin slag mixture. On the other hand, from the volumetric analysis it was observed that the TS-tin slag mixture had a very high porosity that far exceeded the range of values recommended by the Asphalt Institute.

It was also observed that the indirect tensile strength test results gave no indication as to the position of the optimum binder content (see Figure 6.30). However, the results from the indirect tensile stiffness test gave some indication of the value of optimum binder content, although this was not very clear. Also, the stiffness was rather low. Figure 6.29 shows that the optimum bitumen content value was likely to be in between 4% and 4.5% at which point the mixture gave the highest stiffness.

However, after taking into considerations all the analysis of test results, it was decided that the TS-tin slag bituminous mixture was not suitable for use in road pavements on the grounds that the stiffness was very low, the flow very high and the stability very low.

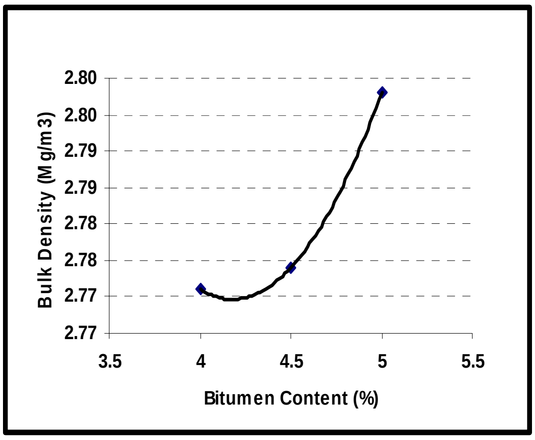



Figure 6.25: Bulk density of TS-tin slag mixture against bitumen content

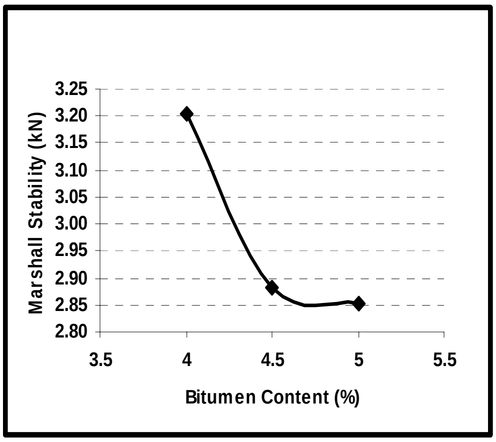



Figure 6.26: Marshall stability test for TS-tin slag mixture

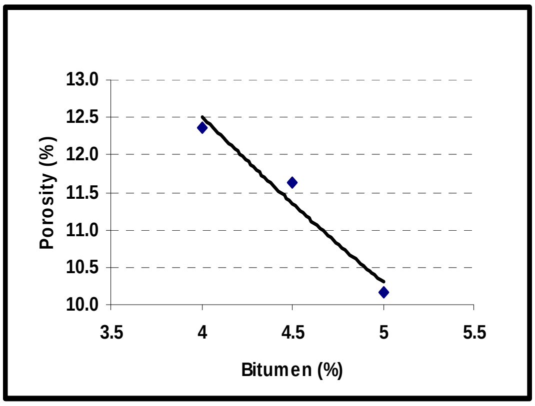



Figure 6.27: Porosity of TS-tin slag mixture measured using rice density method.

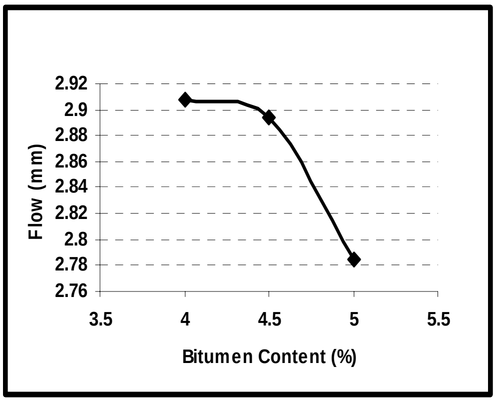



Figure 6.28: Marshall flow test for TS-tin slag mixture

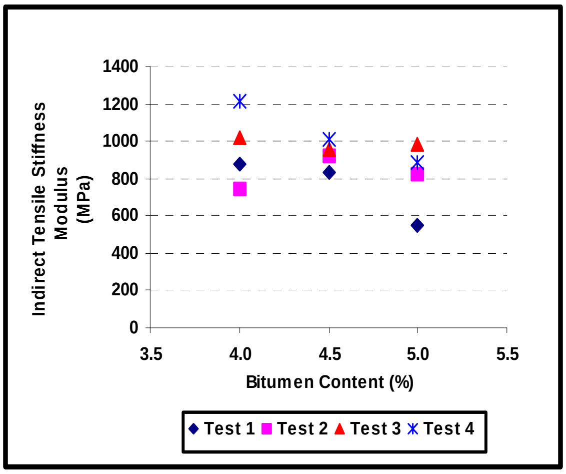



Figure 6.29: Indirect tensile strength test result for TS-tin slag mixture

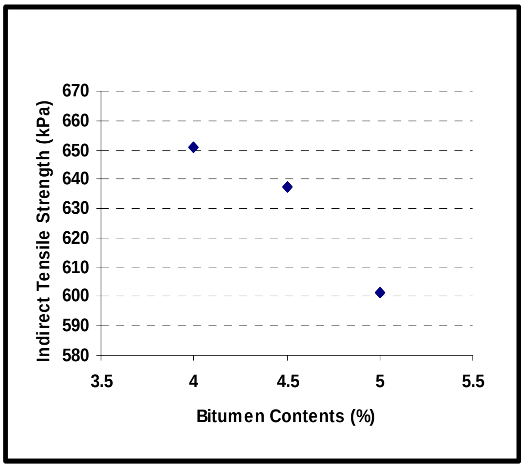



Figure 6.30: Indirect Tensile Stiffness Modulus (ITSM) test results for TS-tin slag mixture

### 6.6.6.3 TSS-tin slag Mixture Design

Based on the mechanical and volumetric test results from TS-tin slag mixture design shown in Section 6.6.6.2, most importantly it was found that the mixture lacked strength, stiffness and had excess flow, therefore the mixture was not suitable for use in road construction. Thus, it was decided that the design mixture required modification. Hint sand with particle sizes ranging between 1.8 mm and 0.075 mm, was added to modify the mixture gradation. For this research purpose the mixture was called TSS-tin slag mixture. Table 6.9 shows the gradation of TSS mixture. A maximum of 20 % sand by mass of aggregate, 10% limestone filler and 70 % of tin slag were blended to make the TSS-tin slag aggregate mixture.

The optimum binder content for TSS-tin slag mixture was determined using the same procedure as for CS-control and TS-tin slag mixtures. However, in this case the range of binder contents used in the study were 4%, 4.3%, 4.6% and 4.9%.

**Table 6.9: Aggregate grading for TSS-tin slag mixture**

| Size (mm) | % passing by mass for n=0.5 | Aggregate mass % |
|---|---|---|
| 10 | 100.00 | 0 |
| 6.3 | 79.68 | |
| 3.35 | 58.50 | 70 (tin slag) |
| 1.18 | 35.31 | |
| 0.075 | 10.00 | 20 (hint sand) |
| Filler | | 10 |
| | | 100 |

Figure 6.31 presents the test results showing the relationship between bulk density and bitumen content for the TSS-tin slag mixtures. The curve gave an indication that the optimum binder content value at maximum bulk density for the TSS-tin slag mixture was in between 4.3% and 4.7%. While Figure 6.32 clearly shows that the optimum binder content value can be achieved at bitumen contents within the range of 4.3% and 4.5% with the peak Marshall stability values between 9.4kN and 9.6kN respectively.

In this study, a 4% porosity value was selected to determine the optimum binder content this was achieved at 4.5% binder content as shown in Figure 6.33. From the above analysis it was decided that the optimum binder content for TSS-tin slag mixture was 4.5% bitumen. This optimum binder content value was then input into Figure 6.34 to determine the Marshall Flow at optimum bitumen content. It was observed that the Marshall Flow value for the TSS-tin slag mixture was within the limits recommended by the Asphalt Institute. Volumetric analysis also shows that the mixture voids in mineral aggregate value at optimum binder content was higher than that recommended by the Asphalt Institute as shown in Figure 6.52. The result can be seen in Figure 6.37.

The use of indirect tensile stiffness modulus results (Figure 6.35) and indirect tensile strength test results (Figure 6.36) in this part of study were not very helpful in determining the optimum binder content. It was not possible to determine a direct relationship between these two parameters and bitumen content.

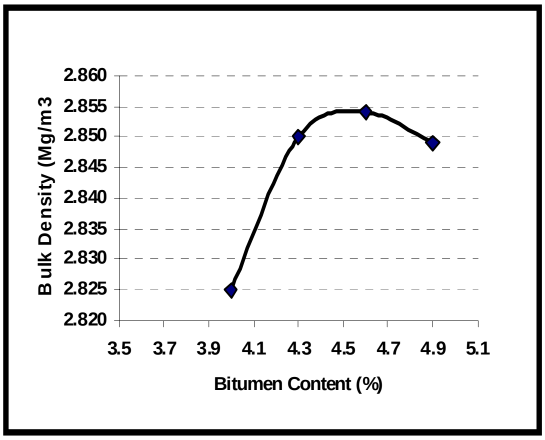



Figure 6.31 Bulk density for TSS-tin slag mixture

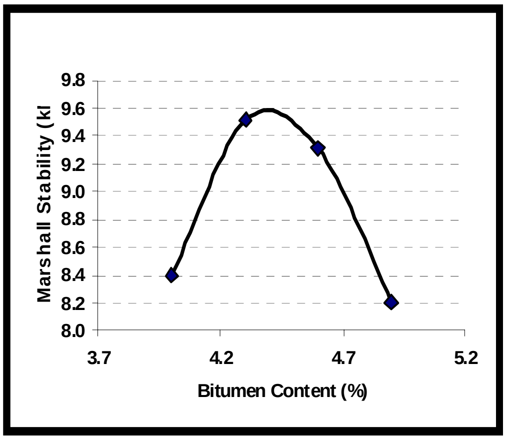



Figure 6.32: Marshall Stability test for TSS-tin slag mixture

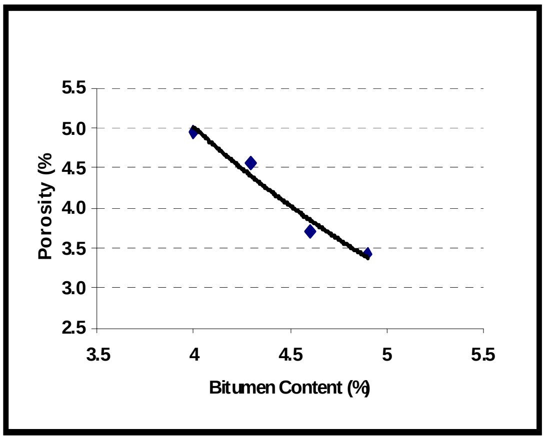



Figure 6.33: Porosity for TSS-tin slag mixture

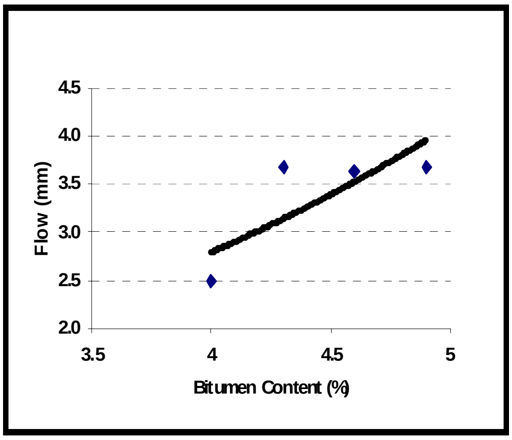



Figure 6.34: Marshall Flow test for TSS-tin slag mixture

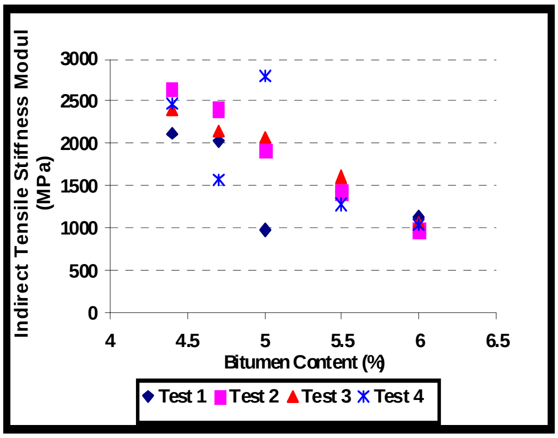



Figure 6.35: Indirect Tensile Stiffness Modulus test for TSS-tin slag mixture

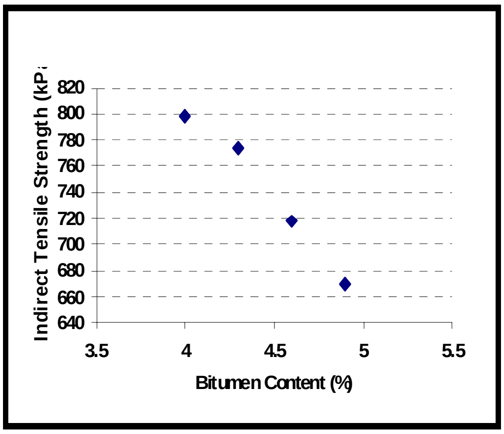



Figure 6.36: Indirect tensile strength test for TSS-tin slag mixture

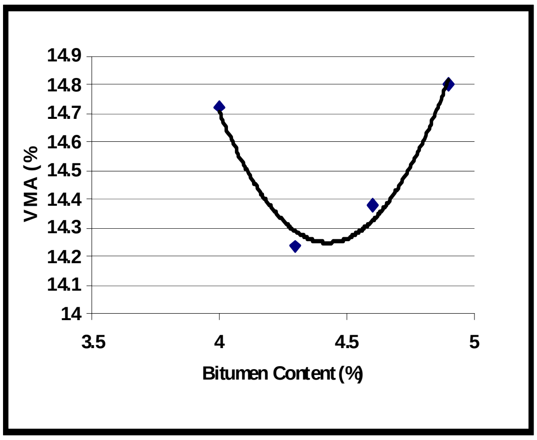



Figure 6.37: Voids in mineral aggregate for TSS-tin slag mixture

### 6.6.6.4 CB-control Mixture Design

As shown in Figure 6.38 the value of optimum binder content for maximum compacted density was between 4.5% and 5%. It can also be clearly seen in Figure 6.40 that the optimum binder content for maximum stability was in the range of 4.5% to 4.8%. Unfortunately, when ultimate stability was achieved at a binder content value in the range 4.5% and 4.8%, it was observed that the porosity value of the resultant mixtures were very low, in between 1% and 2% as shown in Figure 6.39. This porosity range is below the limits recommended by the Asphalt Institute which is between 3% and 5%. After taking into consideration all the above factors, a compromise was reached, and the optimum binder content for the CB mixture was selected at 4.5%. At 4.5% bitumen content the porosity of the mixture was found to be 2%. Although it was still lower than the minimum requirement i.e. 3%, it can still be acceptable if a tolerance of 2% porosity was applied as explained by the Asphalt Institute (1989).

With regard to the Marshall Flow test, it can be clearly noted that the value of flow at the design binder content value of 4.5% was lower than 4.4mm (see Figure 6.41), which was within the recommended Asphalt Institute limits. It was also observed in Figure 6.40 that the stability of the mixture at 4.5% binder content (i.e. 13250N) was higher than the Asphalt Institute minimum specification requirement for heavy traffic i.e. 8006N. The percentage of voids in mineral aggregate (VMA) values were also calculated for all the bitumen contents investigated. The curve in Figure 6.42 shows that the calculated VMA value at 4.5% binder content is acceptable and within the Asphalt Institute minimum requirements.

Figures 6.43 and Figure 6.44 show the ITSM and ITS test results respectively for the CB-control mixture. Unfortunately, due to the large scatter of ITSM results and the profile of the ITS results, it was not possible to use these two parameters to help identify the optimum binder content. In conclusion and after taking into consideration all the available test data it was decided that the optimum binder content for the CB mixture was 4.5%.

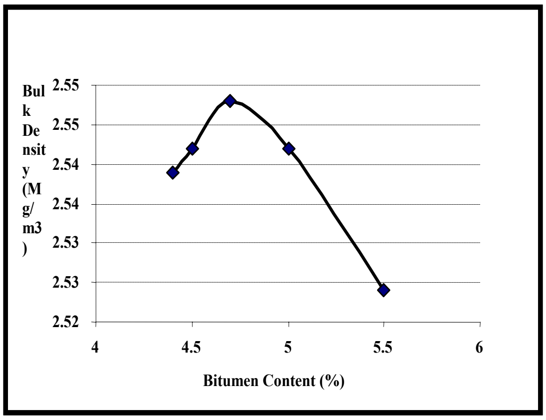



Figure 6.38: Bulk density of CB-control mixture against bitumen contents

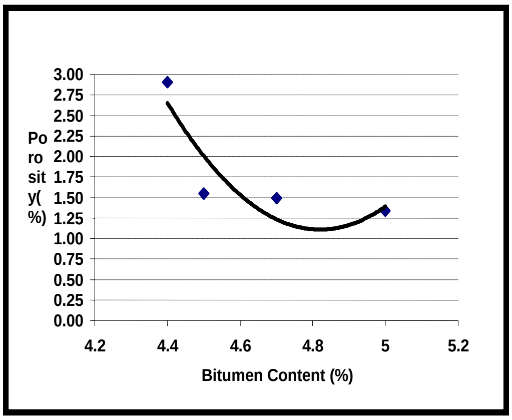



Figure 6.39: Porosity values of CB-control mixtures against bitumen contents

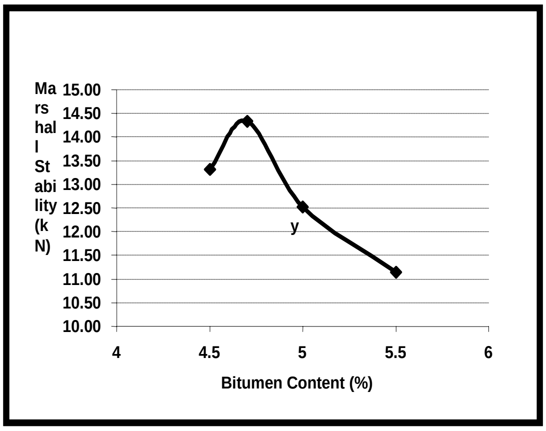



Figure 6.40: Marshal stability for CB-control mixture against bitumen contents

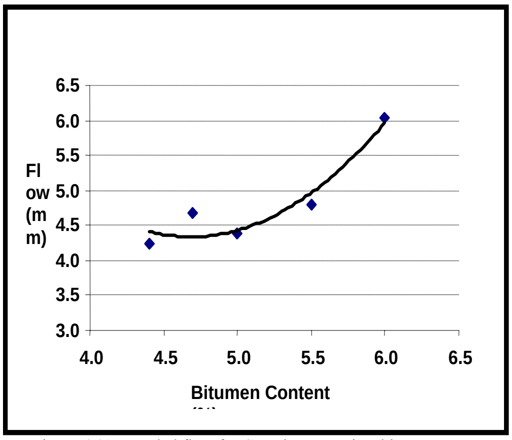



Figure 6.41: Marshal flow for CB mixture against bitumen contents

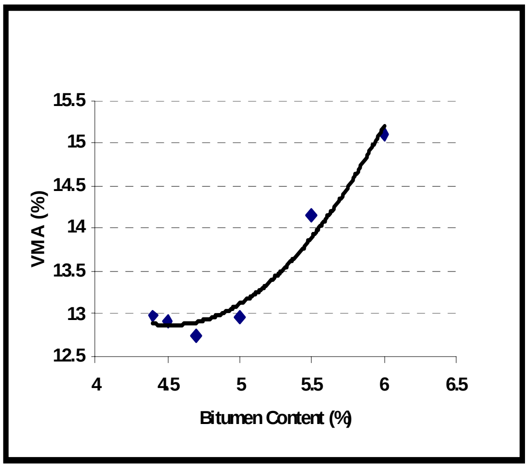



Figure 6.42: Void in mineral aggregate at a range of bitumen content for CB-control mixture

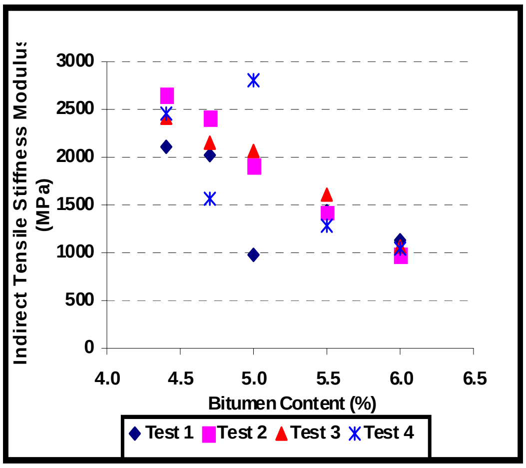



Figure 6.43: ITSM test for CB-control mixture

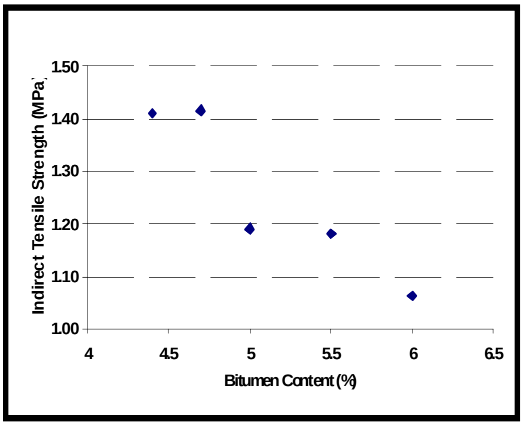



Figure 6.44: ITS test for CB mixture

### 6.6.6.5 TB-tin slag Mixture Design

Once again, the optimum binder content of TB-tin slag mixture was determined using the same criteria as was used when designing the other mixtures. Figure 6.45 shows the results of bulk density determinations for a range of binder contents. The curve clearly shows that the optimum binder content value of the TB-tin slag mixture lies between 4.5% and 4.8%. On the other hand, maximum stability of the mixture was achieved at a binder content of 4.8% as shown in Figure 6.47. In terms of porosity, the optimum binder content at 4% porosity was also found to be 4.8% (see Figure 6.46).

Based on these characteristics, the value of 4.8% binder was selected as optimum binder content for the TB-tin slag mixture. At 4.8% binder content the Marshall Flow value was 3mm as shown in Figure 6.48, which was acceptable under the current recommendations of the Asphalt Institute. The voids in mineral aggregate value achieved at the optimum binder content of 4.8% was 14.3% as shown in Figure 6.49 which exceeded the minimum requirements set by the Asphalt Institute.

Therefore, in conclusion, after taking into consideration all the available test data, the value of 4.8% was selected as an optimum binder content of the TB-tin slag mixture. However, whilst determining the optimum binder content of the TB mixture, the ITSM and ITS test results were not included in the assessment because the results from either test did not display a clear relationship with variations in binder content as was shown in Figures 6.50 and 6.51.

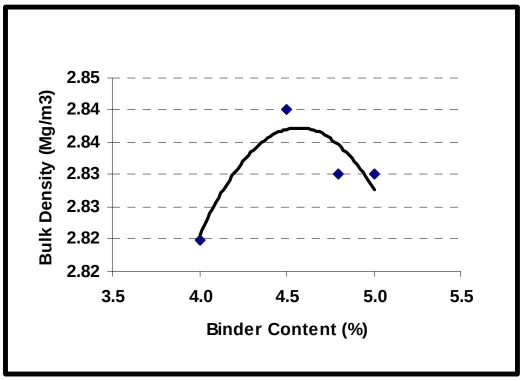



Figure 6.45: Bulk Density for TB-tin slag mixture

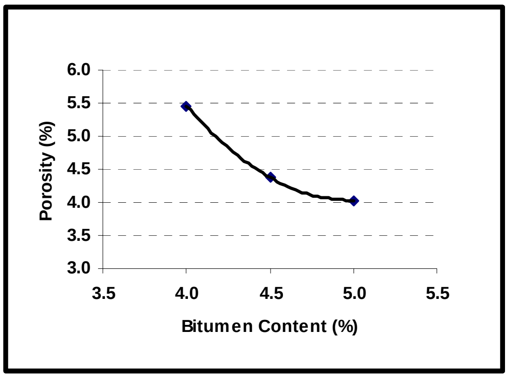



Figure 6.46: Porosity of TB-tin slag mixture

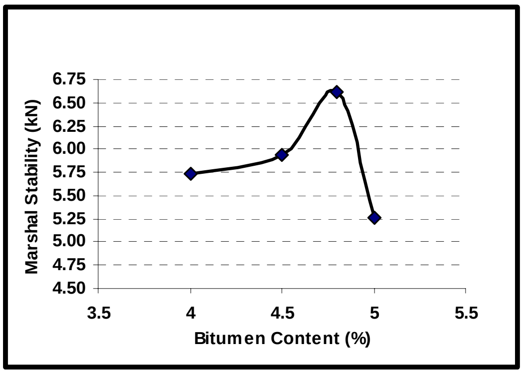



Figure 6.47: Marshall stability test result of TB-tin slag mixture against bitumen contents

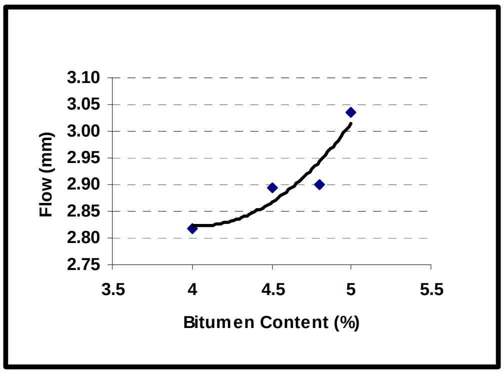



Figure 6.48: Marshall flow test of TB-tin slag mixture

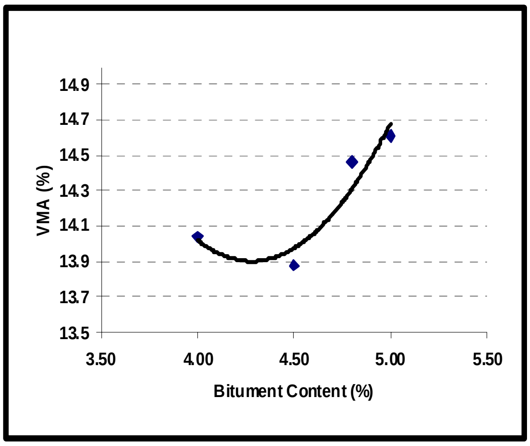



Figure 6.49: Void in mineral aggregate for TB-tin slag mixture

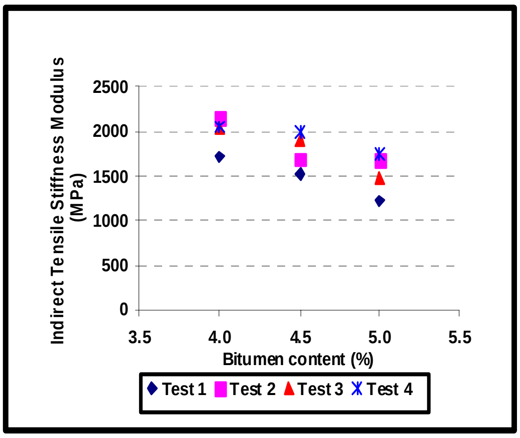



Figure 6.50: Indirect Tensile Stiffness Modulus test result of TB-tin slag mixture

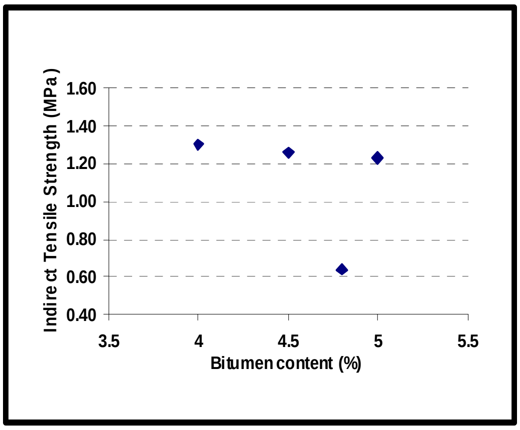



Figure 6.51: Indirect Tensile Strength Test of TB-tin slag mixture

## 6.7 INVESTIGATING THE PERFORMANCE OF THE TIN SLAG AND CONTROL ASPHALT MIXTURES

### 6.7.1 Testing Arrangement

Earlier in Section 6.6 the methodology used to determine the optimum binder content for the CS-control, TSS-tin slag, CB-control and TB-tin slag mixtures was described in detail. Additional specimens from these mixtures at their optimum binder contents were produced to further investigate their mechanical performance in addition to the standard volumetric determinations. The following laboratory tests were conducted to assess in more detail the performance of these mixtures.

