Department of Engineering

Incinerator Bottom Ash Aggregate in Asphalt for Roads: An Investigation into Environmental and Mechanical Characteristics

Thesis submitted in accordance with the requirements of

University of Liverpool for the degree of Doctor in Philosophy

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B.Sc. (Hons), M.Sc.

April 2009

ABSTRACT

This thesis describes an investigation into the feasibility and effects of using Incinerator Bottom Ash Aggregate (IBAA) in asphalt for roads. Incorporation of IBAA into bituminous mixtures suitable for binder course layers was investigated. Furthermore, the effect of such incorporation on the mechanical and environmental properties of the developed mixtures was studied.

Incineration of waste in energy from waste plants results in few by-products. One of the most important by-products is bottom ash which could be processed to produce IBAA. IBAA used to be land-filled. However, due to legislation restrictions, it became very important to find different utilisations for it. One of the promising areas is incorporating IBAA into bituminous mixtures.

A laboratory investigation was undertaken to develop rational IBAA blends which meet the UK grading requirements for binder course layers. Blends contain 0, 30, 60, and 80% IBAA content levels were developed and their volumetric properties were studied. Bituminous mixtures, based on the developed blends, were designed adopting the volumetric mix design approach. The effect of IBAA content on the durability of mixtures, in terms of moisture ingress and ageing resistance, was evaluated.

As IBAA contains some hazardous components, it was necessary to undertake a laboratory investigation into the leaching potential of the developed mixtures. Moreover, the effect of bitumen binding on IBAA leaching potential was tested. Two types of leaching tests were used: agitated extraction and tank tests. The time dependant leaching behaviour in terms of the cumulative release of sulphate, chloride, sodium, and potassium was assessed, for 80% IBAA bituminous mixture. In addition, a diffusion model was applied to the results to predict the leaching potential.

Permanent deformation is one of the major distress modes of asphalt failure. The steady state permanent deformation behaviour of binders integrated in bituminous

mixtures plays an important role in mixtures' permanent deformation behaviour. A Dynamic Shear Rheometer (DSR) was used to study the effect of adding IBAA on the steady state permanent deformation behaviour of binders. The DSR constant strain rate and creep testing modes were used over a wide range of temperatures, strain rates, and stresses to identify the steady state permanent deformation behaviour of three binders. Pure bitumen and two mastics were tested. These mastics are the respective binders for the developed IBAA bituminous mixtures. A Modified Cross Model (MCM) was used successfully to model the results.

Uniaxial constant strain rate and creep tests were undertaken to study the steady state permanent deformation behaviour of the developed IBAA bituminous mixtures. The axial, radial and volumetric deformations were investigated. Moreover, the dilation of asphalt was explored. Furthermore, triaxial constant strain rate tests were undertaken and the effect of confining pressures on the permanent deformation of IBAA mixtures was investigated. A MCM was used successfully to model the results. In addition, the effect of IBAA content level on this behaviour was considered.

Asphalt cracking is as one of the major distress modes in asphalt pavement. Linear Elastic Fracture Mechanics approach was used to evaluate crack resistance of the tested mixtures aiming to investigate the effect of IBAA content on these properties. Fracture toughness, fracture energy and fracture resistance of these mixtures were determined using monotonic mode loading tests. Cyclic mode loading tests were undertaken to monitor crack propagation. Crack propagation was modelled using the stress intensity factor and J-Integral to obtain Paris Law constants. Moreover, a new approach was used to obtain damage parameters of bituminous mixtures using fracture tests instead of fatigue tests.

The implication of this work is that IBAA should be considered as cheap alternative for natural aggregate in hot mix bituminous mixtures without significant concerns with regard to the environmental and mechanical characteristics covered in this study.

ACKNOWLEDGMENT

The project was suggested and supervised by **Dr Hussain Khalid**. I am very indebted to him for his invaluable advices and encouragement throughout this research. I learned so much from him; his door was always open for me. I consider myself fortunate to have had him as my advisor. Also, I would also like to thank Dr Ignacio Artamendi for his many helpful discussions and recommendations. Moreover, I'd like to thank the University of Liverpool pavement group members and the Engineering Department Laboratory technical staff for their assistance and advice with the experimental work.

I'm very grateful to the Egyptian government for the award of a study scholarship to pursue this research. This project was also supported by three industrial partners: Ballast Phoenix Ltd, Tarmac Ltd and Nynas Bitymen to whom I am very much indebted.

I would like to express my gratitude to all persons and organisations that contributed to this work with their support, encouragement and advice. In particular, Mr George Overfield, Dr Nizar Ghazireh, Dr Howard Robinson, Mr Jukka Laitinen, Mr Steve Armstrong, Mrs Patricia Hough, Dr Jackie Taylor, Mr John Wilkinson, and Dr Muhamed Hayuti. Moreover, let me thank all those who have helped in some way but have not been mentioned above by name.

Special thanks are due to my supportive and infinitely patient wife, **Wafaa**, who has continually flooded me with her endless love and support during the compilation of this work. I'd like also to thank my kids, **Nour** and **Hussain**, who provided a much needed diversion which ensured that I always had something else to think about. Moreover, I can not forget **my father** and **my mother** to whom I'm very much indebted and grateful. They are the main reason beyond all my achievements. Thanks are also due to the rest of my family for their interest and support.

Before all and after all, praise to Allah from him we gain knowledge.

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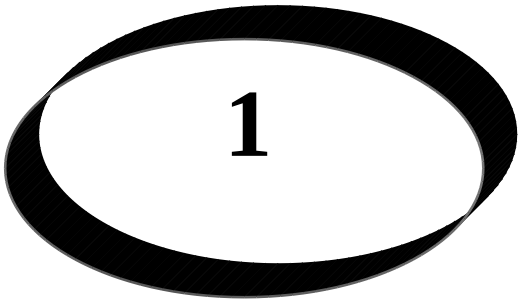


INTRODUCTION

1.1 RESEARCH PROBLEM

The United Kingdom is well supplied with rock suitable for quarrying to produce high-quality aggregate for pavement constructions. However, extraction is becoming increasingly difficult because of environmental and public constraints. Thus, it is expected that engineers should turn to other sources for aggregate supplies. In THE UK, the majority of the demand for aggregates is met by natural aggregates with only about 25% being supplied by aggregates from alternative sources (UKQAA 2008). The majority of alternative aggregates are derived from recycled materials, mainly construction and demolition waste, whereas lightweight aggregates manufactured from industrial by-products have only a minor share of the alternative aggregates market. This minor share needs to be grown as an effective tool to reduce natural aggregates dependency.

Moreover, the 1999 European Union Landfill Directive, which has brought legislation into force in the UK with effect from July 2004, ensures more responsible waste disposal and increased use of recycling and composting. The Directive marks a huge change in the regulation of the waste industry but also puts pressure on society to take

responsibility for its waste. The Directive requires at least 40% of household waste to be recycled by 2010 and a minimum of 15% to be composted. This is equivalent to 80% of the UK population having to recycle 80% of their waste by 2010.

Co-disposal of hazardous and non-hazardous waste in landfill sites was banned in the UK from July 2004. The 10-fold drop in the number of hazardous landfill sites has led to this ban across England and Wales. In 2004, in England and Wales, the total number of public landfill capacity was 182 sites; this was dropped to 14 sites with effect from July 2004. This means that waste has to be transported long distances for disposal. The cost of waste disposal is set to increase significantly, with the cost of landfill forecast to double as the space available in existing sites is used up. Incineration of some waste is the key method to resolve the disposal of municipal waste. This incineration, in energy from waste plants, results in few by-products.

One of such by-products is Incinerator Bottom Ash Aggregate, known as IBAA. IBAA is classified in most countries as inert. Currently in THE UK, the annual production of IBAA is about 1.0 million tonne. Various researchers and highway agencies have used IBAA; however, larger and wider uses are still needed. Dwindling supplies and rising costs of natural resources used in road construction as well as concerns over shrinking landfill spaces prompt researchers to investigate the feasibility of using waste by-products, such as IBAA, as substitute materials in highway constructions. This research is concerned with studying the feasibility of using IBAA in bituminous mixtures for flexible pavement.

1.2 SCOPE OF THE RESEARCH

Numerous waste materials result from manufacturing operations, service industries, sewage treatment plants, households and mining. Legislation has been enacted by several agencies in recent years to either mandate the use of some waste materials or to examine the feasibility of such usage. The asphalt industry has been pressured in recent years to incorporate a wide variety of waste materials into pavements. This has raised the following legitimate concerns, as shown in Fig 1-1:

- Engineering concerns such as effects on the engineering properties (e.g., strength and durability), impact on production, and future recyclability.
- Environmental concerns such as emissions, fumes, leaching and handling and processing procedures.
- Economic concerns such as life cycle costs, salvage value, and lack of monetary incentives.

In this research both engineering and environmental issues, related to usage of IBAA in bituminous mixtures, are considered. With regard to engineering concerns, aspects including incorporating IBAA in hot and cold bituminous mixtures, moisture ingress effect, ageing effect, permanent deformation behaviour, and fracture and crack propagation properties were studied. With regard to environmental concerns, the leaching potential of IBAA bituminous mixtures is set as one of the main aims of this research area as it was shown in the literature that leaching is the main environmental concern related to IBAA. Economic concerns are beyond the scope of this research.

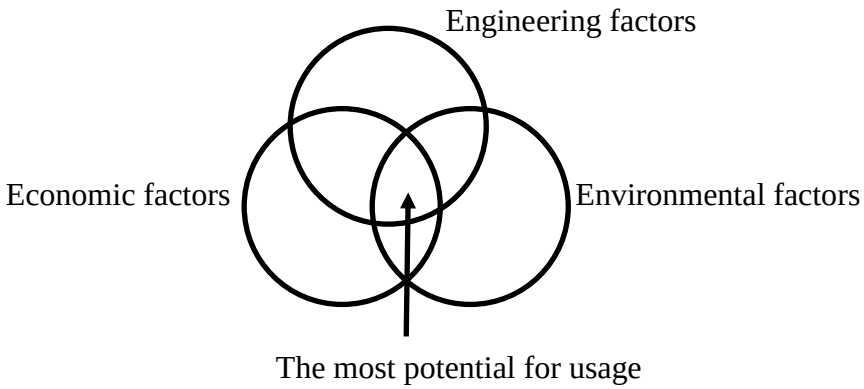


Figure 1-1: Legitimate concerns of IBAA utilisation.

1.3 RESEARCH OBJECTIVES

Specific objectives of the research can be summarised as follows:

- To assemble worldwide literature review on the use of IBAA as a construction material, with special emphasis on its use in asphalt, including environmental criteria.
- To develop rational aggregate blends comprising different IBAA content levels from two sources, and limestone, suitable for binder course layers in flexible pavements.
- To adopt a mix design procedure based on the aggregates' internal void structure and aiming to optimise performance related properties, for bituminous mixtures, containing high content levels of IBAA.
- To evaluate the effect of IBAA content level on the moisture sensitivity and ageing effect of the developed mixtures.
- To study the leaching properties of selected aggregate blends containing different IBAA contents, before and after coating with bitumen in bituminous mixtures.
- To undertake a fundamental study of steady state permanent deformation behaviour of selected bituminous mixtures including an investigation on the effect of IBAA on binder permanent deformation properties.
- To adopt fracture mechanics analysis to characterise fracture and crack propagation properties of bituminous mixtures containing IBAA.

1.4 BENEFITS TO INDUSTRY

The outcome of the research may enhance the level of confidence of the paving industry in these relatively novel materials, which in turn could help to reduce the problems of waste disposal and natural aggregate consuming. The benefits accrued from the success of this research are summarised as follows:

- Enhanced use of IBAA in higher value applications than current practice.
- Reduction in the quantity of ash to be land-filled so reducing the load on landfill sites.
- Recovery of additional value from waste.

- Replacement of virgin aggregate with a material which is otherwise a waste.
- Reduced traffic requirements for urban road construction as travel distances for aggregate will in general be reduced thus reducing the pollution load.
- Increased use of recycled and secondary materials.
- Economic gains to the industry generating the by-products as they can sell IBAA instead of paying for landfill it.
- Enhance the knowledge of binding effect on leaching potential of IBAA.
- Enhance the knowledge of permanent deformation and fracture and crack propagation behaviour of bituminous mixtures containing secondary aggregates.

Against these positive benefits, pavement engineers must acknowledge the uncertainties in behaviour of the replacement aggregate and their environmental effects. Currently, there is little experience in the use of by-product aggregate. Thus, extrapolation from conventional to by-product aggregate behaviour may appear imprudent.

1.5 THESIS ORGANISATION

1.5.1 Chapter 2: Incinerator Bottom Ash Aggregate

This chapter introduces the problems associated with waste arisings, management and incineration and, consequently, IBAA production. Particular attention was given to the physical, mineralogical and chemical properties of IBAA. Furthermore, the chapter provides background information about the use of IBAA in civil engineering applications with special concern with road constructions.

1.5.2 Chapter 3: Mix design

In this chapter, the materials used in the research were identified. Then, an approach to develop rational aggregate blends containing high content level of IBAA was presented. Selected aggregate blends were designed, for bituminous mixtures suitable for binder course in UK flexible pavement layers, using the volumetric mix design

approach. The mix design included a conventional and three IBAA mixtures. The designed mixtures were studied for the effect of moisture ingress and ageing. The results were included in this chapter.

1.5.3 Chapter 4: Environmental aspects

This chapter includes a background on leaching process, mechanisms, factors, parameters, and tests. Furthermore, the results of two types of leaching tests were included. An agitated extraction and a tank tests were undertaken on selected bituminous mixtures containing IBAA and their respective non-bituminous blends. The results were presented and modelled.

1.5.4 Chapter 5: Rheology

This chapter presents a review on deformation behaviour of bitumen and the results of an investigation into the effect of IBAA on binders' permanent deformation behaviour. A Dynamic Shear Rheometer (DSR) was used to study the steady state permanent deformation behaviour of pure bitumen and two mastics including a mastic incorporates IBAA. Two different DSR modes were used: constant strain rate and creep modes. The results were modelled using a Modified Cross Model (MCM).

1.5.5 Chapter 6: Permanent deformation behaviour – Uniaxial tests

In this chapter, the steady state permanent deformation behaviour of bituminous mixtures containing IBAA was investigated. Work undertaken on idealised and conventional bituminous mixtures was reviewed. Furthermore, uniaxial constant strain rate and creep test results were presented. These results included axial and radial deformation behaviour. Moreover, the volumetric deformation and dilation behaviour were also presented. The results were modelled using MCM. Finally, the effect of IBAA content level on this behaviour was discussed.

1.5.6 Chapter 7: Permanent deformation behaviour – Triaxial tests

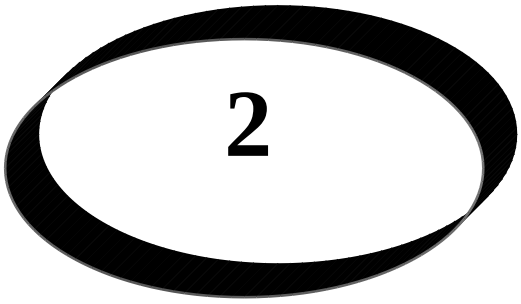
This chapter included the results obtained from triaxial constant strain rate tests. The results included axial, radial, volumetric and dilation behaviour. Again, the MCM was used to model the results.

1.5.7 Chapter 8: Fracture and crack propagation

In this chapter, work undertaken on using fracture mechanics principles to study fracture and crack propagation properties of IBAA bituminous mixtures was presented. Moreover, the results obtained from monotonic and cyclic Semi Circular Bending (SCB) tests were presented. The effect of IBAA content level on fracture strength and Paris Law constants was investigated.

1.5.8 Chapter 9: Conclusions and recommendations

This chapter summarises the findings of the research project; and suggests further areas to be investigated in the topic of IBAA bituminous mixtures.



INCINERATOR BOTTOM ASH AGGREGATE

2.1 INTRODUCTION

When Municipal Solid Waste (MSW) is incinerated in Energy from Waste (EfW) plants, approximately 25% by weight (Zeng and Ksiabati 2003) and 10% by volume (Izquierdo et al 2008) of the waste burnt remains as an inert gravely like ash known as Incinerator Bottom Ash (IBA) or simply Bottom Ash (BA). In 1997, the incineration of MSW resulted in an annual production of 2.5 million tonnes of IBA in France (Bruder-Hubscher et al 2001); 3.0 million tonnes in Germany in 1999 (Vrancken et al 2000); and 1.2 million tonnes in the Netherlands in 2004 (Dijkstra 2007). In the UK, 1.0 million tonnes are produced annually (UKQAA 2008). This ash has traditionally been land-filled but in recent years it has been increasingly further processed and used as a secondary aggregate. Highway agencies throughout the world, especially in the

UK, face the challenge of safe usage of various waste products in pavements and earth structures. Various researchers and highway agencies have used Incinerator Bottom Ash Aggregate (IBAA); however, larger and wider uses are still needed. Dwindling supplies and rising costs of natural resources used in road construction as well as concerns over shrinking landfill spaces have prompted researchers to investigate the feasibility of using waste by-products, such as IBAA, as substitute materials in highways.

In this chapter a brief overview of municipal solid waste is given; its definition, quantities, management, and incineration plants. This is followed by a review of using IBAA in roads. Special emphasis is given to bituminous applications. In addition, the environmental aspects related to such usage are discussed.

2.2 MUNICIPAL SOLID WASTE

2.2.1 Definition

Municipal Solid Waste (MSW) is defined in the UK as "household waste and any other wastes collected by a Waste Collection Authority, or its agents, such as municipal parks and gardens waste, beach cleansing waste, commercial or industrial waste and waste resulting from the clearance of fly-tipped materials" (defra 2008). It is also defined in the USA as "any garbage, refuse, sludge from a waste treatment plant, water supply treatment plant, or air pollution control facility and other discarded material, including solid, liquid, semisolid, or contained gaseous material resulting from industrial commercial mining, and agricultural activities, and from community activities" (US Environmental Protection Agency 1992). The most popular definition is presented as "the solid waste generated at residence, commercial establishments and institutions, but not including construction/demolition debris, automobile scrap or medical/pathological waste" (IAWG 1997).

2.2.2 MSW arising and management

2.2.2.1 MSW arisings

MSW is heterogeneous materials comprise several components. Paper and card usually presents the higher percentage component. Percentages of the other components may vary from country to country or even from region to region. This variation depends on many factors which are very difficult to identify. These factors include human, cultural, environmental, economic, and municipal factors. Generally, these components include plastics, metals, glass, wood, dust, textiles, misc inorganic matters, organics, household hazardous waste, household biomedical, and others. The size of these components varies from fine dust to large bulky items.

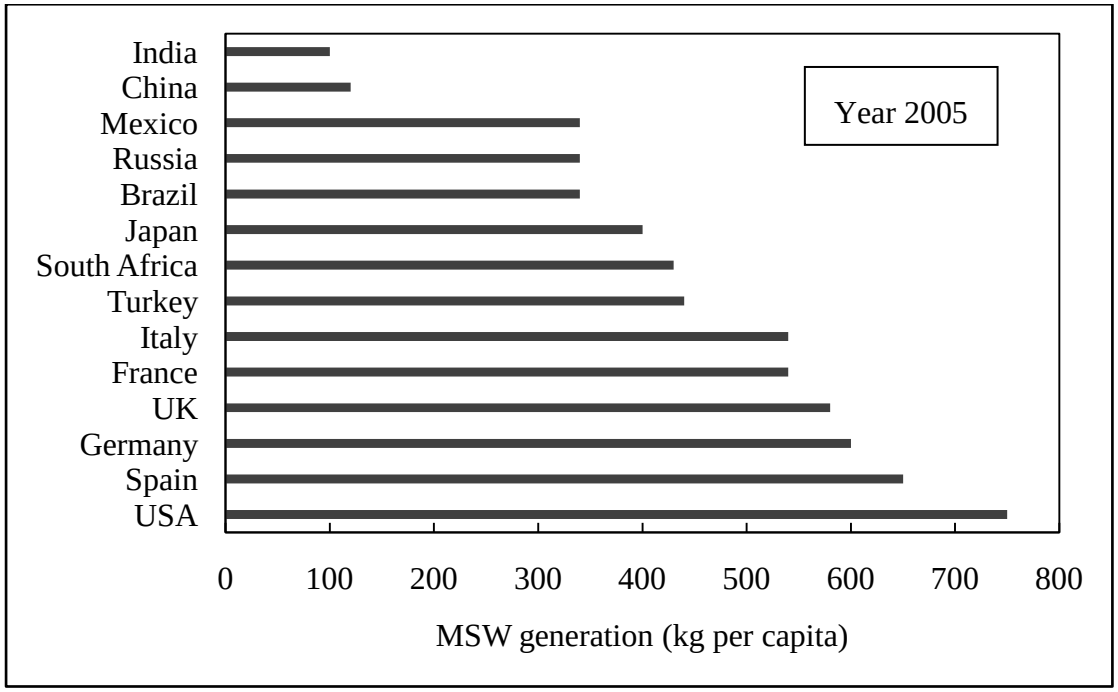


Figure 2-1: MSW generation worldwide (OECD Factbook 2008).

Like its composition, the quantity of MSW generated differs from a country to another. This difference may be attributed to factors such as rate of urbanisation, the types and patterns of consumption and household revenue and lifestyles. Fig 2-1 shows the variation of MSW generation in different countries as recorded in the year 2005. From this figure, it is clear that rich countries, except Japan, produce more waste. However, the total MSW quantity which is produced annually did not always

increase with the increase in population. Fig 2-2 shows the development in MSW arisings for different countries during the period 1980-2005. It is obvious that some countries managed to reduce the MSW arising in 2005 comparing with the year 2000. Other countries exhibited a general increase in MSW quantity.

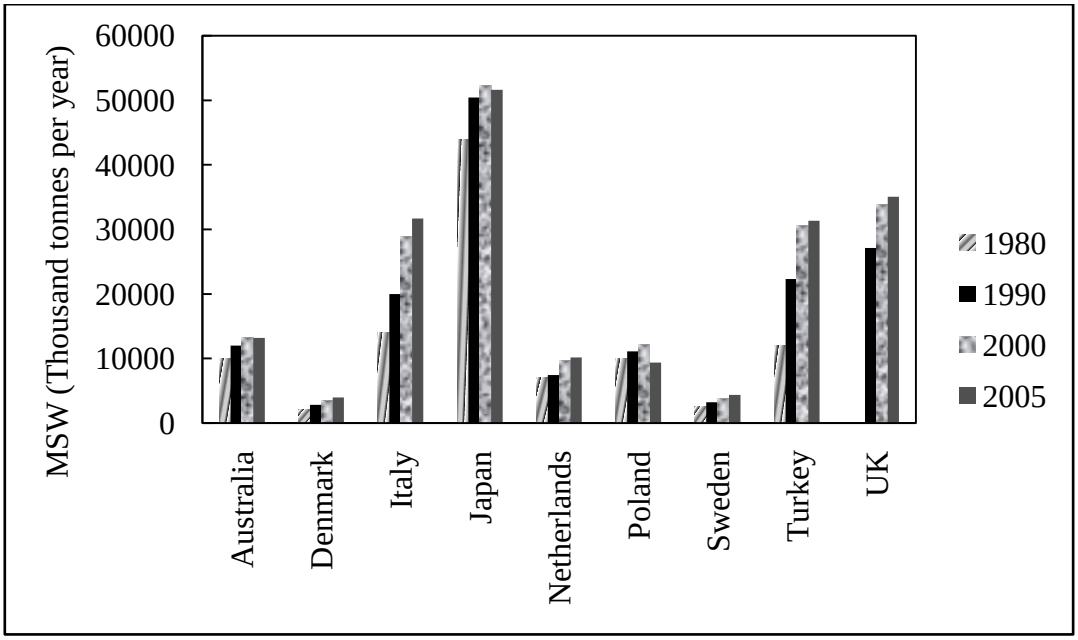


Figure 2-2: MSW development worldwide (OECD Factbook 2008).

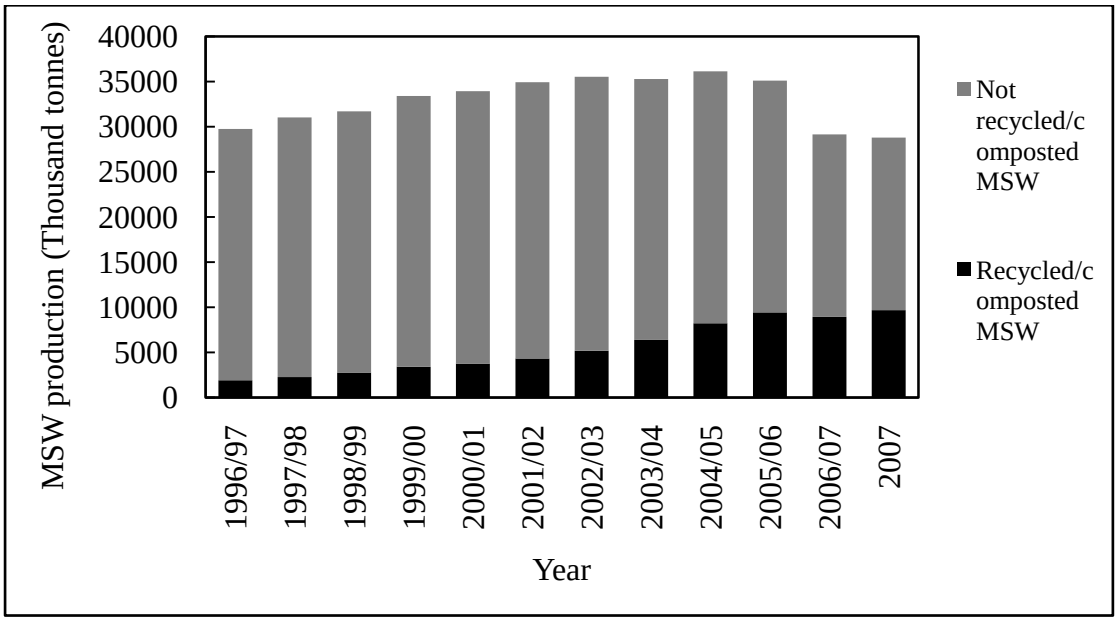


Figure 2-3: MSW arisings in 1996 – 2007 in the UK (defra 2008).

If UK is considered as an example it is shown from Fig 2-2 that generally there is an increase in the MSW arisings from 1980 to 2005. Fig 2-3 shows details for MSW arisings from 1996 to 2007. This figure shows a slight decrease in MSW arisings in the last two years. This decrease may be attributed to the increase in recycling rate accompanied with a decrease in the total household waste production (defra 2008). However, it is expected that this decrease will turn into another increase. This expectation is based on the general trends of MSW arisings observed in Figs 2-2 and 2-3. Moreover, the lack of legislations which controls MSW arisings may support this expectation not only in the UK but also in Europe.

2.2.2.2 MSW management

MSW management strategies include three main options: disposal to landfill, reuse, or recovery through recycling, composting or incineration for energy. Direct landfilling usually is the most utilised means of disposal. However, due to legislation restrictions, especially in Europe, the available landfill sites decreases. This decrease causes an increase in the quantity of MSW which needs to be recycled or incinerated. In 1999, the European Union Landfill Directive (EU 1991/31/EC) was adopted. The Directive aims to improve standards of landfilling across Europe, through setting specific requirements for the design, operation and aftercare of landfills, and for the types of waste that can be accepted in landfills. The Directive requires at least 40% of household waste to be recycled by 2010 and a minimum of 15% to be composted.

In the UK and in 2007, as shown in Fig 2-4, 55% of MSW was landfilled compared with 34% recycled and 11% incinerated. In 2000, the incinerated portion was only 9%. However, due to landfill shrinkage, it is expected that this figure will increase in the following years. Consequently the available quantity of IBAA, as incineration by-product, is expected to increase. Obviously, there is a need to develop novel reuse applications for IBAA as well as to expand the current usage levels.

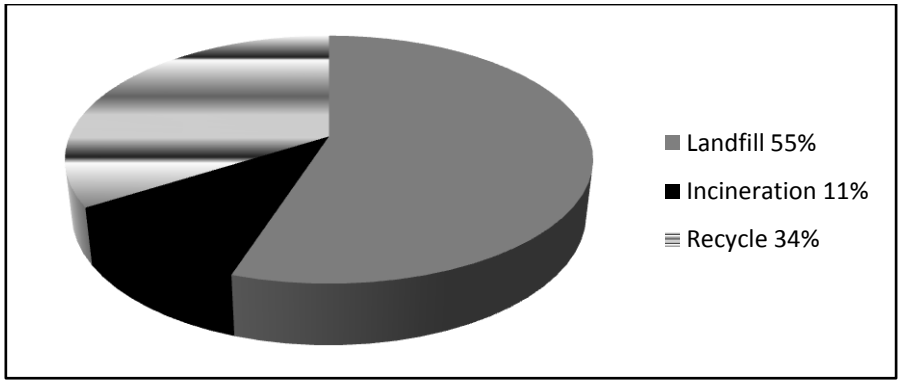


Figure 2-4: MSW management in 2007 in the UK (defra 2008).

2.2.3 MSW incineration

The main target of MSW incineration is reducing the amount of space required for disposal of waste in landfills. Moreover, it is a tool to generate energy which can be used for beneficial purposes e.g. electricity generation. There are various systems available for the incineration of MSW and the generation of usable energy, however these systems can be divided into three main categories:

- Mass burning: the as-received MSW is fed directly into the furnace and burned on a grate or fireplace without any pre-treatment such as size reduction, shredding or material separation prior to burning (Rand et al 2000).
- Refuse derived fuel: a fuel of a more homogenous nature is prepared on-site and either burned in a dedicated furnace at the same location or sold outside to customers who utilise the fuel in their furnaces (Williams 2005).
- Fluidized bed combustion: it burns refuse derived fuel injected into a hot fluidized bed of non-combustible granular material. Sometimes this system is considered as a refuse derived fuel system (Oka 2003).

Mass burning technologies were developed in Europe at the turn of the previous century and have undergone substantial advancement over the past 20 years. A typical mass burn facility is shown schematically in Fig 2-5. By installing advanced combustion control systems, altering the configuration of the furnace and the location of the air injection ports, the combustion performance of these systems has been greatly improved. These improvements have lowered emissions of trace organics and

raised the thermal efficiency of the furnaces. In addition, some facilities now process waste (e.g. by mixing) before feeding it to a mass burn plant and consider the extra cost to be warranted because the system runs more smoothly. In this system a typical MSW incinerator facilities may contain several process sections:

- A waste receiving and storage area.
- A waste feed system to charge the incinerator.
- A combustion system.
- A boiler to convert the heat of combustion to usable energy.
- An air pollution control system.
- An ash handling system.

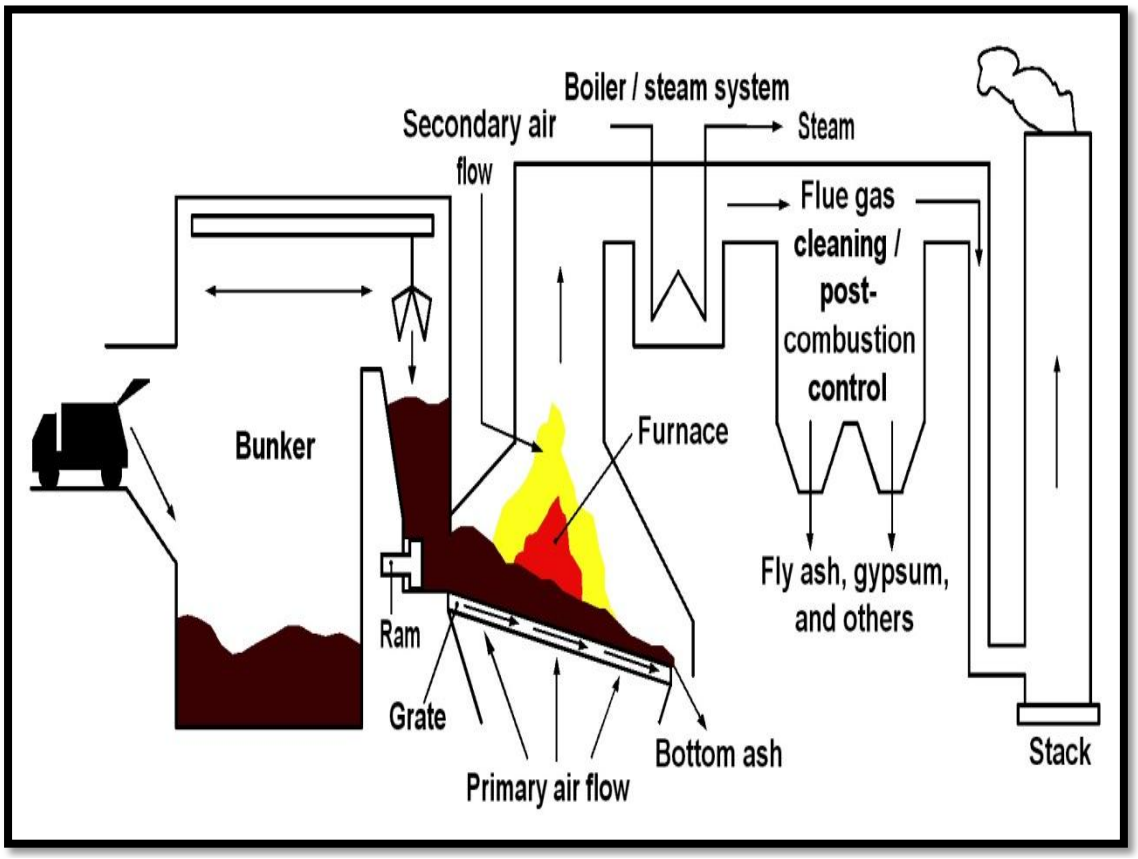


Figure 2-5: Schematic diagram for a typical EFW plant (Anon 2008).

The incineration process not only transforms MSW into energy but also yields residues. These residues can be broadly classified as fly ash, combined with air pollution control (APC) residues, and incinerator bottom ash. The former is the solid

light residues derived from the combustion chamber and are collected from the top of the furnace. This fly ash has important boundaries for utilisation in the civil engineering applications thanks to the high content of potentially hazardous elements (Izquierdo et al 2002) and its fine particle size. IBA is the most significant by-product of the incineration process. It comprises 25% by weight (Zeng and Ksiabati 2003) and 10% by volume (Izquierdo et al 2008) of the waste burnt.

Incinerator bottom ash is collected at the grates of furnaces and removed in a manner that minimise the ingress of air. Usually, a column of water is sealing on the furnace. This water serves to extinguish any remaining combustibles and to cool the IBA. Furthermore, the quenching process reduces large sizes of ash. After being discharged from a quench tank IBA is moved to an ash storage bunker where further dewatering takes place.

Incinerator bottom ash is moved to processing plants. Usually, this step takes place when IBA is still wet to minimize dust emissions. Inside the processing plant, IBA is usually stockpiled outdoors, for three months, as shown in Fig 2-6. This natural weathering is the most cost-effective method of IBA treatment, since it results in the chemical stability of the bottom ash (Wiles 1996). One-to-three month exposure to natural weathering is enough to reduce the release of heavy metals from the residue, promote carbonation, reduce pH, and allow enough time for stabilisation reactions (Chimenos et al 2000). In this period, some of the chemical and mineralogical characteristics of the material undergo significant changes including: oxidation of some metals (e.g. aluminium, iron, copper), dissolution and precipitation of the hydroxides and salts of the main cations, carbonation, and neutralisation of pH and neo-formation of clay-like minerals from glass (Wiles 1996, Meima and Comans 1997, 1999).

After weathering, the raw IBA is put through a process that removes ferrous and non-ferrous metals, organic matter, paper, and the small percentage of raw material that is unusable (York 2000). The remaining material is screened into varying size fractions

of Incinerator Bottom Ash Aggregate (IBAA). A typical IBA processing plant is shown in Fig 2-7.

Figure 2-6: IBA stockpile before processing (Billingham, Teesside, UK).

Figure 2-7: A processing plant to produce IBAA (Billingham, Teesside, UK).

2.3 GENERAL PROPERTIES OF IBAA

IBAA has a lot of definitions which varies from agency to other. The most utilised definition is "It comprises heterogeneous material discharged from the burning grate of the incinerator and material that falls through the burning grate to be collected in hoppers below the furnace" (IAWG 1997). This definition matches with the properties and characteristics of IBAA used in this study.

2.3.1 Physical properties

Visual classification of IBAA fractions, as seen in Fig 2-8, shows presence of metals, slag material, stones, ceramic, glass, and organic material. After any large items are removed, it has the appearance of porous, greyish, silty sand and gravel. IBAA has moisture content frequently dependent upon the type of quenching that is utilise to cool the IBA (Mackay et al 1992). Some incineration facilities use spraying devices to quench the BA and this tends to reduce moisture content, while others use quench tanks with drag chains which increase the moisture content (IAWG 1997). However, no significant differences were found in the literature between the properties of the produced IBAA using either of the two methods. IBAA is a highly porous aggregate, which enhances its tendency to absorb water. The water absorption is also a useful predictor of the potential use for IBAA to absorb bitumen during the manufacturing of bituminous mixtures (Nunnes et al 1996). This property was noticed and supported by the findings of this research as will be shown in Chapter 3. The Young's modulus, as a function of Proctor compaction, is high so IBAA is considered as a strong material when it is in a compacted state (Hartlen and Elander 1986, Pihl 1997). As specific gravity is a very important factor when utilising IBAA in civil engineering construction, it was reported that the specific gravity for IBAA ranges from 2.1 to 2.7 (TFHRC 2008). From this viewpoint IBAA, considering its relatively low unit weight, could be classified as lightweight aggregate. IBAA's mean Proctor density and optimum moisture content values confirm its classification into lightweight aggregates (Eighmy et al 1992). Moreover, IBAA is classified as well-graded material from gradation viewpoint (Zhang 1994, Izquierdo et al 2002, Forteza et al 2004). Based on these properties it is expected that incorporating IBAA in bituminous mixtures may enhance their stiffness however, will require more binder content.

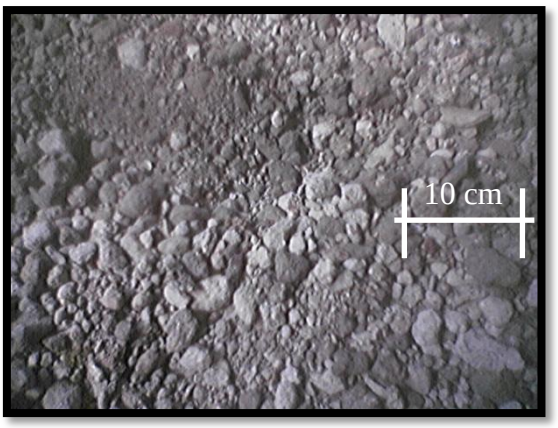


Figure 2-8: IBA.

IBAA property	Its range
Specific gravity	2.1 – 2.7
Unit weight	955 – 1420 kg/m^3
Mean Proctor density	1530 – 1739 kg/m^3
Optimum moisture content	9.6 – 20 %
10% fines value (TFV)	20 - 65 kN
Los Angeles Coefficient (LAC)	38 - 45 %
Shear strength friction angle	38 – 42 ^{o}
Permeability coefficient	10^{-2} – 10^{-3} cm/sec
California Bearing Ratio (CBR)	40 – 70%
Loss on ignition	2.6 – 9.8%
Water absorption	3.5 – 17.1%

Table 2-1: Physical properties of IBAA (Nunnes et al 1996, Rogbeck and Knutz 1996, IAWG 1997, Izquierdo et al 2002)

The concentration of fine materials in BA is an important consideration when used as an aggregate substitute. The percent fines can frequently create problems because that fraction is highly absorptive for water, bitumen, or cement. High fine contents create a material that has a tendency towards freeze-thaw susceptibility and durability failure. These values range from 1.9 to 7.4% (IAWG 1997). Therefore, care should be given to these properties when using IBAA in asphalt. To conclude, the main IBAA physical properties are presented in Table 2-1.

2.3.1.1 Influence of aging on bottom ash physical properties

Studies in Sweden (Hartlen and Rogbeck 1989, Hartlen and Lundgren 1992) and Germany (Vehlow 1992, Pfrang-Stotz and Reichelt 1997) had been conducted on the influence of aging on some physical properties of IBAA. It was found that its E-modulus increased with aging when compacted at optimum moisture content under Proctor compaction conditions. This increase in modulus was attributed to the formation of mineralogical phases that increased particle interlocking within IBAA. In addition, it was found that the Proctor compaction characteristics were much better for IBAA aged for a year than when ash was freshly collected. This was attributed to an increase in the durability which resulted from mineralogical phases changes. However, these studies used natural ageing only while accelerated ageing effect was not investigated and consequently no comparisons between the two procedures have been undertaken.

2.3.2 Mineralogical properties

The mineralogical properties of IBAA play a very important role in the mechanical and mainly in the leaching behaviour of this material. IBAA is known to contain numerous crystalline phases which complicate its leaching behaviour (Izquierdo et al 2001). This also affects the aging and strength development. The International Ash Working Group (IAWG 1997) briefly described the usefulness of morphologic and mineralogical petrography analysis of IBAA as well as provided an overview of MSW bottom ash particle morphology and mineralogy based upon the research of Vehlow et al (1992), Kirby and Rimstidt (1993) and Eighmy et al (1992). They

identified the most likely mineral phases of IBAA as: Quartz (SiO_2), Calcite ($CaCO_3$), Magnetite (Fe_3O_4), Hematite (Fe_2O_3), Gypsum ($CaSO_4\_2H_2O$), Cristobalite (SiO_2), Melilite group minerals ($Ca_2Al_2SiO_7$), Feldspars group ($(K, CA, Na)AlSi_4O_8$), Lime (CaO), Magnetite (Fe_3O_4), Iron oxide (Fe_2O_3), and Sodium Chloride (NaCl). More details about IBAA mineralogical properties can be found in IAWG (1997) as these properties are beyond the scope of this research.

2.3.3 Chemical Properties

Many studies were interested in chemical composition of IBAA e.g. Selinger et al (1997), Bergfeldt et al (1997), Dijkestra et al (2002), Forteza et al (2004). IBAA was found to be reasonably consistent over time with ranges for most detected elements. The concentrations of most heavy metals were found to be lower than those found in fly ash however, higher than those typically found in soils (Musselman et al 1994). The concentrations of elements generally found in IBAA are summarized in the Table 2-2. From this table it is noticed that some elements like Aluminium, Carbon, Ferrous, Sodium, and Silicon are found in high concentrations and consequently, special concern should be given to their leaching potential. However, the reviewed literature on BA leaching highlighted different elements as of high leaching potential, e.g. Arsenic, Boron, Cadmium, Chromium, Copper, Mercury, Lead, and Zinc. This confliction shows the complexity of IBAA behaviour.

With respect to the presence of organic matter in IBAA, the organic carbon content tends to range from 2 to 4%. The majority of this carbon is not burned well; it is possible that this carbon is cellulose, plant fibre, or plastic in nature. Trace organics of potential human health concern have been quantified in bottom ash. These include polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzo-p-furans (PCDFs) as well as potential precursors for these compounds under certain reaction conditions. These precursor compounds include; chlorinated benzenes (CBs), chlorinated phenols (CPs), polychlorinated biphenyls (PCBs), and poly-aromatic hydrocarbons (PAHs) (Lovell and Huang 1990).

Element	Concentration (mg/kg)	Element	Concentration (mg/kg)	Element	Concentration (mg/kg)
Ag	0.29 – 36.9	Cu	190 – 8240	O	400000 – 500000
Al	21900 – 72800	F	200 – 1100	P	1400 – 6400
As	0.12 – 189	Fe	4120 – 150000	Pb	98 – 13700
Au	< 0.20	Ga	10	Rb	40 – 50
B	38 -510	Hg	0.02 – 7.75	S	1000 – 5000
Ba	400 – 3000	I	2 - 10	Sc	3 – 6
Br	1.4 – 150.2	K	750 – 16000	Sb	10 – 432
C	10000 – 60000	La	2- 20	Se	0.05 – 10
Ca	370 – 123000	Mg	400 – 26000	Si	91000 – 308000
Cd	0.3 – 70.5	Mn	83 – 2400	Sn	2 – 380
Cl	800 – 4190	Mo	2.5 – 276	Sr	85 – 1000
Co	6- 350	N	110 – 900	Ti	2600 – 9500
Cr	23 – 3170	Na	2870 – 42000	V	20 – 122
Cs	1.0 – 2.0	Ni	7 – 4280	Zn	613 - 7770

Table 2-2: Element concentrations, generally found in IBAA, worldwide (Lindsay 1979)

2.4 IBAA UTILIZATION

2.4.1 Environmental aspects related to IBAA utilisation

Although IBAA is categorised in some countries as non-hazardous material (Bruder-Hubscher, et al 2001) researchers investigated extensively the environmental aspects related to IBAA utilisation. Special concern was given to its leaching potential because of the heavy metals it contains.

IBAA leaching properties were studied in several occasions. Eighmy et al (1995) investigated the leaching potential of a bituminous mixture; 50% bottom ash + 7% binder content. Results suggested that the IBAA leaching behaviour is controlled by the binder. This behaviour did not show a significant change over 1.25 year study period (Eighmy et al 1997). Moreover, it has been shown, in a field study, that the pH value of an IBAA road-base layer, after 10 years, was significantly less than shortly after placement. This decrease was caused by release of mobile pH constituents and carbonation (Schreurs et al 1997). The pH value was found to have a significant effect on the concentrations of toxic elements in IBAA leachates. These concentrations were shown to be greatest in the earlier stages of IBAA use or disposal, while the long-term leaching level was likely to be reduced by the neutralisation of its pH value (Meima and Comans 1997). In contrast, heavy metals concentrations in an IBAA bituminous mix were recorded as limited (Cai et al 2004) and have no significant effect on the ground water (Thayumanavan et al 2001).

The above mentioned researchers presented promising remarks for utilising IBAA in constructions. However, literature focusing on the time dependency of leaching of constituents from municipal solid waste incineration residues is relatively inconsequential. Moreover, these few studies (Talbot et al 1978, Belevi et al 1992, Comans et al 1993, Zevenbergen and Comans 1994, Kirby and Rimstidt 1994, Fallman 1997 and Dijkstra et al 2006) considered bottom ash as an individual material and not as an aggregate in a blend or a mix. Therefore, the potential environmental impact of using high levels of IBAA in bituminous mixtures on the IBAA leaching properties needs to be investigated as will be discussed in Chapter 4.

2.4.2 IBAA in civil engineering applications

Interest in using IBAA in civil engineering applications started in the 1970s and declined in the 1980s then it picked up, again, from 1990 to be a common research field. The physical and chemical nature of IBAA, described earlier in this chapter, suggested many potential uses of it as an ingredient in some products and as a processed replacement for rock products. IBAA is, after all, very much like crushed rock with some residual metal, glass, and soluble salts. Therefore, a wide range of civil engineering applications have been investigated. Studies showed the possibility to incorporate IBAA as aggregate materials in bricks, concrete, and in road construction applications as well. A summary of the research conducted on these usages is presented herein.

Numerous researchers investigated the fundamental knowledge of suitability of IBAA to be used as a substitute material in civil engineering applications. Seals et al (1972) investigated the suitability of IBAA to be used as an aggregate in construction applications. They studied the mechanical, physical and chemical properties of IBAA produced in the USA and found that –at that time- the incinerator type is the only factor which affects the properties of IBAA. Therefore, they predicted that it will be more desirable to utilise IBAA as an aggregate in a more productive manner. Faber and DiGioia (1976) summarized the annual quantities of flay ash and IBAA produced by major electric utilities in the USA. Similar data for England were also presented. Available data on the engineering properties of these materials and on their usages in the USA and England were also presented. The paper described several projects in USA, England, and Scotland in which large quantities of fly ash have been used successfully and economically as a highway embankment fill material. They recommended the same usages for IBAA; however, no experimental or theoretical work was undertaken to support this conclusion. Another research (Pandeline et al 1997) studied the shear strength and deformation characteristics of IBAA and found that these properties are similar to other soils. Therefore, it was predicted that IBAA will perform adequately in many highway applications as long as proper quality control is maintained.

Using IBAA as embankments and fill materials is one of the most common applications for using IBAA in highways. This usage has been investigated in several occasions. Lovell and Huang (1990) studied the physical, chemical characteristics and engineering properties of IBAA and suggested that it could be used as embankments. Later on, this recommendation was tested successfully in a test road in Denmark (Pihl 1997) and in Germany (Kus et al 1999). Simultaneously in Sweden, IBAA was used successfully in several projects as light fill material (Rogbeck and Knutz 1997) while in the USA; IBAA was suggested as suitable material to be used as embankment material in highways (Vipulanandan and Basheer 1998).

A series of attempts were made to evaluate the use of IBAA for production of lightweight bricks, concrete masonry blocks, and cement bound materials. For example, Phillips et al (2005) developed a successful procedure to manufacture concrete masonry blocks using a mix of fly and bottom ashes. In another study, two lightweight aggregates, incorporating IBAA, were manufactured, a carbon-free and a carbon-containing aggregate, and were characterised in terms of physical, mechanical, mineralogical and micro-structural properties. The IBAA-derived aggregate performance was assessed in comparison with commercial synthetic aggregate and the results confirmed the feasibility of the production of lightweight aggregate using significant concentrations of IBAA. Pflughoeft-Hassett et al (2000) found that bottom ash can be used in limited quantities as amendments to soils for rammed earth construction. This usage did not significantly change the soil properties of cohesion and unconfined strength for the soil tested. However, they concluded that the use of bottom ash must be balanced with the specific soil to be used. Paine (2002) tested cement bound material (CBM) containing IBAA contents (0, 40, 70, and 100%) as natural aggregate replacement, with cement content between 2 and 10%. Mixes properties were determined: optimum moisture content, compressive strength, tensile strength, and leaching behaviour. Results showed that using 40% or more IBAA as aggregate in cement bound sub-base and road-base layers is feasible; it gave sufficient strength and no environmental concerns. In another study, Filipponi, et al (2003) presented results of a wider experimental programme project in which different mixes were prepared by blending bottom ash with ordinary Portland cement in different proportions and at different water dosages. The solidified products were tested for

setting time and bulk density, unconfined compressive strength and evaporable water content at different curing times. The results of the experimental campaign were analysed through a statistical procedure. Results showed that for unconfined compressive strength no interactions between the factors were recognisable. Moreover, linear dependence of strength on both IBAA content and water content was assessed. Conversely, for setting times a non-linear, more than proportional, dependence was observed.

In addition, IBAA has been used in concrete manufacture as an aggregate in a few occasions (Wainwright and Robery 1997, Pera et al 1997, Berg and Neal 1998). However, for this utilisation, IBAA required treatment to limit swelling and cracking and to improve its performance. Moreover, using IBAA in concrete manufacture resulted in an increase in mix's water demand (Basheer and Bai 2005). This increase can be attributed, evidently, to the high water absorption property of IBAA. These studies suggested that the concrete containing IBAA could be used in non bearing works or backfilling of excavations (Berg and Neal 1998) or in lightweight concrete blocks (Qiao et al 2008). Naik (2002) tested concrete incorporating a mix of fly and bottom ash for their fresh properties, and for compressive strength, splitting tensile strength, flexural strength, abrasion resistance, and drying shrinkage. Results showed that up to 35% of ashes did not significantly change concrete properties. Kurama et al (2008) investigated the use of IBAA as an aggregate with different replacement amounts in autoclaved aerated concrete and determined the effect of ash incorporation on the final product properties. Results showed that using IBAA in autoclaved aerated concrete formulations caused a unit weight decrease in the produced concrete for all replacement ratios. However, it was observed that for the concrete having 25% and 50% IBAA usage had also beneficial effect on the strength gaining of the concrete. On the other hand, although Dyer et al (2003) and Basheer and Bai (2005) found that using IBAA as a fine aggregate replacement in concrete gave pozzolanic action signs and increased concrete strength, Frantz and Demars (2000) found that it is not suitable as fine aggregate for structural quality concrete due to problems related to cracking and low strength. These mixed opinions show that using IBAA in concrete has not yet approached a platform and research in this area, which is beyond the scope of this research, needs a lot of investigations. However, using IBAA in road construction has

good records and showed promising indicators. This usage will be discussed in the following section.

2.4.3 IBAA in roads

IBAA was used for the first time as road base in 1971 in construction of access road to computer centre of the Law School at West Virginia University in the USA. IBAA was used without screening or treatment (Moulton et al 1973). Currently, in the UK, 450 thousand tonnes of IBAA are recovered annually. 70% of this amount goes into type 1 aggregates, bulk fills and unbound applications of roads. The other 25% is included in the manufacture of foamed concrete, lightweight concrete and binder course asphalts (BPL 2008).

The recent major contracts in the UK, in which IBAA was used, include the pavement constructions at Terminal 5 (T5) at Heathrow airport, London, where all binder course layers had a 10% IBAA, mixed with recycled glass addition (Wrap 2008). M25 widening, junction 28, Sunderland absorbed 40 thousand tonnes of IBAA used as type 1 sub-base aggregates (BPL 2008). In addition, it is known that the London Olympic projects are now using a bulk fill material of IBAA. No data is published yet on these projects. All these projects show the increasing demand in IBAA usage. In the following sections, the usages of IBAA in both non-bituminous and bituminous road construction applications will be reviewed.