- Marshall Stability and Flow Test
- Indirect Tensile Strength (ITS) Test
- Indirect Tensile Stiffness Modulus (ITSM) Test
- Indirect Tensile Fatigue Test (ITFT)
- Repeated Load Axial Test (RLAT)

The Marshall Stability and Flow, ITS, and ITSM tests were performed as described earlier in Section 6.6.

### 6.7.2 Results and Discussion

#### 6.7.2.1 Volumetric Analysis

Results of volumetric analysis consisting of density, porosity and VMA of the CB-control, TB-tin slag, CS-control and TSS-tin slag mixtures at optimum binder content are shown in Table 6.10. With regard to the bulk density of these mixtures, TSS-tin slag and TB-tin slag mixtures have higher bulk density values than the CS-control and CB-control. Such an outcome was expected, as the tin slag has higher specific gravity than conventional aggregate due to the mineral content of the tin slag.

The porosity values of the TSS-tin slag, TS-tin slag and CB-control mixtures were within the values recommended by the Asphalt Institute as shown in Table 6.10. However, it was found from the investigation that the percentage porosity for the CS-control mixture was lower than the 3% recommended by the Asphalt Institute. However, as stated by the Asphalt Institute, a tolerance value of about 2% is still acceptable in some circumstances. Therefore, in this study, the porosity value of 2% at optimum binder content was permitted for the CS-control mixture. Volumetric analysis indicated that all four materials exceed the 13% VMA value recommended by the Asphalt Institute for 20mm maximum nominal aggregate size.

**Table 6.10: Volumetric analysis for CB-control, TB-tin slag, CS-control and TSS-tin slag mixtures with standard deviations**

| Mixture | Bulk Density ($Mg/m^3$) | Porosity (%) | VMA (%) |
|---|---|---|---|
| CB-control | 2.52±0.06 | 3±0.3 | 13.9±0.3 |
| TB-tin slag | 2.76±0.03 | 4±0.97 | 16.1±0.8 |
| CS-control | 2.51±0.01 | 2±0.3 | 13.6±0.2 |
| TSS-tin slag | 2.85±0.01 | 3±0.29 | 14.4±0.3 |

### 6.7.2.2 Marshall Test Assessment

The results of Marshall test analysis are shown in Table 6.11. Clear differences were observed in Marshall Stability value between the control mixtures and tin slag mixtures, with the CB-control mixture having about twice the stability of the TB-tin slag mixture. The result reflects the superior strength and shear resistance of the mineral aggregates in the control mixture. The same trend was also shown for the CS-control and TSS-tin slag mixtures where the average Marshall stability value for the CS-control and TSS-tin slag mixtures were 16kN and 9kN respectively.

It was also observed that there was a significant reduction in Marshall Stability between the mixtures as the maximum aggregate size of the mixture was increased. The CS-control mixture has a Marshall Stability value higher than CB-control mixture while TSS-tin slag mixture has a Marshall Stability value higher than TB mixture. However, all mixtures have Marshall Stability values that exceed the minimum value recommended for Marshall design Criteria by the Asphalt Institute (as shown in Table 6.12). In terms of Marshall Flow values, it was found that all mixtures had lower flow values than the 4mm recommended upper limit except for the CB-control mixture, which had a flow value of 5.3 mm.

**Table 6.11: Marshall Test for results for the CB-control, TB-tin slag, CS-control and TSS-tin slag mixtures with standard deviations**

| Mixture | Average Marshall Stability (kN) | Average Marshall Flow (mm) | Marshall Quotient (kN/mm) |
|---|---|---|---|
| CB-control | 14.25±2 | 5.23±0.8 | 2.79 |
| TB-tin slag | 6.95±0.5 | 1.89±0.02 | 3.68 |
| CS-control | 15.98±0.34 | 3.92±0.4 | 4.10 |
| TSS-tin slag | 8.55±0.9 | 2.55±0.2 | 3.36 |

**Table 6.12: Marshal Design Criteria (Asphalt Institute, 1989)**

| Marshall Method Criteria | Light Traffic | | Medium Traffic | | Heavy Traffic | |
|---|---|---|---|---|---|---|
| | Surface & Base | | Surface & Base | | Surface & Base | |
| | Min | Max | Min | Max | Min | Max |
| Stability (N) | 3336 | - | 5338 | - | 8006 | - |
| Flow (0.25mm) | 8 | 18 | 8 | 16 | 8 | 14 |
| Percent Air Voids | 3 | 5 | 3 | 5 | 3 | 5 |
| Percent Air Voids in Mineral Aggregate (VMA) | See Figure 6.52 | | | | | |

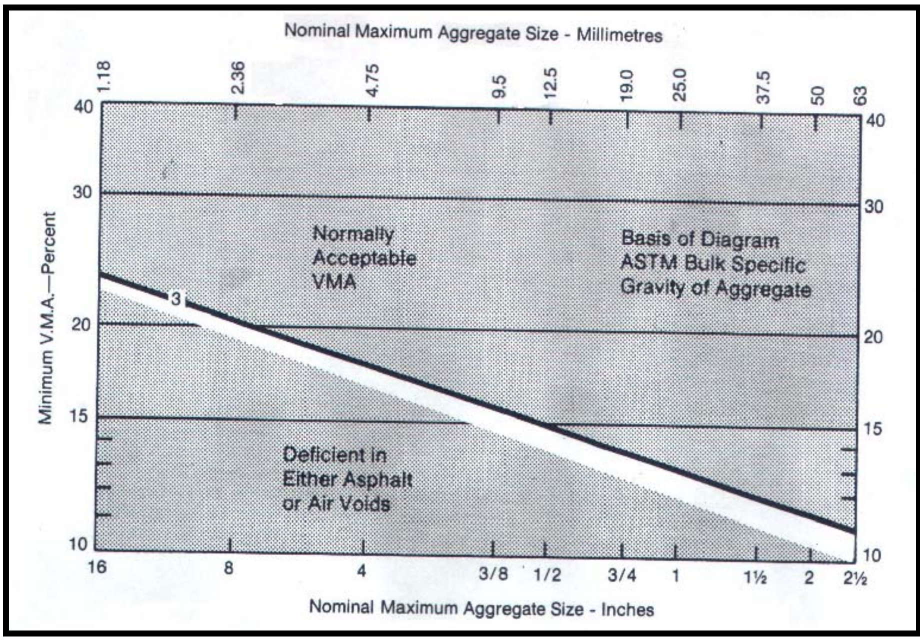



**Figure 6.52: Minimum percent voids in mineral aggregate**

#### 6.7.2.3 Indirect Tensile Strength (ITS) Performance

Table 6.13 presents the results of indirect tensile strength tests conducted on the CB-control, TB-tin slag, CS-control and TSS-tin slag mixtures (three specimens per mixture). The TB-tin slag mixture had an indirect tensile strength value 112% lower than the CB-control mixture. The main reason for such a difference can be attributed to poor grading. Similarly, the indirect tensile strength value for the CS-control mixture was higher than the value obtained for the TSS-tin slag mixture although the difference in tensile strength value between the two mixtures was not very large (only 43%).

Another interesting result which emerged from the investigation was that the measured indirect tensile strength values of the mixture containing 20% sand (i.e. TSS-tin slag mixture) was approximately 54% higher than the indirect tensile strength of the TB-tin slag mixture, although TB-tin slag mixture contained 20mm maximum nominal size aggregate which was larger than TSS-tin slag maximum aggregate size. This finding can be explained in terms of the aggregate packing in the mixture. Sand, which was added to the TSS-tin slag mixture, improved the packing thus increasing the indirect tensile strength value.

**Table 6.13: ITS test results for CB-control, TB-tin slag, CS-control and TSS mixture-tin slag with standard deviations**

| Mixture | Average ITS (kPa) |
|---|---|
| CB-control | 1019±0.1 |
| TB-tin slag | 480±0.01 |
| CS-control | 1060±0.1 |
| TSS-tin slag | 741±0.03 |

#### 6.7.2.4 Indirect Tensile Stiffness Modulus Performance

The indirect tensile stiffness modulus (ITSM) test results for all four mixtures are presented in Figure 6.53. In each case, twenty specimens were tested and the average and 95% confidence limits are shown. The figure indicates that the stiffness of the TB-tin slag is just over half the stiffness of the CB-control mixture. The figure also reveals that the value of indirect tensile stiffness modulus for the TSS-tin slag mixture is slightly lower, by about 39%, than the value obtained for the CS-control mixture. The results thus indicate that the TB-tin slag and TSS-tin slag mixtures are not as stiff as the control mixtures. This may be due to a combination of lack of bond between the tin slag aggregates and the bitumen and the reduced aggregate interlock caused by the spherical shape of the tin slag particles. Therefore, both TB-tin slag and TSS-tin slag will have lower ability to spread traffic loading compared to the control mixtures.

The test results also revealed that the indirect tensile stiffness value for the TB-tin slag mixture (i.e. 1471MPa) was slightly higher than the TSS-tin slag mixture (i.e. 1304MPa). Such a difference can be attributed to the fact that the TB-tin slag mixture contains mineral aggregate with maximum nominal aggregate size bigger than the TSS-tin slag. The maximum nominal size of aggregate added to the tin slag mixture may have played a part in enhancing the stiffness of the mixture.

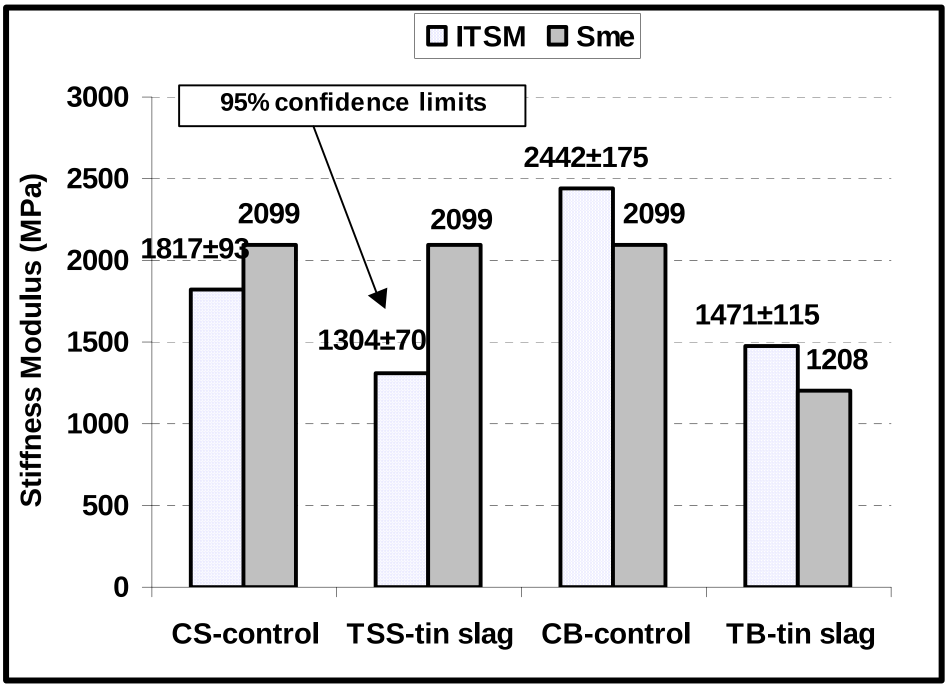



**Figure 6.53: Indirect tensile stiffness modulus test for CS-control, TSS-tin slag, CB-control and TB-tin slag mixtures**

Stiffness modulus of mixtures can also be predicted using the VMA values tabulated in Table 6.10 and Equation 6.9. The stiffness values calculated from the VMA are shown in Figure 6.53. It was found that there were no significant difference between stiffness values determined by the ITSM test and those calculated using VMA except in the case of the TSS-tin slag mixture which had a low ITSM value compared with the Sme value. This may suggest that tin slag is slightly less compatible with this particular bitumen than the conventional aggregate.

$$S_{me} = S_b \left[ 1 + \frac{257.5 - 2.5(VMA)}{n(VMA - 3)} \right]^n \qquad \ldots(6.9)$$

where,

$$n = 0.83 \log \left[ \frac{4 \times 10^4}{S_b} \right]$$

### 6.7.2.5 Indirect Tensile Fatigue Performance

The results from indirect tensile fatigue test determinations for the CB-control and TB-tin slag mixtures are presented in Table 6.14 and 6.15 respectively. Based upon these results, the relationships between the initial tensile strain and the number of cycles to failure ($N_f$) for both mixtures are shown in Figure 6.54. Figure 6.54 also shows the regression lines, which have been placed through those experimental data points. The equations of the two regression lines are given in Table 6.16 along with the relevant results. Based on these regression lines it can be seen that the indirect tensile fatigue test is a suitable test for analysing these mixtures due to the reasonable value of correlation coefficient obtained from the experiments compared to the BS DD ABF (2000) recommended $r^2$ value of 0.90. The results presented in Table 6.16 and Figure 6.54 clearly demonstrate that the TB-tin slag mixture fatigue life performance is far lower than the CB-control mixture.

**Table 6.14: Indirect tensile fatigue test results for CB-control mixture**

| Specimen | Thickness (mm) | Diameter (mm) | Target Stress (kPa) | No of Cycles to Failure ($N_f$) | Stiffness | Maximum Tensile Strain |
|---|---|---|---|---|---|---|
| CB45-2 | 58 | 102 | 300 | 743 | 871 | 706 |
| CB45-3 | 60 | 100 | 270 | 730 | 914 | 606 |
| CB45-4 | 58 | 101 | 250 | 1985 | 1296 | 395 |
| CB45-6 | 61 | 100 | 220 | 7808 | 1684 | 268 |
| CB45-7 | 61 | 100 | 210 | 6088 | 1925 | 224 |
| CB45-8 | 59 | 102 | 200 | 9066 | 1401 | 293 |

**Table 6.15: Indirect tensile fatigue test results for TB-tin slag mixture**

| Specimen | Thickness (mm) | Diameter (mm) | Target Stress (kPa) | No of Cycles to Failure (Nf) | Stiffness | Maximum Tensile Strain |
|---|---|---|---|---|---|---|
| TB48-11 | 57 | 100 | 250 | 606 | 1426 | 359.40 |
| TB48-12 | 55 | 100 | 240 | 716 | 1467 | 335.38 |
| TB48-14 | 54 | 100 | 220 | 1036 | 1750 | 257.71 |
| TB48-15 | 55 | 100 | 210 | 1379 | 1712 | 251.46 |
| TB48-16 | 55 | 100 | 200 | 1754 | 1791 | 228.92 |
| TB48-17 | 54 | 100 | 190 | 883 | 1367 | 284.93 |

**Table 6.16: Fatigue line equations for CB-control, TB-tin slag, CS-Control and TSS-tin slag mixtures**

| Mixture Type | Equation for Strain | Equation for Cycles to Failure | $R^2$ |
|---|---|---|---|
| CB-Control | $\varepsilon = 7577.9\ N_f^{-0.3777}$ | $N_f = 3.4974 \times 10^9\ \varepsilon^{-2.3653}$ | 0.89 |
| TB-Tin slag | $\varepsilon = 5246.6\ N_f^{-0.42}$ | $N_f = 2.4246 \times 10^8\ \varepsilon^{-2.1979}$ | 0.93 |
| CS-Control | $\varepsilon = 2882.6\ N_f^{-0.2732}$ | $N_f = 6.9302 \times 10^{11}\ \varepsilon^{-3.3066}$ | 0.91 |
| TSS-Tin slag | $\varepsilon = 2980.2\ N_f^{-0.2753}$ | $N_f = 3.3103 \times 10^{12}\ \varepsilon^{-3.60}$ | 0.98 |

Table 6.17 shows a few calculations based on the fatigue line equations obtained from the tests (as shown in Figure 6.54 and Table 6.16). It was found that, for the TB-tin slag

mixture, the life at an initial tensile strain value ε of 100 microstrains is 9746 load applications and the initial strain required to achieve a life of $10^6$ cycles is 16 microstrains. While for CB-control mixture, the life achieved at an initial strain of 100 microstrains is 65035 load applications and the strain expected to achieve a life of $10^6$ cycles is 41 microstrains. Such data therefore, clearly indicate that the TB-tin slag mixture can only withstand a tensile strain value about two times lower than that sustainable by the CB-control mixture, indicating poor fatigue performance compared to the CB-control mixture. The low fatigue life for TB-tin slag mixture may have been at least partly due to high air voids. TB-tin slag's open gradation would also have contributed to the lower fatigue life of the mixture. The fact that there was no alteration to the tin slag gradation (size below 10mm) made it open (gap) graded and not continuously graded.

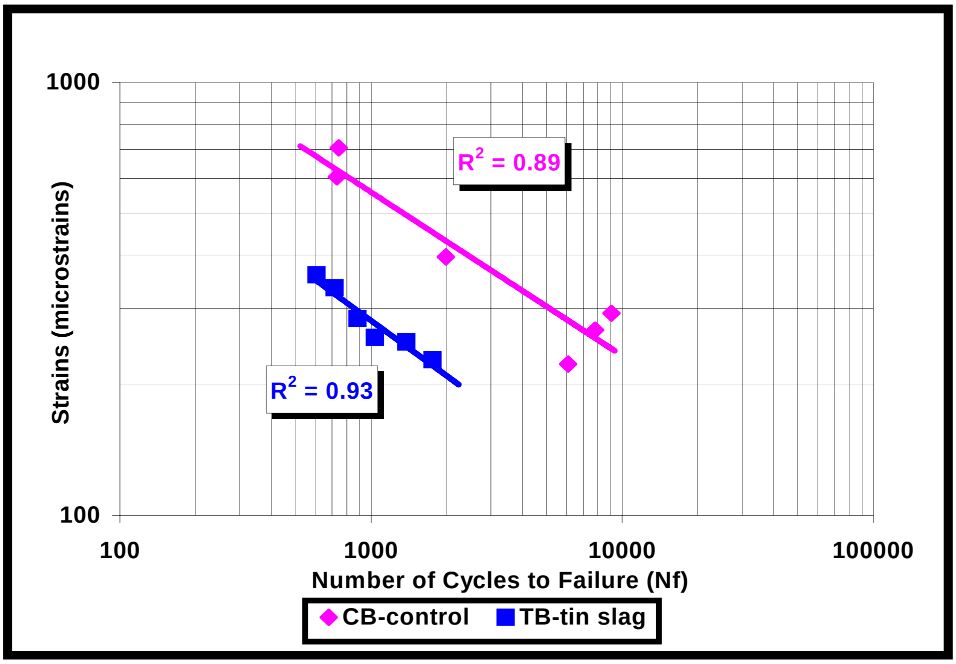



**Figure 6.54: Indirect tensile fatigue test (ITFT) for CB-control and TB-tin slag mixtures**

A similar assessment was also carried out on the TSS-tin slag and CS-control mixtures to evaluate the indirect tensile fatigue test performance and the results are presented in Tables 6.18 and 6.19 respectively. The spread of stiffness results gave a good indication of the consistency of the materials and thus the fatigue life as shown in Figure 6.55. Once again, the regression lines were plotted for both mixtures and high values of correlation coefficients were observed. It seems that the ITFT is a suitable test method for evaluating the TSS-tin slag mixture performance. Table 6.16 presents the equations of fatigue lines obtained from the regression analysis. The results indicate that the TSS-tin slag mixture has a very similar fatigue life performance when compared to the CS-control mixture.

From Table 6.17 it can be seen that the life of the TSS-tin slag mixture at an initial tensile strain of 100 microstrains is 208866 load applications and the initial strain for a life of $10^6$ cycles is 66 microstrains. Such results are comparable to the performance to the CS-control mixture, which has a life of 168867 load applications at 100 microstrains and 66 microstrains for a life of $10^6$ cycles.

**Table 6.17: Application of fatigue line equations for CB-control, TB-tin slag, CS-Control and TSS-Tin slag mixture**

| Mixture | $\varepsilon$ for $N_f = 10^6$ cycles (Microstrains) | $N_f$ at $\varepsilon$ = 100 microstrains (Cycles) |
|---|---|---|
| CB-control | 41 | 65035 |
| TB-tin slag | 16 | 9746 |
| CS-control | 66 | 168867 |
| TSS-tin slag | 66 | 208866 |

**Table 6.18: Indirect tensile fatigue test results for TSS-tin slag mixture**

| Specimen | Thickness | Diameter | Target Stress (kPa) | No of Cycles to Failure | ITSM (MPa) | Maximum Tensile Strain |
|---|---|---|---|---|---|---|
| TSS45-09 | 54 | 100 | 230 | 1144 | 1120 | 421 |
| TSS45-10 | 54 | 100 | 210 | 2694 | 1217 | 354 |
| TSS45-12 | 54 | 100 | 190 | 4977 | 1379 | 282 |
| TSS45-14 | 53 | 100 | 170 | 5929 | 1286 | 271 |
| TSS45-16 | 54 | 100 | 150 | 14609 | 1465 | 210 |
| TSS45-17 | 54 | 100 | 140 | 5432 | 1093 | 263 |
| TSS45-19 | 54 | 100 | 250 | 1030 | 1213 | 423 |

**Table 6.19: Indirect tensile fatigue test results for CS-control mixture**

| Specimen | Thickness | Diameter | Target Stress (kPa) | No of Cycles to Failure | ITSM (MPa) | Maximum Tensile Strain |
|---|---|---|---|---|---|---|
| CS47-09 | 62 | 100 | 300 | 1480 | 1704 | 391 |
| CS47-10 | 62 | 100 | 250 | 6171 | 2004 | 256 |
| CS47-11 | 62 | 100 | 240 | 9710 | 1978 | 249 |
| CS47-12 | 62 | 100 | 230 | 13893 | 2055 | 229 |
| CS47-13 | 62 | 100 | 220 | 7067 | 1569 | 287 |
| CS47-16 | 62 | 100 | 190 | 15279 | 2137 | 182 |
| CS47-18 | 61 | 100 | 170 | 29904 | 1978 | 176 |

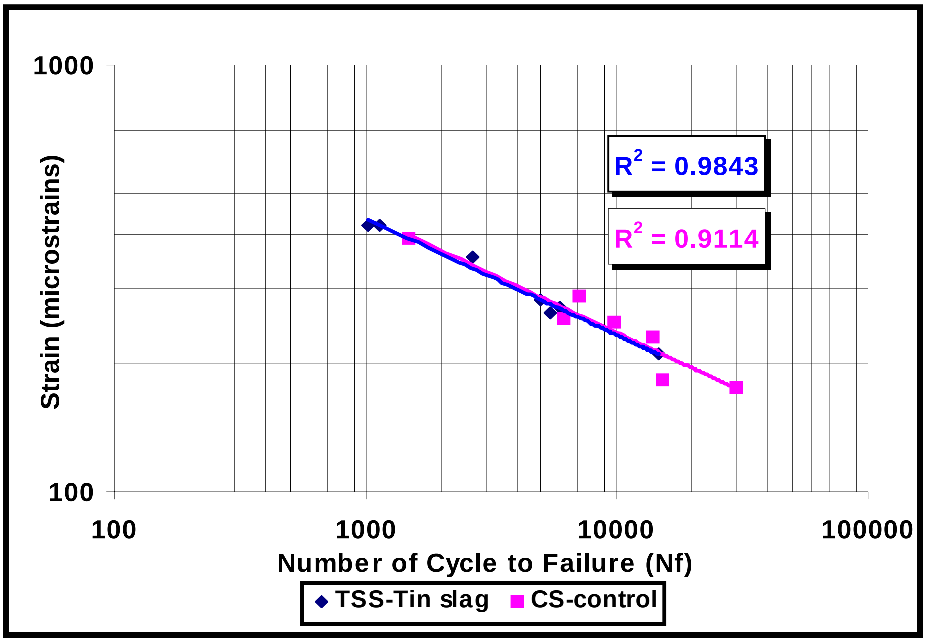



**Figure 6.55: ITFT) for CS-control and TSS-tin slag mixtures**

### 6.7.2.6 Permanent Deformation Performance

Permanent deformations of the mixtures were determined using the repeated load axial test (RLAT). An average of three test results showing the accumulation of axial strain with increasing number of load cycles for TB-tin slag and CB-control mixtures is presented in Figure 6.56. For each material, there was little difference in the results from individual specimens. Based on the results obtained from the tests, it was found that the TB-tin slag mixture suffered higher strains at the end of the test compared with the CB-control mixture. It is clear from the plot that TB-tin slag mixture performance was inferior to the CB-control mixture. The TB-tin slag mixture began to fail as a result of large irrecoverable deformations at about 500 load cycles. On the other hand, the test on the CB-control mixture showed that the mixture was resistant to permanent deformations as the cumulative vertical deformations at the end of the test were small and the mixture was able to withstand 3600 load cycles without collapse.

One of the possible explanations for this behaviour is the stiffness modulus of the mixtures. According to the test results shown earlier in Section 6.7.2.4, it was found that there was significant difference in the stiffness modulus values between the TB-tin slag mixture and CB-control mixture. According to Uzan (2004), increasing the maximum stiffness has the effect of reducing the permanent deformation.

With regard to binder content, specimens with higher binder contents generally failed more quickly than those with lower binder contents due to higher volume of binder resulting in greater lubrication. Lundy and Sandoval-Gil (2004) found that increasing the binder content increases the rutting potential. Therefore, it is possible that the unsatisfactory creep behaviour of the TB-tin slag mixture was caused by excess bitumen content.

Figure 6.57 shows the average of three results of repeated load axial creep tests on TSS-tin slag and CS-control mixtures. Again, there was little difference in performance between individual specimens. TSS-tin slag mixture had significantly better performance than the TB-tin slag mixture (compare Figures 6.56 and 6.57), although its permanent deformation performance was inferior to the CS-control mixture. The figure shows that TSS-tin slag mixture failed at about 2800 number of cycles while the CS-control mixture was able to withstand the full 3600 load cycles without sustaining excessive permanent deformation.

Another interesting result emerges when comparing TB-tin slag mixture and TSS mixture. The results suggest that the TB-tin slag mixture performance was inferior to the TSS-tin slag mixture. This can be explained in terms of void in mineral aggregate content and the size of aggregates in the mixture. The TB-tin slag mixture was made from aggregate with maximum nominal size of 20mm and 16.1% void in mineral aggregate while the TSS-tin slag was made from aggregate with maximum nominal size of 10mm and 14.4% voids in mineral aggregate. According to Anderson and Bentsen (2001), increasing the voids in mineral aggregate from 13% to 15% in coarse aggregate mixtures appeared detrimental to the mixture shear performance properties. Such conclusions are in agreement with results from this investigation. The results were also supported by other work carried out by Lundy and Sandoval-Gil (2004) who recommended that the effect of voids in mineral aggregate depends on both the maximum aggregate size used and the fines content.