2.4.3.1 IBAA in non-bituminous applications

Early studies (Usmen et al 1978, Mackay et al 1992, Levie et al 1994, Izquierdo et al 2001) on IBAA utilisation in roads were conducted to determine if beneficial use could be made of this material. Potential uses were presented as aggregate for road sub-bases, bases, asphalt, and concrete. Results revealed potential use of the ash for road applications with certain treatment recommendations. These promising results encouraged researchers to perform direct investigations on IBAA as natural aggregate substitutions in road base layers. Nunes et al (1996) studied the use of IBAA, among other secondary materials, in road construction as unbound base layers. The study

covered technical aspects, mechanical properties of tested materials and environmental implications of their use. Two methodologies were presented for the mechanical and environmental assessment of secondary aggregates. Fundamental tests such as repeated load triaxial tests and repeated load indirect tensile tests were undertaken. Results represented a good concession in complexity, field simulation and reliability of results. Real road trials were also studied. Pihl (1997) studied the application of IBAA in road construction in Denmark as fill embankment and granular sub-base. A full scale test road was provided. Results showed that after three years of heavy traffic, no significant differences in road properties were noticed. Heath et al (1999) presented road trial sections constructed using IBAA as base coarse material. These sections were subjected to accelerated testing with a heavy vehicle simulator. High deflections were measured in the pavement structure, however, it was concluded that the road performance was acceptable.

To compare IBAA properties with common properties of conventional materials used in road bases, Izquierdo et al (2001, 2002) studied the characterization of bottom ash from MSW in Spain. They found that IBAA studied was fairly similar to bottom ashes from other countries which indicate the consistency of its properties. This result means that there is no source effect on IBAA properties. Similar findings were arrived at in this research and will be presented in Chapter 3. In addition, the same researchers found that these properties were similar to values of common natural materials used in roads. The tested properties included the grain size distribution, sand equivalent, density, absorption degree, Los Angeles abrasion value, compaction degree, and CBR index. Same results were obtained by Forteza et al (2004). They presented the physical and chemical characterization of IBAA. They studied water resistance, compactability, CBR, sand equivalent, plasticity, and fracture faces. They concluded that the engineering properties are close to those of natural aggregates used in road bases. More IBAA fundamental engineering properties were investigated. Arm (2004) studied the mechanical properties of IBAA with the aim of investigating the variation in deformation properties of the ash, expressed as resilient modulus and permanent deformation, for use in unbound road layers using cyclic load triaxial tests. The results showed that there was a variation in the deformation properties. This variation was attributed to the organic matter content. However, it was concluded that

this variation could be kept low by decreasing the organic matter content. Moreover, it was found that the deformation properties were reasonably uniform for each incinerator plant patch. For the cyclic stress levels used in the study, the resilient modulus ranged between 60 and 140 MPa, which are comparable to that of sand; however, the plastic permanent deformation was lower than for sand. Based on these results, it can be seen that IBAA was used successfully in non-bituminous applications for road constructions. These successes encourage a search for more types of applications especially bituminous mixtures.

2.4.3.2 IBAA in Bituminous mixtures

Research on using IBAA in bituminous mixtures is considered to be limited. Early investigations on IBAA utilisation in bituminous mixtures (Moulton et al 1973, Anderson et al 1976) showed limited success. Based on this limited success, bottom ash was suggested to be used in wearing course layers or in bituminous stabilised base layers only.

The engineering properties of bituminous mixtures containing IBAA were studied and compared with bituminous mixtures made of natural aggregates in few occasions. Ormsby and Fohs (1990) found that the properties of IBAA bituminous mixtures were dependant on ash content where the optimum binder content increased and mixture's density decreased with increasing ash content. Same results were obtained by Kandhal (1993). These results were emphasised by the findings of this research as will be discussed in Chapter 3. In addition, the resilient modulus of IBAA mixtures was low where Poisson's ratio was approximately equal when compared with standard bituminous mixtures. However, fatigue life and fracture toughness of these mixtures increased with the increase of ash and binder content, and rutting susceptibility and plastic deformation also increased at higher ash and binder content. On the other hand, Johnson (2000) summarized the disadvantages of using IBAA in bituminous mixtures, where a significant increase in Calcium content was observed. However, all studies, including this work, on IBAA leachability did not raise any concerns regarding Calcium leaching. Moreover, blending IBAA with bitumen in a bituminous blend led to decreasing permeability and porosity and consequently influenced the

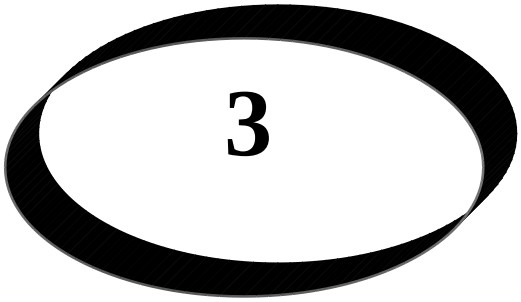
mobility of a number of heavy metal cations. This result is very important as it may explain any observed increase in leaching of certain elements.

IBAA has been used in bituminous mixtures at different content levels up to 50% natural aggregate replacement. It was found that substitution of up to 20% IBAA for virgin aggregates yielded mixtures having aggregate structures that are well developed to resist compaction and rutting (Ogunro et al 2004). A study by Vassiliadou and Amirkhanian (1999) showed that up to 30% substitution of natural aggregates with IBAA resulted in lower resilient modulus values and a moderate effect on short-term tensile strength. Moreover, using 32% IBAA has been suggested as satisfactory for a bituminous base course or for wearing course under low traffic conditions (Garrick and Chan 1993). High IBAA replacement has been attempted in the US, where 50% was used in a binder course asphalt mixture on a major road trial. The used mix met the design requirements, had lower unit weight, and required more binder than conventional mixes. However, it was economic to use (Musselman et al 1994, Zhang et al 1999). The same IBAA level, 50%, was suggested, but using sulphur modified bottom ash, for bituminous mixtures in a field demonstration projects. After 2 month in service, under hot weather conditions, no distress was noticed (Estakhri and Saylak 2000). The moisture susceptibility was investigated for 15% IBAA bituminous mixtures. It was found that the incorporation of IBAA into bituminous mixtures met the Superpave volumetric mix design requirements of tensile strength ratio after one freeze-thaw cycle and did not reduce their resistance to moisture damage (Zeng and Ksiabati 2003).

It is known that there are two main distress modes for bituminous mixtures: permanent deformation and cracking. The latter has not been studied previously for bituminous mixtures containing IBAA. Therefore, it worth investigation as will be shown in Chapter 8. With regard to permanent deformation resistance of mixtures containing IBAA there were mixed observations. On one hand, using 20% IBAA improved rutting resistance for bituminous mixtures. (Ogunro et al 2004). On the other hand, the rutting resistance of bituminous mixtures containing different levels of IBAA, as fine aggregate substitution, was studied (Huang et al 2006). The results of a

wheel tracking test showed that the rut depth increased when IBAA was added. The more the IBAA the deeper the rut depth was. This study concluded that the testing machine reached its maximum depth measurement ability when 75% IBAA was used. These mixed observations may be attributed to different technologies adopted in the EFW plants as the former study was undertaken by a US group while the later study was undertaken in Taiwan. These different technologies may result in different IBAA. However, these mixed observations indicate the need for further studies on the response of bituminous mixtures containing different levels of IBAA to applied loading.

Based on the literature, it is noticed that there is a need to expand the area of incorporating IBAA in road construction. The properties of bituminous mixtures incorporating high content level of IBAA needs further investigation. Moreover, a lack of knowledge was observed with regard to time dependency leaching potential of bituminous mixtures containing IBAA and on permanent deformation behaviour of these mixtures. Crack resistance of such mixtures has not been studied before and it needs an exploration.



MIX DESIGN AND EVALUATION

3.1 INTRODUCTION

This chapter presents rational bituminous mixtures which contain different levels of Incinerator Bottom Ash Aggregate (IBAA). Firstly, a control blend of limestone, which meets British Standards gradation requirements for binder course layers in flexible pavement, has been presented. Then, rational blends containing IBAA at different content levels have been achieved. These blends have been designed, using a volumetric mix design approach, to produce bituminous mixtures. The selected mixtures, which include a conventional limestone mixture and three IBAA bituminous mixtures, have been evaluated in terms of stiffness, moisture sensitivity and ageing effect.

3.2 MATERIALS

Three types of materials were used in this research. Limestone was used to develop traditional blends. Conventional bitumen was added to limestone in order to produce conventional control bituminous mixture. Limestone, was then, replaced by incinerator bottom ash aggregate to develop rational IBAA bituminous mixtures.

3.2.1 Limestone

Limestone is an important raw material and it is often said to be the world's most versatile mineral. It has a wide variety of applications; however its primary use is in the construction industry with a special focus on road constructions. Limestone is widely used in the UK as a natural road material from a total of 6.9 million tonnes industrial demand in 2004 (Hetherington 2007). Therefore, it was chosen to be the control material for this study. Limestone was supplied by Tarmac LTD and sourced from Pant quarry, Flintshire, North Wales. It was supplied in six different commercial sizes namely: 20, 14, 10, 6, 3 mm to dust, and filler. The particle size distribution for these sizes is shown in Fig 3-1. The used limestone typical physical and chemical properties, as received from the supplier, are shown in Table 3-1.

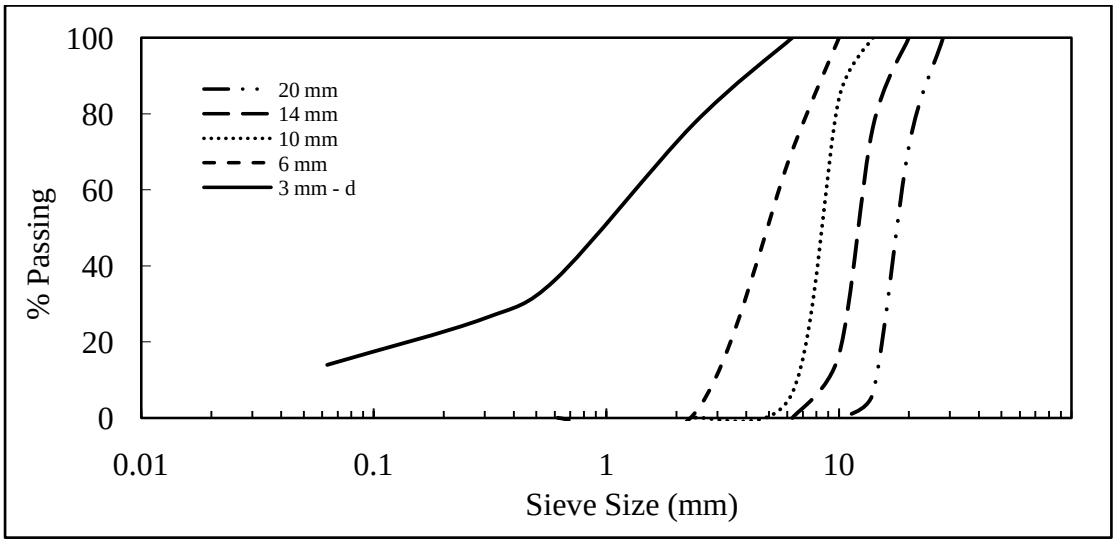


Figure 3-1: Limestone particle size distribution.

Typical aggregate and chemical properties	
Particle apparent density	2.71 Mg/m^3
Water absorption	1.1 %
Aggregate abrasion value	14
Polished stone value	37
Los Angeles coefficient	29
10 % Fines value	180 kN
Acid soluble sulphate	0.01 %
Water soluble sulphate	< 0.01 %
Oxidisable sulphides	< 0.03 %
Loss on ignition	42.9

Table 3-1: Limestone typical properties

3.2.2 Incinerator Bottom Ash Aggregates, IBAA

IBAA used in this study was supplied by Ballast Phoenix LTD and sourced from Teesside and Castle Bromwich, UK. It was supplied in two commercial sizes; 20 - 10 mm and 10 mm down. Particle size distribution, shown in Fig 3-2, was undertaken for all samples to determine the gradation and to study the source effect. It was noticed that the IBAA 20 - 10 mm contained particles smaller than 10 mm and down to 0.063 mm with the latter being less than 5% of the total mass. On the other hand, the 10 mm – down size was confined between 10 and 0.3 mm. Therefore, it was obvious that the larger size contained some very fine aggregates at a higher content than that in the smaller size. In respect of source effect on IBAA gradation, no significant source effect was found. Using visual inspection, no significant difference in the composition

was found. As a result, it was decided to depend on one source for IBAA. Teesside was then chosen for IBAA supply due to logistical reasons.

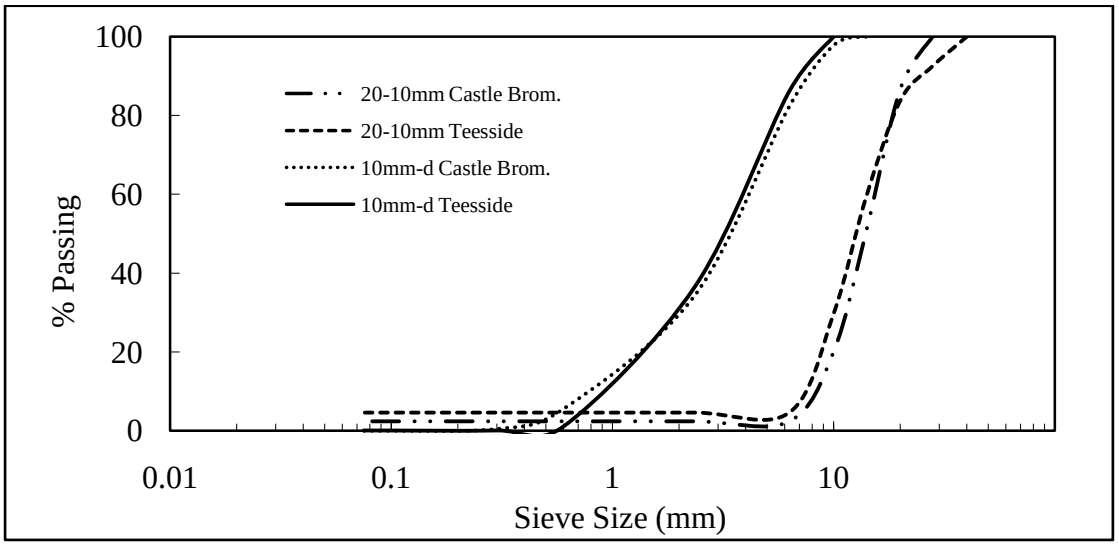


Figure 3-2: IBAA particle size distribution.

Moisture content of IBAA was determined as supplied by drying 1 kg samples for 24 hrs at 100 oC. Then the obtained moisture content values were compared to the IBAA moisture content values quoted by the supplier and collected from literature. Results are presented in Table 3-2. From this table it was noticed that the larger size 20 - 10 mm had a moisture content which was less than the normal range advised by the supplier while the smaller size, 10 - d, had an expected normal range moisture content value. This indicated that the larger particles dried quicker than the smaller sizes and that was eye witnessed over time. IBAA typical physical and chemical properties, as received from the supplier, are shown in Table 3-3.

Sample	Moisture content (%)	Supplier range (%) (BPL, 2005)
20 – 10 mm	0.79 – 4.43	8 – 15
10 mm - d	15.43 – 15.56	10 - 28

Table 3-2 IBAA moisture content

IBAA properties	
Particle apparent density: 20 - 10 mm size 10 mm -d	 2.45 Mg/m^3 2.42 Mg/m^3
Water absorption	5-7 %
10 % Fines value	50 – 65 kN
Polished stone value	45
Los Angeles coefficient	38-42
Acid soluble sulphate	1 – 2.5 %
Water soluble sulphate	0.2 – 1.6 g/l
Oxidisable sulphides	98 mg/kg
Organic content	2-5 %

Table 3-3: IBAA characteristics

3.2.3 Binder

Binder used in this study was bitumen 100/150 pen. It was supplied by Nynas Bitumen and sourced from Venezuela. This bitumen was tested to determine its softening point (BSI 2000a) and penetration grade (BSI 2000b). These values were 43 ^{0}C and 114 Pen respectively. Bitumen was supplied in 5 litres metal tins with density ranged between 1.0 and 1.071 g/cm^3 at 25 ^{0}C.

3.3 NON-BITUMINOUS BLENDS

A control blend of Limestone, which meets the gradation requirements for a UK binder course layers (BSI 2003), was explored. This blend, blend CB, was achieved

using the composition shown in Table 3-4. Fig 3-3 shows blend CB in conjunction with the BS binder course layers gradation limits. From this figure it was clear that blend CB contained 60% of coarse and medium aggregate size, i.e. > 3 mm, and 40% fine aggregates, i.e. ≤ 3 mm, and filler, ≤ 0.063 mm.

Size (mm)	20	14	10	6	3-d	Filler
Content (%)	25	12	8	18	37	3

Table 3-4: Control blend, CB, composition

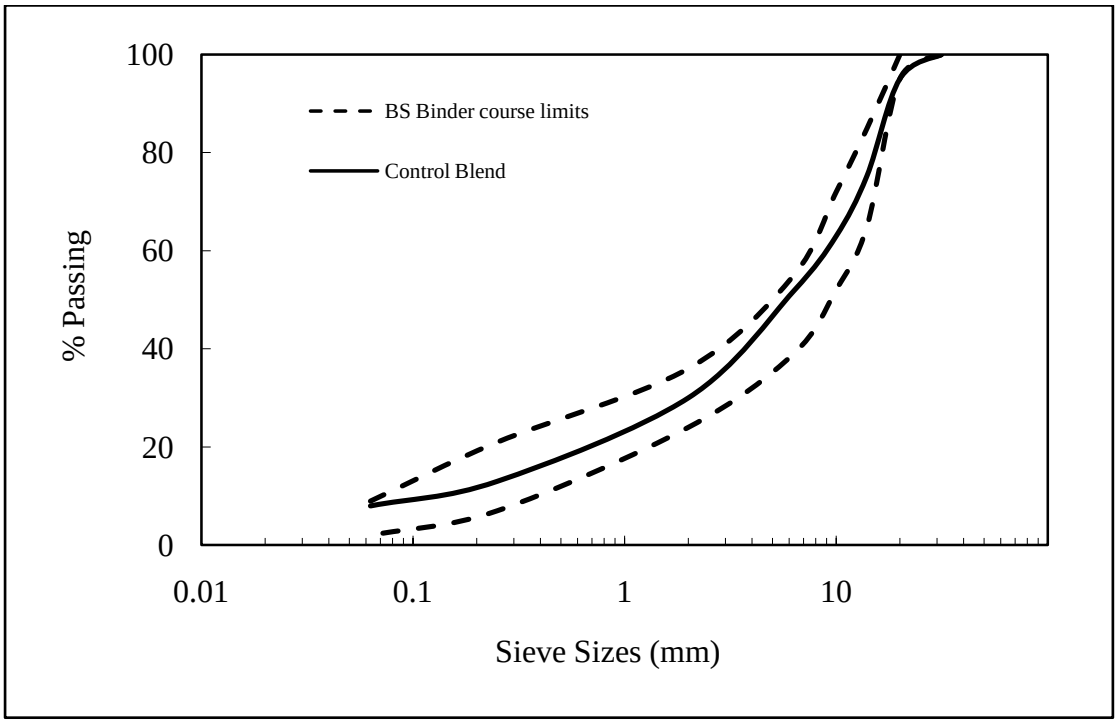


Figure 3-3: Limestone control blend in conjunction with BS binder course layers gradation limits.

The achieved control blend was used to find out blends which contain high IBAA content levels. A procedure for limestone replacement, with IBAA, was sought based on the limestone control blend achieved before. It was noticed that IBAA contained neither fine aggregates nor filler. Consequently, it was decided to use fine aggregates and filler from limestone aggregates only. As a result, replacement would be for the

coarse and medium aggregates, i.e. sizes > 3 mm, which made up 60% of the control blend.

One way of arriving at a rational replacement methodology was to produce an equivalent IBAA blend, EQ-IBAA, to a limestone blend comprising only the coarse and medium aggregates found in the control blend. Figure 3-4 shows that an IBAA blend based on an 80% : 20% ratio of 20 – 10 mm : 10 mm - d sizes closely resembles that of the coarse and medium limestone sizes. Based on this principle, a target blend that contains up to 60% IBAA content could be achieved. From this replacement curve, it was obvious that there was a similarity between the limestone coarse and medium part and the EQ-IBAA blend. Thus, this curve can be used to replace limestone with IBAA at any content level up to 60% of the control blend. Fig 3-5 shows the grain size distribution curve for an IBAA – Limestone blend, TB1, which contains 40% limestone filler and fine aggregate plus 60% IBAA. The composition of this blend is shown in Table 3-5.

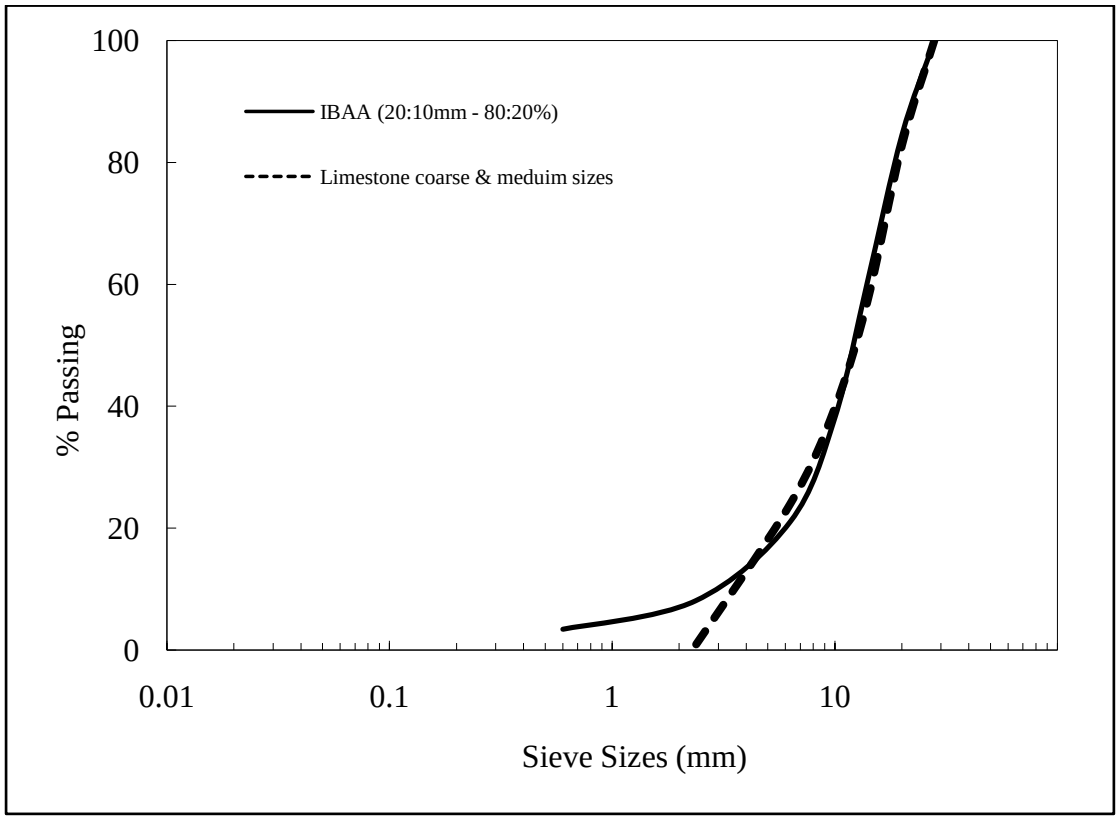


Figure 3-4: Limestone coarse and medium replacement curve.

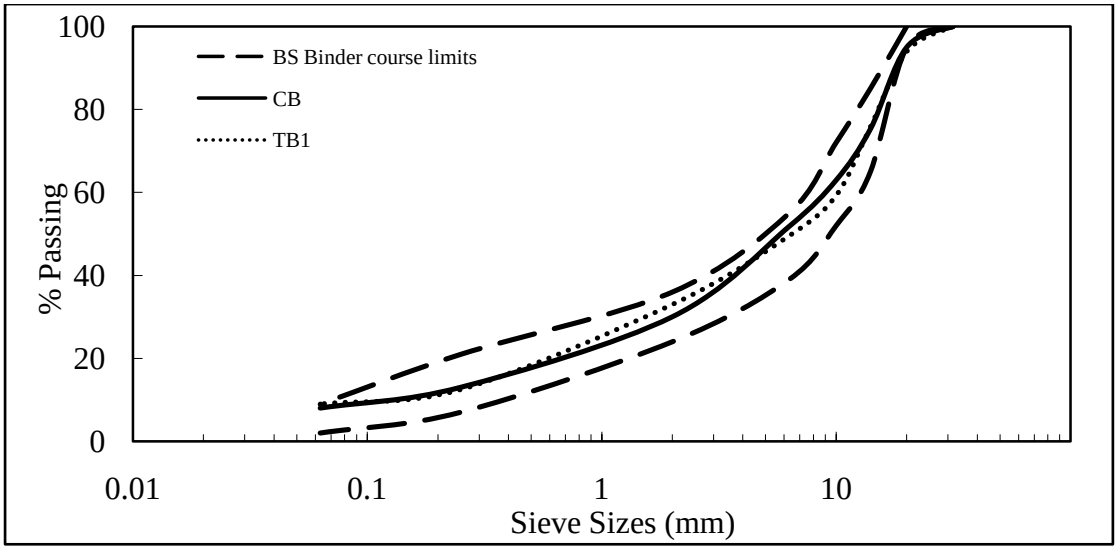


Figure 3-5: IBAA – Limestone blend, TB1.

Material	Size (mm)	Content (%)		
		CB	TB1	TB2
Limestone	20	25	0-25	0
Limestone	14	12	0-12	0
Limestone	10	8	0-8	0
Limestone	6	18	0-18	0
Limestone	3 - d	37	37	17
Limestone	Filler	3	3	3
IBAA	20 - 10	0	48-0	48
IBAA	10 - d	0	12-0	32

Table 3-5: Non-bituminous blends composition

The used methodology considered the replacement of limestone by IBAA using the masses only. However, considers volumes may not result in significant differences as the difference in specific gravity between the two types of aggregate is relatively small. Moreover, any difference in the produced blend comparing to the control blend was accepted as long as the resulted blend meets the requirements of the gradation limits.

A second target blend, TB2, was sought that allows limestone fine aggregate to be replaced with IBAA, in addition to the coarse and medium fractions, to achieve higher IBAA content. Another objective was to utilize the two IBAA sizes at roughly equal proportions. This blend comprised 80% IBAA and 20% limestone fine aggregate and filler. The IBAA content comprised 60% of size 20 - 10mm and 40% of size 10 mm – d. The composition of the second IBAA blend, TB2, as well as TB1 and CB are shown in Table 3-5. Fig 3-6 shows the developed blends compared to specification limits for binder course layers (BSI 2003).

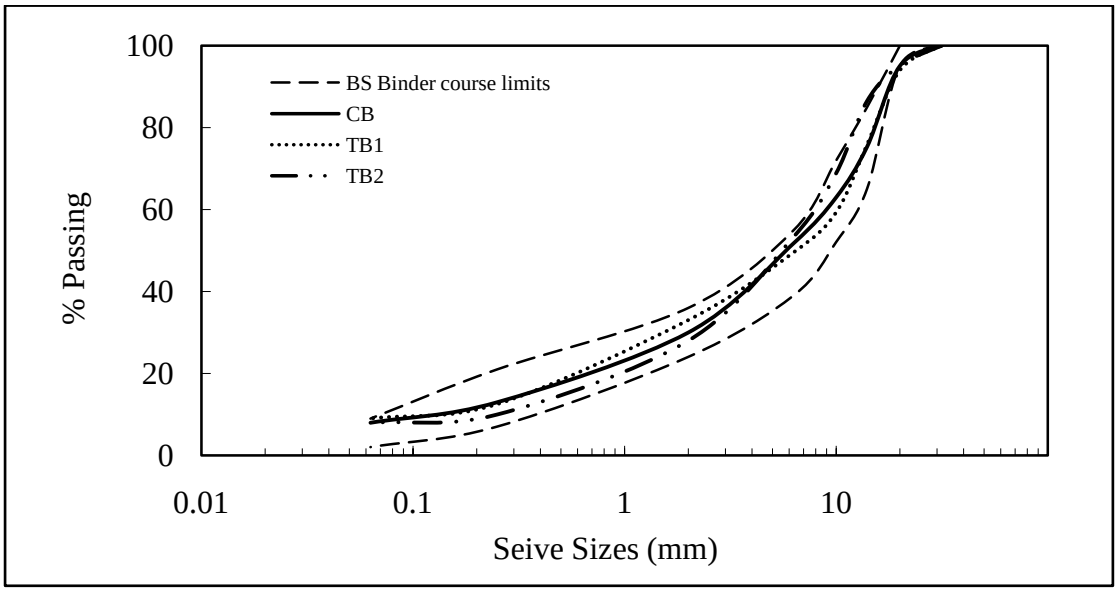


Figure 3-6: The developed blends.

To sum up, limestone replacement with IBAA could be achieved using two methodologies. The first one considered the replacement of some or all coarse and medium limestone sizes in the control blend with IBAA, without any fines

replacement. This technique would allow up to 60% replacement of the total blend. In terms of IBAA material usage, any blend produced in accordance with this method would tend to overuse of the larger size and under-use of smaller size. The second technique allowed replacement of all coarse and medium as well as some of the fine limestone aggregates. It also gave the opportunity to use both IBAA sizes in approximately equal amounts and, in addition, allows up to 80% IBAA content by mass to be used in the total blend.

3.4 VOIDS PROFILE

Compacted densities of the developed blends were determined with a target of developing a voids profile. To determine the compacted density of the blended aggregates, cylindrical moulds of 100 mm diameter and a fixed amount of 1250 gm of aggregates were used. Aggregate blends of different proportions were prepared and placed in the moulds. The blends were then compacted, as dry, using a vibratory hammer, working at 50 Hz frequency, at different compaction times. The effect of compaction time on the resulted compacted densities was investigated. Two different blends were used to study compaction time effect. Firstly, the control blend was tested and then, the EQ-IBAA blend. It was found, as shown in Fig 3-7, that no significant increase in the compacted density after 10 seconds compaction.

Aggregate crushing during compaction was assessed by sieving compacted samples through a 2.36 mm sieve, at each tested compaction time, and determining the amount of material passes as percentage of the original mass of the sample (BSI 1990). These amounts were compared to those obtained by sieving samples before compaction. The ratio of the increase passing was referred to as crushing effect value. Fig 3-8 shows the influence of compaction time on the aggregate crushing. From this figure it was observed that crushing increased rapidly during the early stages of compaction, and then continued to increase at a steady small rate in case of CB and at a high rate in case of EQ-IBAA. Subsequently, 10 seconds was found to be adequate in reaching refusal level without undue amount of crushing undergone. Hence, 10 seconds compaction time was picked up to establish voids profile.

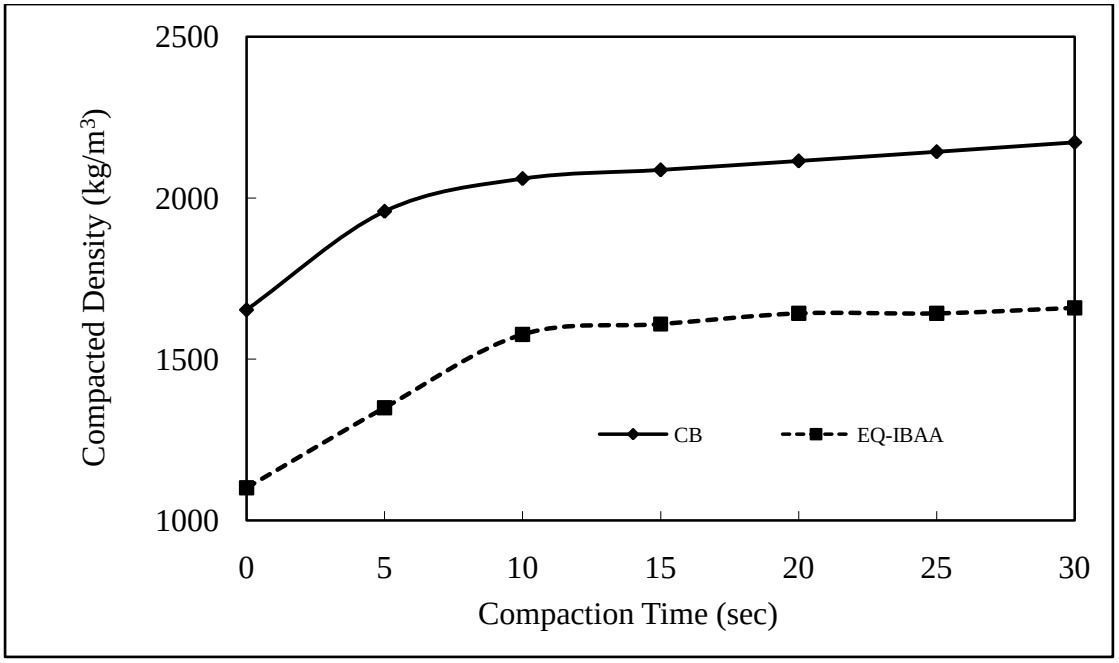


Figure 3-7: Compaction time effect.

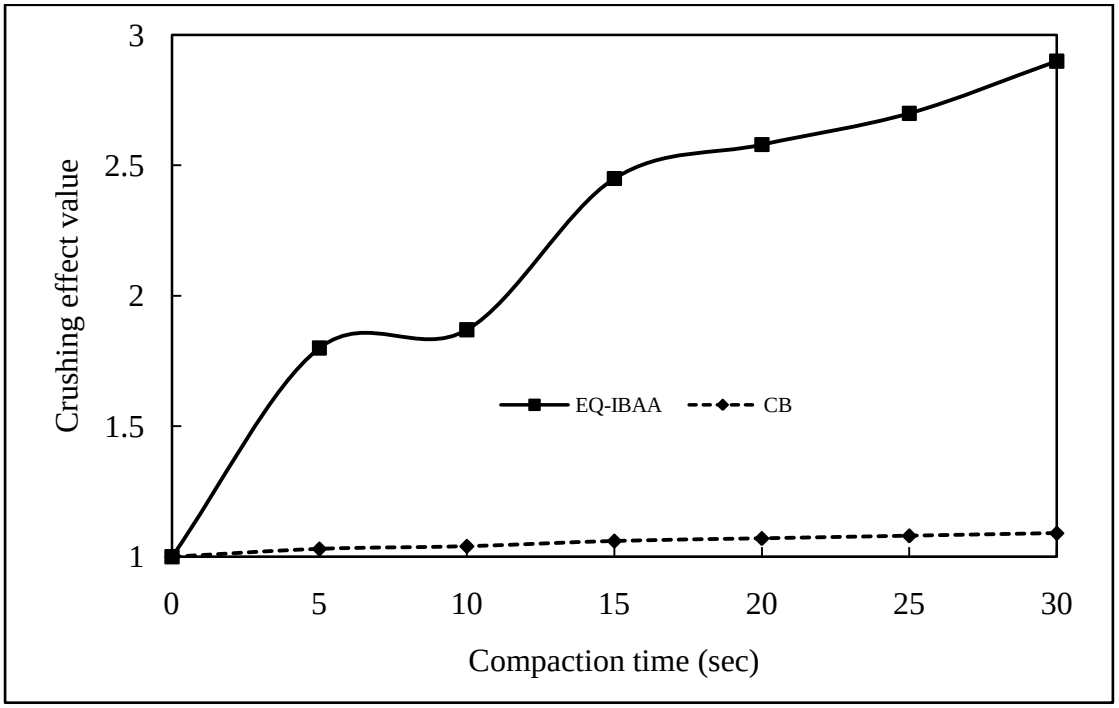


Figure 3-8 Influence of compaction time on aggregate crushing.

The compacted density for CB and TB1, with IBAA content between 10 and 60%, was determined and is presented in Fig 3-9. Measurements were made on triplicate specimens and the average value of the compacted density was taken. From the figure

it was obvious that the compacted density is at the maximum level at 0% IBAA content, which is the control blend. This is due to the fact that IBAA is similar to lightweight aggregates and its density is lower than limestone density. Addition of IBAA led to a decrease in density, as expected. Further addition of IBAA up to 30% showed the compacted density to remain almost constant. This is due to the effect of coarse and medium limestone, which constituted the majority of the aggregates in the coarse part of the blend, which reduced the effect of added IBAA. Then a drop occurred in the compacted density values at 40 to 50% IBAA content, due to the transfer of the effective majority from limestone, which has higher density, to IBAA which has lower density. Thus, the compacted density continued to decrease with IBAA content increase. This reduction is followed by a slightly increase in the compacted density at 60% IBAA content. This was due to the increase in the homogeneity of the blend, which at this point contained only 4 different sizes, but at any point before it contained 8 different sizes. This increased homogeneity helped the smaller particles to fill more of the voids created by the larger particles during compaction, which increased the compacted density of the total blend. The bulk densities were found to have the same trend of behaviour as the compacted densities as shown in Fig 3-9.

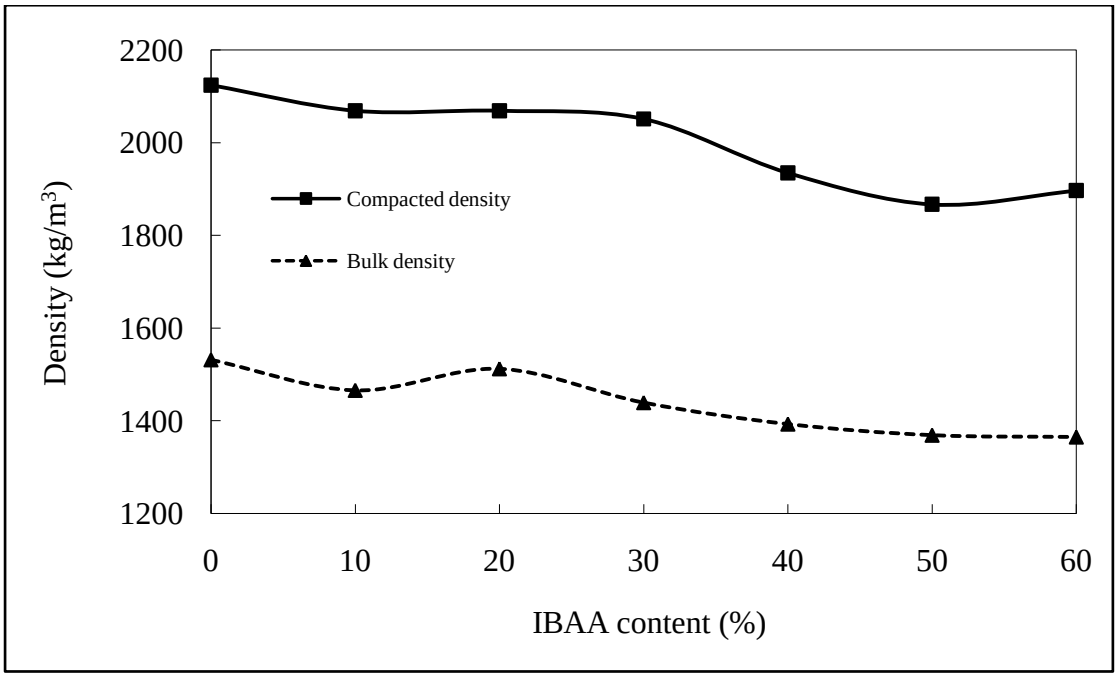


Figure 3-9: Compacted densities for CB and TB1.

The method used to determine voids content in an aggregate blend was based on the knowledge of the compacted density of the aggregate blend and its specific gravity. The specific gravities of aggregates were provided by the limestone and IBAA suppliers. Therefore, voids in mineral aggregates (VMA) were calculated using Eq 3-1. The resulted VMA were used to establish a voids profile. From voids profile, Fig 3-10, it was noticed that voids increased at the beginning of IBAA addition due to the disturbance caused by particle interlock and the difference in particle shape between limestone and IBAA. Then with increasing IBAA content, the effect of particle interlock is reduced due to the increase in fines content. This is due to the fact that IBAA contained more fines than coarse and medium limestone, as noticed from the replacement curve, Fig 3-4. These fines filled the voids up to a certain point. From this point forward fines no longer found room to fill in void spaces. Then, the highest packing occurred. This point was found to allocate between 20 and 30% IBAA content. After this point, voids increased with further IBAA addition when fines found no more room to fill and particle interlock effect began to override. At 60% IBAA content, a sudden drop in voids occurred again due to the increase in homogeneity of the blend which reduced the effect of particle interlock and allowed the effect of fines in IBAA to play a new role in filling voids. From the resulted voids profile, three blends were picked up to be designed as bituminous mixtures. These blends contain 0, 30, and 60% IBAA content. On the other hand for TB2, the compacted density and the voids content were calculated. This led to one more blend, which contains 80% IBAA content, to be designed as a bituminous mixture.

$$VMA = \left[\frac{SGMA - CDMA}{SGMA}\right] \times 100 \quad (3\text{-}1)$$

where: VMA is the voids in mineral aggregate; $CDMA$ is the compacted density of mixed aggregate; and $SGMA$ is the specific gravity of mixed aggregate.

$$SGMA = \frac{100}{\sum_{i=1}^{n} \frac{P_i}{SG_i}} \quad (3\text{-}2)$$

where: P_i is the percentage by weight of the total mix of each aggregate constituent; SG_i is the specific gravity of each aggregate constituent; and n is the number of aggregate constitutes.

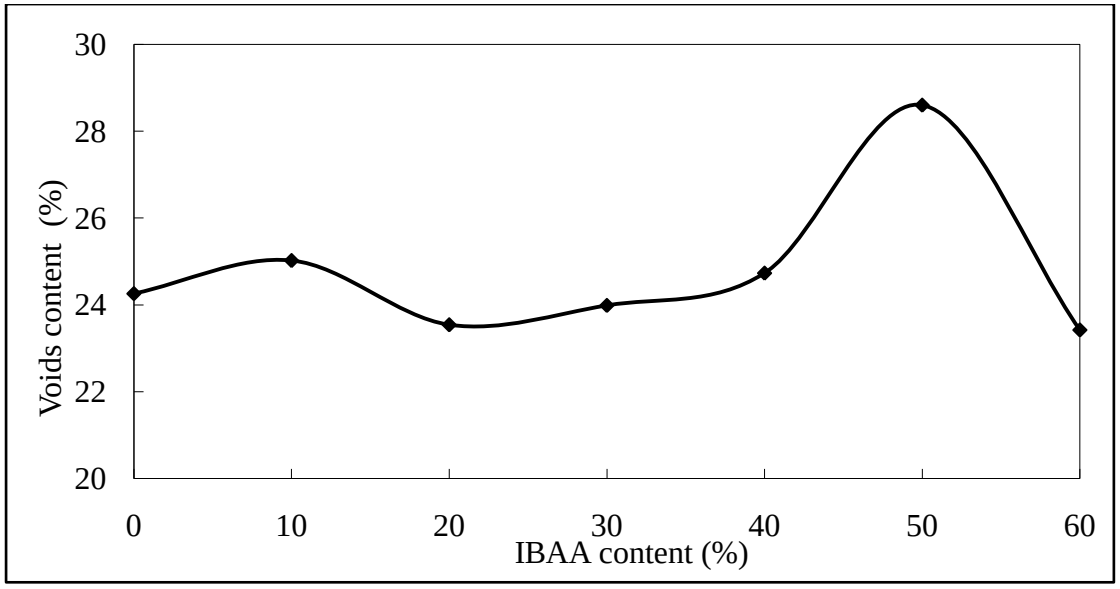


Figure 3-10: Voids profile for CB and TB1.

3.5 HOT MIX DESIGN

The objective of mix design is to produce an economic material using certain resources that meet the required level of pavement performance. Pavement performance is commonly assessed in terms of permanent deformation and fracture resistance. Furthermore, performance requirements are dictated by factors such as volume of traffic, climate conditions and structural section of the pavement. Therefore, bituminous mixtures have to fulfil a wide range of requirements such as stability, flexibility, durability, workability, safety, impermeability, and structural strength.

3.5.1 Mix design procedures

Worldwide, there are many mix design procedures which may be dated back to the 1860s. Some of these procedures are: Pat test method, large stone mixes method, Hubbard-Field method, Recipe specifications, Hveem mix design method, Marshall mix design method, Superpave mix design method, and the volumetric approach mix design method (Roberts et al 1996). Details of the most common procedures are as follow:

3.5.1.1 Recipe specifications

Recipe specifications provide a description of a particular bituminous mix in terms of aggregate type and grading, and the composition of each of the components, together with the method of production, laying and compaction. Recipe specifications are relatively simple to use and are applicable to most types of mixtures. They are based on experience of known compositions, which have performed successfully in practice. The recipe approach to mix design is still the basis of most specifications worldwide.

Recipe specifications have, however, a number of limitations. Main restrictions are traffic and climate condition changes as those, to which the mix is to be subjected, may not be the same as those existing when the experience, on which the recipe is based, was obtained. Moreover, the component materials may have different properties. These differences may require modifications to the recipe but there is no means of assessing what these modifications should be. In addition, the recipe does not take into account aggregate properties, shape, texture and packing characteristics. Finally, recipe specifications are very restrictive towards new materials, since they can not be used until experience has been sufficient to allow a specification to be written.

3.5.1.2 Hveem mix design method

The first step in Hveem mix design method (Vallerga and Lovering 1985) is aggregate selection. Aggregates must meet all the requirements as specified by local highway agencies. These requirements typically include limits on Los Anglos (L.A.) abrasion loss, soundness loss, sand equivalent, percent of deleterious substance, percent of natural sand, percent of particles with crushed faces, and percent of flat or elongated particles. The gradation of the aggregate blend to be used must meet the gradation requirements set by local highway agencies.

The procedure for the Hveem method uses standard test specimen of 64 mm height by 102 mm diameter. Theses specimens are prepared using specified procedure for heating, mixing and compaction. To estimate the required binder content of a

bituminous mix, a Centrifuge Kerosene Equivalent test is used. This test is followed by a stabilometer test, a cohesiometer test, a swell test, and a density voids analysis.

The Hveem mix design method has been used successfully to design asphalt mixtures against rutting. The possible disadvantage of the Hveem method is that the Hveem Stabilometer test is an empirical test and is not widely known and used.

3.5.1.3 Marshall method of mix design

The Marshall mix design method and criteria were originally developed for airfield pavements, but were later also adopted for use in highway pavements. Due to its simplicity, the Marshall method of mix design was the most commonly used mix design method in the U.S. before the introduction of the Superpave design system, and it is still the most commonly used mix design method worldwide.

The first step in Marshall mix design method (White 1985) is aggregate selection which should be performed as described earlier in Hveem mix design method. The binder content should be chosen in accordance with local highway agencies requirement. Mixtures are to be prepared at a temperature at which the binder kinematic viscosity is at a specified value. The mixture is to be compacted in a 101.6 mm diameter cylindrical mould by a Marshall compaction hammer, which is 4.5 kg in weight and dropped from a height of 457 mm for a specified number of blows per side of the specimen. The number of blows to be applied per side is 35, 50 or 75 for light, medium or heavy designed traffic, respectively. Compaction of the mixtures is done at a temperature at which the binder kinematic viscosity is at a specified value. The resulted compacted specimens are to be tested to determine the bulk specific gravity, which is used to calculate the maximum specific gravity of the mixture and the bulk specific gravity of the aggregate. The percent air voids and VMA of the specimen are determined as well. Then, Marshall stability test, which measures the Marshall stability and Marshall flow, is to be performed.

The disadvantage of the Marshall mix design method is that the aggregate orientation in the compacted Marshall specimens is not representative of that in the field-compacted mixtures. The aggregates in the Marshall compacted specimens tend to get crushed at high compaction levels.

3.5.1.4 Superpave mix design method

The Superpave mix design method (Cominsky et al 1994) was introduced as a result of the Strategic Highway Research Program (SHRP) conducted from 1988 through 1993. The Superpave mix design procedure consists of the following main elements:

- Selection of binder is made to meet the requirements of AASHTO.
- Selection of aggregate to meet the requirements for nominal maximum size, gradation control points, gradation restricted zone, consensus aggregate property requirements, and aggregate source property requirements.
- Preparation of mixture: Aggregate and asphalt are mixed at the temperature at which the kinematic viscosity of the asphalt is at a specified value. The loose asphalt mixture is then cured in a forced-draft oven at 135 ^{0}C for 4 hours before compaction.
- Compaction of mixture is performed using a gyratory compactor.
- The design asphalt content is the asphalt content at which the asphalt mixture has an air voids content of 4%.

The possible merit of the Superpave mix design method is that the Superpave gyratory compactor applies a fairly high compaction effort to the mixture. A mixture which cannot withstand a high compaction effort is usually weak and would be eliminated in this process. Another merit of the Superpave mix design method is that it requires the use of high quality aggregates, which would usually result in better performance of the asphalt mixtures. Unfortunately, no performance related test was used in the Superpave mix design method. Mixtures which meet the Superpave mix design criteria may have drastically different field performance. Mixtures which meet the Superpave mix design criteria could have poor performance.

3.5.2 Volumetric mix design approach

The concept behind the volumetric mix design approach is to produce mixes that give maximum density or minimum porosity (Cabrera 1996). This method determines the required binder content through an assessment of the aggregate blend voids when the aggregate system is in a state of full interlock i.e. analysis of the volumetric properties of the bituminous mixture in its compacted state (Preston 1997).

The first step in the volumetric mix design approach is the selection of the aggregate grading and determining the voids profile. From voids profile, different aggregate blends could be selected based on the voids in the dry aggregate blend and on the proportion of each aggregate constituent. Then, the binder content is estimated for a target mixture void content before mixing trials are conducted. Mix design parameters are determined. These parameters include compacted density of the mix (CDM), voids in mineral aggregate (VMA), voids in mix (VIM) and mix stiffness (ITSM). Finally, the optimum binder content, which allows maximum CDM and ITSM with minimum VMA and VIM, is determined.

3.5.3 Control mix design

A control mix (OA) is designed based on the control blend composition. The binder contents selected, based on typical binder course requirements, were: 3.5, 4.0, 4.5, and 5.0%. Therefore, calculations were made to find out the amount of aggregates and bitumen required to produce compacted slabs of 305 x 305 x 65 mm dimensions at all binder contents.

3.5.3.1 Specimen preparation

To manufacture specimens, a laboratory asphalt mixer shown in Fig 3-11 was used. Before being mixed, the aggregates and bitumen were heated at the same mixing temperature which was 160 ^{0}C. Aggregates were heated overnight while bitumen was heated for four hours prior to mixing commence. After mixing, mixtures were poured into steel moulds sprayed with silicon grease to prevent adhesion and, then, covered with oiled papers to prevent adhesion with the compactor. Then, samples were

compacted to slabs using a laboratory roller compactor, shown in Fig 3-12. The roller compactor was heated to a very high temperature to prevent sudden cooling of mixtures. Four different pressures were applied on the produced slabs. These pressures were 25, 40, 50, and 72 psi. Each pressure was applied for 10 passes over each slab. The compacted slabs were left for 24 hours, to cool at ambient temperature, before de-moulding. Cylindrical specimens of 100 mm diameter and 65 mm height were cored from the slabs. Each slab produced five samples.

Figure 3-11: The laboratory asphalt mixture.

Figure 3-12: The laboratory roller compactor.

3.5.3.2 Volumetric parameters determination

The produced cylindrical specimens, five samples, were weighed in air and in water after being covered with cling-film (Del Valle 1985) to prevent water penetration. From the two weights, the compacted density of the mix (CDM) was calculated using Eq 3-3.

$$CDM = \ weight\ in\ air/(weight\ in\ air - weight\ in\ water) \quad (3\text{-}3)$$

Furthermore, the compacted density of mixed aggregate (*CDMA*), theoretical specific gravity of mix (*SGM*), voids in mixed aggregate (*VMA*), and theoretical voids in mix (*VIM*) were calculated using Eqs 3-4 to 3-7.

$$CDMA = \ CDM\ (100 - b)/100 \quad (3\text{-}4)$$

where b: the percentage of bitumen by weight of the total mix.

$$SGM = \ 100/(\sum_{i=1}^{n} \frac{P_i}{SG_i} + \frac{P_{binder}}{SG_{binder}}) \quad (3\text{-}5)$$

where: P_{binder} is the percentage by weight of the total mix of binder; and SG_{binder} is the specific gravity of binder.

$$VMA = \frac{SGMA - CDMA}{SGMA} \times 100 \quad (3\text{-}6)$$

$$VIM = \frac{SGM - CDM}{SGM} \times 100 \quad (3\text{-}7)$$

3.5.3.3 Mix stiffness

Although bituminous mixtures are viscoelastic materials, at typical traffic speeds and road temperatures their behaviour is practically elastic (Nunn 1997). The elastic stiffness modulus of a bituminous mixture is a good indicator of its load spreading

ability. Moreover, it controls the level of tensile strain induced in the underlying layers. Therefore, a material with a high elastic stiffness modulus will have a good load spreading ability which reduces the deflections due to the passing traffic and the tensile strains in the underlying layers.

Figure 3-13: ITSM test using NAT machine.

The Indirect Tensile Stiffness Modulus (ITSM) (BSI 2004) was determined using a Nottingham Asphalt Tester (NAT), Fig 3-13. In this test, specimens, five of 100 mm diameter and 65 mm height, were subjected to a transient load pulse across their vertical diametric axis and the resultant transient deformations along the horizontal diametric axis were measured using Linear Variable Differential Transducers (LVDTs). Measurements were taken in two orthogonal orientations. The test was carried out at a test temperature equals 20 ^{0}C which is the standard ITSM test

temperature. To ensure that the specimens were at the right temperature, specimens were conditioned overnight inside the temperature controlled cabinet of the NAT machine.