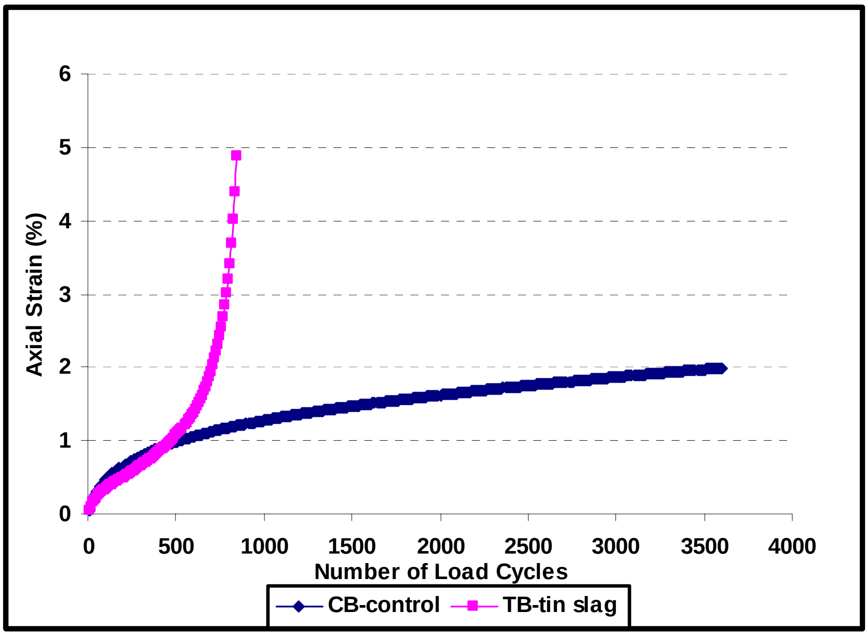



**Figure 6.56: Repeated Load Axial Creep Test (RLAT) for CB-control and TB-tin slag mixtures**

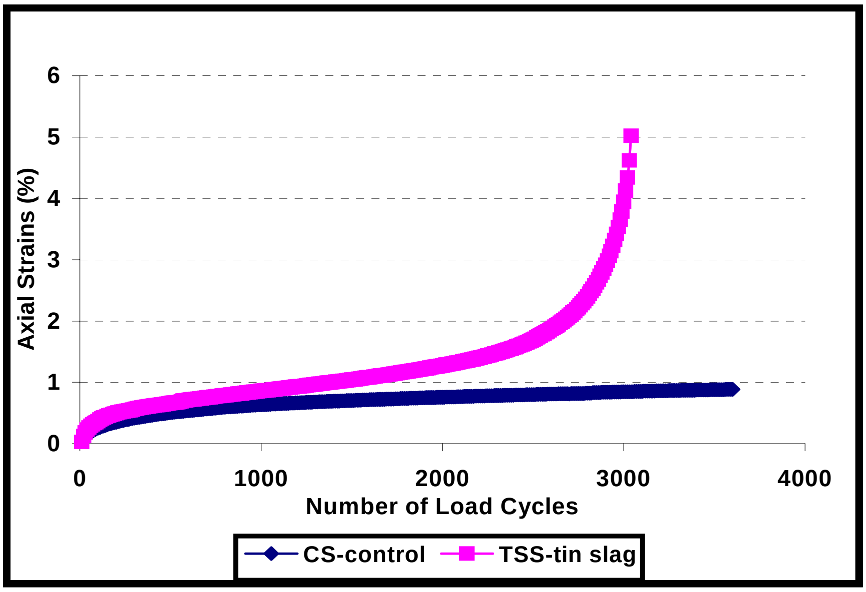



**Figure 6.57: Repeated Load Axial Creep Test (RLAT) for CS-control and TSS-tin slag mixtures**

## 6.8 DURABILITY EVALUATION OF TIN SLAG MIXTURE

For the purpose of this research, only the best performing tin slag mixture was studied to evaluate durability. Thus, on the basis of the work reported in Section 6.7.2, the TSS-tin slag mixture was selected for a further study of durability and the CS-control mixture was used as a control. Two methods were used to evaluate the durability of asphalt mixtures. The first method is termed "Long-term Oven Ageing" (LTOA) and the second method was referred to as the water sensitivity. Both methods have been discussed earlier in Section 6.5.

### 6.8.1 Long-Term Oven Ageing (LTOA)

Three specimens from both control and tin slag mixtures were tested to determine long term ageing effects. The results from the LTOA tests are given in Figures 6.58 and 6.59. Generally, the ageing process resulted in an increase in the tensile strength and stiffness modulus values. Figure 6.58 indicates that conditioning of specimens from both mixtures i.e. control and tin slag, had minimal effects on the tensile strength after ageing. For example, the average ITS value for the conditioned control mixture was about 1.4MPa whilst the unconditioned value was 1.3MPa. In a similar manner there was no significant effect following LTOA on the conditioned tin slag mixture in terms of the indirect tensile strength, which is clearly shown in Figure 6.58.

With regard to the effect of ageing on stiffness, it is clearly shown in Figure 6.59 that the ageing process obviously affected the stiffness of both control and tin slag mixtures. It was observed that the stiffness modulus of tin slag mixture and control increased by about 56% and 40% respectively. When comparison is made between the control and tin slag mixtures as shown by the ITS ageing index or ITSM ratio index in Table 6.20, it is observed that the tin slag mixture was more affected by LTOA than the control mixture in terms of ITS and ITSM. This suggests that the tin slag mixture is likely to have more interconnected air voids which may allow a greater degree of oxidation (Scholz, 1995).

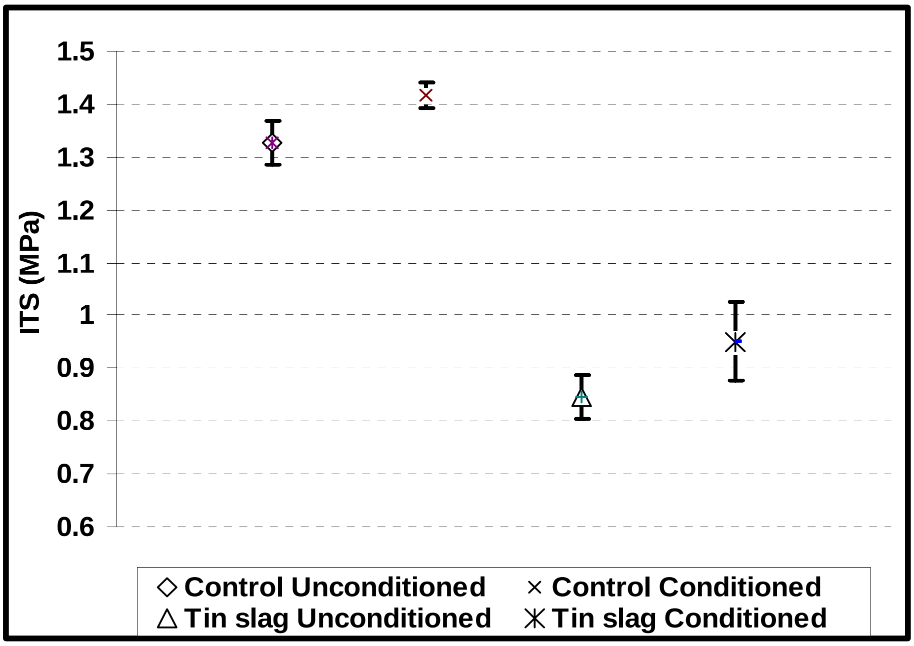



**Figure 6.58: ITS test results for LTOA of unconditioned and conditioned control and tin slag mixture showing 95% confidence limits**

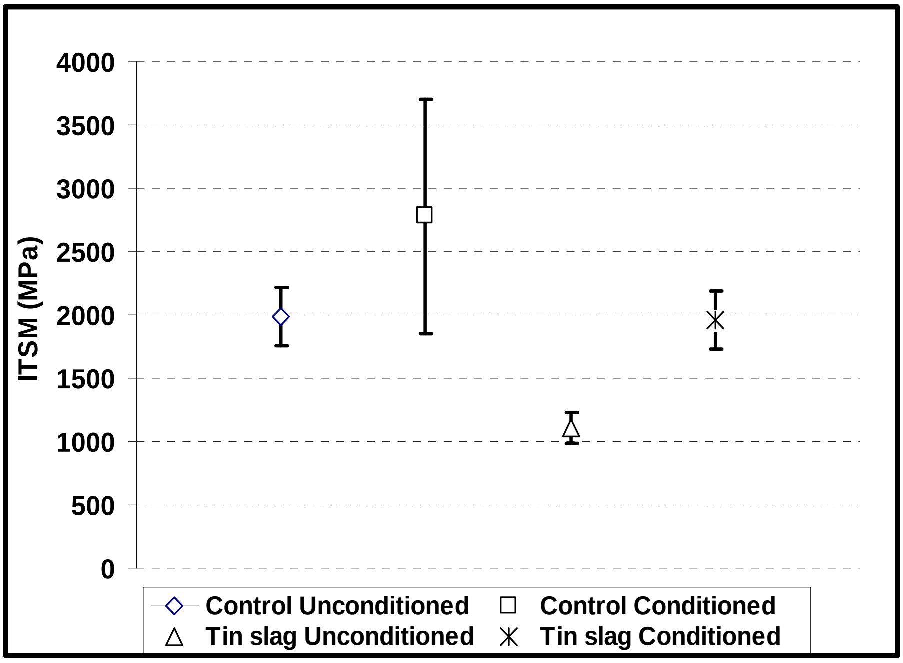



**Figure 6.59: ITSM test results for LTOA of unconditioned and conditioned control and tin slag mixture showing 95% confidence limits**

**Table 6.20: LTOA Durability Index for control and tin slag mixture**

| | LTOA Durability Index | |
|---|---|---|
| | **Control Mixture** | **Tin Slag Mixture** |
| ITS | 106.8 | 115.29 |
| ITSM | 139.74 | 156.16 |

## 6.8.2 Water Sensitivity

Evaluation of water sensitivity of bituminous mixtures in this study was primarily based on the change in the tensile strength and tensile stiffness moduli of the mixtures. In this study, three specimens from both control and tin slag mixtures were tested to assess moisture effects. The results obtained from the tests are presented in Figure 6.60, Figure 6.61 and Table 6.21. The results indicate that there was no significant difference in terms of tensile strength between the conditioned and unconditioned specimens for both mixtures i.e. control and tin slag (see Figure 6.60). Therefore, the tin slag mixture was not materially affected by moisture conditioning. The indirect tensile strength ratio value for the tin slag mixture obtained from this investigation was 97.7% as shown in Table 6.21. This value is above the minimum value recommended by ASTM D 4867 (1996).

Similarly, Figure 6.61 shows that there was no significant change in ITSM upon water conditioning of the tin slag specimen although there was a small reduction for the control mixture specimen. The findings from the investigation are shown in Table 6.21, revealing that tin slag has an ITSM ratio (durability index) value of nearly 100% compared to the control mixture with an index value of about 90%. Overall, it seems that the tin slag mixture has very little moisture sensitivity.

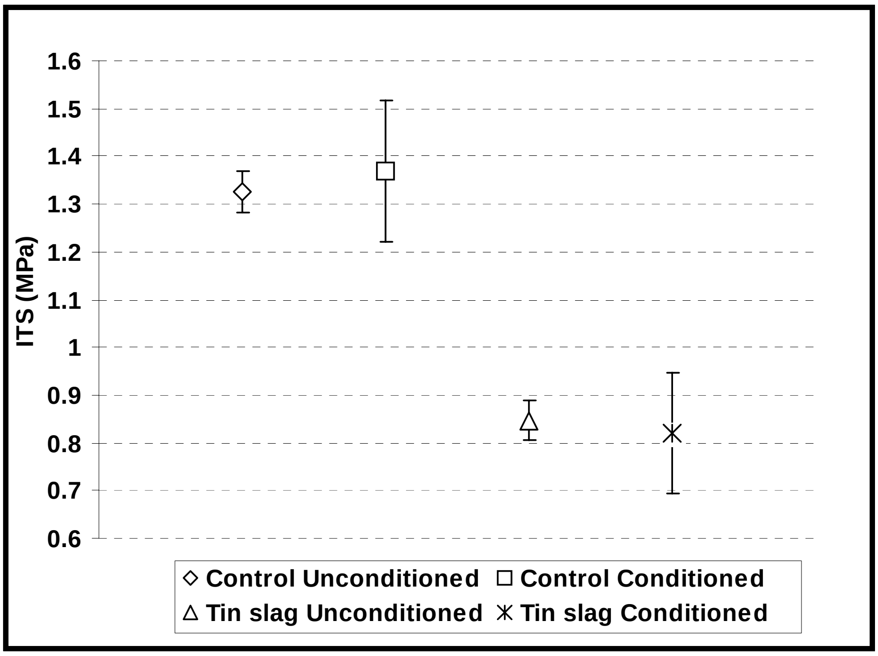



**Figure 6.60: ITS results for water sensitivity of unconditioned and conditioned control and tin slag mixture showing 95% confidence limits**

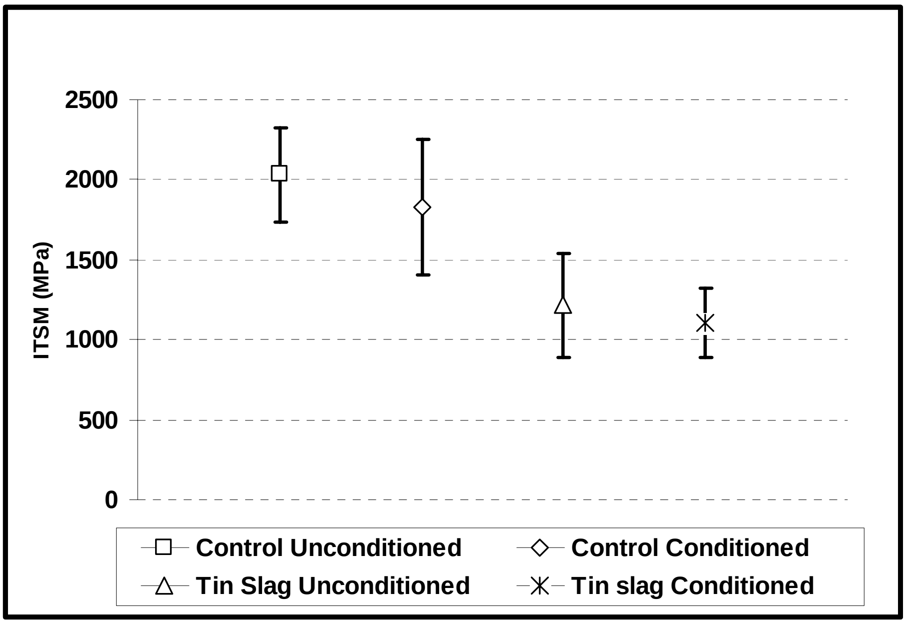



**Figure 6.61: ITSM results for water sensitivity of unconditioned and conditioned control and tin slag mixture showing 95% confidence limits**

**Table 6.21: Water Sensitivity Durability Index for control and tin slag mixture**

| | Water Sensitivity Durability Index | |
|---|---|---|
| | **Control Mixture** | **Tin Slag Mixture** |
| ITS | 103.8% | 97.7% |
| ITSM | 90.1% | 99.2% |

## 6.9 SUMMARY

The investigation in this chapter could be summarised as follows:

- Three mixtures, TB-tin slag, TS-tin slag and TSS-tin slag asphalt mixtures were selected for optimum binder determination. CB-control and CS-control mixtures were used as control mixtures.
- It was found that TS-tin slag which consisted of tin slag aggregate only without any alteration was not suitable for use in road pavements on the basis that the stiffness and Marshall stability values were very low and the Marshall flow value

was very high.

- Both the TB-tin slag and TSS-tin slag mixtures had Marshall stability values that exceeded the minimum requirement recommended by the Asphalt Institute although they were inferior compared to control mixtures.
- It was found that the TSS-tin slag mixture has a higher Marshall Stability value than TB-tin slag mixture.
- With regard to the indirect tensile strength performance, it was found that TSS-tin slag was 54% stronger than TB-tin slag mixture. However, the stiffness modulus test results showed that TB-tin slag mixture has a slightly higher stiffness than the TSS-tin slag mixture.
- In terms of fatigue performance, the results indicated that the TSS-tin slag mixture has a fatigue life performance comparable to the performance of the CS-control mixture. However, it was clearly indicated that TB-tin slag has a poor fatigue performance compared to the CB-control mixture.
- The repeated load axial creep test showed that TSS-tin slag mixture had significantly better performance than the TB-tin slag mixture. However, its permanent deformation performance was inferior to the CS-control mixture.
- In terms of durability, the tests showed that tin slag mixture seems to be less sensitive to moisture damage while the Long Term Oven Ageing test showed that tin slag mixture was more affected than the control mixture but only by a small amount.
- Overall, the laboratory investigation showed that the TSS-tin slag mixture has potential to be incorporated in a pavement structure as an asphalt layer.

Chapter 7

# ENVIRONMENTAL EVALUATION OF USING TIN SLAG IN ROAD PAVEMENTS

## 7.1 INTRODUCTION

Extensive investigations are necessary to assess the environmental compatibility of slag utilisation in road construction. This is not the case for natural aggregates where extensive investigations concerning environmental compatibility are not so critical. Different schemes and protocols have been introduced by researchers to evaluate the environmental impact of using industrial by-products or wastes in road pavements. Fallmann (1990) has introduced one scheme to evaluate the potential impact and effect of slag materials used in road construction (Figure 7.1). Nunes et al (1999) proposed a new protocol for the environmental assessment of unbound and bound secondary materials (by-products or wastes). In their proposal, the secondary materials were assessed in four stages:

- The first stage - basic information, a descriptive stage to characterise the physical properties and chemical composition of the secondary material as received. At this stage, all basic information about the secondary material is gathered to construct a database of material properties.
- The second stage - initial assessment of the performance of two preliminary leaching tests on finely ground secondary material samples in order:
  - (i) To obtain an initial leachate concentration for comparison with minimum discharge quality objective (DQO).
  - (ii) To assess the amount of contamination available for leaching long term, by a simple “availability” test.

  If the secondary material fails at stage two, it has to go on to stage three.

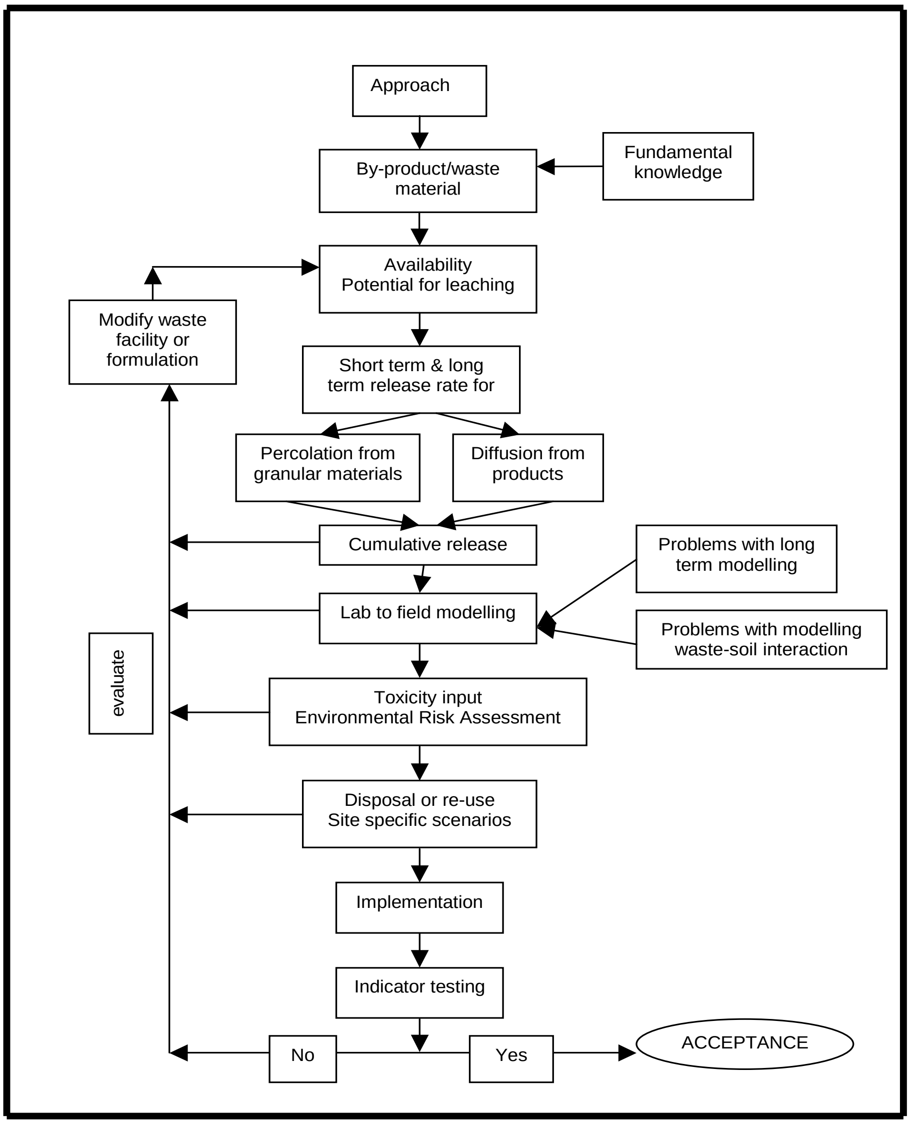



**Figure 7.1: Scheme to evaluate the potential risk of utilising/disposing a residue (Fallman, 1990)**

- Stage three - performance assessment testing of unbound or bound material at a realistic grading in a leaching test that approximates to the road conditions. Here, the release of contaminants is examined by diffusion in the specific case of a fully saturated road foundation. The release of contaminants from bound and unbound materials is also examined under flow-through conditions comparable to an extreme road drainage system. If at this stage the secondary materials fail to achieve the required discharge quality result, they would be required to be treated with a binder such as cement or bitumen. After this, the secondary material will be tested again. This performance assessment is used to ascertain the suitability of a secondary material for controlled utilisation in road construction or whether the material requires more treatment.

- Stage four – Basic information on treated secondary material, the description of the secondary material (if necessary after treatment with binder). The product should be described by the process that formed it. The assessment at this stage should include an economic analysis.

The primary objective of performing an environmental evaluation on alternative materials used in road construction is to enable an assessment of the potential or actual impact on the environment caused by these materials. The results of the tests can be used (Reid *et al*. 2001):

- To develop a rationale for setting of criteria for material quality and road design, which will ensure adequate protection to the environment.
- For quality control purposes to ensure that alternative materials to be used in road construction comply with certain environmental standards or regulations as set by the authorities.
- To assess the risk of any hazardous constituents to public health.

In assessing the environmental impacts of using slag materials, information on environmental properties is needed. The potentially relevant information on the environmental properties of the slag materials to be used in road construction may be summarised as follows (Reid et al. 2001):

- Total composition/content of selected elements;
- Leaching behaviour, which includes;
  - (i) Determination of potential leaching of selected contaminants.
  - (ii) Determination of actual or expected leaching of selected contaminants as a function of liquid to solid ratio (L/S) and/or time.
  - (iii) Influence of pH, redox potential, complexing, etc. on the leaching behaviour.

## 7.2 WATER MOVEMENT IN ROAD PAVEMENTS

When discussing the leaching of constituents from by-products or wastes used in the road environment, one cannot avoid discussing water movement therein. Although, road pavements are designed to prevent the infiltration of water into their structure and the underlying sub-grade, in practice it is impossible to prevent some water from infiltrating into the pavement. The water moves into, through and out of the pavement structures variously throughout the construction, service life and maintenance of the road. This movement largely controls the migration of contaminants from the pavements. Therefore, the movement of water in a pavement structure is very important information for assessing of environmental impact. A small amount contaminant may move by diffusion irrespective of the water movement, but this movement of contaminant is expected to be very small compared to that moved by advection in flowing water.

Dawson (1985) discussed the possible methods of water ingress into the pavement structure. He suggested that water enters the pavement structure by several mechanisms including:

- Water infiltration through the pavement surface.
  This includes through construction joints, cracks due to thermal, traffic loads or pavement failure and penetration through intact bound layers.
- From the sub-grade
  This includes entry by artesian head in the sub-grade, pumping action at formation level and capillary action in the sub-base.
- From the road margins
  This includes entry by reverse falls at formation level, lateral/median drain surcharging, and capillary action in the sub-base and through unsealed shoulder-collecting run-off.

Baldwin, et al. (1997) explained that water could enter and leave road structures during construction, reconstruction and pavement life. The typical movement of water in a road environment is illustrated in Figure 7.2.

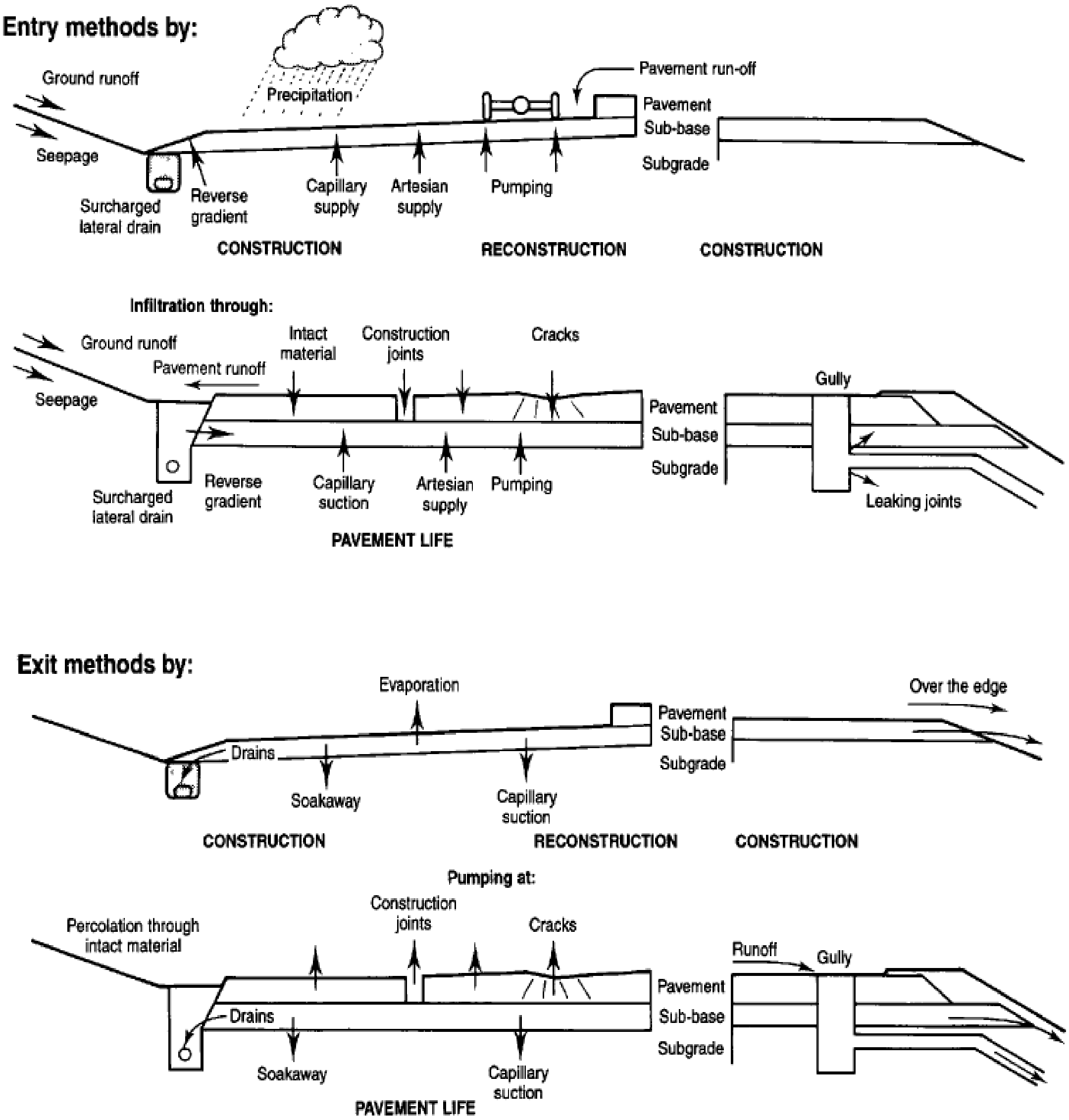



**Figure 7.2: Schematic of water movement in road environment (Baldwin et al., 1997)**

## 7.3 LEACHING OF CONTAMINANTS

The potentially adverse impact of slag materials on the environment should be given full attention, in particular those associated with surface water and groundwater pollution due to the leaching of contaminants. Alternative materials in road construction such as slags contain varying concentrations of chemical compounds, which could potentially cause pollution of surface and groundwater if they leach. It is unlikely that contact between liquid and road construction materials can be completely avoided. Consequently leaching of soluble material into infiltration rainwater, surface water and groundwater may be a significant route for potential contaminant release.