The ITSM test is controlled by a computer which recorded measurements taken by LVDTs and calculated stiffness (Schmidt 1972) using Eq 3-8.

$$E = Q\,(0.273 + \upsilon)/(\Delta h \times t\,) \quad (3\text{-}8)$$

where: E is the stiffness modulus of the material (N/mm^2); Q is the peak vertical load (N); Δh is the peak transient deformation along the diameter axis (mm); t is specimen thickness (mm); and υ is Poisson's ratio (assumed to be 0.35).

3.5.3.4 Control mix design results

Results corresponding to the binder contents investigated are presented in Fig 3-14. These results are based upon the average value obtained from five specimens. Average values and standard deviation of these measurements are presented in Table 3-6. These mix design parameters indicated that:

- The VIM decreased with the increase in binder content. This is due to the fact that adding more binder fills more voids.
- The CDM reached its maximum value at approximately 4.5 % binder content.
- The VMA were at their lowest at, again, 4.5% binder content.
- The ITSM decreased with increasing of binder content, as expected, due to the lubricating effect of the bitumen.

As a result, 4.5% binder content was chosen to be the optimum binder content (OBC) for the control mix.

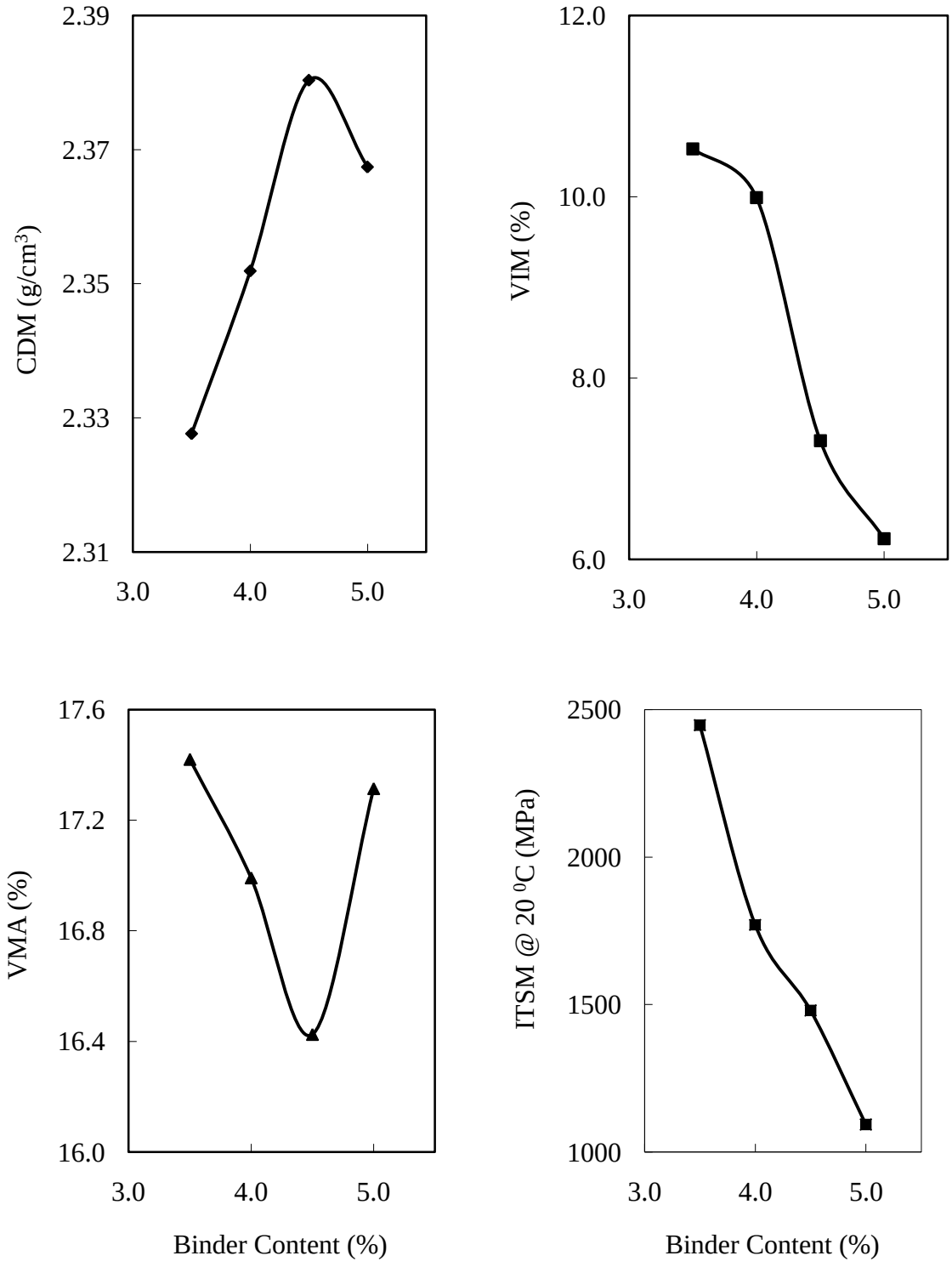


Figure 3-14: Control mix design parameters.

Binder content (%)	CDM (g/cm3)		VMA (%)		VIM (%)		ITSM (MPa)	
	Average	STD	Average	STD	Average	STD	Average	STD
3.5	2.327	0.012	17.42	0.43	10.53	0.46	2447	212.8
4.0	2.352	0.009	16.99	0.35	9.99	0.38	1770	122.6
4.5	2.380	0.008	16.42	0.31	7.31	0.35	1480	190.9
5.0	2.367	0.013	17.31	0.48	6.23	0.54	1093	71.9

Table 3-6: Control mix design parameters

3.5.4 IBAA mixtures design

Like the control mix, bituminous mixtures containing 30, 60 and 80% IBAA content were manufactured and tested. Theses mixtures were called AA, BA and CA respectively. The results, shown in Fig 3-15 and Table 3-7, indicated that the recommended optimum binder contents are 5.5, 6.5 and 7.5% respectively. The following are relevant remarks on these outcomes:

- Mixes AA and BA had the same trends as the control mix. These trends are typical for bituminous mixtures. However, mix CA, which contained 80% IBAA, had its own unique trend as the CDM increased at binder content higher than OBC and the VMA decreased. The VIM and ITSM values were acceptable. The reasons for this behaviour are not entirely known. One of the reasons may be that this mixture may have undergone some crushing. The reason of this crushing could be due to the behaviour of some metallic and glass components which exist in high proportions in mix CA. At low binder contents, the behaviour of mix CA was not uncommon except the ITSM value which was low compared with that at higher binder content. The reason for this was clearly due to the poor coating of bitumen. This poor coating was very noticeable during manufacture. The main reason for this phenomenon is

the presence of ceramic, glass and porcelain in high proportions. These materials have very weak adhesion properties with bitumen.

- It was obvious that the more IBAA added, the higher the optimum binder content was and more voids resulted owing to the high absorption properties of IBAA. At the OBCs the voids filled with bitumen (VFB) were found to be 64, 56 and 53%, for mixes AA, BA and CA respectively, which indicated the high absorption properties of the IBAA.
- Adding IBAA significantly improves the mixtures' ITSM values, excluding mix CA.
- Mix design results showed that, in terms of IBAA mixtures, mix AA, with 5.5% OBC and 7.4% voids content, is suitable for binder course layers in flexible pavements according to the UK specifications, which allow up to 8% VIM. On the other hand, mixes BA and CA were found to have high OBC and voids content values.

Binder content (%)	**ITSM (MPa)**		**CDM (g/cm3)**		**VIM (%)**		**VMA (%)**	
	Average	**STD**	**Average**	**STD**	**Average**	**STD**	**Average**	**STD**
5.5	1805	103.1	2.293	0.017	7.421	0.71	20.35	0.61
6.5	2138	96.7	2.177	0.016	11.01	0.66	25.17	0.55
7.5	1700	165.8	2.063	0.009	14.04	0.38	29.85	0.31

Table 3-7: IBAA bituminous mixtures design parameters

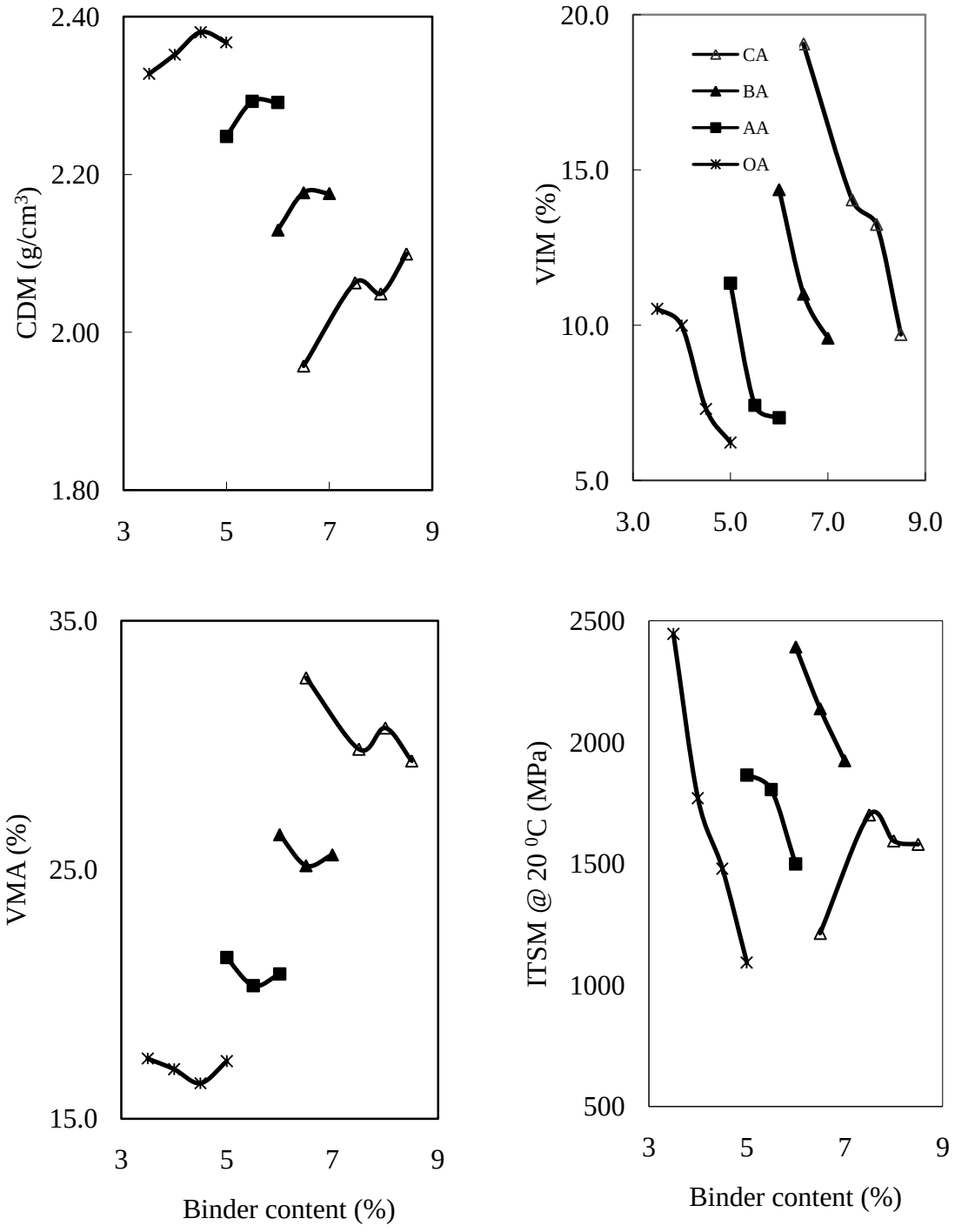


Figure 3-15: IBAA bituminous mixtures design parameters.

3.5.5 Moisture sensitivity study

Bituminous mixtures should have an acceptable resistance to changes caused by the ingress of moisture. Water entry may result in loss of adhesion between bitumen and aggregates, which can lead to a reduction in the strength of the mixture. In most moisture sensitivity tests, coated aggregate is soaked in water under controlled conditions of time and temperature. Stiffness measurements are taken before and after immersion in order to assess the mixture susceptibility to water ingress. One of these regimes is that stipulated by the British Board of Agrément (1998).

The test procedure used can be summarised as follows. Firstly, ITSM of the unconditioned specimens, cylindrical specimens of 100 mm diameter and 65 mm height, was determined. The specimens were then placed in a vacuum desiccator, shown in Fig 3-16a, at 20 ^{0}C, covered with distilled water and a partial vacuum of 510 mm Hg was applied for 30 minutes. After which the specimens were moved from the desiccator and placed in a water bath, shown in Fig 3-16b and c, at 60 ^{0}C for 6 hours. After that, the specimens were immediately moved to a cold water bath at 5 ^{0}C for 16 hours, and then placed in a moderate temperature bath at 20 ^{0}C for 2 hours. This thermal conditioning cycle, 6 hrs at 60 ^{0}C, 16 hrs at 5 ^{0}C and 2 hrs at 20 ^{0}C, was repeated three times one after the other immediately. During these steps, temperatures were monitored using a computer. At the end of the last cycle, the specimens were conditioned overnight at 20 ^{0}C and their retained stiffness was measured.

The retained stiffness results, shown in Fig 3-17, indicated that all mixtures suffered a loss in their stiffness values due to moisture effects. However, the retained stiffness ranged from 60 to 70% over a narrow range of binder contents. As a result, the IBAA could be considered as having a minor reduction effect on ITSM due to water ingress.

Figure 3-16: Moisturizing assembly.

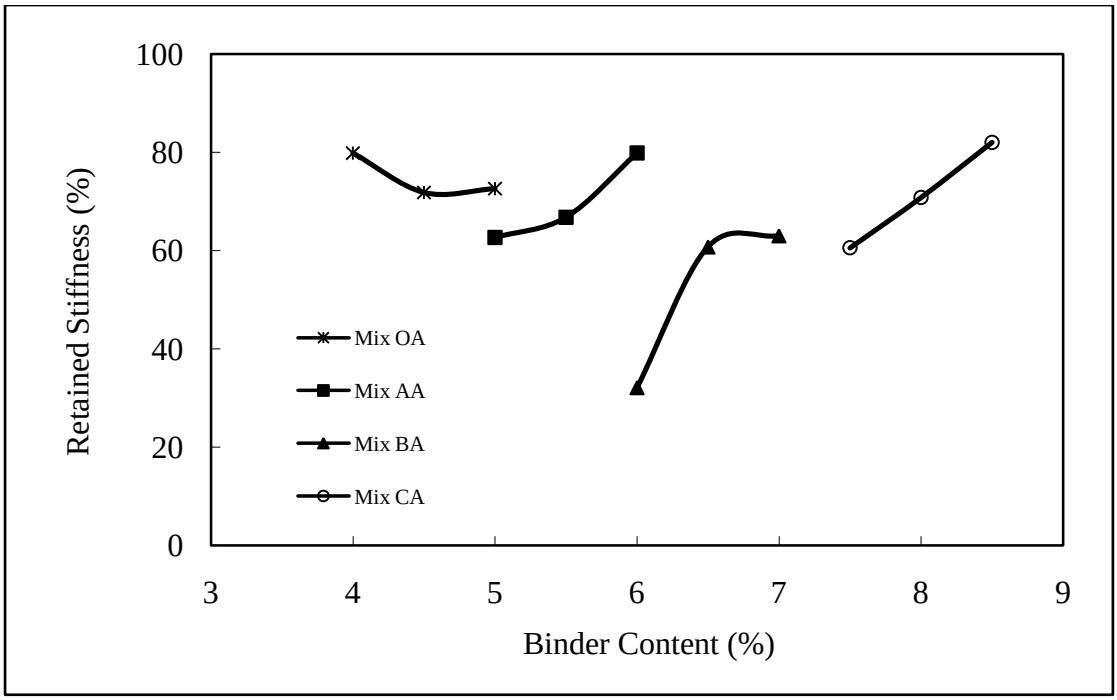


Figure 3-17: Retained ITSM values after moisturizing.

3.5.6 Ageing effect study

A laboratory accelerated curing test was conducted with the aim of relating the in-service stiffness modulus of bituminous mixtures to their initial stiffness modulus measured soon after manufacturing. The protocol used was presented by the UK Highways Agency (Chaddock and Pledge 1994) in which the specimens were mounted in the percentage refusal density (PRD) moulds and then heated in a temperature controlled forced air draft oven at 60 ^{0}C for 48 hrs. Ordinary 100 mm diameter compaction moulds, which are similar to PRD moulds, were used in this study, as shown in Fig 3-18. The stiffness of the specimens was measured before and after curing to predict the stiffness after one year in service.

Fig 3-19 shows that mix AA, at its OBC, underwent least change in stiffness due to ageing, followed by mixes CA, OA and BA at their OBCs. This can be attributed to the fact that mix AA had the lowest voids, which aided in resisting ageing effects. Mix CA, although had highest voids, the binder film was thinnest as most of the binder was absorbed by the large proportion of IBAA in the blend. Thus, the effect of the binder was minimal. Mixes OA and BA showed commensurate ageing to their respective voids contents. The increase in ITSM was in the range of 5 to 15% for mixes OA, AA and CA, at their OBCs, which is good from the durability viewpoint. On the other hand, for mix BA, the increase was about 30%, at its OBC, which is not good at low temperature conditions as the material may become more brittle and fracture can happen earlier. However, the rate of increase of the ITSM is expected to diminish after the first year of mixture production (Said 2005).

Figure 3-18: Samples in moulds during ageing.

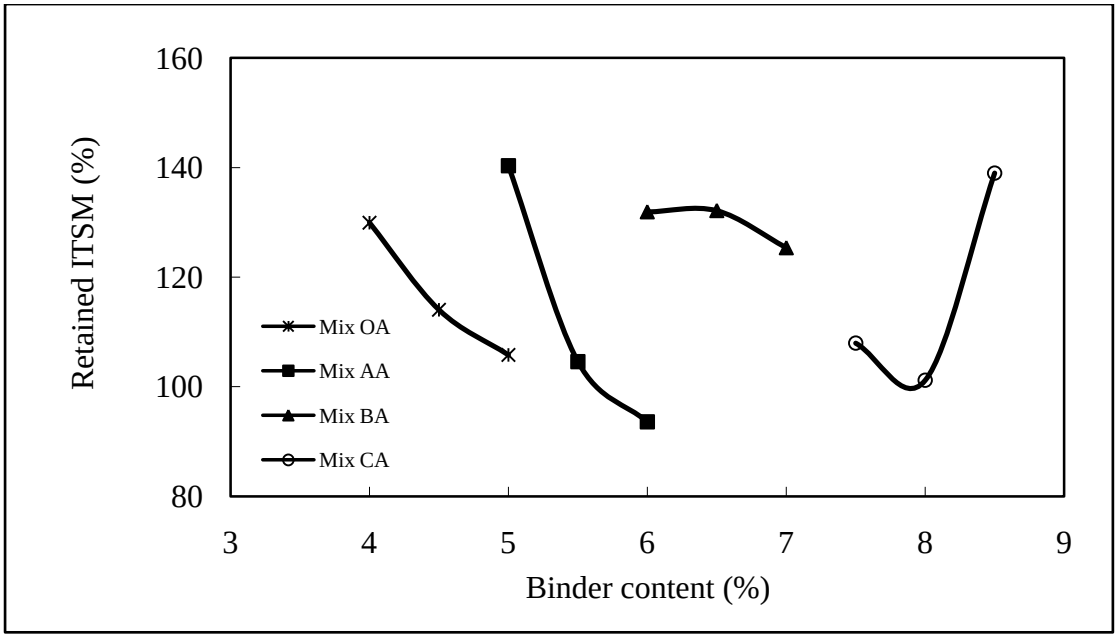
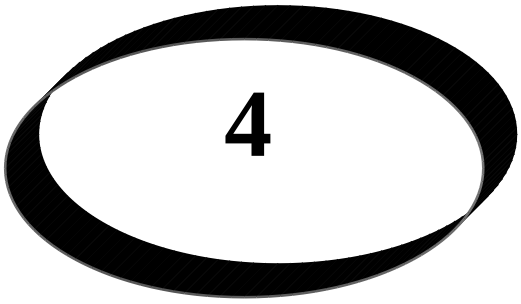


Figure 3-19: Retained ITSM values after ageing.

3.6 CONCLUDING REMARKS

From the preceding sections, the following remarks could be made:

- Using IBAA at 30 and 60% in bituminous mixtures is suitable for binder course in flexible pavements. Nonetheless, relatively high binder contents of 5.5 and 6.5%, respectively, were required for adequate performance.
- The 80% IBAA mixture seemed to have a unique trend regarding the effect of binder on design parameters. Despite this, using 7.5% as optimum binder content seems to fulfil the mixture requirements.
- Addition of IBAA significantly improves the mixtures' ITSM values, excluding mix CA, at 80% IBAA.
- The control mix retained stiffness, after a water immersion regime, was found to be 70%. Using IBAA led to a reduction in the retained stiffness of the mixtures. However, this reduction was less than 10% compared with the control mix.
- IBAA mixtures' stiffness increased when subjected to an ageing protocol. This increase was significantly high at 60% IBAA, whereas the 0, 30 and 80% mixtures underwent insignificant changes to their stiffness values.



ENVIRONMENTAL ASPECTS

4.1 INTRODUCTION

This chapter considers the leaching process and the mechanisms and factors controlling leaching. The fundamental leaching parameters are discussed and the common tests to examine leaching are categorised and reviewed. Then, data obtained from agitated extraction, tank and availability tests are presented and analysed. Finally, theoretical leaching models are reviewed and the applicability of a diffusion model to leaching tests obtained data is examined.

4.2 LEACHING IN THE ENVIRONMENT

Leaching is the process by which soluble components, such as salts, nutrients, chemicals, organic matter, metals, or any other contaminants, are released from the solid phase into the water phase. This process is due to the action of a number of factors, e.g. rain, which lead to washing the releases into a lower layer or dissolve and carry them away. The fluid which initiates the leaching process is called leachant while the resulting fluid which contains the released contaminates is called leachate or

elute. The released elements are known as constituents. The process itself is universal, as any material exposed to contact with water would leach.

Generally, it is known that one of the most important environmental risks related to the use of materials, especially new ones, for road construction purposes is the leaching potential of their contaminants, and subsequent transport of released constituents from the material into the environment. The constituents may be released due to the action of water, rain or groundwater movements. These constituents are transported, again, by water to affect the surrounding environment such as groundwater, surface water and/or verge vegetation and subsequently humans.

In pavements, it is not uncommon to monitor vertical drainage to road bases. Water penetrates the pavement mainly via surface cracks and broken bitumen films. However, sometimes it passes through construction imperfections (Peploe and Dawson 2006). This phenomenon, which is a leaching process in reality, is causing contaminates release. The released constituents may target lower layers, the groundwater table, surface waters, i.e. rivers, streams, lakes, etc., or other elements around the road such as verge vegetation. The final target is the human beings who may use pavement release receptors. Therefore, it is fundamental to assess the leaching potential for any aggregate materials when used in a road construction. This importance may be considered as vital when using a new material. This is the case in this study when IBAA is used as aggregate materials for roads. The potential impact on the environment of such release due to the use of IBAA as a road construction material is vital and requires due attention.

4.3 LEACHING MECHANISMS

There are two leaching mechanisms for the release of constituents from pavement to the water phase. These mechanisms are chemical and physical and in most cases a combination of both controls the constituents' release. A summary of these two mechanisms is presented in the following paragraphs.

Chemical mechanism may take place by dissolution of minerals, adsorption or availability (van der Sloot and Dijkstra 2004). In general, metal oxides leach by dissolution while heavy metals are controlled by adsorption. A number of inorganic constituents, such as very soluble salts, are not very reactive and show neither solubility control (dissolution) nor sorption control (adsorption). Upon contact with water they will dissolve instantaneously and quantitatively. Those elements are availability controlled, as the total available concentration can be released from the product.

Physical mechanism may take place by diffusion, percolation, dispersion, or surface wash off. In compacted non porous materials, such as bituminous mixtures, diffusion is the most potential physical mechanism (IAWG 1997). In diffusion, the constituents move solely as a result of the movement of molecules in the absence of flow, i.e. constituents move regardless of the motion of the medium they are in, headed for a less concentrated medium. Diffusion can be an end process or a step prior to percolation which takes place usually in granular materials. In granular permeable materials, rainfall causes water percolating. Rain water takes the constituents through the material to the soil or groundwater, i.e. contaminants will be transported by the liquid medium they are in, with a flow rate the same as that of the medium. This process is sometimes called advection (Conner 1990). Surface wash off is similar to advection; however, it is related to the outside surface of materials. Dispersion is close to advection; however, the path of the constituents will depend on the solid they are moving through, e.g. in soils, the constituents will move around particles and through pore spaces with different flow rates depending on the route they take (Taylor 2004).

4.4 FACTORS CONTROLLING LEACHING

Leaching from any material is dependant on many factors which affect the quantity of released constituents from a solid phase. These factors may be categorised as physical, chemical and biological factors (IAWG 1997, Hill 2004, Abbott et al 2003, Townsend et al 2003, van der Sloot and Dijkstra 2004). A summary of these factors is

presented in Table 4-1. The most important factors are discussed in the following sections.

4.4.1 Physical factors

4.4.1.1 Particle geometry

Particle geometry of a material determines the surface area exposed to the leachant. The larger the surface area is, the higher the chance of contaminants to leach. Moreover, particle size plays an important role in determination a constituent travel distance as constituents travel from the centre of a particle to its surface to come in contact with the water phase. This means that small particles may leach more than large particles.

4.4.1.2 Porosity

The ratio of the pore space to the total volume, which is known as material porosity, controls the rate of constituents transport in the direction of the water phase. It is obvious that higher porosity enables higher, or at least faster, leaching.

4.4.1.3 Tortousity

Tortousity of a material is the ratio between the actual leaching path length between two points and the theoretical straight line between the same points. Tortousity is a function of diffusion coefficient, which will be discussed later in this chapter, and the material porosity. High tortousity means lower release.

4.4.1.4 Permeability

Permeability is a well known material parameter which shows the velocity by which a constituent may release with a leachant. Permeability is related to material compaction and binding and it is affected significantly by compaction time, energy, method, and binder properties. Material with high permeability may release higher

constituents i.e. low permeability results in lowering the leaching potential of any material.

4.4.1.5 Sorption

Sorption is a term which describes a weak bonding of a constituent in leachant into the material surface via sorption reactions. The strength of this bond is dependant on the chemical nature of the constituent and surface as well as the general environment such as pH and reduction oxidation (redox) potential; see sections 4.4.2.1 and 4.4.2.2. These factors have a significant effect on the likelihood of a sorbet constituent to be desorbed again.

4.4.1.6 Erosion

Erosion of monolith material may transpire on account of the action of weathering or service life. Erosion leads to an increase in the surface area, in addition to exposure of new surfaces to leachant. Erosion results in increasing release potential.

4.4.1.7 Time

Time is a very important factor in leaching. Time of use of a material in an application affects its cumulative release. Time of contact between a material and a leachant, leaching time, affects the release rate unless equilibrium is established. In addition, material and leachant properties may vary with time. Therefore, it is very important to specify a time scale when studying leaching.

4.4.1.8 Liquid to solid ratio

The liquid to solid ratio (L/S) is defined as the ratio of the amount of leachant in contact with solid to the amount of this solid. This ratio plays an important role in controlling the quantity of the released constituents. Generally, chemical concentrations are higher at low L/S and decrease as L/S increases because of less dilution of material with leachant.

4.4.1.9 Temperature

Generally, temperature increase results in an increase in solubility and chemical reaction rates. This may, subsequently, result in an increase in the leaching potential.

4.4.2 Chemical and biological factors

4.4.2.1 pH

The pH of the material and that of its environment and, subsequently, the pH of the overall system are fundamental in determining the release of many constituents. This is valid for all sorts of materials. The system's pH is controlled by the solid material at low L/S and by the leachant at high L/S. The pH value of the surrounding fluid determines the maximum water phase concentration. The strong influence of pH on leaching is because of the dissolution of most minerals, as well as sorption processes, is pH dependent. This means that the release of almost all contaminants that are solubility controlled or sorption controlled, show pH dependent release.

4.4.2.2 Reduction oxidation potential

The potential of reduction oxidation, also known as redox, shows the possibility for material conversion between its oxidation states. Redox affects material solubility and its leaching potential. For heavy metals, the oxidation of an initially reduced material usually enhances leached amounts while later reduction will have the opposite effect. Under oxidising conditions, i.e. in the presence of oxygen, metal oxides are poorly soluble and hence are important for immobilising metals in the solid phase. Under reducing conditions metal oxides have an increased solubility. Conversely metal sulphides are generally insoluble under reducing conditions. However, they are more soluble under oxidising condition, although solubility is in general low for metal sulphides under all conditions.

4.4.2.3 Equilibrium conditions

When a constituent concentration in the water phase approaches the same level as its concentration in the solid phase, an equilibrium condition is achieved. This may occur as a result of long leaching time or rapid dissolution kinetics. At equilibrium, theoretically, release rate approaches zero.

4.4.2.4 Complexation

Complex formation occurs when a number of molecules are bonded to ions, usually metal. A common example of such complexation is metal/organic acid complexes. This enhances the dissolution of the constituents as the resultant complexes does not contribute to the normal equilibrium balance. Therefore, more release is required to approach equilibrium. Organic compounds are the most common comlexing agents and copper is the most susceptible metal to be complexed. Therefore, high organic matter levels render high leaching potential in terms of metals especially copper.

4.4.3 Effect of binding on IBAA leaching properties

There are three known techniques to achieve waste materials stabilisation. These are physical immobilisation, chemical stabilisation and thermal vitrification. In the UK, only the first technique is used for IBAA in roads while the latter techniques are used for pre-treatments for landfill purposes (Abbott et al 2003). The IBAA physical immobilisation takes place using either cement or bitumen. Cement is used in the production of cement bound materials (CBM) which are not uncommonly used as base layers in UK pavements. Binding IBAA with cement, although it is a physical immobilisation, could be considered as chemical binding as well. This is due to the constituent binding with the calcium silicate hydrate formed by cement hydration (Allerman et al 1982). Leaching of IBAA in CBM was suggested to be controlled by physical mechanism (diffusion control) rather than chemical mechanism (Abbott et al 2003). This is because of the high pH of cement matrices which leads to overall low leaching (Glasser 1994, Gerdes and Wettmann 1994, IAWG 1997, Frantz and Demars 2000). This conclusion was confirmed by several studies found in the literature. Pain et al (2002) studied the leaching potential for CBMs which contained IBAA between

0 and 100% with a range of cement between 2 and 10%. This study concluded that the leaching potential for IBAA CBMs was lower than regulatory drinking water limits. However, using 2.5% cement provided much higher leaching potential when compared with a reference concrete mix (Cai et al 2004). Regarding the long term leaching impact, it was shown that mixing IBAA with cement caused insignificant problems (Johnson 2000).

Binding IBAA with bitumen is not common in the reviewed literature. However, it is known that such incorporation may provide physical isolation of IBAA via surface coating by bitumen which reduces the contact with the leachant. In addition, this binding reduces the pore space and permeability and causes changes in water chemistry which affect the mobility of many components of IBAA. All these factors lead to less leaching potential. This fact was concluded in a number of studies. (Knights and Johnson 1995, Plaue et al 1996, Eighmy et al 1997, Frantz and Demars 2000, Thayumanavan et al 2001). Moreover, Musselman et al (1996) showed that no detectable difference was found between the leaching of asphalt made with ash or traditional natural aggregates. The environmental impacts of bituminous mixtures containing IBAA have not been covered abundantly in the literature. From those studies that have tackled this subject it was found that encapsulating IBAA by bitumen in a well compacted asphalt mix created very little environmental risks. However, concerns with effects of the engineering properties and their subsequent effects on leaching were raised (Huang et al 2006). One key aspect to the use of bitumen is the good sealing of the ash within the asphalt matrix. The use of advanced mixing arrangements, such as foaming, have been found to improve the particle coating and results in greater than 95% reduction of leaching (Dijkink 1994). In contrast to the former studies, Hill and Dawson (2000) found that, sometimes, binders have the potential to introduce further and more significant constituents to the environment. Examples for such constituents were zinc and iron (Min et al 2003), and chloride (Bai and Basheer 2003). Therefore, it can be concluded that, at present, there is a limited understanding of how bitumen can control leaching of contaminates form IBAA in road construction.

Physical factors	Chemical and biological factors
➢ Material related factors: • Particle geometry • Material porosity • Material tortousity • Material permeability • Material sorption • Density differences between material components • Material homogeneity degree • Material sensitivity for erosion ➢ Leachant related factors: • Percolation occurrence • Leachant flow rate ➢ Material/leachant contact related factors: • Rate of contact between material and leachant • Time of contact between material and leachant • Liquid to solid ratio (L/S) ➢ Leaching environment related factors: • Temperature • Hydrological conditions • Saturation degree	➢ Material related factors: • Material pH • Contaminates leachability • Internal chemical interactions • Surface dissolution • Reaction kinetics • Organic matter • Dissolved organic carbon • Reduction oxidation (redox) potential • Material degradation • Pore clogging ➢ Leachant related factors: • Leachant pH • Leachant chemical composition • Complexation ➢ Leaching environment related factors: • Environment pH • Redox potential • Changes in the chemical properties • Equilibrium conditions

Table 4-1: A summary of the factors controlling leaching

4.5 FUNDAMENTAL LEACHING PARAMETERS

One of the most popular approaches to assess the leaching potential of a material is through studying its fundamental leaching parameters. These parameters can be used to measure the constituent release under different environmental conditions. There are three main fundamental leaching parameters, namely availability, solubility and diffusion.

4.5.1 Availability

Availability can be expressed as the quantity of each component that may be leached from a material under environmentally extreme conditions. Availability can be also perceived as the maximum quantity of a constituent that can be released into elutes under aggressive leaching conditions. These conditions may express, in theory, leaching over a 1000 to 10000 years (Netherlands Normalization Institute 2004a). Once the available quantity of specified constituents has been leached, no more release will occur. Under availability controlled conditions, which occur at high L/S, the resulting leachate is at a lower concentration than the saturation condition for the constituents. It is noteworthy to mention that the higher the L/S the lower the concentration of a constituent in a leachate (Hill 2004). The availability of a constituent may reach up to 100% of the total quantity present in the solid material. This is, however, not common as the availability, usually, is significantly less than that, even under sever leaching conditions (van der Sloot 1991). Availability may be measured using different tests, e.g. the availability test EA NEN 7371 (Netherlands Normalization Institute 2004a).

For bottom ash, the availability of specified constituents, e.g. Si, Ca and Mg, can be considered insignificant while other constituents, such as Pb, have availability which is significantly less than the total content of the element. The case is different with some other constituents, such as Cl, which have availability approximately equal to the total content. Determination of availability, however, does not indicate whether or not this maximum quantity of a particular constituent will be released, or over what precise time interval the release will occur for the environmental exposure scenario of

interest (IAWG 1997). The availability of bottom ash constituents is presented in Table 4-2. The data in this table summarises results from Canada, Denmark, Germany, The Netherlands, Sweden and USA.

Element	Total content (mg/kg)		Availability (mg/kg)	
	min	max	min	max
Ca	50000	90000	20000	70000
Cl	1000	3000	1000	3000
K	7000	20000	1000	4000
Mg	10000	30000	1000	6000
SO_4	12000	30000	8000	18000
As	5	40	0.3	5
B	80	300	50	200
Ba	500	1800	50	200
Cd	2	25	0.5	5
Cr	200	1000	2	10
Cu	1200	2500	50	200
Hg	0.5	1	0.01	0.1
Mo	5	30	1	4
Pb	1500	3000	50	300
Sb	30	200	1	2
Zn	2000	4000	50	500

Table 4-2: Typical bottom ash availability (IAWG 1997)

4.5.2 Solubility or pH dependency

Solubility can be defined as “the maximum quantity of one phase dissolved by another under specified conditions” (Sharp 1990). In terms of leaching, the solubility of a constituent will control the maximum concentration of that constituent in a leachate. Solubility control occurs when the leachate is saturated in terms of the constituents. This occurs, usually, at low liquid to solid ratio. The most important

factor which controls the solubility is the pH of the leachant. The relationship between the pH and the L/S has found to have a significant effect on the solubility as the initial pH of a leachant may differ from that of the resulting leachate which changes the solubility. This change of the pH value was reported to be very significant at low L/S while the initial pH value is the important factor in high L/S leaching process (van der Sloot et al 1997).

Different constituents showed different trends in terms of their solubility. The solubility of most heavy metal, e.g., Pb, Cd, Zn, is a function of the solution's pH value (Kosson et al 1996). Solubility of a particular element can also be increased by the presence of agents such as chloride or acetate, or reduced by the presence of sulphate or sulphide. Solubility is typically measured using a low L/S. Two test methods which have been used to measure solubility as a function of solution pH are the pH-stat test (Commans et al 1993) and the acid neutralization capacity leach test (Environment Canada 1986).

The solubility of many IBAA constituents was studied, as a function of pH, on numerous occasions (Versluijs et al 1990, Sawell et al 1989, Eighmy 1993, Johnson et al 1996, Kosson et al 1996, van der Sloot 1996, IAWG 1997, Miema and Comans 1997, Dijkstra et al 2006). For example, Kosson et al (1996) reported that the minimum solubility for lead was observed at pH between 8 and 10 while chloride was relatively independent of pH and released rapidly as a function of L/S. Moreover, it was noticed that, for Zn and Cd the plateau is reached at pH 5-6 and for Pb at around pH 4. For Cu the trend is less obvious. The maximum concentrations of Cu, Pb, Cd and Zn represent 25, 50, 45 and 70% respectively of the total metal content of the IBA. This is shown in Fig 4-1 (Johnson et al 1996).

4.5.3 Diffusion

Diffusion is defined as the process by which matter is transported from one part of a system to another as a result of random molecular motions (Artamendi and Khalid 2006). Constituent release is categorised as diffusion controlled (Kosson et al 1996) when it is limited by the rate at which the constituents leach from the material. The

leaching of constituents with low mobility is most likely to be diffusion controlled while the leaching of constituents with high mobility is most likely to be availability or solubility control. The diffusion control leaching is more likely to occur in monolithic materials, e.g. bituminous mixtures, CBMs and well compacted granular materials.

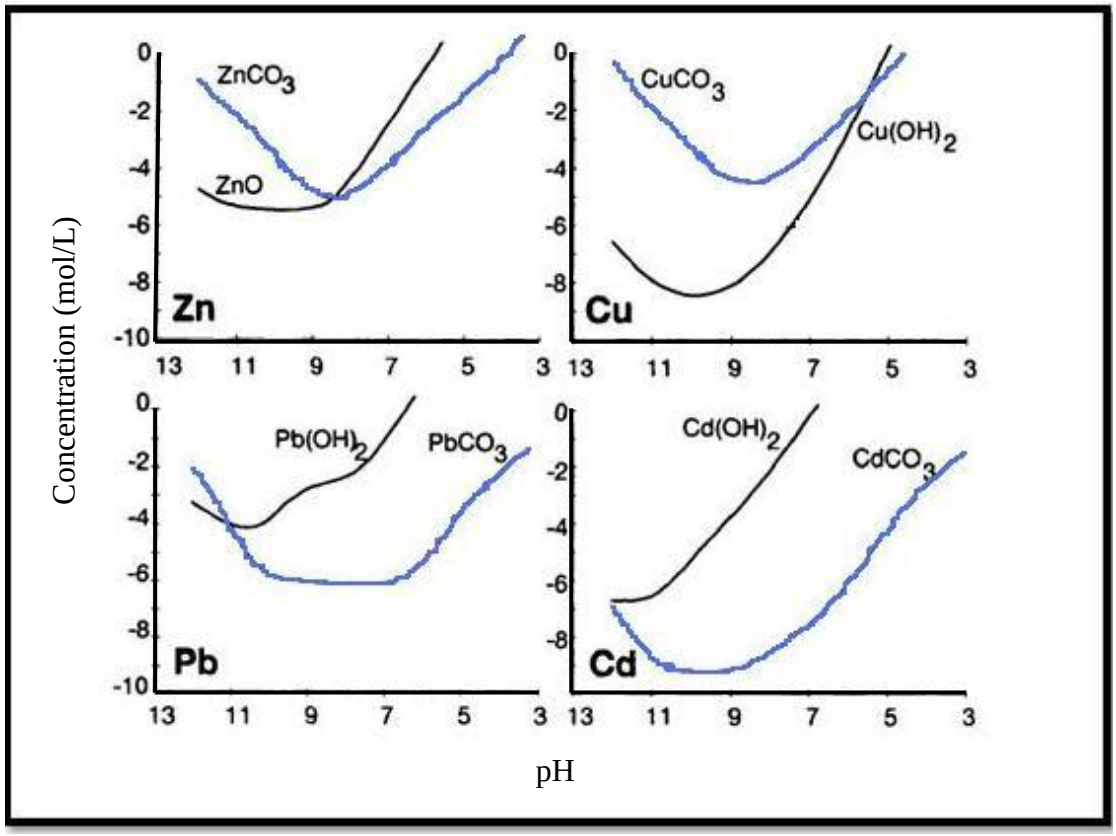


Figure 4-1: Laboratory determined Zn, Cu, Pb and Cd concentrations, with respect to carbonates and hydro-oxides, presented as a function of pH for IBA (Johnson et al 1996).

The mathematical theory of diffusion (Crank 1975) is based on Fick's second law (1855). This law is used in non-steady or recurrently changing state diffusion, i.e. when the concentration within the diffusion volume changes with respect to time. This may be expressed mathematically as shown in Eq 4-1. To estimate the diffusion coefficient, it is possible to apply a one-dimensional semi-infinite linear diffusion model (IAWG 1997) under a few principles. These principles assume uniform material in composition, no depletion occurs, i.e. no decreasing in the available level

of constituents, and maximum concentration gradient between the solid and the water phases.

$$\frac{\partial \phi}{\partial t} = D \frac{\partial^2 \phi}{\partial X^2} \quad (4\text{-}1)$$

where: ϕ is the concentration of a constituent (mol/m^3); t is the leaching time (sec); D is the diffusion coefficient (m^2/s); and X is the distance from the leaching region in the material to its surface (m).

The solution of Fick's second law, in accordance with the above-mentioned assumptions for diffusion of semi-infinite materials, is presented in Eq 4-2. It was found (IAWG 1997) that using C_1 = 0 for t > 0 is appropriate for monolith experimental conditions. Thus, the resulting diffusion equation is shown in Eq 4-3.

$$\frac{C - C_1}{C_i - C_1} = \text{erf}\left(\frac{X}{2\sqrt{Dt}}\right) \quad (4\text{-}2)$$

where: C is the concentration as a function of location within the solid phase (mg/kg); C_0 is the initial concentration at zero time (mg/kg); and C_i is a constant concentration at material surface (mg/kg).

Note: erf is a mathematical jargon that expresses the "error function" (Crank 1975).

$$D = \pi M_t^2 / 4t(\rho C_0)^2 \quad (4\text{-}3)$$

where: M_t is the cumulative release for a constituent (mg/m^2); C_0 is the constituent's experimentally determined availability (mg/kg); and ρ is the bulk density of the material (kg/m^3).

For IBA, the diffusion coefficient was determined for compacted granular materials and paving blocks (IAWG 1997) and the values, for the different constituents, are presents in Table 4-3 in terms of leachability index values, *pD*. The leachability index values are the common way to express the diffusion coefficient as $pD = -logD$.

Element	*pD* (-log m²/s)	
	IBA (Granular materials)	**IBA (Paving blocks)**
Al	14.06	N/A
Ba	12.3	3.9
Br	10.05	N/A
Ca	12.86	14.1
Cd	>15	>14
Cr	11.73	>13
Cu	>14.8	13.6
Fe	14.2	N/A
K	10.12	10.4
Li	11.93	N/A
Mg	14.66	N/A
Na	10.24	10.4
Ni	>13	N/A
NO_3	11.73	N/A
Pb	16.17	16.4
Si	14.49	N/A
SO_4	15.62	N/A
Zn	15.71	16.1

Table 4-3: Leachability index for compacted granular materials and paving blocks comprising bottom ash (After IAWG 1997)

4.6 LEACHING TESTS

Leaching tests were originally developed to consider the short term environmental impact of solid waste disposal to landfill and for contaminated land purposes. In any leaching test, the tested material is brought into contact with a fluid known as leachant under specific test conditions. The analysis of the resultant leachate should be used as indicator for the leaching potential of the material contaminates. The main aspect in a leaching test is its ability to provide data from which a reasonable assessment for the material risk, to human or environment and prior to utilisation, could be interpreted. The range of leaching tests is vast as a large number of leaching tests have been developed worldwide. However, there are a limited number of common, well recognised and credited tests which were found in the literature. These tests have been discussed in the following sections.

It is noteworthy to highlight that none of these tests, however commonly used, can give data similar to the actual construction. This is mainly due to the fact that construction comprises different parts and materials which are impossible to simulate in leaching tests. Moreover, the water movement in a construction is completely different, in flow rate and chemical effects, from the leachant in leaching tests. In addition, the time scale is completely different. A leaching test may last for hours or days while a real construction should perform satisfactorily for decades.

4.6.1 Objectives of leaching tests

The basic objectives of leaching tests are:

- Classify materials as hazardous or non hazardous.
- Assess the leaching potential of contaminates.
- Simulate site leaching scenarios.
- Assess treatment effectiveness of a material.
- Identification of leachable constituents.
- Determine the fundamental leaching parameters.
- Establish a risk assessment for a material.

4.6.2 Categories of leaching tests

Leaching tests could be classified into two broad categories (Environment Canada 1990, Lewin et al 1994, IAWG 1997) on the basis of whether or not the leachant is renewed.

- Extraction tests:

  In these types of tests, the leachant is contacted with the material for a specified time without leachant renewal. The leachate is analysed, in most tests, at the end of the test. A fundamental assumption in this type of tests is that an equilibrium condition is achieved at the end of the test. Four extraction subcategories were found in the literature as shown in Table 4-4.
- Dynamic tests:

  In these types of tests, the leachant is continuously or intermittently renewed. The purpose of this renewal is to maintain a driving force for leaching. The leachant is analysed at the end of each test period to provide information about kinetics of solid phase dissolution and contaminant fluctuation as a function of time. These types of tests are very helpful in investigation of the complex leaching mechanisms. Again, four dynamic test subcategories were found in the literature, Table 4-4, and have been discussed, in addition to the extraction tests, in the following sections.

4.6.2.1 Agitated extraction tests

Agitated extraction tests are performed to achieve steady state conditions as speedily as possible. These tests measure the chemical properties of the material-leachant system. The test duration is short and an equilibrium condition is assumed to be reached by reducing the sample size and using agitation. These factors ensure higher exposed surface area and homogenous mixture. Moreover, it enhances the L/S. The most common tests of this type are:

- Extraction procedure toxicity test, EP-Tox (US environment protection agency 1990a).
- American society for testing and materials extraction test (ASTM 2006a).
- Toxicity characteristic leaching procedure, TCLP (US environment protection agency 1990b).

- Synthetic precipitation leaching procedure, SPLP (US environment protection agency 1990c).
- Waste extraction test, WET (California Code of Regulations, 1985).
- Multiple batch leach testing, MBLT (Environment Canada 1990).
- Availability test, EA NEN 7371 (Netherlands Normalization Institute 2004a).
- French leach test, AFNOR (AFNOR 1988).
- German leach test, DIN 38414 S4 (Institut fur normung 1984).
- National rivers authority interim test for contaminated soils, NRA (Lewin et al. 1994).

4.6.2.2 Non-agitated extraction tests

Non-agitated extraction tests are performed to measure the leaching physical mechanisms. They are similar to the agitated extraction tests nevertheless without using agitation. The main crisis with these tests is that the time required to reach an equilibrium condition may be much longer than that used for an agitated extraction test. Examples for these tests are:

- Small scale shaking leaching test, SLT (Belevi and Baccini 1989).
- Waste package interactions test, Sandia (Molecke 1983).
- Static leach test, MCC-1 (Krivovochev et al 2006).
- High temperature static leach test, MCC-2 (Krivovochev et al 2006).

4.6.2.3 Sequential chemical extraction tests

A sequential chemical extraction test is composed of a series of non-agitated extraction tests. It involves performing sequential leaching steps using one of two different methods. In the first method, different samples of the same material are exposed to different leachants which are increasingly more aggressive in terms of chemical attack to the material. In the other method, the same material sample is exposed to different leachants. The amount extracted in whichever method is related to a certain chemical form of mineral phase in the solid phase. It means that the results are not associated with chemical constituents' release. Examples for these tests are:

- Sequential chemical extraction test (Stegemann and Cote 1990).
- Standard test Method for sequential batch extraction of waste with water, D4793 (ASTM 2004a).
- Standard test Method for sequential batch extraction of waste with acidic extraction fluid, D5284 (ASTM 2004b).

4.6.2.4 Concentration build-up tests

Concentration build up tests endeavour to simulate the leachate contained within water extracted from a large body of material or the internal pore water concentration. This is achieved by carrying out successive leaching processes with the liquid however new batches of sample. This will allow build up of the salts and other materials such that they reach saturation. An example of this test is presented by Sawell and Constable (1993).

4.6.2.5 Serial batch tests

A serial batch test is conducting by mixing and agitating the material with the leachant for a specified duration prior to replacing the leachant with a fresh leachant until the number of leaching periods has been completed. The data obtained from this test can be used to establish an extraction profile. Examples for this test are:

- Multiple extraction procedure, MEP (US environment protection agency 1990d).
- Cascade availability test, NEN 7341 (Netherlands Normalization Institute, 1995).

4.6.2.6 Flow-around tests

In flow-around test, the flow of fresh leachant around the sample provides the driving force to keep leaching. Agitation is generally not performed while leachant flow may be continuous or intermittently renewed. Examples for this type are:

- Monolithic diffusion test, NEN 7345 (Netherlands Normalization Institute, 1994).

- Standard Test Method for Static Leaching of Monolithic Waste Forms for Disposal of Radioactive Waste, ASTM C1220 (ASTM 2004c).

4.6.2.7 Flow-through tests

In a flow-through test, leachant is passed, either continuously or intermittently, through an open cylinder, either small or large, of packed test materials over a long time, weeks or years. The results are used to assess the contaminant removal. Examples for this type are:

- Standard test method for leaching solid waste in a column apparatus, D4874 (ASTM 2006b).
- Column test, NEN 7343 (Netherlands Normalization Institute 1993).
- Tank test, EA NEN 7375 (Netherlands Normalization Institute 2004b).
- Lysimeter studies (Cranell and Eighmy 1988, Stegemann et al 1995, Kus et al 1999, Corwin 2000, Hansen et al 2000, Bruder-Hubscher 2001a, Takamatsu et al 2006).

4.6.2.8 Soxhelt tests

This type of leaching tests is conducting by continuously contact the material with fresh leachant without adding or removing leachant from the apparatus. It is used to obtain the maximum amount of a constituent leachable from a material. An example for this type is:

- Soxhlet leach test method, MCC-5 (Leroy et al 2000).

4.6.3 Variables of leaching tests

Designing of a leaching test should take into account some important variables. These variables are associated with the material, leachant, testing time and temperature. These variables could be summarised as:

- Sample preparation variables which take into consideration sample size reduction, surface wash, curing, and aging.

- Material properties such as porosity and permeability.
- Leachant composition which consider the pH, redox potential and the chemical composition.
- L/S which is associated with leachant volume, material mass and material surface area.
- Material leachant contact which consider shaking, stirring, tumbling, or gas bubbling.
- Leaching period which consider the time needed for equilibrium condition development.
- Leachate separation.
- Test temperature.

Test type	Extraction tests	Dynamic tests
Tests subcategories	➢ Agitated extraction tests ➢ Non-agitated extraction tests ➢ Sequential chemical extraction tests ➢ Concentration build-up tests	➢ Serial batch tests ➢ Flow-around tests ➢ Flow-through tests ➢ Soxhelt tests
Test duration	Short	Long
Operation	Relatively easy	Relatively difficult
Cost	Relatively low	Relatively high
L/S	Relatively high	Relatively low
pH	Easy to control	No pH control

Table 4-4: Summary of leaching test categories (After Environment Canada 1990, Townsend 2003)

4.7 IBAA LEACHING TEST RESULTS

Reviewing the literature showed plenty of studies concerned with IBA leaching properties in its raw nature or as a secondary material in non bituminous mixtures. Leaching of IBAA bituminous mixtures was investigated in very few occasions. The former and the latter studies were reviewed in Chapter 2. The shortage in the available data on leaching potential of IBAA in bituminous mixtures, especially when used at high content level, inspires the necessity to investigate this area. The leaching potential for mixes AA, BA and CA, presented in Chapter 3, was investigated by means of conducting two different leaching tests. Firstly, an agitated extraction test was carried out on mixes AA, BA, CA and their original non bituminous blends. Two main aspects were investigated from this test. The first was to detect the leachability of IBAA, when used in bituminous mixtures with different content levels, in terms of release quantity at equilibrium conditions. The second aspect was to examine the effect of bitumen content on this leachability. Secondly, a tank leaching test was conducted on mix CA and its non bituminous blend. The target of this test was to observe the cumulative IBAA release over a long time. Lastly an availability test was conducted to enable studying the fundamental leaching parameters.

4.7.1 Agitated extraction test results

A National Rivers Authority (NRA) recommended leaching test (Lewin et al. 1994) was adopted and used, in this study, to test mixes AA, BA and CA which were discussed previously in Chapter 3. Three samples of each mix were tested at three different binder contents, including their OBCs. Leachates for the unbound loose blends, originally used to develop the bituminous mixtures, were also tested to examine the effect of bitumen binding on IBAA leaching properties.