### 7.3.1 Leaching Mechanism

The mechanism of leaching into the environment is a very complex process. Mass transfer in porous materials is highly dependent on the morphology of the porous structure. This morphology is defined by pore diameter, tortuosity and constrictivity. Long-term studies to characterize the behaviour of different chemical species in porous materials have often shown that two kinetic mechanisms come into play (Ligia, et al., 2000). During the first, relatively short phase of contact i.e. material-water, the kinetic rate is rapid and a state of pseudo-equilibrium seems to be reached. The second phase is characterized by a slow kinetic rate; low fluxes of substances entering or leaving the matrix can be demonstrated and only low variations in flux can be noted in time (residual flux). Kosson et al. (1996) and van de Sloot (1996) explained that leaching mechanism of materials is controlled either by constituent availability, solubility or diffusion.

#### 7.3.1.1 Availability Controlled Leaching

Availability is defined as the maximum soluble fraction of a residue constituent that can be released into solution under aggressive leaching conditions (Hill, 2003). Under availability-controlled leaching conditions, the leachate concentration is less than the saturation condition for the element or species of interest. The volume of element finally leached can be significantly less than the total content of that element. Determination of availability, however, does not indicate whether or not this maximum quantity of a particular constituent will be released in practice or over what precise time interval the release will occur for the environmental exposure scenario of interest.

Availability-controlled leaching is most likely to occur in systems with a high liquid to solid ratio, where solubility limits are not reached. Also, the degree of availability varies considerably for different chemical species and solid matrices. Figure 7.3 illustrates typical availability-controlled leaching (Hill, 2003).

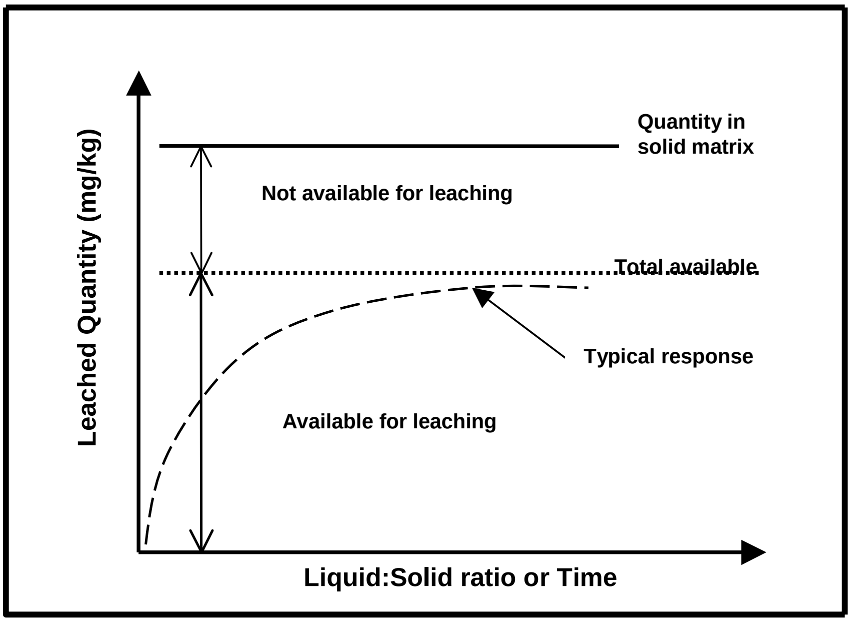



**Figure 7.3: Typical example of availability controlled leaching (Hill, 2003)**

### 7.3.1.2 Solubility Controlled Leaching (for loose granular residue)

Solubility-controlled leaching is one of the most common forms of contaminant release to the environment. The solubility is defined as the maximum mass of the solute that can be dissolved in a specified mass of the solvent. Solubility-controlled leaching occurs when the solution in contact with a waste is saturated with respect to the species of interest. Under the solubility-controlled conditions, the leached quantity is proportional to the ratio of the volume of leachant to the mass of solid matrix (liquid to solid ratio). It is prevalent at low liquid to solid ratio (which typically occurs in the field) when percolation dominates the means of water contact with contaminated materials.

The solubility of most heavy metal cations (e.g. Pb, Cd, Zn, etc) is strongly influenced by the equilibrium pH. This has been reported by van der Sloot (1996) as shown in Figure 7.4 for Cadmium and Zinc (note the minimum solubility at pH 10 – 11). Solubility of a particular species can also be increased by the presence of significant concentrations of complexing agents such as chloride or acetate, or reduced by the presence of co-precipitating species such as sulphide or sulphate.

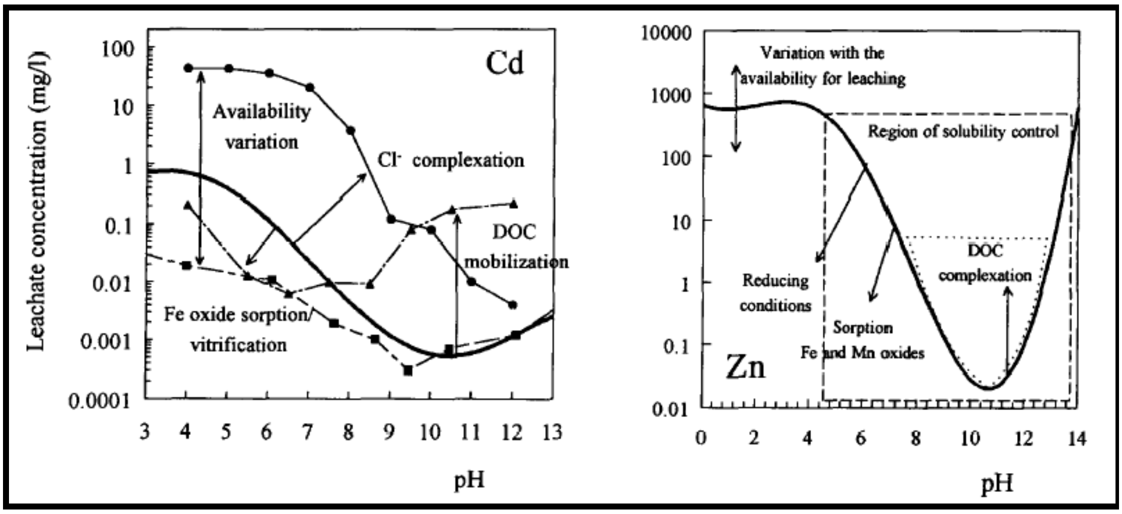



**Figure 7.4: Main factors influencing the leaching of Cadmium and Zinc in specific pH domains (van der Sloot, 1996)**

### 7.3.1.3 Diffusion from the Solid Phase (compacted granular or monolithic materials)

Mass transfer-controlled release occurs when the rate of constituent release from the solid matrix is the release limiting step and thus saturation or equilibrium with the leachate is not achieved. The molecules will move down the concentration gradient to try to establish equilibrium conditions. Diffusion can occur at three levels:

- Solid state diffusion
- Solid-liquid diffusion
- Liquid diffusion

In solid-state diffusion the chemical exists across the solid. This can result from irregular chemical make-up or leaching from specific areas of the material. Solid-state diffusion will eventually lead to a chemically homogenous material. However, this process is too slow to be of any real significance in leaching.

Solid-liquid diffusion results when a leaching solution around a solid is not in chemical equilibrium with the solid. The concentration gradient between the two media results in the chemical speciation and migration of contaminants until equilibrium conditions are reached.

As a result of leaching, the highest concentration of contaminants occurs in the solution adjacent to the solid. Movement of the leached species across the pore water or the bulk leachant to equalise the concentration is achieved by liquid diffusion.

## 7.4 CATEGORIZATION OF ALTERNATIVE MATERIALS

Leach test data should be interpreted very carefully. It cannot be used to estimate directly the contaminant load or the amount of dilution required in the mixing zone. Nevertheless, the test data allows inter-comparison to be made or assessments relative to primary aggregates. Baldwin, et al. (1997) proposed that alternative materials can be categorised into three types with respect to leaching characteristics relative to primary aggregate (e.g. limestone). Under this proposal, alternative materials are categorized into three groups. Group 1 are materials that should have no restriction for use in road pavements (based on potential effect on water quality). On the other hand, materials in Group 2 may need some restrictions based on their potential to affect water quality. Materials in Group 3 need some restrictions based on their potential to affect water quality. Box 8.1 shows the summary of categories proposed by Baldwin et al.

**Box 7.1: Categorization of alternative material (Baldwin, et al., 1997)**

Group 1 - No restrictions based on potential to affect water quality

In laboratory leach tests

- No black list species over the EQS or WQS
- No grey list species requiring more than a 100x dilution to meet EQS or WQS
- No other species requiring more than a 100x dilution to meet EQS or WQS

Group 2 - may need some restrictions based on potential to affect water quality

In laboratory leach tests

- No black list species over the EQS or WQS
- No grey list species requiring more than 1000x dilution to meet EQS or WQS

Group 3 - will need restrictions based on potential to affect water quality

In laboratory leach tests

- No black list species requiring more than 1000x dilutions to meet EQS or WQS
- No grey list species requiring more than 1000x dilutions to meet EQS or WQS

EQS - Environmental Quality Standard

WQS - Water Quality Standard

## 7.5 ENVIRONMENTAL EVALUATION OF HEAVY METALS IN TIN SLAG

The evaluation of environmental impacts of heavy metals from tin slag involved two assessments, the assessment of chemical composition of the solid fraction and the assessment of constituents leached from the materials. The first assessment is important in order to get information on the total chemical composition of the slag material. Knowledge of the chemical composition is considered important for the following reasons:

- Identification of the presence of any constituents that have the potential to cause pollution.

- To assist in determining the proportion of the solid fraction of each constituent that has been leached.
- To enable comparison to be made with established data.

Generally, the determination of chemical compositions of solids involves x-ray fluorescence (XRF) spectrometry and inductively coupled plasma mass spectrometry (ICP-MS). The selection of using these techniques depends on detectability of elements. Some elements such as Mercury (Hg), Arsenic (As) and Selenium (Se) exhibit a high detection limit when using XRF technique thus ICP-MS is used to determine these elements.

However, the determination of heavy metals in the solid fraction was not performed in this investigation. As discussed before, the determination of chemical composition is used for identification of the presence of any constituents that may be harmful to the environment. However, because the aim of the study was to assess the possible effect of tin slag on the environment when using tin slag in road pavements, the determination of chemical composition of the solids was not crucial. Also, the cost of determining the constituents in the solid fraction is very high, therefore, it is enough to determine the constituents leached from tin slag. For these reasons, the environmental evaluation of tin slag focused on the leaching test only.

### 7.5.1 Leaching Test Programme

Two methods were used for the leaching test programme; the compliance test and the tank-leaching test. The tests were used to compare the properties of the slag materials and a reference material. Natural aggregate, as a reference material, was used as a benchmark against which to compare the slag materials.

#### 7.5.1.1 Compliance Leaching Test

The compliance test is used to determine whether the materials comply with published or specified reference values normally produced by the regulatory authorities. A compliance test provides information on leaching of materials under experimental conditions, specifically a liquid to solid ratio of 2 l/kg in the first step and subsequently of 10 l/kg in the second step.

The test is applicable to waste, which has a particle size below 4 mm without, or following size reduction. The small size of the particles increases the surface area to volume ratio of the materials thereby increasing the surface area in contact with the leachate and shortens the diffusion path length. In this test, the tested material and leachate are mixed continuously by agitating. However, the test generates a leaching environment that is much harsher than in a real road pavement as abrasion may occur between individual material particles and between the leaching vessel and material particles. Such harsh conditions represent an extreme worst scenario. If the constituents of the solid fraction do not leach under such harsh conditions then they are unlikely to leach under the real situation environment.

The test does not take into account the effect of pH, redox condition, temperature changes and speciation which probably occur during the leaching process. So, this test cannot be used alone to determine the leaching behaviour of a waste. The compliance test is applicable to waste material and sludge having a high solid content therefore suitable for alternative aggregates. The necessary quantity of eluate in each step shall be obtained to perform the physical and chemical characterisation of the eluate. This test may not be applicable to materials, which react with leachant. British standard BS EN 12457-3 "Compliance test for leaching of granular waste materials and sludges" (BSI, 2002) provides details of the test methodology. This method was chosen in this investigation for the following reasons:

- At present it is the most up-to-date test being developed in Europe.
- The test method was recommended by Baldwin et al. (1997) and Reid et al.

(2001). Therefore, by using this method, results obtained here may be compared with previous results from other authors.

- The results of the tests can be directly compared with the regulatory limits on a pass/fail basis.
- The test also can be used to determine the effects on leaching of conditions such as pH, conductivity and redox potential. These parameters can be very important in determining the actual leaching behaviour of the wastes. These parameters are measured immediately during the collecting of the eluate.
- On a more practical level, the compliance test is also considered a suitable test because it is easy to perform and very rapid to get the result.
- In addition, the test is economic and quick to perform.

The compliance tests were performed on tin slag and conventional aggregate having particle sizes below 4mm without any treatment. The tests were performed using the University of Nottingham shaking/tumbling equipment as shown in Figure 7.5. To minimize cross contamination, the containers used in these tests were made from polypropylene and thoroughly washed with distilled water before each use.

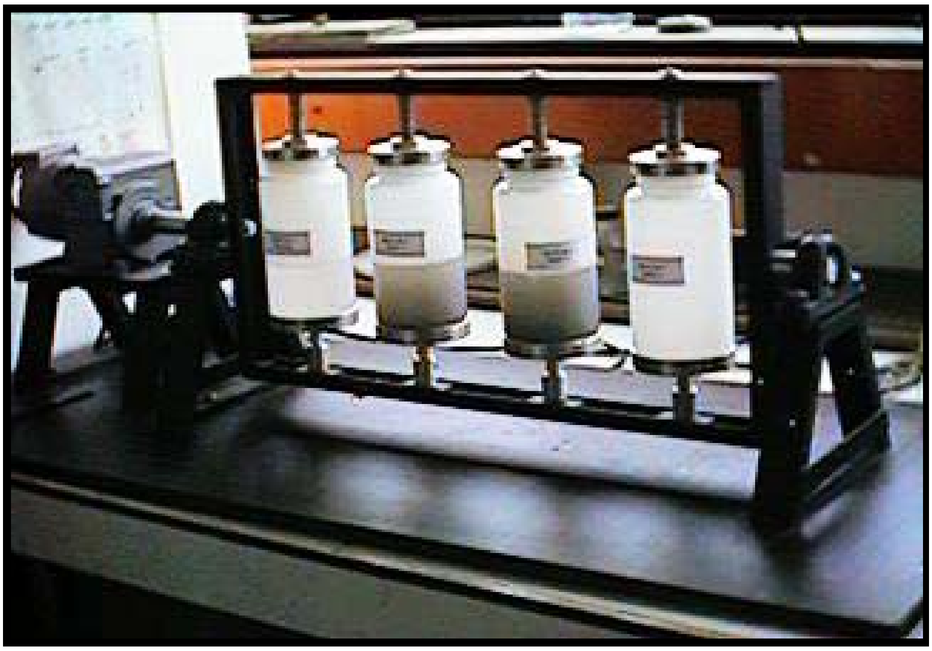


**Figure 7.5: Compliance test equipment setting**

#### 7.5.1.2 Tank Leaching Test

The tank-leaching test is used to investigate the leaching behaviour of monolithic specimens. The development of the leached concentration with time can be observed from the tank-leaching test. The effect on pH and electrical conductivity from the leaching process can also be observed. One of the advantages of this technique is the leaching sample is not moved (only the water), thereby being more simulative of typical usage. Furthermore, this technique also enables a larger and more representative sample to undergo leaching (thus larger particle sizes may be included).

In this investigation, the tank leaching tests were performed in accordance with BS EN 1744-3 (BSI, 2000) on the following specimens:

- Compacted unbound tin slag (UTS)
- Compacted unbound conventional aggregate (UG)
- Bitumen bound tin slag with 4.5% bitumen (TAM)
- Bitumen bound conventional aggregate with 4.7% bitumen (GAM)
- Cement bound tin slag with 3% cement (CBM)

Compacted unbound tin slag and compacted unbound conventional aggregate were prepared in accordance with BS 1924-2. The standard rig and vibrating hammer were used for compaction. A specially designed mould, which was proposed by Nunes et al (1997) as discussed in Chapter 5, was used for this purpose. In the case of unbound tin slag and conventional aggregate, a geosynthetic sleeve was used to prevent the specimen from disassociation after compaction. For the leaching test, immediately after compaction, the specimen was put on a geosynthetic screen and then placed together into a tank containing distilled water where the liquid to solid ratio was established at 10 l/kg. The design of tank-leaching test set-up and the screen where the sample was positioned during the test is shown in Figures 7.6 and 7.7.

Bitumen bound tin slag and conventional asphalt were prepared in accordance with the standard protocol which was previously discussed in Chapter 6. The TSS-tin slag and

CS-control mixtures were selected for the environmental evaluation study based on their performance in the previous study. In this study the weight of the specimens used was between 2 kg and 3 kg. The distilled water used in the study had a pH between 5 and 7 and a conductivity less than 0.5μS/m.

A control test, comprising about 20 litres of distilled water and the geosynthetic screen, was performed in a similar manner but without any specimen. The test was designed to determine any cross-contamination that originates from the test procedure. A 150ml sample eluate was collected after 24 hours. All components involved in this testing were made from polyethylene or polypropylene thus minimising cross-contamination, as in these tests, only non-organic contaminants were determined.

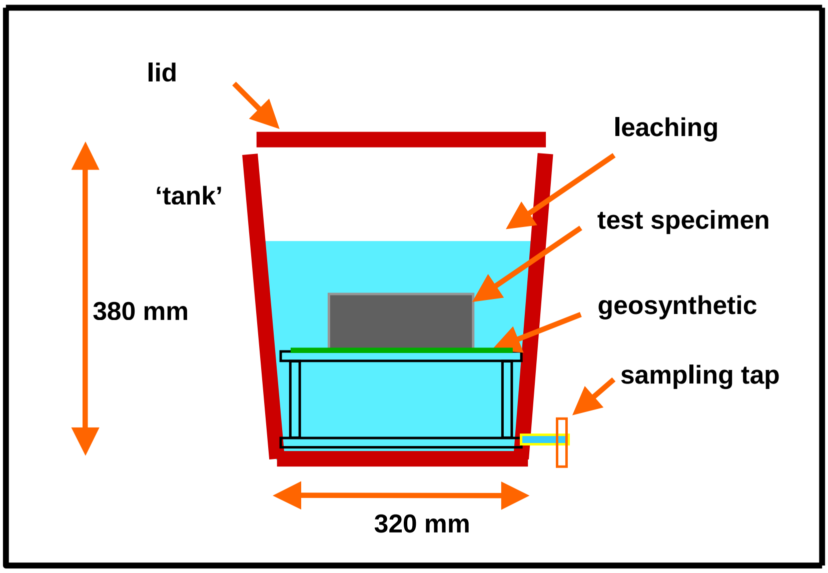



**Figure 7.6: Schematic diagram of the equipment used for the tank-leaching test**

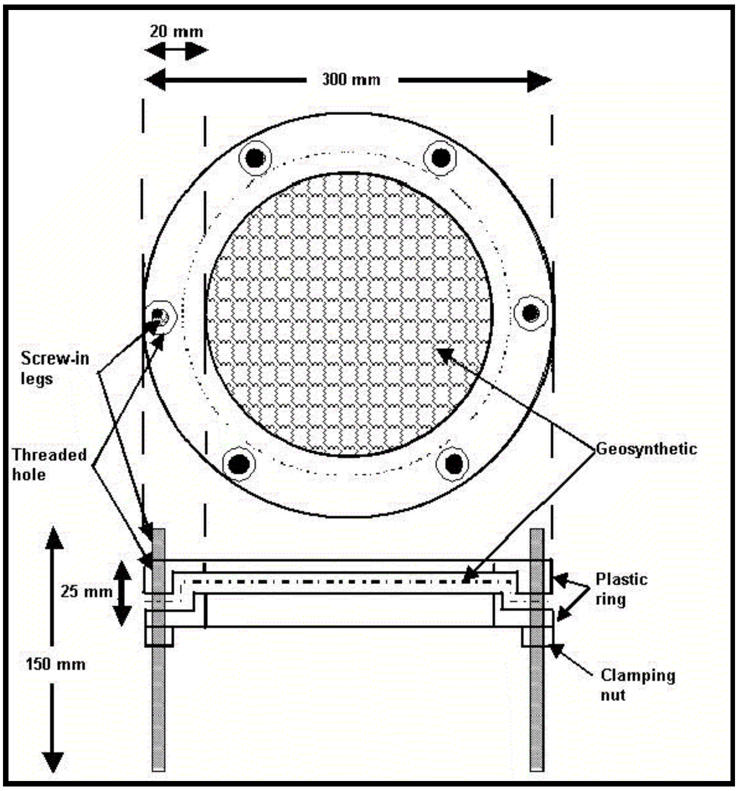



**Figure 7.7: Schematic diagram of screen used in the tank-leaching test**

#### 7.5.1.3 Determination of Constituents

Eluate or leachate samples collected from both compliance and tank leaching tests were sent to a third party laboratory for inorganic constituents determination. The organics content in the eluate was not determined since it was not very important in this study as tin slag is produced via a high temperature process (i.e. more than 1200$^{o}$C). Thus, the organic content was likely to be insignificant and undetectable since most of the organic material would have been vaporised.

### 7.5.2 Results and Discussion

#### 7.5.2.1 Compliance Leaching Test

The test results obtained from the compliance test allow a direct comparison with regulatory limits on a pass/fail basis. The results obtained from the tests were compared with the water quality guideline values produced by the World Health Organisation (WHO, 2004).

Table 7.1 shows the concentration of constituents observed from the compliance test. In general, most of the concentrations of constituents in Step 1 were higher than in the corresponding Step 2 of the test. This is to be expected because the material in the second stage has already been subject to one leaching test and a larger volume of liquid was used in the second stage. The compliance-leaching test shows that most of the concentrations of constituents leaching from the tin slag were lower than World Health Organization water quality guideline values except Arsenic. However, Arsenic falls under the “Grey” list, which tabulates those materials deemed less hazardous than others (“black” list) (Baldwin, at al., 1997).

When the tin slag data were compared with the test results from the conventional aggregates (i.e. reference material), it was observed that the amount of Arsenic and Boron (in Step 1) and Manganese (in Step 2) leached from the tin slag, were higher than in the reference material, but not significantly so. However, the Boron and Manganese levels were still far below the water quality guideline values. For other elements such as Iron, Magnesium, Zinc, Tin and Potassium, there are no WHO guideline values for drinking water because all these elements do not present any risk to health.

It should be stressed here that the test conditions of the compliance test are significantly harsher than the in-situ environment. The WHO water guideline values may also not be those applied by regulators in any given situation. Also, when the constituents leach from pavements, they will be filtered and sorbed before the water enters drinking water resources. Therefore, even though the value of Arsenic was a little bit higher than the guideline value, it is not expected to have any significant adverse effects on drinking water. Furthermore, regulators set site-specific environmental limits, which for roads may be less stringent than drinking water guideline values.

Comparison of the concentration of constituents in the two stages of the compliance test provides information on the relative solubility of the leached constitutions. In some cases the concentration of the constituents from the test may be very low, which means the retention of constituents in the sample matrix is very high or indicates that the constituents in the test materials are fairly immobile. It is also probably an indication of the availability of the constituents in the sample matrix. The comparison of the concentrations can be interpreted by calculating the Q value (Baldwin et al., 1997) from Equation 7.1.

$$Q = \frac{A_{2-10}}{A_2} \qquad \ldots(7.1)$$

where:

$A_2$ is the release of a constituent at L/S = 2 (mg/kg of dry matter)

$A_{2-10}$ is the release of a constituent at L/S = 10 (mg/kg of dry matter)

The values of $A_2$ and $A_{2-10}$ can be calculated using the following equations.

$$A_2 = C_2 \times [(L_2 / M_D) + (MC / 100)] \qquad \ldots(7.2)$$

where

$A_2$ is the release of a constituent at a L/S = 2 (in mg/kg of dry matter)

$C_2$ is the concentration of a particular constituent in the eluate (in mg/l)

$L_2$ is the volume of leachant used (in l)

$MC$ is the moisture content ratio expressed as a percentage of the dry mass

$M_D$ is the dry mass of the test portion (in kg)

and

$$A_{2-10} = C_2 \times (V_{E1} / MD) + C_8 \times [(L_2 + L_8 - V_{E1}) / M_D + (MC / 100)] \qquad \ldots(7.3)$$

where

$A_{2-10}$ is the cumulative release at a cumulative L/S of 10

$C_8$ is the concentration of a particular constituent in the eluate from the second extraction (in mg/l)

$L_8$ is the volume of leachant applied in the second extraction (in l)

$V_{E1}$ is the volume of eluate recovered from the first extraction (in l)

and other parameters as before.

According to Baldwin et al (1997), if the $Q$ value is low (e.g. $Q<2$) the constituents are either washed out largely within L/S = 2 or the retention of the constituents in the sample matrix is extremely high. A high $Q$ value (i.e. $Q>6$) indicates that leachability increases after leaching solubility-controlling constituents, or that there are major changes in the leachability controlling mechanisms (e.g. pH, redox potential etc.). If the $Q$ value is intermediate (e.g.$2<Q<6$), the leachability is largely solubility controlled and the behaviour is reasonably well defined. In this case, the concentration in the eluate is constant and the release as a function of L/S increases proportionally to L/S until the leachable fraction is depleted, unless leachability-controlling parameters are changing with time.

Table 7.2 presents a summary of Q values calculated for tin slag and conventional aggregate. Generally, most of the analytes for tin slag and conventional aggregate show intermediate Q values (i.e. 2<Q<6). This suggests that the leaching of the contaminants is solubility controlled where solubility will limit the concentrations leached unless the controlling parameter (e.g. temperature, pH) change with time (Baldwin, et al., 1997).

Only a few contaminants with high Q values were observed, such as Se, Mn and Zn for tin slag and Zn, for conventional aggregate. This indicates increased leaching at higher L/S ratios in response to changes in the factors controlling leachability. Also, a limited number of contaminants show lower Q values (i.e. Q<2). This suggests that the contaminants have low mobility where the retention of the contaminants in the materials is high or contaminants are leached within the first stage (L/S = 2).

Using the data reported in Tables 7.1 and 7.2, tin slag was categorised as explained in Box 7.1. It was found that tin slag could be categorised into Group 1, which indicates no restriction on use, based on the potential to affect water quality. This indicates that the tin slag could be used in road pavements without giving adverse effects on surface water or ground water.

#### 7.5.2.2 Tank-Leaching Test

Table 7.3 presents the results obtained from the tank-leaching test for all specimens. The results of the test show that the quantities of constituents leaching from the specimens were very small. Most of the constituents leached from the specimens were below the detection limit. It was observed that the concentration value Arsenic leaching from compacted unbound tin slag was higher than the water quality guideline value proposed by World Health Organisation although not significantly so. However, this constituent could not be detected when tin slag was bound with bituminous material and cement.

A similar result was observed for Aluminium where the value of leached Aluminium from the unbound tin slag and the cement bound conventional aggregate specimens was a little bit higher than the water quality guideline value but was not detectable when bound with bituminous material. However, it was observed that the binder played an insignificant effect in preventing Aluminium leaching from cement bound material as the quantity of Aluminium leaching from the tin slag cement bound material (CBM) was higher than from the untreated compacted tin slag. It is suggested that the leached Aluminium came from cement which agreed with Hill’s (2004) findings. This behaviour also occurred for Potassium. In general, the tank-leaching test showed that cement binder treatment does not significantly modify the leaching behaviour of the tin slag tested.