A leachant was prepared by adding 100 gm of sample material to one litre of distilled, de-ionised water to obtain a liquid/solid ratio of 10:1 (L/S_{10}). This was then mechanically agitated, at 10 cycles per second, for 24 hrs. This duration was sufficient for the sample to reach the steady state or near equilibrium leaching conditions (Bialucha 2000). After equilibrium was achieved, leachates were filtered through 0.45

µm filter paper. The solution was then analysed for the determinants. The pH and the electrical conductivity were analysed using a calibrated electrode. In addition, metals were analysed using Inductively Coupled Plasma - Optical Emission Spectrometer (ICP-OES). The analysed metals were: Arsenic – Boron – Cadmium – Chromium – Copper – Iron – Mercury – Nickel – Lead – Zinc.

Moreover, cyanides, sulphides and chlorides were analysed using liquid ion chromatography. Ammonia and Chemical Oxygen Demand (COD) were analysed using colour-metric determinations. Phenol was analysed using gas chromatography. These constituents were picked up based on the recommendations of the reviewed literature (Eighmy et al 1995, IAWG 1997, Kus et al 1999, Schreurs 2000, Bruder-Hubscher 2001b, Paine et al 2002, Abbott et al 2003, Cai et al 2004, Dijkstra et al 2006, Huang et al 2006).

Constituents found in the leachates from loose blends were compared to the corresponding environmental quality standard or water quality standard (EQS/WQS) reference limits (Baldwin et al 1997). Table 4-5 shows that the concentrations of the constituents, found in the leachates, were at very low levels. This result highlighted that use of IBAA, in bituminous and non bituminous mixtures, gave an environmentally accepted material.

The effect of bitumen binder coating on the leaching characteristics of a bituminous mixture containing IBAA was also investigated. The constituents found in the loose samples were compared to those found in the bituminous mixtures. Results indicated that coating IBAA with bitumen had a significant affirmative effect on its leaching properties. It was found that the pH and the electrical conductivity values for bituminous mixtures leachates were less than those for the loose blends. Moreover, most of the constituent concentrations found in the leachates were found to decrease with increase in bitumen content. The only immunity was the sulphate which had higher values in some bituminous mixtures compared with the loose blend. Table 4-6 shows these results.

Constituent	EQS/WQS (mg/l)	Mix AA (mg/l)	Mix BA (mg/l)	Mix CA (mg/l)
As	0.05	<0.01	<0.01	<0.01
B	2	<1	<1	<1
Cd	0.005	<0.001	<0.001	<0.001
Cr	0.05	<0.05	<0.05	<0.05
Cu	3	<0.02	<0.02	<0.02
Fe	0.2	<0.1	<0.1	<0.1
Hg	0.001	<0.001	<0.001	<0.001
Ni	0.05	<0.02	<0.02	<0.02
Pb	0.05	<0.01	<0.01	<0.01
Zn	5	<0.1	<0.1	<0.1
C_6H_5OH	0.0005	<0.0001	<0.0001	<0.0001
CN	0.05	<0.05	<0.05	<0.05
SO_4	250	14	42	25
CL	400	10	95	47
COD	200	10.5	76.8	48.4
NH_3	0.5	0.06	0.28	0.38

Table 4-5: Loose blends leaching results compared with EQS/WQS standards

Mix AA	Constituent		Loose blend	Binder content (%)		
				5	5.5	6
	pH value		12	9.8	10.5	10
	Sulphate	mg/l	42	15	12	11
	Chloride	mg/l	95	21	10	<10
	COD	mg/l	76.8	6.4	3.3	6.5
	Electrical conductivity	µScm	1192	151	106	88
Mix BA	**Constituent**		**Loose blend**	**Binder content (%)**		
				6	**6.5**	**7**
	pH value		12	9.1	9.8	10.1
	Sulphate	mg/l	42	63	33	45
	Chloride	mg/l	95	49	35	36
	COD	mg/l	76.8	23.8	6.3	7.4
	Electrical conductivity	µScm	1192	396	254	304
Mix CA	**Constituent**		**Loose blend**	**Binder content (%)**		
				7.5	**8**	**8.5**
	pH value		12	8.6	10.3	9.6
	Sulphate	mg/l	42	41	33	27
	Chloride	mg/l	95	68	46	31
	COD	mg/l	76.8	11.6	12.6	5.2
	Electrical conductivity	µScm	1192	404	314	238

Table 4-6: Effect of bitumen binding on IBAA bituminous mixtures leaching properties

4.7.2 Tank test results

Diffusion control leaching is the most expected scenario for constituent release from IBAA bituminous mixtures as explained earlier in this chapter. Measuring the constituents' release required running a leaching test which can simulate this regime. One of these sort of tests is the tank leaching test which was undertaken in accordance with EA NEN 7375 (Netherlands Normalization Institute 2004b). This test is sometimes known as diffusion test; schematic diagram for this test is shown in Fig 4-2. Two different materials were tested: mix CA and its loose non bituminous blend. This mix, which contains 80% IBAA, was chosen as a worst case scenario in long term leaching as it contains the highest IBAA content amongst the mixtures produced in this study.

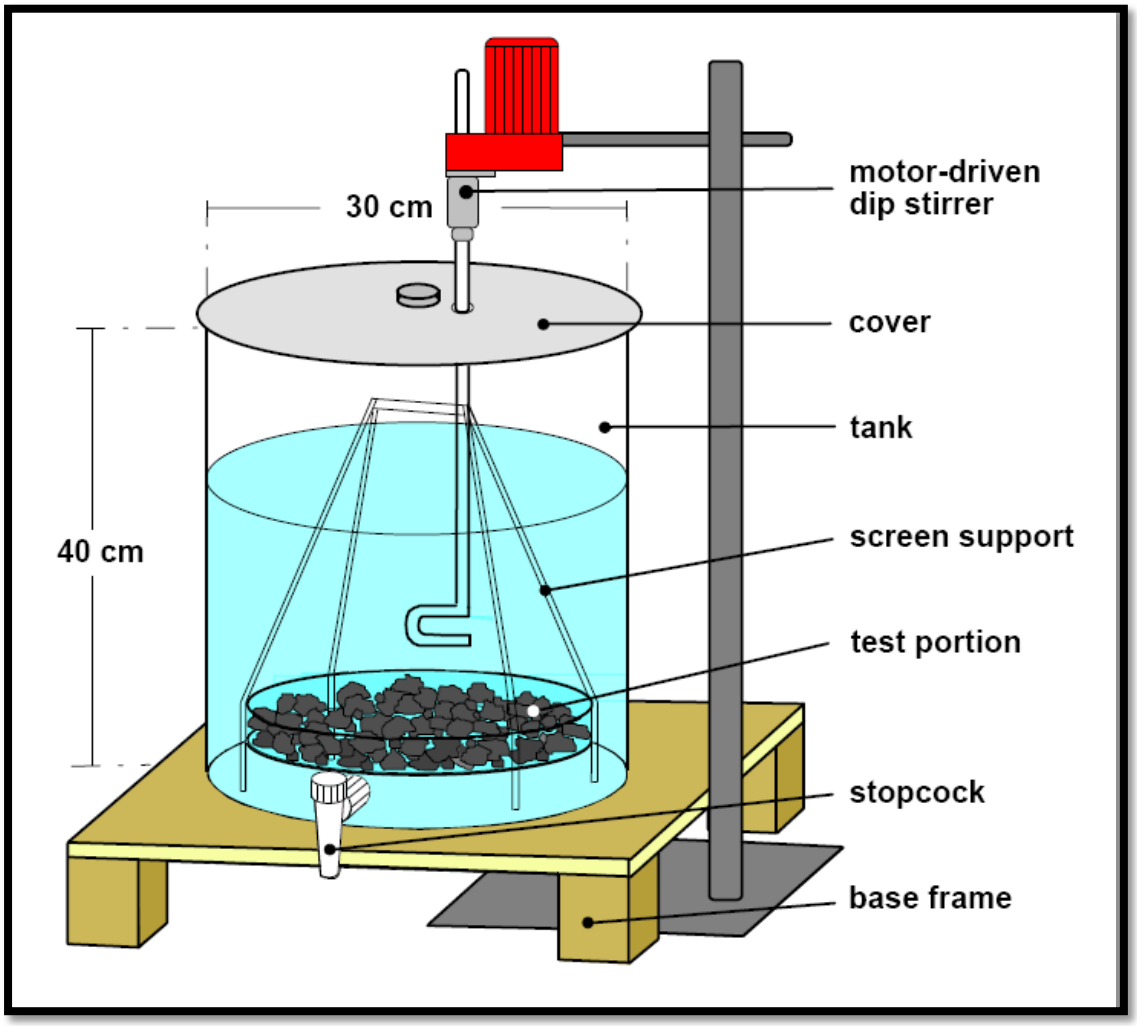


Figure 4-2: Schematic diagram of tank test arrangement (Bialucha 2000).

The tested samples were placed in plastic sealable tank, rinsed with nitric acid and then with water, and covered with demineralised water so that sample is surrounded by at least 2 cm from all sides. Water volume was five times the sample volume. Then, the diffusion test was carried out in several stages at a temperature of 20±2 ^{0}C. Firstly, the tank was sealed before elute being drained. After drainage, the tank was refilled with water. Elute was subsequently filtered using membrane filters of size 0.45 µm and kept in sealable storage plastic bottles for analysis. This procedure was repeated at different time intervals; 0.25, 1, 2.25, 4, 9, 16, 36, and 64 days. At each replacement time, elute was analysed to measure the release of the following components: cooper, lead, zinc, sulphate, potassium, sodium, and chloride using the same chemical processes mentioned earlier.

Tank test results, shown in Figs 4-3 & 4-4, implied that, for all constituents, release was higher in the non bituminous blends, i.e. bitumen coating significantly improved IBAA leaching characteristics. This resembles the result obtained from the agitated extraction test. Sulphate was found to be the highest release from both the non bituminous blend and the bituminous mix. Therefore, much attention has to be drawn to its effects on the environment if this high IBAA level bituminous mixture were to be adopted in any construction. Even though chloride, sodium and potassium recorded noticeable releases, their cumulative releases were within EQS/WQS policy limits. Nonetheless, the time dependent leaching behaviour had the same pattern for all these four constituents. This pattern showed a rapid concentration increase followed by a slow release. Steady state was not attained during the various phases of the experiment, lasting 64 days. In respect of copper, lead and Zinc, the recorded cumulative releases, were low and below detection values, thus complying with EQS/WQS environmental regulations. These results acknowledged the environmental impact of using IBAA in bituminous mixtures at high levels.

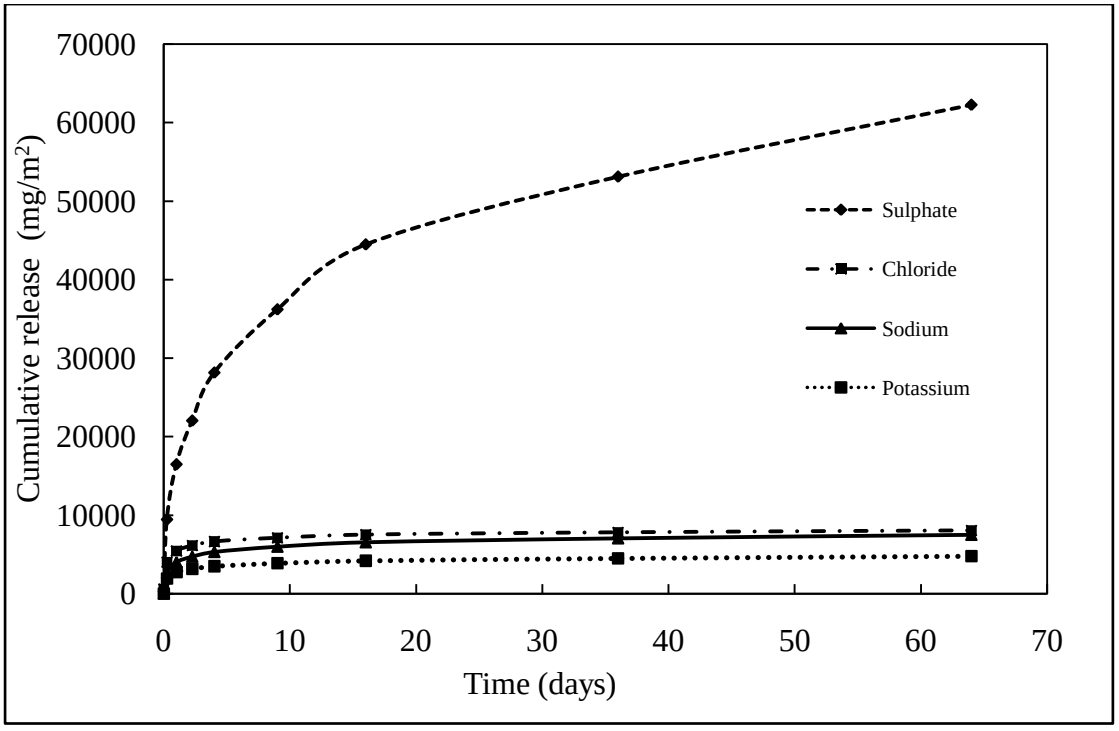


Figure 4-3: Cumulative release from a non bituminous 80% IBAA blend from tank test.

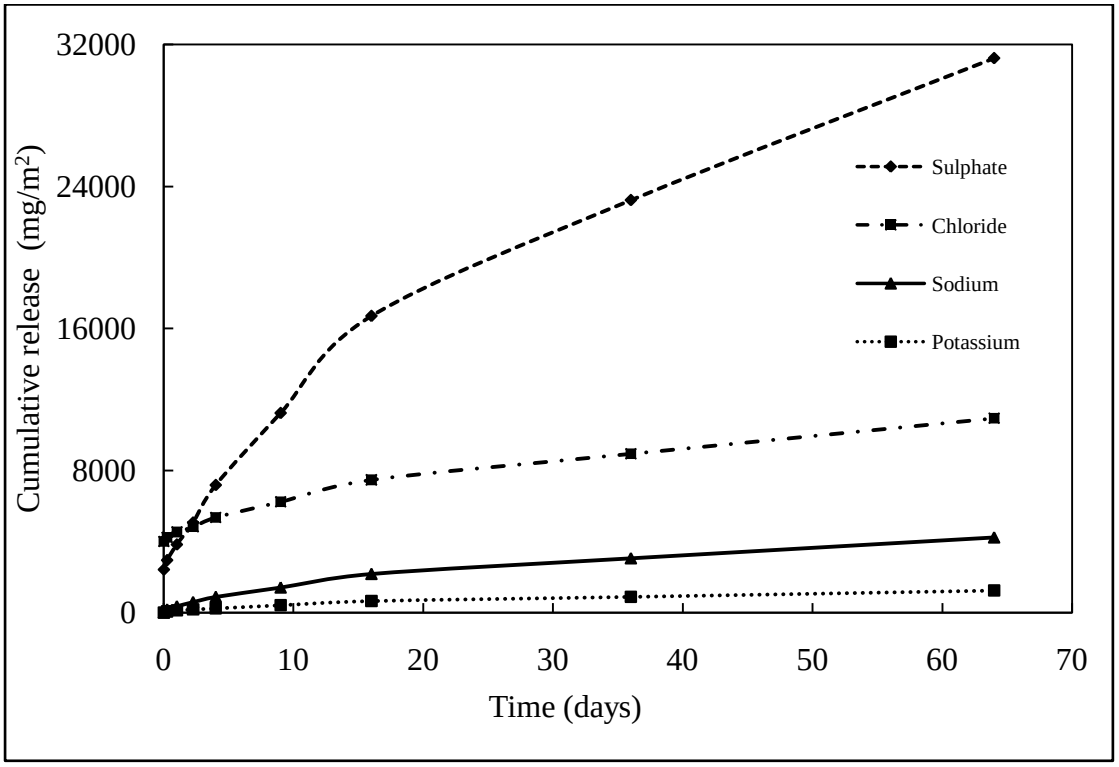


Figure 4-4: Cumulative release from Mix CA (80% IBAA) from tank test.

4.7.3 Availability test results

An availability test, according to EA NEN 7371 (Netherlands Normalization Institute 2004a) was conducted to measure the availability IBAA constituents, which is necessary for diffusion coefficient calculations. The availability test, adopted in this study, was developed in the Netherlands (de Groot and Hoede 1994). In this test 16±2 gm samples, from mix CA and its loose non bituminous blend, were subjected to two leaching stages, using L/S_{50}, and were agitated magnetically. In the first stage, leaching was continued for three hours at a constant pH of 7. The second stage comprised leaching for three hours at a constant pH of 4. The pH values were monitored and measured using a pH-meter and were adjusted to the desired levels using nitric acid additives. After the first stage, elutes were filtered within one hour of collection and stored for analysis. Then, the residue was returned to the testing beaker. More water was added to keep the L/S ratio and, hence, the second leaching stage commenced. The test was conducted at a temperature of 20±2 ^{0}C. The availability for each component was calculated using Eq 4-4 and results are presented in Table 4-7.

Component	**CA Loose blend**	**Mix CA**
	Availability (mg/kg)	
Cooper	1700	650
Lead	120	400
Zinc	840	520
Sulphate	7300	8800
Potassium	2500	1500
Sodium	9900	4500
Chloride	1300	900

Table 4-7: Availability values of the components analysed from mix CA and its loose blend

$$C_0 = c\,(2V_0 + V_1 + V_2)/(m_0 \times f) \quad (4\text{-}4)$$

where: C_0 is the availability of a component; c is the concentration of that component in the elute; V_0 is the volume of the first step added water; V_1 is the volume of the first

step added nitric acid; V_2 is the volume of the second step added nitric acid; m_0 is the dry weight of the analytical sample; and f is a factor = 1000.

4.8 LEACHING MODELS

The various theoretical modelling approaches in current use to describe leaching are discussed in this section with specific concern to bottom ash leaching. These models can be categorised into two main categories as follows:

- Equilibrium models.
- Dynamic multi-components models.

4.8.1 Equilibrium models

The equilibrium modelling approach is derived from geochemical thermodynamics models that require the leaching system to be at, or at least near, equilibrium condition. Consequently, this approach models the leaching at no flow condition, which requires specific leaching parameters, i.e. specific pH, L/S etc.

Abundant studies have been published on equilibrium models (Kincaid et al 1984a & 1984b, Nordstrom and Ball 1984, Morrey et al 1986). These studies explained that the equilibrium models attempt to find the point at which the system-dissipated energy is nil or at least minimum. At that point, the equilibrium condition should be reached. These attempts can be achieved by two ways. In the first, the model tries to minimise a free energy function while in the second, the model tries to solve a set of non-linear equations consisting of equilibrium constants and constrains on the mass balance of an element in the system.

4.8.2 Dynamic multi-components models

The dynamic multi-component modelling approach is derived from the dynamic multi-component flow-through models that do not require equilibrium conditions. These models use boundary conditions and initial conditions for constituents. This

approach, in contrast with the equilibrium approach, can predict longer term leaching and can work under changing scenarios, i.e. change in pH or L/S. This model, which was developed by Theis et al (1992), can incorporate some complex parameters such as dissolution, sorption, desorption, and mass transfer.

4.8.3 Diffusion modelling for IBAA in bituminous mixtures

A simple one-dimensional model can be used to envisage the release of constituents over a specified period of time. This model, even though approximate, gives a conservative estimate of the cumulative release (Mt), based only on the constituent availability (C_o), diffusion coefficient (D) and the exposed geometric surface. This model is based on Eq 4-3, (Crank 1975). This equation was used in this study to calculate the diffusion coefficient, as pD for t = 64 days. Results for all detected constituents, as shown in Fig 4-5, showed that the mass transfer of sulphate, by diffusion, was relatively more mobile than the other detected constituents which had higher pD values. This result, once more, emphasised the importance of investigating sulphate potential effects on the environment when IBAA is used at high levels in bituminous mixtures.

The diffusion coefficient calculations showed that pD values ranged between 6 and 12 for IBAA bituminous mixture which contained 80% IBAA. Based on this result, an estimate of the cumulative release in the field may be predicted using a three-dimensional diffusion model based on Eq 4-3 (Crank 1975) and presented in Eq 4-5 (IAWG 1997). Fig 4-6 presents a predicted cumulative release for a binder course layer of 30 cm thickness over a 100 year period. This shows the effect of the diffusion coefficient on constituent release over time. It is obvious that constituents with high diffusivity, i.e. low pD value, leach more rapidly. This is in line with the experimental results discussed earlier. Despite that, this model can be considered as a worst case scenario as it gives a higher release than that of the experimental results. Moreover, it assumes constant saturation of the layer, thus, not considering the effect of wetting and drying, which leads to lower releases (Kosson et al 1996).

$$M_t = \int_0^t \left\{ \left(\frac{64}{\pi^3}\right)^2 \rho C_i abc \sum_{l=0}^{\infty} \sum_{m=0}^{\infty} \sum_{n=0}^{\infty} \frac{\alpha e^{-t\alpha}}{(2l+1)^2(2m+1)^2(2n+1)^2} \right\} dt \quad (4\text{-}5)$$

where: *a, b, c* are the dimensions used in the model and *l, m, n* are integers.

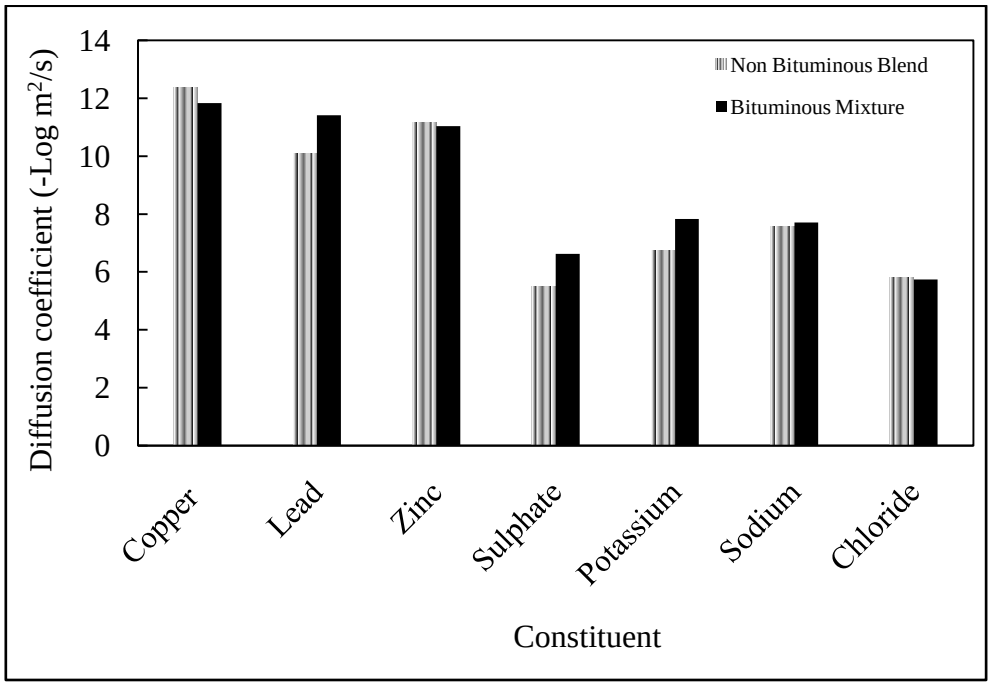


Figure 4-5: Diffusion coefficient values for the constituents calculated at 64 days.

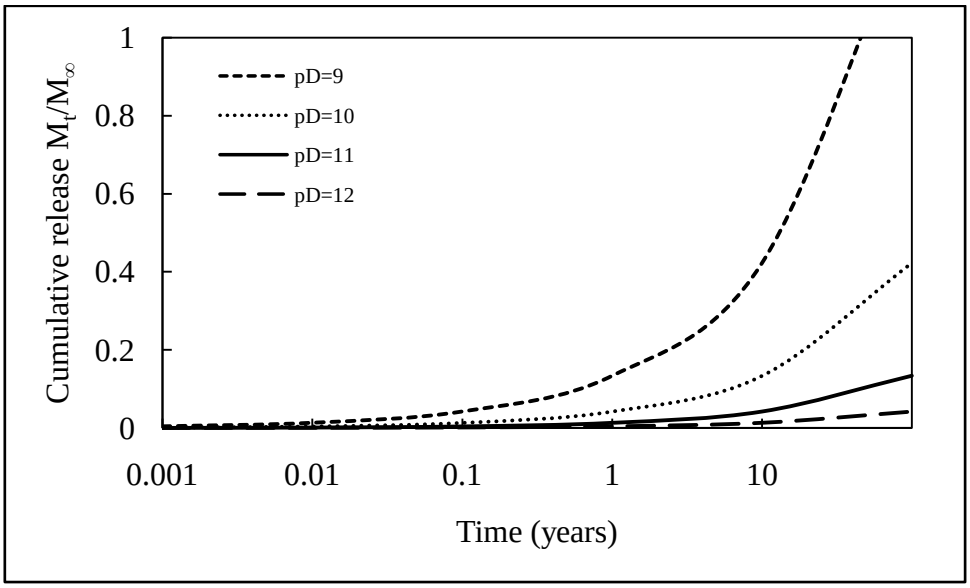
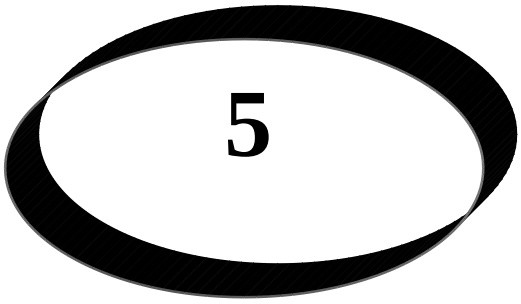


Figure 4-6: Cumulative release model for diffusion from a binder course layer (0.3m thick).

4.9 CONCLUDING REMARKS

From the preceding sections, the following remarks could be made:

- The pH and electrical conductivity values were found to decrease dramatically after coating IBAA with bitumen, whereas the sulphate was found to increase, with no known reason, in some mixtures.
- The time-dependent leaching behaviour showed that sulphate was found to be of the highest release from both the non bituminous blend and the bituminous mixture. Therefore, much attention has to be drawn at its effects on the environment if this high IBAA level bituminous mixture were to be adopted in any construction.
- The diffusion coefficient emphasised that the mass transfer of sulphate was relatively more mobile than the other detected constituents.
- A three-dimensional model can be used to predict the cumulative release from a binder course layer considering a worst case scenario.



RHEOLOGY

5.1 INTRODUCTION

Rheology is the science of "*the study of the deformation and flow of matter*". This term was presented for the first time by Professor Bingham (1920) and was taken from the Greek word "*rheos*" which means everything flows. Rheology, although a very wide term, deals mainly with materials which obey neither Hooke's law for elastic solids nor Newton's law for viscous fluids. Rheological behaviour of materials is assessed using rheometers. Generally, there are two types of rheometers, namely: rotational and capillary rheometers. Each has a range of test modes. Rotational test modes are: Viscometry (shear), Creep and Recovery, Oscillation, Stress Relaxation, and Multiple Frequency Oscillation. Capillary test modes are: Viscometry (shear and extension), Die Swell, Degradation, Melt Fracture, and Stress Relaxation. These types of rheometers are used to study the rheological behaviour of a wide range of materials, e.g. paints, plastics, rubber, and lubricants. Bitumen, because of its nature which allows high deformations and its stress and temperature dependency, belongs to the group of materials which can be studied using Rheology.