**Table 7.1: Concentration of constituents observed from the compliance test**

| Constituent | WHO Guideline ($\mu$g/l) | Tin slag | | Conventional Aggregate | |
|---|---|---|---|---|---|
| | | Concentration $C_2$ ($\mu$g/l) | Concentration $C_8$ ($\mu$g/l) | Concentration $C_2$ ($\mu$g/l) | Concentration $C_8$ ($\mu$g/l) |
| Arsenic | 10 | 88.9 | <10 | <10 | <10 |
| Boron | 300 | 125 | <100 | 104 | <100 |
| Cadmium | 3 | <3 | <3 | <3 | <3 |
| Copper | 2000 | <10 | <10 | 104 | <10 |
| Lead | 10 | <1 | <1 | <1 | <1 |
| Mercury | 1 | 0.0694 | <0.053 | 0.112 | 0.0948 |
| Nickel | 20 | <10 | <10 | <10 | <10 |
| Selenium | 10 | <0.75 | 2.85 | 19.9 | <0.75 |
| Manganese | 500 | <50 | 78.1 | <50 | <50 |
| Aluminium | 200 | <100 | <100 | 798 | 624 |
| Cyanide | 70 | <50 | <50 | <50 | <50 |
| Iron | * | <100 | 3610 | 1100 | 262 |
| Chromium | 50 | <10 | <10 | <10 | <10 |
| Magnesium | * | 576 | 274 | 2710 | 2110 |
| Zinc | * | <20 | 156 | <20 | 127 |
| Tin | * | <100 | <100 | <100 | <100 |
| Potassium | * | 280 | 156 | 3970 | 1820 |

* There is no WHO guideline value

**Table 7.2: Comparison of amount leached for the different constituents**

| Material | Release increases Q>6 | Solubility controlled 2<Q<6 | High solubility or low mobility Q<2 |
|---|---|---|---|
| Tin Slag | Se, Mn, Zn | B, Cd, Cu, Pb, Hg, Ni, Al, Cn, Fe, Cr, Sn, K | As, Mg |
| Conventional aggregate | Zn | As, B, Cd, Cu, Pb, Hg, Ni, Mg, Al, Cn, Cr, Mn, Sn, K, | Se, Fe |

**Table 7.3: Tank leaching test results**

| Constituents | WQS ($\mu$g/l) | UG Concentration ($\mu$g/l) | UTS Concentration ($\mu$g/l) | GAM Concentration, ($\mu$g/l) | TAM Concentration ($\mu$g/l) | CBM Concentration, ($\mu$g/l) |
|---|---|---|---|---|---|---|
| Arsenic | 10 | <10 | 49.6 | <10 | <10 | <10 |
| Boron | 300 | <100 | <100 | <100 | <100 | <100 |
| Cadmium | 3 | <5 | <5 | <5 | <5 | <5 |
| Copper | 2000 | <10 | <10 | <10 | <10 | <10 |
| Lead | 10 | <1 | <1 | <1 | <1 | <1 |
| Mercury | 1 | 0.0784 | 0.056 | 0.0606 | 0.0547 | 0.0749 |
| Nickel | 20 | <10 | <10 | 17.6 | <10 | <10 |
| Selenium | 10 | 1.2 | <0.75 | 1.18 | 0.843 | 1.11 |
| Manganese | 500 | <50 | <50 | <50 | <50 | <50 |
| Aluminium | 200 | <100 | 267 | <100 | <100 | 474 |
| Cyanide | 70 | <50 | <50 | <50 | <50 | <50 |
| Iron | * | 206 | 627 | <100 | <100 | 557 |
| Chromium | 50 | <10 | <10 | <10 | <10 | <10 |
| Magnesium | * | 434 | 119 | <114 | <114 | <114 |
| Zinc | * | <20 | <20 | <20 | <20 | <20 |
| Tin | * | <100 | <100 | <100 | <100 | <100 |
| Potassium | * | 420 | 551 | <114 | <114 | 2550 |

WQS – Water quality standard

UG – Unbound compacted conventional aggregate

UTS – Unbound compacted tin slag

GAM – Conventional aggregate asphalt mixture

TAM – Tin slag asphalt mixture

CBM – Cement bound mixture

* There is no WHO guideline value

## 7.6 RADIOLOGICAL IMPACT ASSESSMENT

All building materials contain various amounts of natural occurring radioactive nuclides. Materials derived from rock and soil contain mainly natural radionuclides of the Uranium 238 series, Thorium 232 series and Potassium 40. When radionuclides, which are naturally present in rocks, soils and minerals, have been concentrated by human industrial activities, they are classified as technologically enhanced naturally occurring radioactive materials (TENORM). Some of the TENORMs such as fly ash from coal fired power stations, by-product gypsum and certain slags, which are produced in large volumes, may be recycled in or used in building materials. Estimation of concentrations of Uranium 238, Thorium 232 and Potassium 40 in some building materials are shown in Table 7.4.

**Table 7.4: Estimates of concentrations of Uranium, Thorium and Potassium in building materials (NCRP, 1988)**

| Material | Uranium mBq/g | Thorium mBq/g | Potassium mBq/g |
|---|---|---|---|
| Granite | 63 | 8 | 1184 |
| Sandstone | 6 | 7 | 414 |
| Cement | 46 | 21 | 237 |
| Limestone concrete | 31 | 8.5 | 89 |
| Sandstone concrete | 11 | 8.5 | 385 |
| Dry wallboard | 14 | 12 | 89 |
| By-product gypsum | 186 | 66 | 5.9 |
| Natural gypsum | 15 | 7.4 | 148 |
| Wood | - | - | 3330 |
| Clay Brick | 111 | 44 | 666 |

### 7.6.1 TENORM in pavement structure

In some countries, it is common practice to use the slag from industrial processes like steel making or slags from other non-iron processes like copper-making as a primary aggregate. The slag is mostly used as aggregates in road building or building projects where, for technical reasons, it is usually mixed with other building materials (EC, 2001). Most of the slags contain TENORMs. Table 7.5 shows the activity concentration of some slags produced from certain industries.

**Table 7.5: TENORM activity concentration in varies slags (EC, 2002)**

| TENORM Residue | Activity Concentration [Bq/kg] | |
|---|---|---|
| | U-238 chain | Th-232 chain |
| Iron slag from blast furnace process in iron production | 64 - 368 | 30 - 98 |
| Slag from steel production | 5 - 31 | 0 - 5 |
| Copper slag from primary process | 35 | 13 |
| Aluminium slag from secondary production | 17 | 15 |
| Tin slag | 1100 | 300 |
| Phosphor slag (thermal process) | 1000 | 50 |
| Slag from incineration plant for domestic waste | 18 | 15 |
| Lead slag | 270 | 36 |

### 7.6.2 Generic Exposure Scenarios for the Radiological Assessment

The concentrations of TENORM residues in slag depend on the type of production process and the origin of the ore. So each will lead to typical TENORM residues with certain physical and chemical properties as well as different activity concentrations. In assessing the degree of exposure, besides the concentration of slag involved in the material of concern, the chemical and physical processing (such as grinding up material) should be taken into account as these processes may increase the radiological hazard of slag material. In addition, the transmission pathways of leachate or other emissions, the later re-use of the materials, and detailed possible exposure scenarios which apply to various steps of recycling or disposing of the material are also important parameters for radiological assessment.

In order to relate the dose received by individuals to a specific practice, or to the levels of radioactivity involved in a practice, a set of exposure scenarios, which relates the activity content to an individual dose, is constructed. The strategy is to develop a set of simple but realistic scenarios which represent average exposure situations to the critical groups of exposees. With regards to road pavements, the scenarios include the following (European Commission, 2002):

- Disposal or recycling option

  It is important for the derivation of scenarios to bear in mind that material, which is at one time recycled, will later become waste which might be disposed of (in a landfill or on a heap). Disposal at a later time is fully covered by the disposal scenarios, because any disposal after an initial reuse or recycling will lead to further mixing which will reduce the specific activity. The other option, to recycle material after it has been disposed of, is not explicitly taken into account in the scenarios. This recycling would, however, be covered by the recycling scenarios which could be applied in cases where material from disposal sites or heaps will be recycled once again.

- Transportation

  The exposure is predominantly by external radiation. During loading and unloading the driver is exposed to dust therefore the exposure pathway inhalation and ingestion have to be taken into account. For the short-distance transport of TENORM residues, the exposure time of the driver is shorter than for long-distance transport. The other assumptions are similar to the long distance transport.

- Dilution Scenario

  Slag used in road pavements is likely to be mixed with uncontaminated materials such as binder or natural aggregate as discussed in Chapters 5 and 6. Therefore, at the end of the process or in the end product there is almost always a reduction in specific activity compared with the TENORM residue material. Dilution factors allow for the content of TENORM residues in the mixture, which cause the radiological exposure, and therefore should be taken into consideration with regard to the dose calculation.

- External exposure

  External exposure is due to gamma emissions, inhalation of dust containing radioactivity and ingestion of material by workers and members of the general public. The exposure scenario may, for example, be relevant in the case of storage of TENORM in a yard area of a slag processing company.

- Groundwater pathway:

  Dispersion of radionuclides via seepage from e.g. a heap or pavement layers to a groundwater layer and from there to a private well is taken into account by assuming that the groundwater is used for irrigation of crops. In cases where the ground water is used as drinking water, it is necessary to evaluate on a case-by-case basis.

Based on the above generic exposure scenarios, the European Commission has produced guidelines on general clearance or exemption levels for slag to be used in road construction. The exemption or clearance level for U-238 and Th-232 for slag to be used in road construction is 12Bq/g and 14Bq/g respectively.

## 7.7 RADIOLOGICAL ASSESSMENT PROGRAMME

### 7.7.1 Assessment of Radioactive Residues in Solid Fraction

Information on radionuclide concentration of the solid fractions of slag materials is very important, as it is the basis of classification of radionuclides. For regulatory control purposes, each of the radionuclides has its own concentration limit that would classify the source as a radioactive material. The radionuclide concentration of the solid fraction of a slag material can be used to:

- Identify the presence of any constituents that have the potential to cause a radiological impact.

- Make a comparison of each constituent of the solid fraction with a regulatory limit.
- Enable radiation dose calculations to be made so that the radiological risk can be assessed.

For the purpose of this investigation, only Uranium 238, and Thorium 232 were taken into account in assessing radiological hazards. With regard to the Potassium 40, it was not considered in the radiological assessment in this study because, in the human body, Potassium is under homeostatic control, thus small increases in the intake of Potassium via drinking or eating will have no significant impact on the levels of Potassium in the body (UNSCEAR, 1988). In other words, when the Potassium level, necessary for the body is reached, no Potassium 40 can be taken up under this condition. Additionally, the inhalation dose to the lungs can be neglected due to the relatively low dose coefficient. Therefore, Potassium 40 present in the slag will not result in increased radiation doses. A previous study (Camplin, 1980) also indicated that external doses from Potassium-40 were very low. Hence Potassium-40 is not considered, in general, to be radiologically significant, therefore it was not included in this assessment.

Neutron Activation Analysis (NAA) methodology was used to determine U-238 and Th-232 concentration. NAA was performed at the Malaysian Institute of Nuclear Technology Research (MINT). The radioactivity determined from the NAA was compared with the clearance level stipulated by the European Commission and the UK's Ionising Radiation Regulations, 1999.

The dose caused by nuclides of the U-238 and Th-232 decay chain can rise independently of each other. A simple summation formula (see Equation 7.4) was used to determine the radioactivity level of mixed radionuclides.

$$\sum_{i=1}^{n} \frac{C_i}{C_{Li}} \leq 1 \qquad \ldots(7.4)$$

where

$C_i$ is the total activity in the material per unit mass of radionuclide *i* (Bq/g)

$C_{Li}$ is the clearance level of radionuclide *i* (Bq/g)

$n$ is the number of radionuclides in the mixtures

In the above equation, if the sum is less than unity, the material complies with the clearance requirements.

### 7.7.2 Leaching Tests Programme

In order to study the radiological impact of slag materials, leaching test methods, compliance tests and tank-leaching tests were used to determine the release of radionuclides. The leaching test methods used in this study were the same as the leaching test methods described in Section 7.5 but in this study the radionuclides were determined from the eluate.

The eluate from the leaching tests was sent to a third party laboratory for determination of gross alpha ($\alpha$) and gross beta ($\beta$) activity. Gross alpha and beta activity is the total radioactivity due to alpha or beta particle emissions as inferred from measurements on a dry specimen. At this stage, the concentration of radionuclide was not determined, in line with the recommendations of the World Health Organisation (see Figure 7.8). This is to reduce unnecessary cost as it is very costly to determine each radionuclide's concentration compared to the cost of determining gross alpha ($\alpha$) and gross beta ($\beta$) activity. It is also unnecessary to determine radionuclide concentration if the level of the radionuclides is too low to detect.

## 7.8 RESULTS AND DISCUSSION

### 7.8.1 Radioactivity in Solid Residue

The radionuclide concentrations of the solid fraction of the slag materials obtained in this study were compared with clearance level values established by the Regulatory Authority and European Union standard of practice. If the concentration of the radionuclide is found below or at the clearance value, the sources of radiation may be released from regulatory control.

Table 7.6 presents the concentrations of U-238 and Th-232 in tin slag. It was found that the radioactivity levels for both the U-238 and Th-232 in tin slag were below the clearance level stipulated by the European Commission and the UK's Ionising Radiation Regulations 1999. However, this assessment involved two radionuclides. Therefore, the clearance level of tin slag should be calculated from a summation of the overall radionuclides in the tin slag using Equation 7.4. It was found from the summation that the radioactivity level in the tin slag was 0.43 compared to the EU clearance levels and 0.57 compared to the UK's clearance levels. Thus, individually and in combination, the results show that tin slag may be used in road pavements without any regulatory control. Consequently, work with tin slag is not required to be notified to the authority as stipulated in the UK's Ionising Radiation Regulations 1999. The results from the tests also suggest that there is no necessity to determine the external radiation level of tin slag and of mixtures made from it, since the radioactivity level of the slag was less than the clearance value and the slag can be released from regulatory control.

The test results also indicate that tin slag contained higher U-238 and Th 232 concentrations than in granite. However, this was not conclusive evidence because radioactivity concentration in granite depends on its source.

**Table 7.6: Concentration of U-238 and Th-232 in tin slag compared with granite**

| Material | Bq/g | |
|---|---|---|
| | U-238nat | Th-232 |
| Tin slag | 2.75 | 2.82 |
| Granite+ | 0.063 | 0.008 |
| UK clearance level* | 10 | 10 |
| EU clearance level** | 12 | 14 |

+ NCRP (1998)

* HMSO (2002)

** European Union (2002)

### 7.8.2 Leaching Test

The results observed from the leaching tests were compared with World Health Organisation (WHO)'s drinking water guideline. The screening levels recommended by

WHO are 0.1Bq/litre for gross alpha (α) activity and 1.0Bq/litre for gross beta (β) activity. If the radioactivity concentrations are below this guideline value, then no further action is required (see Figure 7.8). If either the gross alpha activity concentration of 0.1Bq/litre or the gross beta activity concentration of 1.0Bq/litre is exceeded, then the specific radionuclides should be identified and their individual activity concentrations need to be measured.

The results of the leaching tests are shown in Table 7.7. It was found that the gross α and gross β values for all specimens tested for the leaching test were below the guideline values recommended by the World Health Organisation. Therefore, there was no further action necessary and the water was suitable for drinking. It was concluded that the slag will have no undesirable effects on water quality.

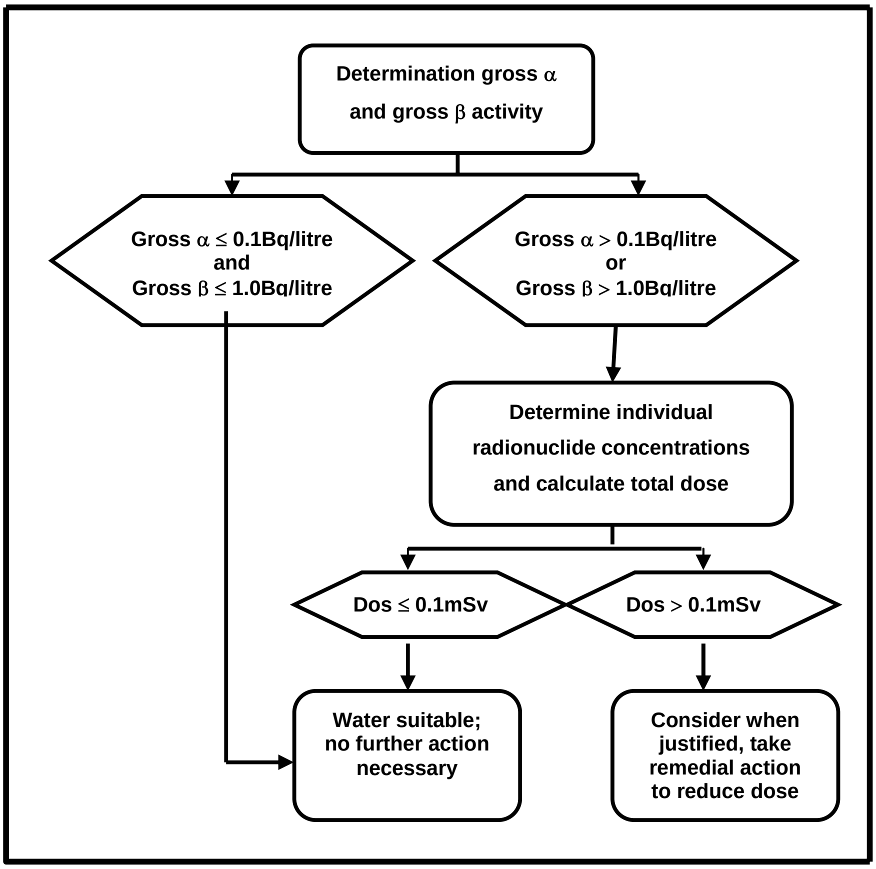



**Figure 7.8: Application of recommendations on radionuclides in drinking-water based on an annual reference level of dose of 0.1 mSv**

**Table 7.7: Leaching test results for determination of gross α and gross β**

| | Gross Alpha (Bq/l) | Gross Beta (Bq/l) |
|---|---|---|
| WHO guideline value | 0.1 | 1.0 |
| Distilled water | <0.010 | <0.025 |
| Tin slag VE1 | 0.019 | <0.025 |
| Tin slag VE2 | 0.020 | 0.031 |
| Granite VE1 | <0.010 | 0.159 |
| Granite VE2 | <0.010 | 0.091 |
| Bitumen bound Tin slag | <0.010 | <0.025 |
| Bitumen bound conventional aggregate | <0.010 | <0.025 |
| Cement Bound Tin slag | <0.010 | 0.127 |
| Unbound compacted tin slag | <0.010 | <0.025 |
| Unbound compacted conventional aggregate | <0.010 | <0.025 |

## 7.9 SUMMARY

This investigation has examined the environmental effects of using tin slag in road pavements which can be summarised as follows:

- The work on the compliance-leaching test revealed that most of the leached heavy metal constituents, except Arsenic, Iron and Magnesium, had concentrations lower than World Health Organisation water quality guideline values.
- The higher concentrations of Arsenic, Iron, and Magnesium may not pose any adverse effects on the environment because those materials fall under the “Grey” list indicating less hazard than posed by other elements.
- The tests show that most of the constituents cannot be detected when bound with bituminous material. However, the binder plays an insignificant role in preventing Aluminium leaching from cement bound materials as the quantity of Aluminium leaching was higher than that from untreated compacted tin slag. It was suggested that the excess Aluminium came from the cement.

- The radioactivity level of tin slag in the solids fraction was well below the clearance levels stipulated by the European Union and the UK's Ionising Radiation Regulation 1996. Therefore, tin slag may be used without any regulatory control.
- The investigation also revealed that the gross alpha and beta levels determined from the leaching test eluate were lower than the screening levels recommended by the World Health Organisation (WHO). Therefore, there was no need to do further tests to assess the radioactivity level on discharge water.
- It can be concluded that tin slag may be used in road pavements without any significant deleterious effects on the environment.

Chapter
8

# PAVEMENT STRUCTURAL DESIGN

## 8.1 INTRODUCTION

The main objective of pavement structural design is to provide a structure that will be suitable in a specific environment and able to sustain the anticipated traffic loading (European Commission, 2000). To enable the pavement structure to carry the applied traffic load, which is the main factor influencing the structural design condition, the design process involves the selection of materials for each of the pavement layers and an assessment of the thickness requirement for each layer. The thickness of pavement necessary to provide the desired load-carrying capacity is a function of the following principal variables – magnitude of vehicle wheel load or axle load, configuration of vehicle wheels or tracks, volume of traffic during the design life of pavement, pavement material properties and soil strength.

There exist two main approaches to the design of road pavement structures, an empirical method and an analytical design method. The empirical method is based on experience accumulation and is not based on engineering principles. By contrast, the analytical design approach uses theoretical analysis and incorporates the mechanical properties of both bound and unbound materials. It provides more reliable performance predictions and should be capable of dealing with any design situation.

In the previous chapters, laboratory investigations of the mechanical properties of unbound granular tin slag, tin slag cement-bound and tin slag asphalt mixtures have been reported. The primary objective of this chapter is to demonstrate how tin slag materials, as described in earlier chapters, might be implemented in pavement structures. In order to achieve this objective, an analytical design method has been employed to assess the impact in terms of performance of using tin slag materials in a pavement structure when compared to conventional materials.

## 8.2 PAVEMENT DESIGN PROCESS

In this Chapter the analytical design method used is described. The basic procedure of the method can be summarised in the main following points (Brown and Brunton, 1986):

- Specify the loading.
- Estimate the size of components.
- Consider the materials available.
- Carry out the structural analysis using theoretical principles.
- Compare critical stresses, strains or deflections with allowable values to assess whether the design is satisfactory.
- Make adjustments to materials or geometry until satisfactory design is achieved.
- Consider the economic feasibility of the result.

Figure 8.1 shows the pavement design process. The design is based on three basic assumptions as follows:

- The pavement layers extend indefinitely in the horizontal direction
- The bottom layer i.e. the sub-grade extends indefinitely downwards.
- Materials are not stressed beyond their elastic range.

The design procedure requires the determination of the asphalt and subgrade stiffnesses together with the allowable strains. The minimum thickness of asphalt is also determined during the design process. The designs considered here are relatively simple and comprise a three layered pavement structure of an asphalt pavement on a base of unbound granular material overlaying the existing sub-grade (see Figure 8.2).

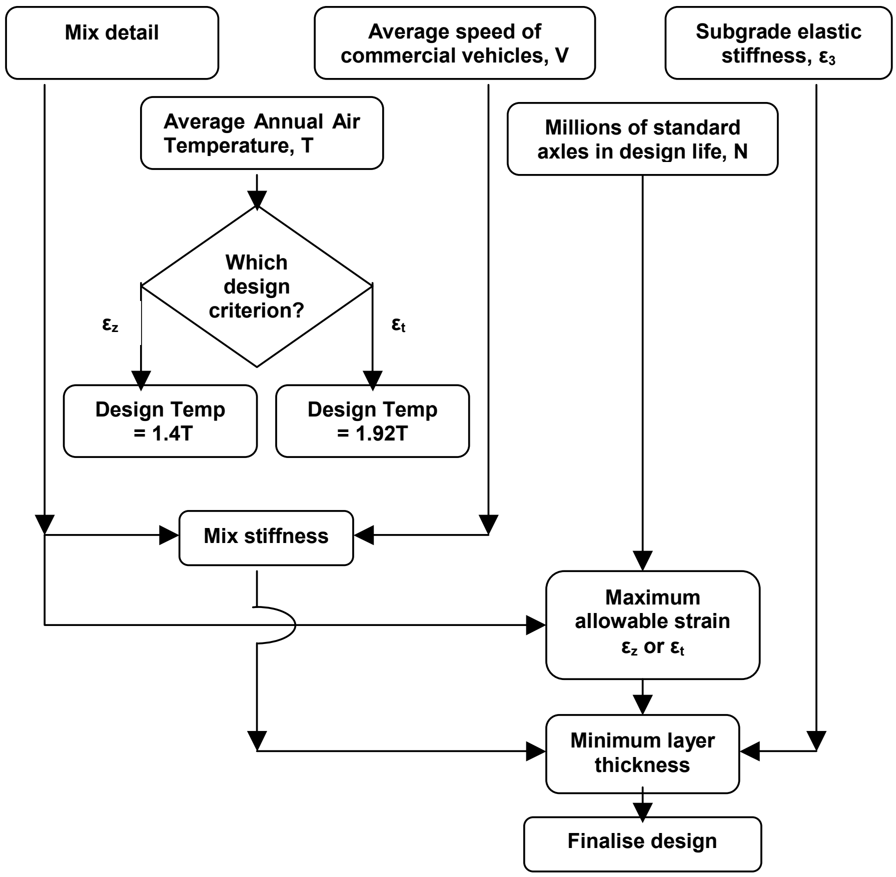



**Figure 8.1: Flow diagram of design procedure (Brown and Brunton, 1986)**

### 8.2.1 Criteria of Pavement Failure

Two criteria of pavement failure were considered in this design analysis as follows:

#### 8.2.1.1 Fatigue Failure Criterion

The design criterion for fatigue failure is tensile strain at the bottom of the asphalt layer, mid-way between the dual wheels in the direction of trafficking. A maximum allowable horizontal tensile strain at the bottom of the bituminous layer induced by the load application, i.e. $\varepsilon_t$ can be determined using the following equation (Brown and Brunton, 1986):

$$\log \varepsilon_t = \frac{14.39 \log V_b + 24.2 \log SP_i - 46.06 - \log N}{5.13 \log V_b + 8.63 \log SP_i - 15.8} \qquad \ldots(8.1)$$

where:

$\varepsilon_t$ is the maximum allowable tensile strain

$V_b$ is the volumetric proportion of binder

$SP_i$ is the initial softening point of binder

$N$ is the life in millions of standard axles

$V_b$ is calculated using the Equation 8.2 and in the calculations presented in this chapter, data from Chapter 6 were used to calculate the $V_b$ values. The $V_b$ values for TSS-tin slag and CS-control mixtures were calculated at 12.4% and 11.7% respectively.

$$V_b = \frac{(100 - V_v)(M_B / G_b)}{(M_B / G_b) + (M_A / G_a)} \qquad \ldots(8.2)$$

where:

$V_v$ = volume of voids.

$M_B$ = binder content per cent by mass of total mix

$M_A$ = aggregate content per cent by mass of total mix

$G_b$ = specific gravity of binder

$G_a$ = specific gravity of mixed aggregate

$SP_i$ value was assumed as 45.7°C for bitumen with 100pen (Brown and Brunton, 1986). Based on the above equation, it was calculated that the $\varepsilon_t$ values for TSS-tin slag and CS-control mixtures were 244 microstrains (με) and 239 microstrains (με) respectively. Based on the test results in Chapter 6, which proposed that the tin slag-sand (TSS-tin slag) asphalt mixture may be used for low or medium volume roads. It was decided that, for the purpose of this research, the design life, or number of load applications $N$ (in millions of standard axle, msa) was to be 1 msa. It should be noted that the development of Equations 8.1 and 8.2 was based on in-situ conditions and they are therefore suited to pavement analysis computations. The laboratory test situation (as reported in Chapter 6) differs from that in-situ in that no rest periods are allowed and crack propagation time is small. To allow for these differences, in a general way, the life at any strain level has been increased in Equations 8.1 and 8.2 by a factor of 100. This is made up of a factor of 5 for rest periods and 20 for crack propagation (Brown, et al., 1977).

#### 8.2.1.2 Permanent Deformation Criterion

Rutting can be prevented by limiting the maximum vertical strain ($\varepsilon_z$) at the top of the sub-grade layer. For life to failure, the maximum allowable vertical (compressive) strain, $\varepsilon_z$, can be calculated from the Equation 8.3 (Brown and Brunton, 1986).

$$\varepsilon_z = \frac{451.3}{(N / fr)^{0.28}} \quad \ldots(8.3)$$

where:

*N* is life in millions of standard axles, in this case 1msa.

*fr* is a rut factor which is 1.56 for dense bitumen macadam

From the Equation 8.3 it was calculated that the $\varepsilon_z$ value was 511 microstrains.

### 8.2.2 Analytical Modelling

The Shell Bitumen Stress Analysis in Roads (BISAR) computer model was used to analyse pavement design structures along with the Nottingham Pavement Design Method. BISAR allows linear elastic analysis in a multi-layer structure and provides the strain, stress and displacement profile. BISAR uses the following assumptions:

- The system consists of horizontal layers of uniform thickness resting on a semi-infinite base or half space.
- The layers extend infinitely in horizontal directions.
- The material of each layer is homogeneous and isotropic.
- The materials are elastic and have a linear stress-strain relationship.

BISAR calculations require several inputs to characterize a pavement structure and its response to loading. These inputs are:

- The number of layers – for the purpose of this research a three-layered structure was selected. These were the sub-grade, sub-base (granular layer) and surfacing (asphalt layer)
- Stiffness moduli of the layers – the value of stiffness modulus for TSS-tin slag and CS-control asphalt mixtures were chosen from previous test results shown in Chapter 6. A value of 50MPa was chosen for the sub-grade stiffness modulus based on the standard value used in Malaysia (Mudiandy, 2004). In the BISAR program, the stiffness modulus of unbound granular base (sub-base), $E_{gb}$, with thickness $h_2$ is dependent on the stiffness modulus of sub-grade, $E_{sg}$, and can be expressed as follows:

$$E_{gb} = k \times E_{sg} \qquad \ldots(8.4)$$

where:

$k = 0.2 \times h_2^{0.45}$

$h_2$ = thickness of sub-base in mm.

- The Poisson's ratios of the layers – For each asphalt mixture, it was assumed that the Poisson's ratio was 0.35 since this value is routinely used in pavement design. The sub-grade and sub-base were assumed to have Poisson's ratios of 0.4 and 0.3 respectively.
- The thickness of the layers – for the purpose of this study, the thickness of the asphalt layer was fixed while the thickness of the sub-base layer was variable. The objective was to obtain as minimum an asphalt layer thickness as possible so as to minimise the overall cost as the asphalt layer is the most expensive layer in a pavement structure.
- The vertical normal component of the load – in this study a standard 80kN axle was assumed with dual wheels each carrying 20kN. The contact radius of the imprint of each wheel on the road surface was assumed to be 105mm (see Figure 8.2).

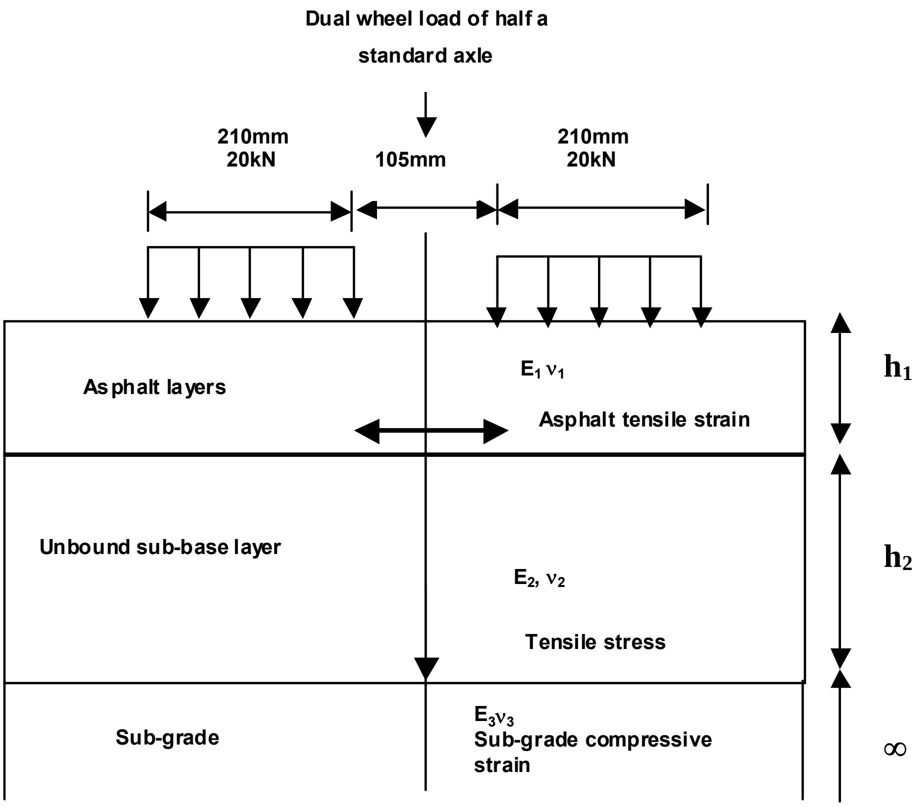



**Figure 8.2: Pavement structure diagram for BISAR analysis**

## 8.3 RESULTS AND DISCUSSION

### 8.3.1 Scenario 1 - The Use of Tin slag as an Asphalt Layer

Figures 8.3 and 8.4 show the results from pavement structural design analysis with tin slag used as an asphalt layer. It was found that at a TSS-tin slag asphalt layer thickness $h_1$=150mm, the minimum sub-base thickness, $h_2$, required to comply with both vertical and tensile strain criteria was 900mm. However, when the thickness of TSS-tin slag asphalt layer, $h_1$, was increased to 200mm, the minimum sub-base layer required for the pavement design, reduced to 450mm. After taking into account the combined thickness of both the asphalt layer made from TSS-tin slag and the sub-base layer, it was suggested that a suitable, more realistic thickness for the TSS-tin slag asphalt layer should be $h_l$=200mm.

Figure 8.5 shows the result of pavement structural analysis when using CS-control as an asphalt layer. It was found that at an asphalt layer thickness, $h_l$=150, the minimum sub-base thickness required to meet both vertical and tensile strain criteria was only 600mm compared to 900mm when 150mm of TSS-tin slag was used as a surfacing. By comparing the pavement design results in Figures 8.3 and 8.4, it is suggested that if TSS-tin slag asphalt mixture is to be used as a wearing course layer, the thickness requirement is around 200mm.

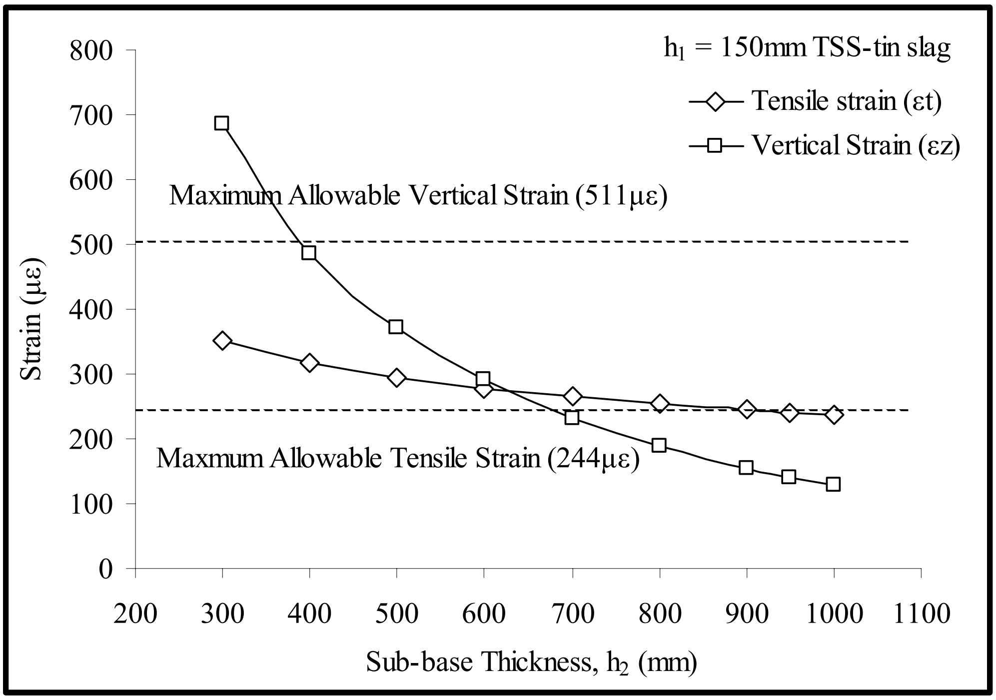



**Figure 8.3: Pavement structure analysis for 150mm TSS-tin slag asphalt layer with granular sub-base**

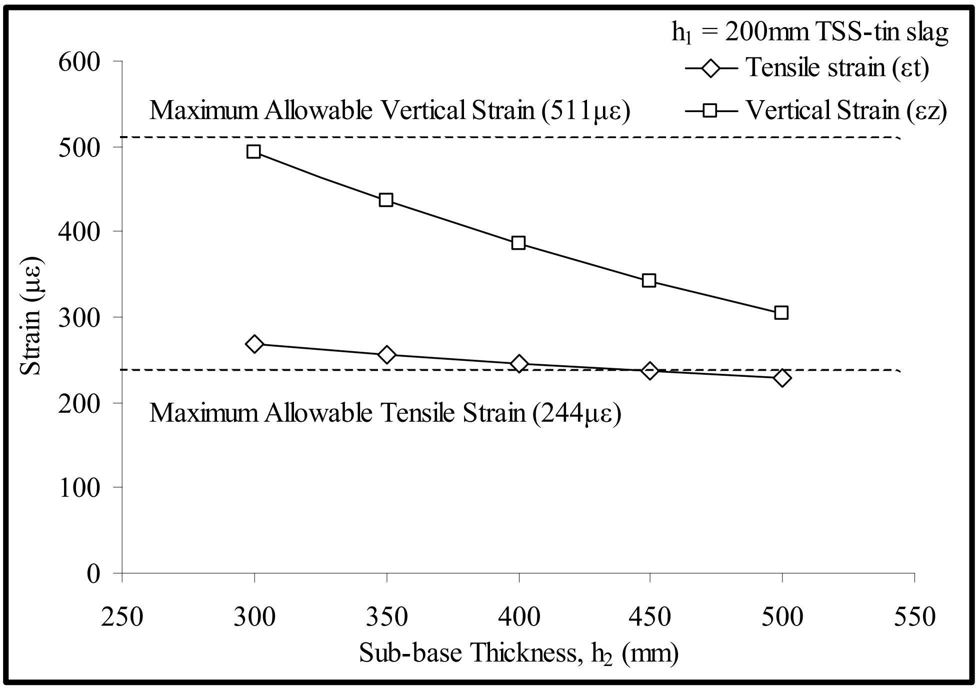



**Figure 8.4: Pavement structure analysis for 200mm TSS-tin slag asphalt mixture with granular sub-base**

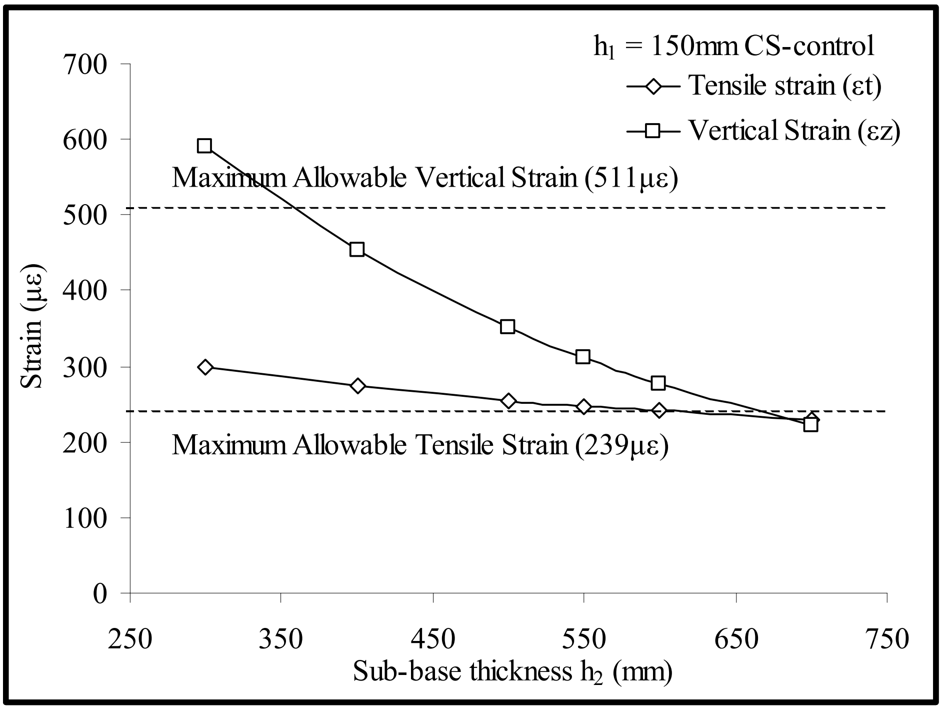



**Figure 8.5: Pavement structure analysis for 150mm CS-control asphalt layer with granular sub-base**

### 8.3.2 Scenario 2 – The use of Tin Slag as Granular Sub-Base Layer

Figure 8.6 shows the pavement design analysis incorporating tin slag as a granular sub-base layer with CS-control as an asphalt layer. It was found that with an asphalt layer of $h_1$=150mm, the minimum tin slag granular sub-base thickness $h_2$ required to comply with both vertical and tensile strain criteria was 800mm. However, the minimum tin slag granular sub-base thickness requirement reduced to only 450mm when the thickness of asphalt layer was increased to $h_2$=200mm (see Figure 8.7). Nevertheless, such analysis is very conservative as the stiffness modulus of the CS-control mixture used in the calculations was 1817MPa (as measured using ITSM test in the laboratory) whereas a conventional DBM layer on the road is expected to have an in-situ stiffness value of around 3100MPa. If a standard DBM was used in the design, the thickness of tin slag needed for the granular sub-base would have been less than the results shown above.

When the same wearing course thickness ($h_1$=150mm) of tin slag was used as an asphalt layer, it was found that the minimum thickness of tin slag required as a granular sub-base was approximately $h_2$=1250mm (see Figure 8.8). This is too thick for a sub-base and is not realistic. Therefore, the thickness of tin slag asphalt layer was increased to $h_1$=200mm and the results of this analysis are shown in Figure 8.9. It was observed that a thickness of $h_2$=550mm of tin slag granular sub-base would be required to comply with both the vertical and tensile strain criteria requirement. If comparisons are made between Figures 8.7 and 8.9, it can be observed that both designs require almost similar thicknesses of asphalt and granular sub-base layers. This shows that in principle, it is possible to use tin slag in the asphalt surfacing layer and sub-base granular layer simultaneously.

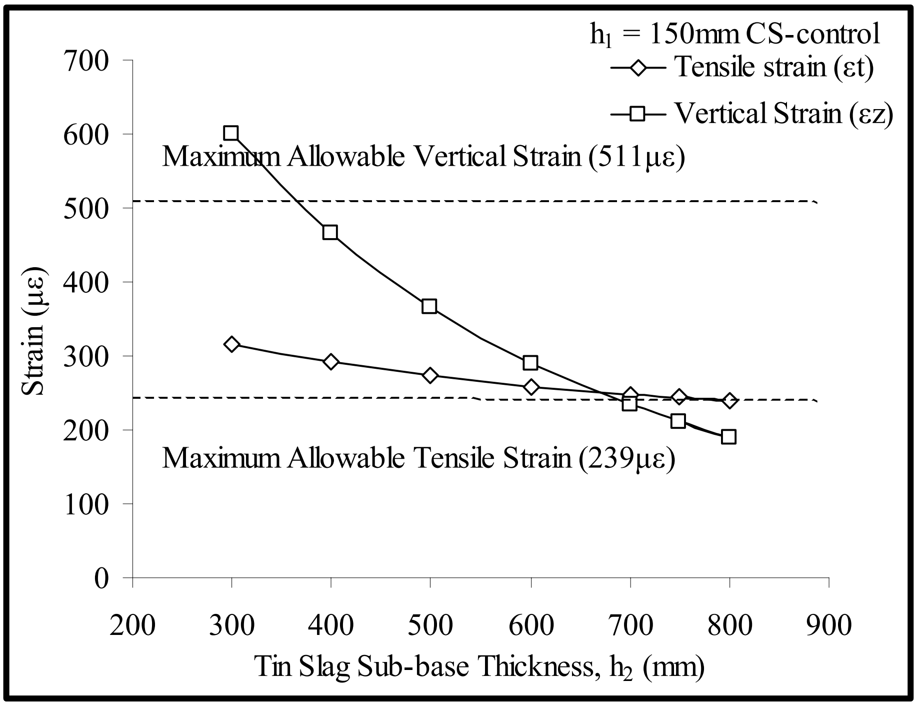



**Figure 8.6: Pavement structure analysis for 150mm CS-control asphalt layer with tin slag granular sub-base**

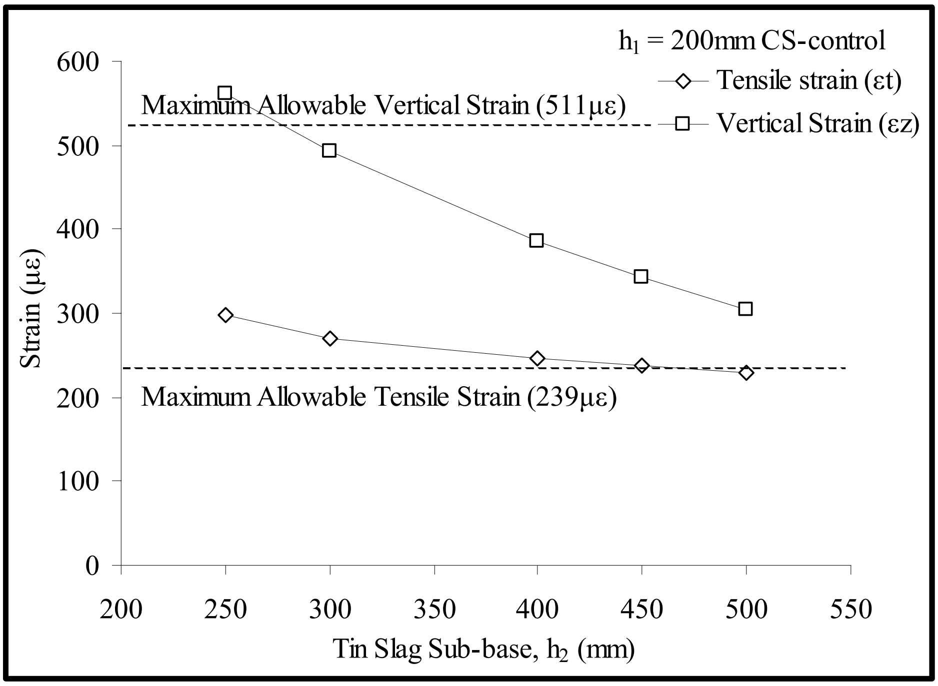



**Figure 8.7: Pavement structure analysis for 200mm CS-control asphalt layer with tin slag granular sub-base**

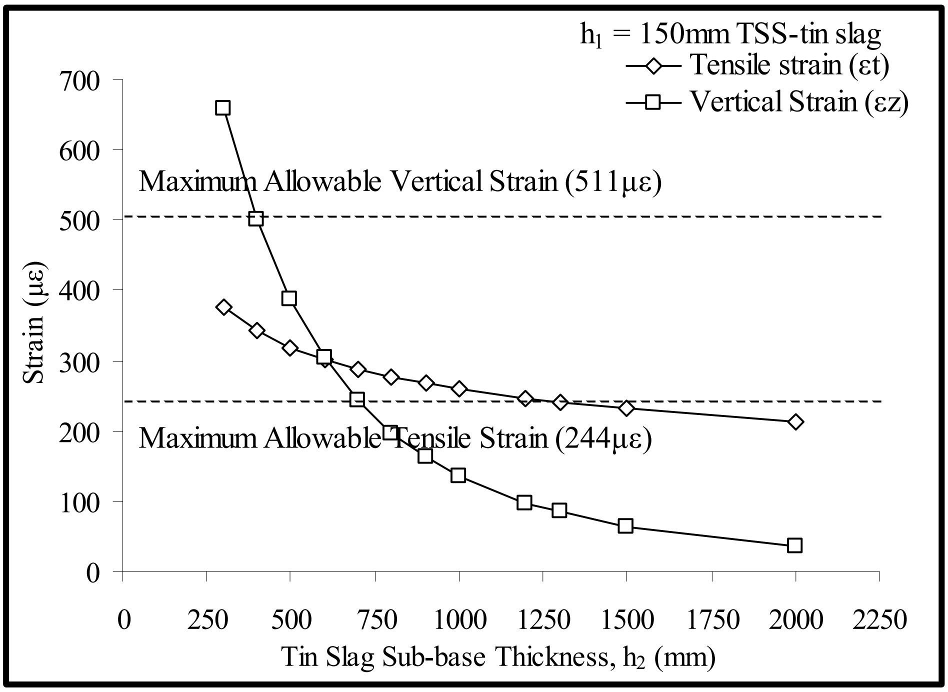



**Figure 8.8: Pavement structure analysis for 150mm TSS-tin slag asphalt layer with tin slag granular sub-base**

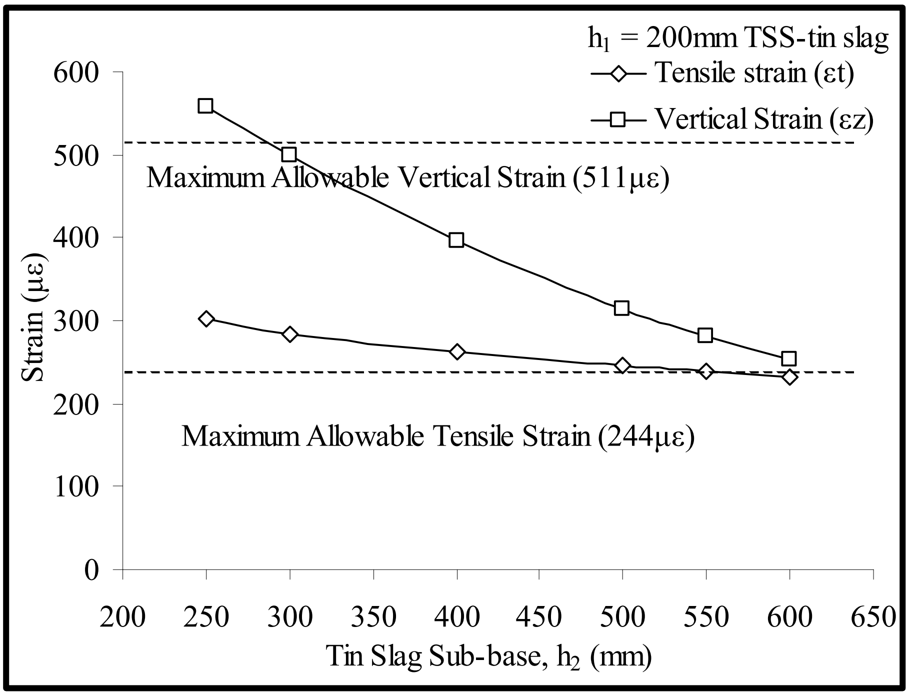



**Figure 8.9: Pavement structure analysis for 200mm TSS-tin slag asphalt layer with tin slag granular sub-base**

## 8.4 PREDICTION OF PERMANENT DEFORMATION

Permanent deformation or rutting of bituminous pavements can be estimated using Equation 8.5 developed by Van de Loo (1978).

$$R_d = \frac{C_m \times h \times \sigma_{avg}}{S_{mix}} \qquad \ldots(8.5)$$

where: $R_d$ = rut depth (mm)

$C_m$ = the dynamic effect correction factor which takes into account differences between static (creep) and dynamic (rutting) behaviour. It depends on type of mix (see Table 8.1).

$h$ = the initial thickness of the bituminous layer (mm)

$\sigma_{avg}$ = the average value of compressive stress under the moving wheel (MPa), dependent upon bituminous layer type and pavement temperature

$S_{mix}$ = the stiffness of the bituminous mix obtained from the creep test (MPa) at a certain value of bitumen stiffness $S_{bit} = S_{bit,\ visc}$.

**Table 8.1: Correction factor, $C_m$, for dynamic effects (Van de Loo, 1978)**

| Mixture type | $C_m$ |
|---|---|
| Sand sheet and lean sand mixes<br>Lean open asphaltic concrete | 1.6 – 2.0 |
| Lean bitumen macadam | 1.5 – 1.8 |
| Asphaltic concrete, gravel sand asphalt, dense bitumen macadam (DBM) | 1.2 – 1.6 |
| Mastic types, hot rolled asphalt (HRA) | 1.0 – 1.3 |

The stiffness modulus of bitumen that corresponds to its viscous component can be estimated from Equation 8.6.

$$S_{bit,visc} = \frac{3\eta}{NT_w} \qquad ...(8.6)$$

where:

$S_{bit,\ visc}$ = viscous component of bitumen stiffness (kPa)

$\eta$ = viscosity of bitumen (Ns/m$^2$) as a function of penetration index (PI) value and softening point of bitumen and can be obtained by using the nomograph presented in Figure 8.10.

$N$ = number of stress applications

$T_w$ = loading time of one load pulse.

For the purpose of this assessment, the following assumptions were used:

- The $C_m$ value selected for a computation of rut depth estimation was 1.4 for dense bitumen macadam, based on the average value stipulated in Table 8.1 for dense bitumen macadam.
- The number of stress applications ($N$) was 1 x $10^6$ based on the suggestions in Chapter 6.
- A $T_w$ value of 0.02 seconds was used for all calculations, which was assumed to correspond to a vehicle speed of 50km/h.

The pavement temperature was taken as 1.47 times the average annual air temperature (Brown et al., 1982). This factor was established using typical monthly air temperature from Croney and Croney (1991) and from a consideration of the effects of diurnal variations in both temperature and traffic loading. In this case, 28.5$^o$C was selected for Malaysia's average annual air temperature. This annual air temperature was obtained from an average of the monthly highest and lowest temperatures for twelve months (December 2003 to November 2004) as published by the Malaysian Meteorological Services (2004). Therefore, the asphalt design temperature was calculated as 41.9$^o$C. The penetration index (PI) was obtained from BANDS computer programme using a softening point value of 45.7$^o$C (Brown and Brunton, 1986).

The viscosity of bitumen, $\eta$, used in the investigation was obtained from a nomograph (Van de Loo, 1978) as shown in Figure 8.10 and this allowed the value of $S_{bit,\ visc}$ to be calculated using Equation 8.6. The $S_{bit,\ visc}$ value was unchanged since the same type of bitumen was used for both TSS-tin slag and CS-control mixtures. Table 8.2 shows the values of viscosity ($\eta$) and stiffness ($S_{bit,\ visc}$) of the bitumen used.

**Table 8.2: Stiffness and viscosity of bitumen at 40°C**

| Bitumen | Penetration index (PI) | T-$T_{SP}$ | Viscosity of bitumen, $\eta$, (Ns/m$^2$) at 40°C | $S_{bit,\ visc}$ (MPa) |
|---|---|---|---|---|
| 100pen. | -0.6 | -3.8 | $4\times10^{3}$ | $8.6\times10^{-7}$ |

In principle, the relationship between stiffness of the mixture ($S_{mix}$) from creep test results from Figure 6.57 in Chapter 6 and stiffness of bitumen ($S_{bit}$), at the same loading time, can be used to characterise the mixture behaviour in terms of deformation performance. In this case, only the central part of the relationship shown in Figure 6.57 has been used, i.e. the region with a constant rate of accumulation of strain approximately between 20 and 2500 load cycles. The strain rate after this region cannot be used since it increased rapidly leading to creep rupture. It is therefore acknowledged that the resulting relationship between mixture and bitumen stiffness is approximate. Stiffness of bitumen values can be determined using BANDS computer program developed by Shell. Figure 8.11 shows the relationship between $S_{mix}$ and $S_{bit}$ on a logarithmic scale for the TSS-tin slag and CS-control mixtures both at 40°C. From Figure 8.11, at $S_{bit}$ $8.6\times10^{-7}$ MPa, it was found that the values of $S_{mix}$ for TSS-tin slag and CS-control mixtures are 2.3MPa and 4.5MPa respectively. Using Equation 8.5, the rut depth for TSS-tin slag and CS-control mixtures can be estimated as shown in Table 8.3. The result shows that TSS-tin slag mixture suffers approximately four times the amount of rutting predicted for the CS-control mixture.

According to Croney and Croney (1991), experience shows that when permanent deformation exceeds 15mm, there is an increasing probability of cracking in the wheel tracks. In Britain, for flexible pavements, a maximum deformation of 25mm in the wheel tracks has been defined as the failure condition. A maximum deformation of 15-20mm is regarded as optimum condition for remedial work. Using such criteria it can be seen that TSS-tin slag mixture can survive beyond one million standard axle wheel passes before rutting becomes critical.

The rut depth estimation shows that the TSS-tin slag mixture can withstand one million applications of standard axles without a problem. However, beyond one million standard axles, the TSS-tin slag pavement may suffer from excessive rutting. Therefore, it is recommended that if TSS-tin slag wearing course mixtures are to be used in pavements carrying in excess of 1msa, the tin slag asphalt mix should not be used as a wearing course. In such cases, it would be advisable to use the tin slag asphalt in binder or base course layers.

The above rutting prediction was based on the repeated load creep test results. However, to get more accurate results for characterising asphalt mixture and to validate the above creep tests, the use of wheel tracking is recommended. This can assess the resistance to rutting of an asphalt mixture under conditions which simulate the effect of traffic under a rolling tyre. Under the wheel tracking test, a loaded wheel tracks a sample under specified conditions of speed and temperature while the development of the rut is monitored continuously during the test.

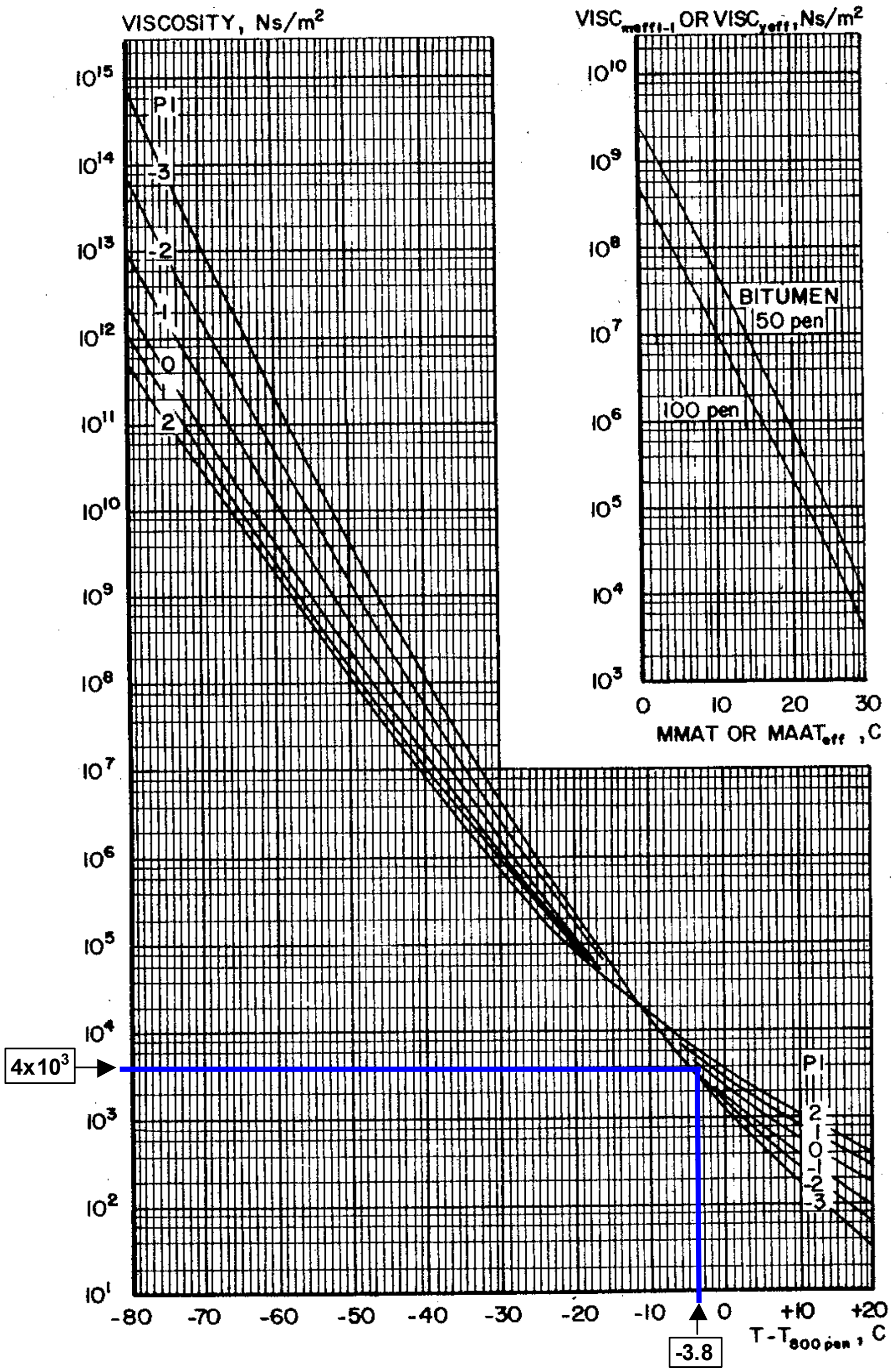



**Figure 8.10: Viscosity as a function of the temperature difference T-$T_{SP}$ (Van de Loo, 1978)**

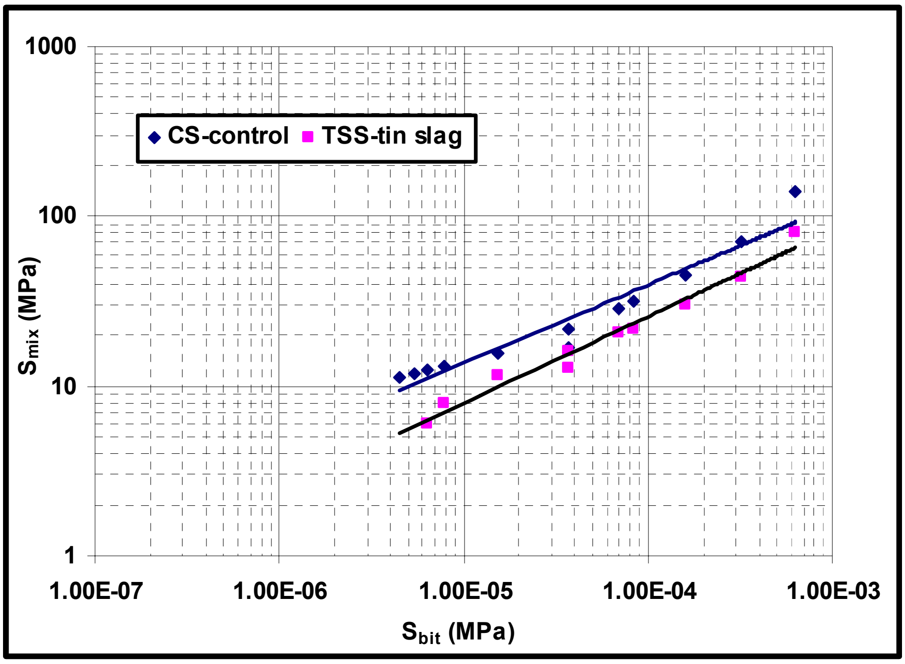



**Figure 8.11: Relationship between $S_{mix}$ and $S_{bit}$ of TSS-tin slag and CS-control mixtures**

**Table 8.3: Estimated rut depth value for TSS-tin slag and CS-control mixtures after one million applications of standard axles.**

| Mixture | Rut Depth (mm) |
|---|---|
| TSS-tin slag | 12.67 |
| CS-control | 3.88 |

## 8.3 SUMMARY

- In this study, both the Nottingham Pavement Design Method and Bitumen Stress Analysis in Roads (BISAR) have been used for pavement structural design to determine the possibility of using tin slag in asphalt and granular base layers.
- During the pavement structural design process, the thickness of the asphalt layer was minimised considering that the asphalt layer was the most expensive layer for road making.
- Pavement structural design analysis showed that it was possible to construct a road to withstand 1msa using tin slag asphalt mixture with a thickness 200mm as

a surfacing layer, laying on top of a sub-base of thickness 450mm.

- Tin slag could also be used as a granular sub-base layer. It was found that with a conventional asphalt layer 200mm thick, the minimum thickness of tin slag granular sub-base required was 550mm. However, this design was very conservative since the standard DBM was not used when designing the asphalt layer.
- Tin slag could also be used simultaneously in both the asphalt and granular sub-base layers. It was observed that when tin slag asphalt and tin slag granular sub-base layers with thickness 200mm and 550mm respectively were used, analysis using BISAR showed that there was not much difference in thickness requirements when conventional asphalt and tin slag granular sub-base layers were used.
- The predictions of permanent deformations in the asphalt layers show that TSS-tin slag mixture can withstand one million applications of standard axles without suffering from excessive rutting. However, it was recommended that for pavements carrying in excess of 1msa, the TSS-tin slag asphalt mixtures are more suitable as binder or base courses.

Chapter

9

# OVERALL DISCUSSION, RECOMMENDATIONS FOR FUTURE RESEARCH AND CONCLUSIONS

## 9.1 INTRODUCTION

This chapter presents a summary and an overall discussion of the research conducted in the previous chapters, including physical and mechanical properties of tin slag, hydraulically bound materials, bituminous bound materials, environmental evaluation and pavement structural design. The chapter also includes recommendations for future research related to these works and the overall conclusions on the possibilities available for utilisation of tin slag in road pavements.

## 9.2 BASIC PHYSICAL AND MECHANICAL PROPERTIES OF TIN SLAG

A study of particle shape and surface structure of tin slag showed that most of the tin slag particles had angular shape. However, a small amount of tin slag, especially with particle sizes greater than 6mm, had well-rounded shapes. Needle-shaped particles were also present in small quantities. The proportion of elongated and flaky particles was insignificant. The Aggregate Crushing Value (ACV), Aggregate Impact Value (AIV) and Ten-percent Fines Value (TFV) tests showed that in terms of toughness, tin slag has a satisfactory performance and meets most of the requirements stated in the UK's Specification for Highway Works and has potential for use in road pavements, although the TFV test results seemed to be unduly low.

*Recommendation for future research*

- Since there is very little information available in the literature about tin slag properties, the programme of testing should be extended beyond the scope of this investigation to include other tests such as expansion test, magnesium sulphate test and California Bearing Ratio (CBR). This is important to help establish a more comprehensive database on the properties of tin slag.

## 9.3 INVESTIGATING THE POTENTIAL USE OF TIN SLAG AS UNBOUND GRANULAR MATERIAL

Repeated load triaxial tests have been used to study the shear strength and stiffness modulus of unbound tin slag. With regard to the comparative study on shear strength, it can be concluded that tin slag and a granite conventional aggregate with similar maximum particle size, have very little difference in terms of shear strength. In terms of stiffness modulus, it was found that the value for tin slag was 14% lower than that for the granite conventional aggregate with similar maximum nominal aggregate size.

*Recommendations for future research*

- A more detailed investigation of the stiffness modulus and shear strength of tin slag using conventional repeated load triaxial test equipment is recommended to obtain more accurate data. Furthermore, it is recommended that combinations of tin slag and mineral aggregate should be tested for their mechanical properties.
- At present, there is no available information on permanent deformation behaviour of unbound tin slag under repeated loading. Research in this field is recommended as a means of predicting and modelling the rutting behaviour.

## 9.4 ASSESSMENT OF TIN SLAG IN HYDRAULICALLY BOUND MIXTURES

A simple comparative study using a compressive strength test method was used, capable of assessing the pozzolanic property of tin slag. The test results showed that tin slag has a modest pozzolanic property and thus it has little potential for use in forming tin slag-bound materials. Therefore, tin slag was proposed to be utilised in cement bound materials.

Two mixtures, tin slag and tin slag/sand mixtures were selected to study the possibility of using tin slag in cement bound materials. The tests showed that cement bound tin slag/sand mixtures have higher compressive strength values than cement bound tin slag only mixtures at the same quantity of cement (i.e. tin slag/sand mixture required less cement content compared with tin slag only mixture for the same strength). An assessment of tin slag in hydraulically bound materials allowed the following conclusions to be drawn:

- The laboratory tests showed that both cement-bound tin slag and cement-bound tin slag/sand mixtures may be successfully used as cement-bound materials in road-bases.
- There is a possibility of producing tin slag cement-bound materials of higher strength by adding sand. Using sand can reduce the need for cement, thus reducing the cost of producing cement bound mixtures without any compromise in the strength of the mixtures.
- In terms of durability, the tin slag and tin slag/sand mixtures seem to comply with the requirements of the Specifications for Highway Works.

*Recommendations for future research*

- Limited laboratory testing has proved that there is a potential to design cement-bound tin slag/sand mixtures for use as sub-bases. On the basis of satisfactory performance in the laboratory, further testing and pilot trials are recommended to develop a more detailed understanding of these mixtures.

- Further investigation into the best amount and gradation of sand could be undertaken. More work in optimising moisture content for hydration as well as compaction is warranted.
- A more comprehensive study of the compressive strength profile of the mixtures at different curing periods is recommended to gather more information about the potential for using tin slag in cement-bound materials.
- The programme of testing should be extended to determine the chemical composition of tin slag (not considered in this study). This issue becomes more important when a comprehensive assessment of the pozzolanic properties of tin slag is to be undertaken.

## 9.5 INVESTIGATING THE POTENTIAL FOR INCORPORATING TIN SLAG IN BITUMINOUS BOUND MATERIALS

Two mixtures, TB-tin slag mixture (which was composed tin slag and conventional aggregate with 20mm maximum nominal aggregate size), TS-tin slag mixture (which was composed tin slag with 10mm maximum nominal size) and then TSS-tin slag (which was composed of tin slag/sand with 10mm maximum nominal aggregate size) were investigated and compared with control mixtures having similar maximum nominal size mineral aggregate. Optimum binder contents were determined for each mix separately following well-established mix design procedures that included gradation control and volumetric analysis. A series of performance tests including Indirect Tensile Stiffness Modulus (ITSM) Test, Indirect Tensile Fatigue Test (ITFT), Repeated Load Axial Test (RLAT) and mixture durability were carried out to determine the performance of the mixtures. The main conclusions arising from this investigation are as follows:

- Tin slag aggregate without any alteration (TS-tin slag mixture) was not suitable for use in asphalt layers on the grounds that the stiffness and Marshall stability values were very low and the Marshall flow value was very high. Therefore, 20% fine sand was added to the tin slag to alter the gradation.

- With regard to the Marshall stability, both TB-tin slag and TSS-tin slag mixtures showed values that exceed the minimum value recommended by the Asphalt Institute. However, TSS-tin slag mixture has a higher Marshall Stability value than TB-tin slag mixture.
- In terms of indirect tensile strength test performance, the test results indicated that TSS-tin slag was 54% stronger than TB-tin slag mixture. However, TB-tin slag mixture has an indirect tensile stiffness value slightly higher than the TSS-tin slag mixture.
- In a study of permanent deformation performance on the TB-tin slag mixture and the TSS-tin slag mixture, it was found that the TSS-slag mixture performance was superior to the TB-tin slag mixture.
- It was therefore suggested that the TSS-tin slag mixture was very suitable for use in bituminous bound materials.

*Recommendations for future research*

- It is recommended that further investigation be undertaken to determine the optimum amount of sand and filler that should be added to the tin slag asphalt mixture and the optimum bitumen grade.
- Unlike the fatigue performance of the tin slag mixtures which was very comparable to conventional mixes, the creep performance of the wearing course tin slag gradation was not as good as the control mix. Therefore, tin slag may be more effectively used as a component of binder or base courses and further investigation should be carried out on the performance of tin slag asphalt mixtures for these layers.
- On the basis of satisfactory performance in the laboratory, further laboratory testing and a pilot trial are recommended to develop more understanding of the mixture's likely performance in the pavement.

## 9.6 ENVIRONMENTAL EVALUATION OF USING TIN SLAG IN ROAD PAVEMENTS

The possible adverse effects of tin slag on the environment were evaluated based on the constituents leached using the compliance and the tank-leaching test methods. The results of these tests were compared with reference levels for drinking water quality produced by the World Health Organisation. The environmental assessment of the use of tin slag in road pavements has drawn the following conclusions:

- The assessments of tin slag bituminous bound and cement bound materials showed that these treatments gave no significant contribution towards preventing constituents leaching from the mixtures.
- Most of the heavy metals in the leachate were at concentrations less than the limit value given in the water quality guidelines stipulated by the World Health Organization.
- According to the categorisation introduced by Baldwin et al (1997), tin slag could be categorized into Group 1 which has no restriction on use, based on potential to affect water quality.
- An assessment of the radioactivity levels in the tin slag showed that the level was less than the clearance level values stipulated by the European Commission and the UK's Ionising Radiation Regulations 1999. Therefore, tin slag may be used in road pavements without any regulatory control.
- The results of leaching tests showed that the gross $\alpha$ and gross $\beta$ values were well below the guideline values recommended by the World Health Organisation which means that the tin slag will have no undesirable effects on water quality in terms of radioactivity.
- Overall, tin slag may be used in road pavements without any significant deleterious effect on the environment.

*Recommendations for future research*

- It is recommended that the constituents in the solid fraction of the tin slag are determined. This is very important when the behaviour of leached constituents is to be studied.

- The study of behaviour of leached constituents should be extended. A study of duration of leaching and the effect of pH on leaching should be carried-out to provide the necessary information to define leaching behaviour under the wider range of conditions of tin slag compared to conventional aggregate.
- The programme of testing may be extended to determine the levels of radioactive concentration in bituminous and cement bound materials to obtain precise information on the radioactivity level of the composites containing tin slag. Such information is important to convince the authorities and the public of the safety and viability of using tin slag.

## 9.7 OVERALL CONCLUSIONS

Research in this thesis has combined an assessment of physical and mechanical properties of tin slag, an assessment of the potential for using tin slag as unbound material, an investigation into incorporating tin slag in bituminous bound materials, and also the use of tin slag in cement bound materials. The results showed that tin slag has the potential to be used in all the aforementioned applications. However, a decision has to be made as to which applications are best and in particular applications that can utilise the largest quantities of slag, taking into account the environmental effects, economic factors and public acceptance.

To identify which applications will use most of the tin slag, a comparison was made between two applications, a bituminous bound layer and an unbound granular base layer. For the purpose of this calculation, a single two-lane carriageway with 7.2m width with 3m-paved shoulder was assumed, based on the current recommendation in the United States of America (Wright, 2004).

For tin slag to be used as unbound granular material, a thickness of 450mm was used along with 200mm thickness of CS-control asphalt layer as designed in Chapter 8 and detailed in the second scenario. A tin slag bituminous bound material with thickness of 200mm was also used for the purpose of this calculation. In terms of the amount used, it was found that about 19000 tonnes of tin slag is required per one kilometre length of road as an unbound granular base, compared to about 4900 tonnes of tin slag per one kilometre when used in a bituminous bound layer. It must be noted that 200mm of asphalt was the amount required based on the stiffness modulus values obtained in the laboratory which are known to be lower than those that can be achieved in the field. Hence, for a design life of 1msa, a thinner asphalt layer would be required in real life. In terms of tin slag annual supply in Malaysia which currently stands at approximately 24000 tonnes, only 1 kilometre of unbound granular base layer could be built per year but 5 kilometres of tin slag bituminous bound materials can be built per year. With regards to the present tin slag stock (which is estimated about 280,000 tonnes), approximately 50 kilometres length of bituminous bound layer can be built using the present stock of tin slag or about 13 kilometres if the tin slag in used as an unbound granular base material. Given the limited lengths of need which could be constructed each year from current production some further study into use of tin slag in asphalts at lower percentage rates might be considered.

With regard to the environmental effects, generally all the applications investigated have no adverse effects on the environment although more comprehensive studies into this topic must be carried out. Tin slag bituminous bound materials seem to be the most acceptable in terms of the environmental effects compared with other applications. As described in Chapter 7, when tin slag was used as an unbound granular base or cement bound materials, some heavy metals were leached which exceeded the amounts leaching from bituminous bound application. The results show that tin slag bituminous bound materials are more reliable in terms of environmental effects compared to all other road applications.

In terms of leaching concentration, the constituent concentrations in the mixtures depend on the amount of tin slag used in the various applications. In bituminous bound materials, 69.7% of the mixture was tin slag while in cement bound material the figure was 79.6% and 100% for unbound granular materials. For that reason, bituminous bound materials will have the lowest constituent concentrations compared with cement bound materials and unbound granular materials and will thus be more acceptable in terms of environmental effects and safety.

One of the important environmental issues is the future problem of recyclability. In the use of tin slag there are no problems that should arise when the slag is used as unbound granular base, in cement bound materials or in bituminous bound materials.

Based on the above arguments, the present public acceptance and experience gained in using copper slag in road pavements in the USA as discussed in Chapter 2, it is therefore suggested that tin slag is very suitable for use in bituminous bound materials.

## 9.8 FINAL REMARKS

The main objective of this research was to study the possibility of using tin slag in road pavements. This was accomplished through the assessment of the performance of tin slag in various applications such as unbound granular materials, hydraulically bound materials and bituminous bound materials. The leaching and radiological effects of using tin slag have also been studied to ensure these applications have no adverse impacts on the environment. The study undertaken provides new knowledge about the potential applications of tin slag in road pavements. The results of this research show that it is possible to use tin slag in road pavements although further studies are warranted for particular applications. In particular, mix optimisation and pilot-scale trials are recommended.

# REFERENCES

## Abbreviations

| | |
|---|---|
| **BACMI** | British Aggregate Construction Materials Industries |
| **BSI** | British Standards Institution |
| **DETR** | Department of Environment, Transport and Region. |
| **ITDG** | Intermediate Technology Development Group |
| **MSC** | Malaysian Smelting Corporation Bhd. |
| **NCHRP** | National Cooperative Highway Research Program |
| **NCRP** | National Council on Radiation Protection and Measurement |
| **NLA** | National Lime Association |
| **NSA** | National Slag Association |
| **TFHRC** | Turner-Fairbank Highway Research Center |
| **WHO** | World Health Organisation |
| **UNSCEAR** | United Nations Scientific Committee on the Effects of Atomic Radiation |

**Airey, G.D.** *Introduction to mixture design.* Residential course on bituminous pavements – Materials, Design and Evaluation, 8$^{th}$-10$^{th}$ April 2002. Lecture Notes. University of Nottingham, 2002.

**Anderson, R.M. and Bentsen, R.A.** Influence of voids in mineral aggregate (VMA) on the mechanical properties of coarse and fine asphalt mixtures. *Journal of the Association of Asphalt Paving Technologists*, 2001, 70, 1-37.

**Asphalt Institute**. Manual No. 10 Soil Manual. The Asphalt Institute 1993

**Asphalt Institute.** The asphalt handbooks. Manual series no. 4(MS). 1989 Edition, Kentucky: Asphalt Institute, 1989.

**Atkinson, V.M., Chaddock, B. C., and Dawson, A.R.** *Enabling the use of secondary aggregates and binders in pavement foundations.* Crowthorne: Transport Research

Laboratory, 1999, (TRL Report 408).

**Ayano, T., Kuramoto, O. and Sakata, K.** Concrete with copper slag fine aggregate. *Journal of the Society of Materials Science*, 2000, 49(10), 1097-1102.

**BACMI.** *Bituminous Mixes and Flexible Pavements – An Introduction*. London: BACMI Ltd., 1992.

**Baldwin, G., Addis, R., Clark, J., and Rosevear, A.** *Use of industrial by-products in road construction: water quality effects.* London: Construction Industry Research and Information Association (CIRIA), 1997, (R-167).

**Barksdale, R.D. and Itani, S.Y**. Influence of aggregate shape on base behaviour, *Transport Research Board 1227*, Rigid and Flexible Design Analysis, Transportation Research Board, National Research Council, Washington, DC, 1989, pp. 173-182.

**Barksdale, R.D., et al**. *Laboratory determination of resilient modulus for flexible pavement design* [Online]. NCHRP Web Document 14, 1997. Available at: http://books.nap.edu [6 August 2004]

**Botton, J.W., et al**. Correlation of selected laboratory compaction methods with field compaction. *Transportation Research Record 1454*, 1994, 193-201.

**Boyce, J.R.** *The behaviour of a granular material under repeated load*. PhD Thesis, University of Nottingham, 1976.

**British Geological Survey.** *Minerals Planning Guidance Note 6 (MPG6): Guidance for aggregate provision in England. Analysis of data for 1996-97.* 2001. Keyworth, Nottingham, 2001.

**BSI** *BS 812-105.2 Testing aggregates - Methods for determination of particle shape -*

*Elongation index of coarse aggregate* London: British Standard Institution (BSI), 1990.

**BSI** *BS 812-110 Testing aggregates - Methods for determination of aggregate crushing value (ACV)*. London: British Standard Institution (BSI), 1990.

**BSI** *BS 812-111 Testing aggregates - Methods for determination of ten per cent fines value (TFV)*. London: British Standard Institution (BSI), 1990.

**BSI** *BS 812-112 Testing aggregates - Method for determination of aggregate impact value (AIV)*. London: British Standard Institution (BSI), 1990.

**BSI** *BS 812-121 Testing aggregates - Method for determination of soundness* London: British Standard Institution (BSI), 1989.

**BSI** *BS 1047 Specification for air-cooled blastfurnace slag aggregate for use in construction*. London: British Standard Institution (BSI), 1983.

**BSI** *BS 1881-120 Testing concrete - Method for determination of the compressive strength of concrete cores.* London: British Standard Institution (BSI), 1983.

**BSI** *BS 1924-2:1990 Stabilized materials for civil engineering purposes – Part 2: Methods of test for cement-stabilized and lime-stabilized materials.* London: British Standards Institution (BSI), 1990.

**BSI** *BS 3797 Specification for lightweight aggregates for masonry units and structural concrete.* London: British Standards Institution (BSI), 1990.

**BSI** *BS EN 933-1 Tests for geometrical properties of aggregates - Gradation.* London: British Standards Institution (BSI), 1997.

**BSI** *BS EN 933-3 Tests for geometrical properties of aggregates. Determination of* *particle shape - Flakiness.* London: British Standards Institution (BSI), 1997.

**BSI** *BS EN 933-4 Tests for geometrical properties of aggregates. Determination of particle shape - Shape index.* London: British Standards Institution (BSI), 2000.

**BSI** *BS EN 1097-2, Part 2 Tests for Mechanical and Physical Properties of Aggregates, Methods for the Determination of the Resistance to Fragmentation.* London: British Standards Institution (BSI), 1998.

**BSI**. *BS EN 1097-5 Test for mechanical and physical properties of aggregate.* London: British Standard Institution (BSI), 1999

**BSI**. *BS EN 1097-6 Test for mechanical and physical properties of aggregate – Part 6: Determination of particle density and water absorption.* London: British Standard Institution (BSI), 2000.

**BSI.** *BS EN 1367-2 Test for Thermal and Weathering Properties, Magnesium Sulfate Test*. London: British Standards Institution (BSI), 1998.

**BSI.** *BS EN 1744-1 Tests for chemical properties of aggregates - Chemical analysis.* London: British Standards Institution (BSI), 1998.

**BSI.** *BS EN 4987-1 Coated macadam (asphalt concrete) for roads and other paved areas - Specification for constituent materials and for mixtures.* London: British Standard Institution (BSI), 2001.

**BSI.** *BS EN 12457-3 Compliance test for leaching of granular waste materials and sludges.* London: British Standard Institution (BSI), 2002.

**BSI**. *BS EN 13043 Aggregates for bituminous mixtures and surface treatments for roads, airfields and other trafficked areas.* London: British Standards Institution (BSI), 2002.

**BSI.** *BS EN 13286-7 Unbound and hydraulically bound mixtures-Part 7: Cyclic load triaxial test for unbound mixtures.* London: British Standards Institution (BSI), 2004.

**BSI.** *BS EN 13286-41 Unbound and hydraulically bound mixtures-Part 41: Test methods for determination of the compressive strength of hydraulically bound mixtures.* London: British Standards Institution (BSI), 2003.

**BSI**. *BS DD213: 1993 Methods for determination of the indirect tensile modulus of bituminous mixtures*. London: British Standard Institution (BSI), 1993.

**BSI.** *BS DD226:1996 Methods for determining resistance to permanent deformation of bituminous mixtures to unconfined dynamic loading*. London: British Standards Institution (BSI), 1996.

**BSI.** *BS DD ABF:2000 Methods for determination of the fatigue characteristics of bituminous mixtures using indirect tensile fatigue.* London: British Standards Institution (BSI), 2000.

**BSI**. *Draft: BS EN 12697-12 Bituminous mixtures – Test methods for hot mix asphalt. Part 12. Determination of the water sensitivity of bituminous specimens.* London: British Standards Institution (BSI), 2000

**BSI.** *Draft BS EN 12697- 23 Bituminous mixtures – Test methods for hot mix asphalt Part 23. Determination of indirect tensile strength of bituminous specimen.* London; British Standards Institution (BSI), 2000.

**BSI.** *Draft BS EN 12697- 34 Bituminous mixtures – Test methods for hot mix asphalt Part 34.* Marshall test. London; British Standards Institution (BSI), 1999.

**BSI.** *Draft BS EN 13286-53 Unbound and hydraulically bound mixtures - Methods for making test specimens - Part 53: Making cylindrical specimens by axial compression.* London; British Standards Institution (BSI), 2000

**Broadhead, G.E. and Hills J.F.** Aggregates for Bituminous Materials. *In*: Pike, D.C. ed. *Standards for Aggregates*.1st Edition. Chichester: Ellis Horwood Limited, 1990, pp. 142 – 241.

**Brown, S.F.** *Stresses and deformations in flexible layered systems subjected to dynamic loads*. PhD thesis, University of Nottingham, 1967.

**Brown, S.F. and Brunton, J.M.** *An Introduction to the Analytical Design of Bituminous Pavements*. University of Nottingham, 1986.

**Brown, S.F. Brunton, J.M. and and Pell, P.S**. The development of analytical pavement design for British conditions. *Proceeding of the Fifth International Conference on the Structural Design of Asphalt Pavement*. August 23-26, 1982. Vol. 1. Pp. 3-16.

**Brown, S.F., Pell, P.S. and Stock, A.F.** Application of simplified, fundamental design procedures for flexible pavements. *Proceeding of the Fourth International Conference on the Structural Design of Asphalt Pavement*. August 22-26, 1977. Vol. 1. Pp. 327-352.

**Brown, S.F. and Scholz, T.V.** *Development of Laboratory Protocols for The Ageing of Asphalt Mixtures*. 2nd Eurasphalt & Eurobtume Congress, Barcelona 2000.

**Bullas, J.C. and West, G.** *Specifying Clean, Hard and Durable Aggregate for Bitumen Macadam Roadbase. Research Report 284*, Transport and Research Laboratory, Department of Transport, 1991.

**Button, J.W., Little, D.N., Jagadam, V. and Pendelton, O.J.,** Correlation of selected laboratory compaction methods with field compaction. *Transportation Research Record 1454*. Transportation Research Board, 1994, pp 193-201.

**Camplin, W. C**. *Coal-fired power stations. The radiological impact of effluent discharges to atmosphere*. Chilton, NRPB-R107, 1980.

**Cement Australia** Particle size distribution of cement. QCL Group Technical Note [Online]. Available at: http://www.cemaust.com.au/qcl/index.htm [3 May 2004]

**Cheung, L.W.** *Laboratory Assessment of Pavement Foundation Materials*. PhD Thesis, University of Nottingham, 1994

**Cioffi, R., Lavorgna, M. and Santoro, L.** Reuse of secondary lead smelter slag in the manufacture of concrete blocks. *In:* Woolley, G.R., Goumans, J.J.J.M. and Wainwright, P. J. eds. *Waste Materials in Construction*. 1st ed. Vol.1. Oxford: Elsevier Science Ltd., 2000, pp. 741-749.

**Collins, R.J. and Ciesielski, S.K**. *Recycling and Use of Waste Materials and By-Products in Highway Construction*. National Cooperative Highway Research Program Synthesis of Highway Practice 199, Transportation Research Board, Washington, DC, 1994.

***Cooper, K.E. and Brown, S.F.*** Development of a simple apparatus for the measurement of the mechanical properties of asphalt mixes. *Proc. Eurobitume Symposium*, Madrid, 1989, pp.494-498.

**Cooper K.E., Brown, S.F. and Pooley, G.R**. *The design of aggregate grading for asphalt basecourse*. Proc. AAPT. Vol. 54, pp 324 –346. 1985.

**Cooper, K.E., Brown, S.F., Preston, J.J.N, and Akeroyd.** F.M.L. *Development of practical method for the design of hot mix asphalt.* Transportation Research Record 1317. Transportation Research Board, pp. 42 – 51, 1992.

**Coventry S., Woolveridge, C. and Hillier, S.** *The reclaimed and recycled construction materials handbook*. Construction Industry Research and Information Association, (CIRIA), London, 1999.

**Croney, D and Croney, P.** *The design and Performance of Road Pavements*.McGraw-Hill. 2nd Edition, Maidenhead, 1991.

**Curtis, C.W., Ensley, K. and Epps, J.** *Fundamental properties of asphalt-aggregate interactions including adhesion and adsorption*. *SHRP-A-341*, Strategic Highway Research Program, national Research Council, Washington D.C. 1993.

**Das, B.M., Tarquin, A. J., and Jones, A. D**. Geotechnical Properties of a Copper Slag. *Transportation Research Record 941*, Transportation Research Board, Washington, DC, 1993

**Dawson, A.R.** *Water movement in road pavements.* 2nd Symposium on unbound aggregate in roads, UNBAR 2, 16-17 July 1985, Department of Civil Engineering, University of Nottingham, England.

**Dawson, A.R.** The degradation of furnace bottom ash under compaction. *In*: Jones, R.H. and Dawson, A.R eds. *Proceedings of the International Symposium on Unbound Aggregate in Roads*. UNBAR3, University of Nottinghsm: 1989, pp. 169-177.

**DETR** *Secondary and recycled aggregates uses in road construction under existing specifications*, Digest 101, 1999

**Elliot, R.C.** An assessment of some self and slowly cementing industrial by-products for use in roadbases. *In*: Unbound aggregate in roads. Dawson, A.R and Jones, R.H. eds. *The proceeding of the UNBAR4 symposium*, July 17 – 19, 1995. University of Nottingham. Nottingham, 1995.

**Emery, J.J.** *Slag Utilization in pavement construction*. Extending Aggregate Resources, ASTM STP774, American Society for Testing Materials, 1982, pp. 95-118

**European Commission**. *COST 337 Unbound Granular Material for Road Pavements.*

*Final Report for the Action*. Luxembourg, 2000.

**European Commission.** *Practical use of the concepts of clearance and exemption, Part II, Application of the concepts of exemption and clearance to natural radiation sources.* Radiation Protection 122, Luxembourg, 2002.

**Fallman A.M**. *Internatinal Seminar on Leach Test.* Swedish Geotechnical Institute, Swedish Research Council, Report No. 12, Stockholm, 1990.

**Feasby, D. G.** *Mineral Wastes as Railroad Ballast.* Canada Centre for Mineral and Energy Technology, National Mineral Research Program, Mineral Sciences Laboratories Report MRP/MSL 75-76 (OP), Ottawa, Canada, 1975.

**Fookes, P.G., Gourley, C.S. and Ohikera, C**., Rock weathering in engineering time. *Quarterly Journal of Engineering Geology*. Vol. 21, 1988.

**Fordyce, D.** The compaction of asphaltic mixture. *Performance related test procedures for bitumonous mixtures*, *In*:M.D. Gilchrist ans A.M. Hartman. Eds., Boole Press, Dublin, Ireland, 1997.

**Gandhi, P.M. and Lytton, R.L.** Evaluation of aggregates for acceptance in asphalt paving technologies. *Journal of the association of asphalt paving technologies*, Vol. 15, 1984.

**Garrity, L.V. and Krieg, H.F.** Studies of accelerated soundness tests. *Proceeding , Highway Research Board,* Vol. 15. 1935.

**Gibb, J.M**. *Evaluation of resistance to permanent deformation in the design of bituminous paving mixtures*. PhD thesis, University of Nottingham, Nottingham. 1996.

**Hasan, S., Meyer, A.H. and Fowler, D.W.** Establishments of acceptance limit the 4-

cycle MSS and modified wet ball mill tests for aggregates used in seal coats. *Research Report 12222-IF*, 1991.

**Hartman, A. M., Gilchrist, M.D., and Walsh, G.** Effect of mixture compaction on indirect tensile stiffness and fatigue. *Journal of Transportation Engineering*, 2001, 127(5), 370-378.

**Havey, M. et al.** *Radiation Protection-65, Principles and Methods for Establishing Concentrations and Quantities (Exemption values) Below which Reporting is not Required in the European Directive.* Luxembourg: Commission of the European Communities, 1993, (Doc.XI-028/93).

**Harvey, J. and Monismith, C.L.** Effects of laboratory specimen preparation variables on fatigue and permanent deformation test results using Strategic Highway Research Program A-003A proposed testing equipment. *Transport Research Record 1417*, 1993, pp. 38-48.

**Harvey, J., et al.** Effects of laboratory specimen preparation on aggregate-asphalt structure, air-void content measurement and repetitive simple shear test results. *Transport Research Record 1454*, 1994, pp. 113-122.

**Hicks, R.G. and Monismith, C.L.** 1971 Factors influencing the resilient response of granular materials. *Highway Research Record 345*, pp. 15-31.

**Highways Agency**. *Manual of Contract Document for Highway Works, Vol.1 Specification for highway works*. The Stationary Office, London. 1998

**Highways Agency**. *Design Manual for Roads and Bridges*, HD 35/95 HMSO, London: 2001

**Highways Agency**. *Design Manual for Roads and Bridges, Vol.7 Sect. 1 Part 1 HD*

*23/99 General Information*, HMSO, London: 1999.

**Highways Agency**. *Manual of Contract Document for Highways Works, Vol. 2, Specification for highway works, Series NG 900 Road pavements – bituminous bound materials*. HMSO, London: 2001

**Highways Agency**. *Manual of Contract Document for Highways Works, Vol. 1, Specification for highway works, Series 800 Road pavements – unbound, Hydraulically bound and other materials.* HMSO, London: 2001

**Highways Agency**. *Manual of Contract Document for Highways Works, Vol. 1, Specification for highway works, Series 1000 Road pavements – Concrete and Cement Bound Materials.* HMSO, London: 2002.

**Hill, A.R**. Understanding the leaching behaviour of slags, testing and interpretation. Manufacturing and Processing of Iron and Steel Slags. *Proceeding 3rd European Slag Conference*, 2nd – 4th October 2002, Kegworth. Euro slag Publication No. 2. 2003.

**Hill, A.R.** *Leaching of Alternative Pavement Materials.* Ph.D. thesis, University of Nottingham, 2004.

**Hosking, J.R. and Tubey, L.W**. *Research on low grade and unsound aggregate*. Crowthorne: Road Research Laboratory 1969, (Report LR 293)

**Hunt, L. and Boyle, G.E.** *Steel slag in hot mix asphalt concrete.* Oregon: Oregon Department of Transportation, 2000, (Final Report no. OR-RD-00-09).

**ITDG (Intermediate Technology Development Group).** *Testing methods for pozzolanas-Technical brief*. Intermediate Technology Development Group, [Online] Available at: http://www.itdg.org/docs/technical_information_service/pozzolanas_testing.pdf [6

August 2003]

**Kandhal, P. S., and Parker, F.J.** *Aggregate tests related to asphalt concrete performance in pavements*. Washington: Transport Research Board, 1998 (NCHRP Report 405).

**Kalyoncu, R.S.** *Slag – Iron and Steel*, U.S. Geological Survey Minerals Yearbook – 2000, pp.71.1-71.3

**Kennedy, J. and Hopkins, M.** *Cement-bound materials for pavements – material selection, mix design, construction and testing*. Crowthorne: British Cement Association, 2000.

**Kosson, D.S., van der Sloot, H.A. and Eighmy, T.T**. An approach for estimation of contaminant release during utilization and disposal of municipal waste combustion residues. *Journal of Hazardous Materials*, 1996, 47, 43-75.

.

**Lancaster, I.** Bitumens. *In*: Hunter, R.N. ed. *Asphalt in road construction*. 1st ed. Thomas Telford Publishing. London. 2000.

**Lee, A.R.** *Blastfurnace and steel slag – production, properties and uses.* 1st ed. London: Edward Arnold, 1974.

**Lemass, B.,** Slag solutions for heavy duty road pavements, *Proceeding 16th ARRB Conference, Part 2*, 1992

**Ligia, T.B., et al.** Distributed mass transfer rate for modelling the leaching of porous granular materials containing soluble pollutants. *Journal of Chemical Engineering Science,* 2000, 55, pp. 1257-1267.

**Lundy, J.R. and Sandoval-Gil, J.A.** *Permanent deformation characteristics of Oregon* *mixes using the asphalt pavement analyser*. Oregon: Oregon State University, 2004, (Final Report SPR 340).

**Malaysian Meteorological Services.** *Monthly Weather Bulletin* [Online]. Kuala Lumpur. Available at: http://www.kjc.gov.my/publication/mwb.html [21 December 2004].

**MSC.** Personal communication (2001)

**Marek, C.R.** Basic properties of aggregate. *In*: Barksdale, R. D., ed. *The Aggregate Handbook.* Arlington: National Stone, Sand and Gravel Association, USA, 2001, pp. 3-1 – 3-81.

**Markwick, A. H. D. and Shergold, F.A.** The aggregates crushing test. *Journal of the Institution of Civil Engineers*. 1945, 24(6) pp. 125-133.

**McCarthy, D.F.** *Essentials of soil mechanics and foundations – basic geotechnics*, 3rd Edition. New Jersey: Prentice Hall, 1988.

**Mineral Research and Recovery Inc**. [ONLINE].
Available at: http://www.mrrinc.com/ [30.10.2001]

**Muniandy, S.** University Putra Malaysia. Personal communication. 2004.

**Monismith C.L., Epps, J.A. and Finn, F.N**. Improved asphalt mix design. *Proceeding of Association of Asphalt Paving Technologist*, 11-13 February 1985. San Antonio, Texas, 1985. Vol. 54. pp. 340-406.

**Moutier, F., Duan, T.H. and Chauvin, J.J.** The effect of formulation parameters on the mechanical behaviour of mixes. *Association of Asphalt Technology (AAPT).* Volume 57, pp. 213 – 242 (1988).

**Motz, H.** Production and use of air-cooled blastfurnace and steel slag. *In:* Euroslag. *Manufacturing and Processing of Iron and Steel Slags.* Proceeding of 3rd European Slag Conference. Euroslag Publication No. 2, 2003, pp. 7 – 20.

**Motz, T. and Geiseler, J.** Products of steel slags an opportunity to save natural resources. *Waste Management*, 2001, 21(3), pp. 285-293.

**NCHRP.** *Laboratory Determination of Resilient Modulus for Flexible Pavement Design: Final Report (1997).* [Online].
Available at: http://www.nap.edu/books/nch014/html/index.html [28 October 2004]

**NCRP.** *Exposure of the population in the United States and Canada from natural background radiation.* NCRP Report No. 94. 1988

**NLA.** *Using lime for soil stabilization and modification*. National Lime Association, Arlington, Virginia. (2001) http://www.lime.org/soil2.pdf

**Noureldin, A.S. and McDaniel, R.S.** *Evaluation of Steel Slag Asphalt Surface Mixtures*, Presented at the 69th Annual Meeting, Transportation Research Board, Washington, January 1990.

**NSA.** *Report: NSA 181-14-Processed Blast Furnace Slag, The All Purpose Construction Aggregate*, National Slag Association, Alexandria, Virginia, 1985.

**Nunes, M. C.M.** *Enabling the use of alternative materials in road construction*. Ph.D. Thesis, University of Nottingham, 1997.

**Nunes, M.C.M., Bridges, M.G., and Dawson, A.R.** Assessment of secondary materials for pavement construction: Technical and environmental aspects. *In:* Cabrera, J.G. and

**Nunes, M.C.M., Dawson, A.R., Chaddock, B.C.J. and Atkinson, V.** *Enabling the use* *of secondary aggregate and binder in pavement foundations. Results of phase 1 study. PGR96006*. University of Nottingham, 1996.

**Nunn, M.E. and Smith, T.M.** Improvements to indirect tensile stiffness modulus test. *In*: Cabrera, J.G., ed. *Performance and durability of bituminous materials, Proceeding of 2nd European Symposium*, University of Leeds, April 1997. Leeds: pp. 411-426.

**Oxford University Press.** Ox*ford English Dictionary Online* [Online] Oxford University Press, 2002. Available at: http://dictionary.oed.com/entrance.dtl [3 October 2002]

**Pawluk, A.** *The construction & make-up of ancient Roman roads.* Available at: http://www.unc.edu/courses/rometech/public/frames/art_set.html [03 December 2004]

**Ramirez, T.L.**, *Steel slag aggregates in bituminous mixtures.* Final Report. Pennsylvania Department of Transport, Harrisburg, 1992.

**Read, J.M.** *Fatigue Cracking of Bituminous Paving Mixtures*. Ph.D. thesis, University of Nottingham, 1996

**Read, J.M and Whiteoak, D**. *The Shell Bitumen Handbook*, Fifth Edition. London: Thomas Telford, 2003.

**Reid, J.M. and Chandler, J.W.E.** *Recycling in transport infrastructure.* London: TRL Limited, 2001. [Contract reference 39/3/620cc1992]

**Reid, J.M. et al.** *ALT-MAT: Alternative materials in road construction*, Report No. WP6.TRL.002, Delivery D7 Final Draft Report for Publication, 2001. Available at: www.trl.co.uk/altmat [10 December 2001].

**Richardson, J.T.G. and Haynes, D.J.**, *Slag bound materials in composite roads.* Society of Chemical Industry, 2000. [Online].

Avaiablt at: http://www.soci.org/SCI/publications/2001/pdf/pb79.pdf [24 June 2002]

**Roberts, F.L., Kandhal, P.S., Brown, E.R., Lee, D.Y. and Kennedy, T.W.** *Hot Mix Asphalt Materials, Mixture Design, and Construction*. National Asphalt Pavement Association Education Foundation. Lanham, MD (1996).

**Robinson, H.L.** Slag bound material (SBM) – for flexible composite pavement design. *In*: Cabrera, J.G. and Zoorob, S.E., eds. *Proceeding of the third European Symposium on Performance and Durability of Bituminous Materials and Hydraulic Stabilised Composites*, Leeds, April 1999. Aedificatio, 1999, pp. 303 – 310.

**Robinson, H.L and Acock, P.A.B** Low energy materials for trench reinstatement. *In*: Cabrera, J.G. and Zoorob, S.E., eds. *Proceeding of the third European Symposium on Performance and Durability of Bituminous Materials and Hydraulic Stabilised Composites*, Leeds, April 1999. Aedificatio, 1999, pp. 465 – 476.

**Rockliff, D., Moffett, A. and Thomas, N.** Recent development in the use of steel (BOS) slag aggregates in asphalt mixtures in the UK. In: Zoorob, S.E., Collop, A.C. and Brown, S.F., eds. *Proceedings of the Fourth European Symposium on Performance of Bituminous and Hydraulic Materials in Pavements*, Nottingham, 11-12 April, 2002. A.A Balkema Publishers, The Netherlands, 2002, pp.251-260.

**Rogers, C.A., Bailey, M.L., and Price ,B**. Micro-Deval test for evaluating the quality of fine aggregate for concrete and asphalt *Transportation Research Record 1301*, 1991.

**Saeed, A., Hall, J. W., and Barker,W.** *Performance-related tests of aggregates for use in unbound pavement layers.* NCHRP Report 453. National Research Council. Washington D.C. National Academic Press, 2001.

**Scholz, T.V**. *Durability of bituminous paving mixtures*. PhD thesis University of Nottingham, 1995.

**Scott, J.S.** Dictionary of civil engineering. $4^{th}$ Edition. Penguin books. London. 1991

**Senior, S.A and Rogers, C.A**. Laboratory test for predicting coarse aggregate performance in Ontario. *Transportation Research Record 1301*, 1991.

**SETRA** .*La technique francaise des assisses de chaussees traitees aux liants hydrauliques et pouzzolaniques* . Service d'Etudes Techniques des Routes et Autoroutes et Laboratoire Central des Ponts et Chaussees, Ministere des Transports, France. 1980.

**Shell Bitumen.** *The shell bitumen handbook.* Surrey, Shell Bitumen UK. 1990.

**Sherwood, P.T.** *Alternative materials in road construction – A guide to use of waste, recycled materials and by-products*. First Ed. London: Thomas Telford Publication, 1995.

**Sherwood, P.T.** *Alternative materials in road construction – A guide to use of waste, recycled materials and by-products*. Second Ed. London: Thomas Telford Publication, 2001.

**Sousa, J.B., Harvey, J., Painter, L., Deacon, J.A., and Monismith, C.L.** *Evaluation of laboratory procedures for compacting asphalt-aggregate mixtures*. SHRP-A/UWP-91-523, Strategic Highway Research Program, National Research Council, Washington D.C. 1991.

**Sousa, J.B., Pais, J.C., Prates, M., Barros, R., Langlois, P. and Leclerc, A.M.** Effect of aggregate gradation on the fatigue life of asphalt concrete mixes. *Transport Research Record 1767*, TRB, National Research Council, Washington D.C., 1998, pp. 33-39 (1998)

**Suparma, L.B**. *The use of Recycled Waste Plastics in Bituminous Composites*, Ph.D. thesis, University of Leed, 2001.

**Terrel, S. and Al-Swailmi**. *Water sensitivity of asphalt-aggregate mixes: Test Selection. SHRP-A-403*. National Research Council. Washington DC. 1994

**Texas Department of Transport –** *TeDot experience* [Online]
Available at: http://www.dot.state.tx.us/GSD/recyle/xpercnst.htm [5 January 2003]

**TFHRC.** *User guidelines for waste and by product materials in pavement construction [Online*]. Federal Highway Administration, 2001. Available at:
http://www.tfhrc.gov/hnr20/recycle/waste/bfs1.htm
http://www.tfhrc.gov/hnr20/recycle/waste/bfs2.htm
http://www.tfhrc.gov/hnr20/recycle/waste/ssa2.htm
http://www.tfhrc.gov/hnr20/recycle/waste/ssa1.htm
http://www.tfhrc.gov///hnr20/recycle/waste/nfs4.htm
[29 November 2001]

**Thom, N.H. and Brown, S.F.** *The effect of grading and density on the mechanical properties of a crushed dolomitic limestone*. Proc. 14$^{th}$ ARRB Conf. Part 7, pp. 94-100. 1988.

**Thomas Research**. *Instability mechanisms in iron and steel industry slags.* [Online]. Avaiable at: http://www.thomasresearch.freeuk.com/instability.html [23 September 2002]

**Thomson, M.R.** Aggregate as a structural product. *In*: Barksdale, R.D. ed. *The aggregate handbook*. Washington D.C.: National Stone Association. 2001

**Tian, P., Zaman, M.M., and Laguros**, J.G. Gradation and moisture effects on resilient modulus of aggregate bases. *Transportation Research Board*, No. 1619 pp. 75-85, 1998.

**UNSCEAR.** *Sources, effects and risks of ionizing radiation*. New York, United Nations, Report to the General Assembly with annexes. 1982.

**Uzan, J.** Permanent deformation in flexible pavements. *Journal of Transportation Engineering*. American Society of Civil Engineering (ASCE), Vol. 130, Issue 1, pp. 6-13 (2004).

**Van de Sloot, H.A.** Developments in evaluating environmental impact from utilization of bulk inert wastes using laboratory leaching tests and field verification. *In*: Cabrera, J.G. and Woolley, G. R., eds*. Bulk "Inert" Waste: An Opportunity to Use. Waste Management. Special issue*. Vol. 16. No. 1-3. Pp. 65 – 81, Leeds. 1996.

**Van de Sloot, H.A**. Systematic leaching behaviour of trace elements from construction materials and waste materials. *In:* Goumans, J.J.J.M, Van Der Sloot, H.H. and Aalbers, Th.G., eds. *Waste materials in construction. Proceedings of the international conference on environmental implications of construction with waste materials*, Maastricht, The Netherland, 10-14 November 1991.

**Van de Loo, P.J.** The creep test: A key tool in asphalt mix design and in the prediction of pavement rutting. *Proceeding of the Association of Asphalt Paving Technologist*, 1978, Vol. 47, pp. 522-555.

**Verhasselt, A. and Choquet, F**. Steel slags as unbound aggregate in road construction: problems and recommendations. *In:* JONES, R.H. and DAWSON A.R., eds. *Unbound aggregates in roads.* Nottingham: Buttterworth, 1989, pp. 204-211.

**Walsh, I. D.,** The Evaluation and use of slag bound sub-base/roadbase with performance related tests. *In*: Cabrera, J.G. and Zoorob, S.E., eds. *Proceeding of the third European Symposium on Performance and Durability of Bituminous Materials and Hydraulic Stabilised Composites*, Leeds, April 1999. Aedificatio, 1999, pp. 327 – 342.

**Walsh, I.D. and Williams, G**., Use of ash bound material as road base on A259 Ramsgate Harbour approach road. *In*: Zoorob, S.E., Collop, A.C. and Brown, S.F., eds. *Proceeding of the Fourth European Symposium on Performance of Bituminous and*

*Hydraulic Materials in Pavements, BITMAT 4*, Nottingham, 11-12 April 2002, pp.175-180.

**Western Pozzolan Corp.** *Overview* [Online].
Available at: http://www.westernpozzolancorp.com/overview.htm [13 May 2003]

**Whiteoak, D.** The Bitumen Handbook First edition, Surrey, Shell Bitumen UK, 1990.

**WHO (World Health Organisation).** *WHO Guidelines for drinking water – water quality*, Third Edition. Vol.1, Geneva, 2004. [Online]
Available at: http://www.who.int/water_sanitation_health/dwq/gdwq3/en/ [11 November 2004]

**Williams, R. I. T.** Cement Stabilised Materials. IN: Pell, P.S. ed. *Developments in highway pavement engineering-1. Vol. 1*. Essex: Applied Science Publishers Ltd, 1978, pp.163-217.

**Woodward, D.** Assessing the aggregates for use in asphalts. *In:* Hunter, R.N. ed. *Asphalts in road construction*. 1st ed. London, 2000, Thomas Telford Limited, 2000, pp. 1-44.

**Woolley, G. R., eds**. Bulk "Inert" Waste: An Opportunity to Use. *Waste Management. Special issue*, 1996, 16(1-3), pp. 87-96.

**Wright, P.H.** *Highway Engineering*. 7th.ed. Hoboken, N.J.: John Wiley & Sons, 2004.

**Wu, Y., Parker, F. and Kandhal, K.** *Aggregate toughness/abrasion resistance and durability/soundness tests related to asphalt concrete performance in pavements*. NCAT Report No. 98-4. National Center for asphalt Technology, Auburn University, Alabama, 1998.

**Xie, H. and Lekarp, F**. *The influence of moisture content on mechanical performance* *of unbound granular materials* – A literature review. Arbetsrapport Trita-vt ar 01:01, 2001.

**Zoorob, S.E**. Design and construction of hot-mix bituminous surfacing and roadbases. *In*: O'Flaherty, C.A., ed. *Highways-The location, design, construction and maintenance of road pavements*. 4th ed. Oxford, 2002, Butterworth-Heinemann, 2002, pp.325-361