It is known that sound depiction of binder performance leads to good prediction of mixture's performance. One of the most important properties needed to understand

permanent deformation of binders is their steady state behaviour. Steady state behaviour of pure bitumen has been studied, in published literature, in tension and compression. Moreover, the steady state shear behaviour has been studied using coaxial shear and creep tests. This chapter aims to study the steady state shear behaviour of binders using a Dynamic Shear Rheometer (DSR). Moreover, it aims to investigate the effect of using IBAA in bituminous mixtures on their binders' permanent deformation behaviour and consequently on mixtures' deformation behaviour as will be shown in Chapters 6 and 7.

In this chapter, the inelastic flow models and binder steady state permanent deformation behaviour were reviewed. Then, a DSR was used to study the steady state permanent deformation behaviour of three binders over a range of temperatures and loading frequencies. A 100/150 pen bitumen was used in the study, which also included two mastics; the first mastic consisted of bitumen and limestone fines while the second consisted of bitumen, limestone and IBAA fines. These binders are the respective binders for the mixtures produced and presented in Chapter 3. The low frequency constant strain rate mode was used in the DSR at high temperatures to arrive at steady state stress levels for different loading rates, where the material begins to deform plastically. The DSR creep mode was then used at lower temperatures to determine the strain rate of binders at different stress levels. The results were used to identify the steady state deformation behaviour of the three binders and the effect of IBAA content on this behaviour.

5.2 RESEARCH ON BITUMEN

Bitumen is a material which can be naturally occurring or is manufactured from crude oil. Bitumen is produced as a result of processes on organic materials over several millions of years. Natural bitumen has been used as an adhesive and waterproofing material from as early as 3800 B.C. (Read and Whiteoak 2003). Extensive use of bitumen in pavement construction started in the early nineteenth century. Research to understand the behaviour of bitumen as an engineering material was started in the 1920s. Research on bitumen was started with a simple colloidal model and its

corroboration by mechanical tests. This was followed by Van der Poel's work (Van der Poel 1954), in which the stiffness of bitumen was correlated with two experimental parameters, namely the penetration (BSI 2000b) and the softening point (BSI 2000a). After 1960, a wide range of experimental tools were developed to study the chemical composition and mechanical behaviour of bitumen. A more detailed review on progress in research on bitumen since the 1920s can be found in Cheung (1995).

5.3 RHEOLOGY INELASTIC FLOW MODELS

To understand the results obtained from rheometers, it is necessary to recognize and comprehend the assumptions and models that control the rheological behaviour of non-Newtonian fluids. Therefore, these models will be discussed in this section.

There are many models that have been used to characterise the inelastic behaviour of materials. One of such old models is the Bingham model (Bingham 1922) which represents a solid but is used only when flow occurs. It assumes that the material behaves as an elastic solid for stresses less than a yield stress after which it behaves according to its plastic viscosity i.e. as a viscous fluid according to Eq 5-1. This equation assumes a linear relation between shear stress and strain rate with a slope equal to the plastic viscosity of the material. This model was used successfully to study the plastic deformation for some materials specially metals as they have an easy determinate yield stress. This model, which is a very simple rheological model, could not be adopted for bitumen due to the flow linearity assumption after yielding which was not noticed for bitumen.

$$\sigma_{sh} = \sigma_y + \eta\dot{\varepsilon} \qquad (5\text{-}1)$$

where σ_{sh} is the shear stress; σ_y is the yield stress; η is the plastic viscosity; and $\dot{\varepsilon}$ is the strain rate.

The Casson model (Casson 1959) is another inelastic model and it is a semi empirical model usually used for suspensions e.g. inks. It is a commonly used rheological model that quantifies yield stress and high shear viscosity. There are many parallel models such as the Ellis model (Ellis 1927) and Carreau model (Carreau 1972). These two models are nearly similar as they show Newtonian behaviour at low stresses and power law behaviour at high stresses. These models, although widely used in Rheology, have not been reported as bitumen models.

5.3.1 The Power Law Model (PLM)

The power law model is one of the well known models in Rheology and was presented by Ostwald and Auerbach (1926) to represent the behaviour of many polymer solutions and melts. This model can be expressed by Eq 5-2. For a power law fluid with exponent greater than 1, the viscosity decreases with an increase in shear rate due to shear thinning. Alternative term, pseudo-plastic is also used to express a time dependent decrease in viscosity with increase of shear rate. Moreover, the power law model has been used for materials with exponent smaller than 1 which is shear thickening case in which material viscosity increase with increasing the shear rate. This model was used successfully to study the rheological properties of bitumen in a few occasions as will be explained later in this chapter.

$$\dot{\varepsilon}/\dot{\varepsilon_p} = (\frac{\sigma}{\sigma_0})^n \quad (5\text{-}2)$$

where $\dot{\varepsilon}$ is the strain rate; σ is the stress; $\dot{\varepsilon_p}$ is the strain rate at reference stress σ_0; and n is the power law exponent.

5.3.2 The Modified Cross Model (MCM)

As bitumen is classified as non-Newtonian fluids, due to its viscoelastic nature, it generally exhibits pseudo-plastic flow, i.e. a decrease in viscosity with increasing rate of shear. On the assumption of that pseudo-plastic flow is associated with the

formation and rupture of structural linkages, Cross (1965) presented an equation which governs this type of flow. This equation is expressed mathematically as in Eq 5-3. The model was based on the arguement that if a system contains elements that are capable of assuming some structural formation that is wholly or partially disrupted by shear, a viscosity / shear rate dependence of a pseudo-plastic flow nature will be expected.

$$\eta = \eta_\infty + \left(\frac{\eta_0 - \eta_\infty}{1 + \alpha\ (\dot{\gamma})^{2/3}}\right) \tag{5-3}$$

where η_0 is the limiting viscosity at zero rate of shear; η_∞ is the limiting viscosity at infinite rate of shear; α is a constant associated with the rupture of linkage; and $\dot{\gamma}$ is the rate of shear.

In the same year, Sisko (1965) published an empirical viscosity equation for pure bitumen, Eq 5-4. This equation was created using results obtained from a cone-plate viscometer which was used to determine the steady state shear viscosity of three different bitumens. Recently (Garrick 1992) this model was applied empirically to model the shear viscosity of bitumen as a function of shear rate. Results showed that the model reasonably fitted the experimental data.

$$\eta = \frac{\eta_0}{1 + c\ (\dot{\gamma})^n} \tag{5-4}$$

Cheung (1995) jointed the success of Sisko's empirical model (Sisko 1965) and the evidence of flocculation behaviour of bitumen (reviewed and presented by Cheung 1995), and strongly suggested the suitability of the Cross model (Cross 1965) in describing the pseudo-plastic flow behaviour of bitumen at a wider range of stress levels. Therefore, he suggested a modification on the Cross model (Eq 5-3) and presented the Modified Cross Model (MCM) as Eq 5-5 (Cheung 1995). This form was used for bitumen subjected to tensile deformation. In this form the reference stress is considered as the failure strength of the structural linkage beyond which extensive

pseudo-plastic flow occurs. This value was found to be temperature independent. In addition, the MCM suggested non-linear behaviour at high stress levels originates from the strain rate dependence of the linkage rupture mechanism. Cross (1965) suggested a constant exponent of 2/3 for his model. On the other hand, Cheung (1995) concluded that this exponent is variable depending on the physics of the rupture mechanism and could be found by fitting the model to the experimental data.

$$\eta = \frac{\eta_0 T}{1+ (\eta_0 T \dot{\varepsilon}/\sigma_0)^n} \tag{5-5}$$

Eq 5-5 was applied successfully for constant strain rate and creep tensile tests on 50 Pen grade bitumens (Cheung 1995, Cheung and Cebon 1997a, b) before being rewritten in a new form presented in Eq 5-6 (Deshpande 1997). This new form however presented by Deshpande (1997) is known as MCM and usually adopted to Cheung (1995).

$$\sigma = \frac{\sigma_0 \dot{\varepsilon}}{\dot{\varepsilon_p}} \left\{ \frac{1}{1+ \left(\frac{\dot{\varepsilon}}{\dot{\varepsilon_p}}\right)^m} \right\} \tag{5-6}$$

where: σ is the uniaxial stress; $\dot{\varepsilon}$ is the uniaxial strain rate; and σ_0, m, $\dot{\varepsilon_p}$ are material constants for the bitumen.

5.4 DEFORMATION BEHAVIOUR OF BITUMEN

Understanding the mechanical behaviour of asphaltic mixtures relies on an understanding of the behaviour of the constituent materials and their interaction. The mechanical properties of bitumen can play an important role in the deformation behaviour of asphaltic mixtures. Bitumen is a viscoelastic material and its deformation under stress depends on temperature, strain/stress level and loading time.

The behaviour of bitumen under loading is considered as one of the most important areas which require further study. Bitumen behaves as a viscous liquid at high temperatures and/or long loading times, whereas at low temperatures and/or short loading times it behaves as an elastic solid. The behaviour at intermediate temperatures and loading times is viscoelastic (Read and Whiteoak 2003). Both linear and nonlinear behaviour have been studied for bitumen. The quasi-static deformation behaviour of bitumen, defined as the response under static loading, has been described by several theories such as the time dependent modulus, correspondence principles, molecular theories and mechanical analogies. Most of the theories have concentrated on small strains in which the bitumen behaviour is nearly linear (Cheung 1995). The linear behaviour was studied and modelled successfully, using linear viscoelastic theories, for low stress levels (Saal 1950 and Brown, Sparks 1958, Vinogradov et al 1976, Goodrich 1991, Christensen and Anderson 1992). However, it has been reported (Lethersich 1942) that nonlinearities are more significant at higher stress levels. The limit of bitumen linearity was investigated (Airey et al 2002, 2003, 2004) for a range of unmodified and modified binders using a DSR and tension tests. A strain dependent linearity criterion was found for binders at low temperatures while a stress dependent linearity criterion was found at high temperatures as well as a high temperature strain dependent linearity criterion for modified binders.

Empirical approaches have been used to model the transition from linear to non-linear behaviour. For instance, a cone-plate viscometer was used over a wide range of shear rates to arrive at a model for the steady state shear viscosity of pure bitumen (Sisko 1965). This model was expressed as in Eq 5-4.

The behaviour at higher strains where the non-linearity is dominant has not been studied very much e.g. at larger strains, bitumen was found to behave as a power law material (Sisko 1965, Khong et al 1978, Cheung 1995, and Ossa 2004).

5.5 STEADY STATE BEHAVIOUR OF BITUMEN

Few trials have been conducted to study the steady state permanent deformation behaviour of bitumen. Cheung (1995) and Cheung and Cebon (1997a, b) developed various physical models for the steady state behaviour of a 50 Pen bitumen by undertaking a number of monotonic tests in tension, compression and shear, over a range of loading and temperature conditions. Dumb-bell shape axisymmetric specimens with 80 mm in length were tested under tension stresses. Constant strain rate and creep tensile tests were undertaken at temperatures ranged between -10 °C and 30 ^{0}C and stress levels greater than 100 kPa on a 50 Pen bitumen. They concluded that the steady state behaviour of the bitumen at a particular temperature can be represented by a power law equation. Moreover, it was found that the temperature dependence of the steady state deformation behaviour of the bitumen at ambient temperatures can be well described by the Arrhenius equation. By combining these two theories, the Power Law Model (PLM) was presented as in Eq 5-7. The steady state behaviour at these testing levels was reported as non-linear with a creep exponent of 2.3.

$$\dot{\varepsilon}/\dot{\varepsilon_p} = \left(\frac{\sigma}{\sigma_0}\right)^n \exp\left(-\frac{Q_p}{RT}\right) \quad (5\text{-}7)$$

where $\dot{\varepsilon}$ is the uniaxial strain rate; σ is the uniaxial stress; n is the creep exponent; $\dot{\varepsilon_p}$ is the reference strain rate at reference stress σ_0; Q_p is the thermal activation energy (was determined as 228 kJ/mol); R is the universal gas constant (was given a value of 8.13 Jmol^{-1}K^{-1}); and T is the temperature in degree Kelvin.

The steady state deformation behaviour of the bitumen at high temperatures was found to be well described by the Williams–Landel-Ferry equation (Williams et al 1955) WLF equation, which is based on the concept that molecular mobility at any temperature depends primarily on the free volume (Cheung, 1995).

In order to observe the deformation behaviour of the bitumen at lower stress levels, constant strain rate tests were carried out at the same temperatures (-10 to 30 ^{0}C). It was found that the behaviour at lower stress levels is linear (power law creep with a creep exponent of 1). For all temperatures, the transition from linear to non-linear

behaviour took place at approximately the same stress level (100 kPa). The change in deformation behaviour from linear at low stress levels to non-linear at high stress levels was attributed to the rupture of the molecular network formed by the association of asphaltens. They proposed the modified Cross model for describing the uniaxial steady state deformation behaviour of the bitumen. Based on this model, the steady state deformation behaviour changes from linear at low stress levels to non-linear at high stress levels.

Cheung (1995) applied the MCM to the data for 61 types of bitumen from wide range of sources. It was found that the stress level at which the viscous behaviour changes from linear to non-linear power law creep lies between 0.1 and 0.5 MPa. The value of creep exponents in the power law regime was found to be between 2 and 2.5.

Cheung also investigated the effects of ageing on the steady state deformation behaviour of bitumen. By applying the MCM to previous research data for more than 50 types of bitumen, it was found that ageing generally increases the creep exponent and viscosity, and decreases the transition stress level. By carrying out creep tests in tension and compression at temperatures from –35 to 20 ^{0}C on the 50 Pen bitumen, a model was also proposed for describing the bitumen behaviour at temperatures in the vicinity of and below the glass transition. The glass transition is the temperature which divides low temperature behaviour (glassy behaviour) from the high temperature behaviour. For bitumen, the glass transition temperature varies between -40 and 0 ^{0}C (Cheung, 1995).

Based on Cheung's conclusions, Collop et al (2002) used a Dynamic Shear Rheometer (DSR) to characterise two binders: 50 Pen and 100 Pen bitumens. They carried out constant stress creep tests over a range of shear stress levels at 20 ^{0}C. They found that at low stress levels (< 70 kPa) the deformation behaviour of the bitumens was linear, whereas at higher stress levels the deformation behaviour was non-linear, and the effective creep exponent was found to be 2.4. They developed a model, very similar to the MCM, for describing the deformation behaviour of the bitumen subjected to shear loading in the DSR. The model related the steady state shear stress to the steady state shear strain rate. It was concluded that the creep tests performed by

a DSR with parallel plate geometry can be used for characterisation of the value of the power law creep exponent at high stress levels and the shear viscosity at low stress levels in the linear regions. Using correlation factors, the DSR model in the linear and non-linear region agreed well with the uniaxial tensile model. Eqs 5-7 and 5-8, for uniaxial stress and pure shear, respectively, were used to correlate the DSR model to the uniaxial tensile model developed by Cheung (1995) for the same 50 Pen bitumen.

$$\left(\frac{\dot{\gamma}}{\dot{\varepsilon_p}}\right) = \sqrt{3}^{n+1} \left(\frac{\sigma_{sh}}{\sigma_0}\right)^n \tag{5-8}$$

where $\dot{\gamma}$ is the shear strain rate and the rest as defined before.

It can be seen that the behaviour in tension and shear is related by the correlation factor of $\sqrt{3}^{n+1}$, which equals 3 in the linear region (n = 1), and 6.47 in the non-linear region (n = 2.4).

Ossa et al. (2005) investigated the deformation behaviour of 2 pure bitumens by performing static tensile creep, monotonic constant strain rate, continuous cyclic and pulse strain tests in tension over a range of temperatures between -5 and 20 ^{0}C. Similar to the findings of Cheung et al. (1997a, b), they found that the steady state deformation behaviour of the bitumen is non-linear viscous at high stress levels and linear at low stress levels.

Continuous stress controlled cyclic tests were carried out over a range of mean stresses, load ratios, frequencies, and temperatures. It was shown that the response of the bitumens in this test, in terms of strain versus time, was similar to the static creep response. It was found that the effect of load ratio and frequency on the steady state strain rate was negligible, and the behaviour was mainly governed by the mean stress. The cyclic steady state behaviour was found to follow the steady state behaviour in static creep with the creep stress interpreted as the cyclic mean stress.

Cyclic pulse train tests were carried out over a range of temperatures, stress levels and time period ratios. It was shown that, for a fixed value of stress level, the accumulated permanent deformation decreased with decreasing the time period ratio, because larger fractions of the creep strain recovered during zero-load gaps between the pulses. In all tests the MCM was applied successfully and gave the same conclusions as Cheung's.

Based on literature observations, it is clear that the MCM is a very dependable model to study the steady state permanent deformation of bitumen. Moreover, the DSR was found to be a promising tool in this field. However, these two tools have neither been used over a wide range of temperature nor for binder mastics. Therefore, it was decided to use the DSR, in this study, to investigate the steady state permanent deformation behaviour of the 100 Pen bitumen and the mastics used in the previous chapters to develop IBAA bituminous mixtures. Moreover, it wroth widens the temperature range of the study.

5.6 DYNAMIC SHEAR RHEOMETER (DSR)

Dynamic Shear Rheometer (Bohlin DSR II), shown in Fig 5-1, was used in this study. The DSR is a sophisticated piece of equipment that is talented to investigate the rheological performance of small samples of binders under a wide range of temperatures as well as loading rates (frequencies). Compressed air is required to facilitate frictionless movement of the working parts whilst a temperature control unit regulates the thermal conditions during testing. The DSR incorporates a clean, easy to use system which completely immerses the sample in a temperature controlled fluid to control the thermal testing conditions. A computer, through an interface box, governs the execution and control of the test.

The principles involved in the DSR tests are illustrated in Fig 5-2. The specimen is sandwiched, in a specified adjustable gap, between a spindle and a base plate. The spindle, used in this study, is disc shaped and is allowed to rotate. The base plate is a circular disc which remains fixed, using a hexagonal locking nut, during testing. The

angular rotation (θ) and the applied torque (M) are measured electronically and hence the resulted shear strain (ε_s) and stress (σ) are calculated, automatically using the DSR software, using Eq 5-9 and 5-10 respectively (Kennedy et al 1994).

$$\varepsilon_s = \frac{\theta \times r}{h} \tag{5-9}$$

$$\sigma = \frac{2\,M}{\pi\, r^3} \tag{5-10}$$

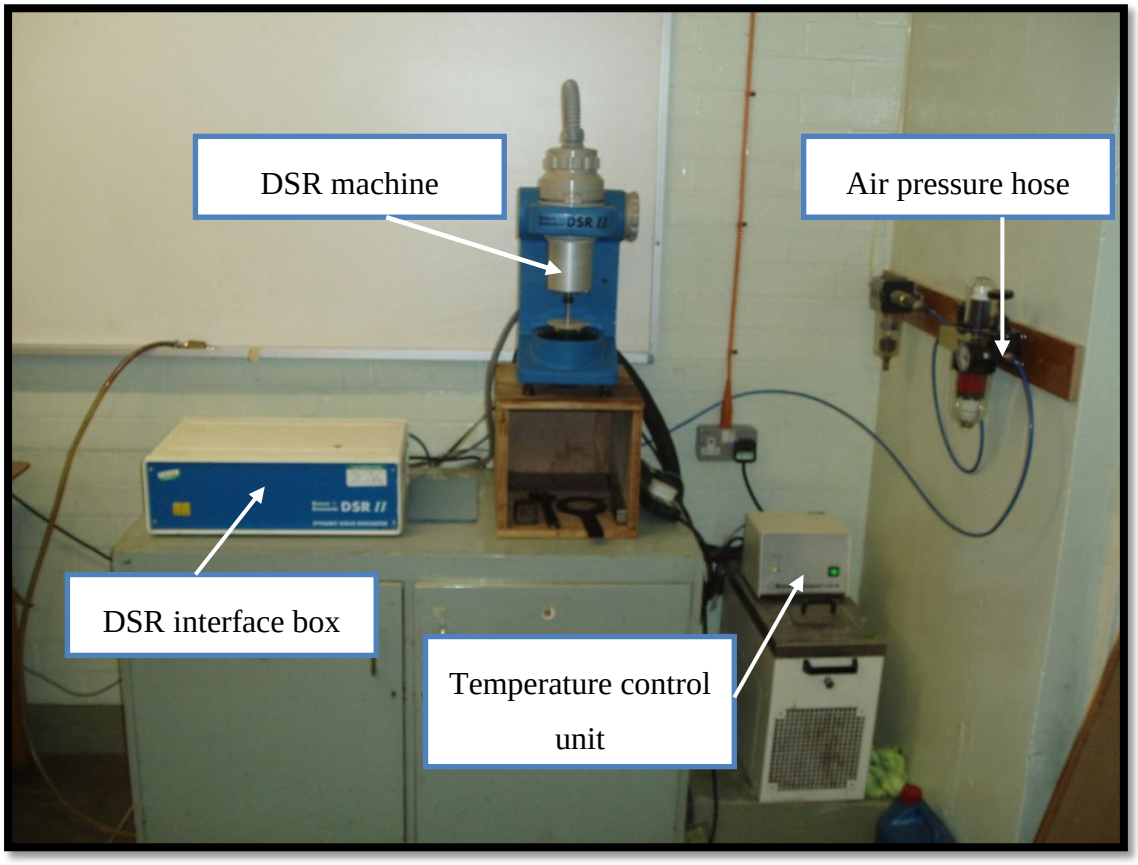


Figure 5-1: DSR assembly.

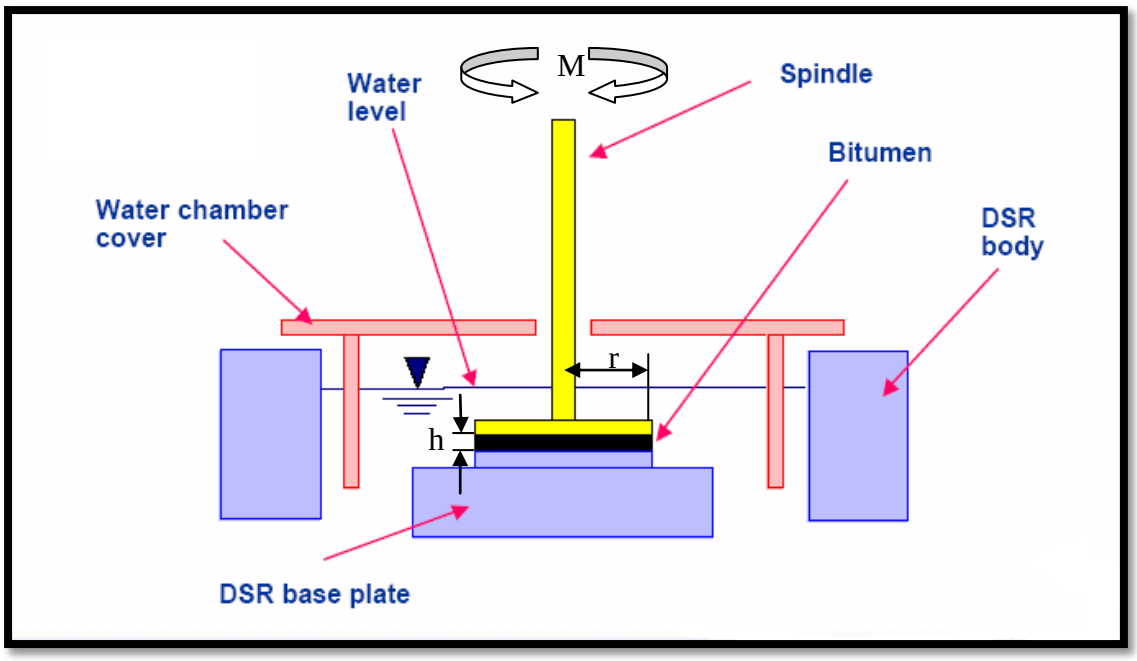


Figure 5-2: DSR constant strain rate testing principle.

5.7 BINDER DEFORMATION USING DSR

5.7.1 Materials

Three bituminous binders were employed in this work. Firstly, a 100/150 pen bitumen (B1), sourced from Venezuela, was tested. This bitumen is the base binder for mixtures discussed in Chapter 3. Secondly, mastic of bitumen and limestone fines (B2) was used. This mastic has been used to manufacture Mix OA which has been discussed in Chapter 3. This mastic included limestone fines < 0.063 mm which were blended with the 100/150 Pen bitumen. The resulted mastic had a filler/binder ratio of 1.5. The third binder (B3) consisted of bitumen, limestone and IBAA fines. This third binder was part of Mix BA. It included limestone and IBAA fines < 0.063 mm, with a ratio of 3.5:1. B3 had a filler/binder ratio of 1.3. To sum up, three binders were tested. These binders included the pure bitumen used to produce the bituminous mixtures which are used in this study. Two mastics which form the mastic part of mixes OA and BA were also tested.

It worth noting that the study does not include the mastics from mixes AA and CA. The reason is these two mastics have a composition which is very close to mastic B3. Trial tests showed that there were no significant differences between the behaviour of these two mastics and that of B3. Consequently, it was decided to test B1 which has no IBAA in its composition and B3 which includes IBAA. These two mastics may give a good indication about the effect of IBAA on binders' behaviour which is one of the targets of this chapter.

5.7.2 Specimen preparation

Specimens were prepared using two different methods. For B1, the pure binder, bitumen was heated to 80~90 ^{0}C above its softening point, which was found to be 43 ^{0}C as shown in Chapter 3. Then it was poured onto a release paper and left to cool at room temperature before storing at 5 ^{0}C for testing, as suggested by Walsh (1999).

Figure 5-3: High shear mixer.

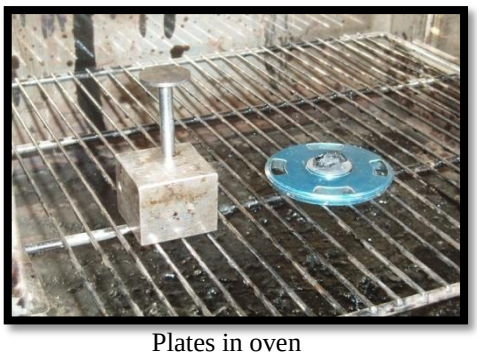
Binders B2 and B3, the mastics, were prepared as follows. Firstly, the blends used to produce the mixes OA and BA were sieved. Fines passing sieve size 0.063 mm were collected, heated to 140 ^{0}C for two hours and then blended with bitumen, heated to the same temperature, using a high shear mixer, shown in Fig 5-3. The resultant mastics were then poured onto a release paper and left to cool at room temperature before storing at 5 ^{0}C for testing.

Prior to testing, samples were cut and placed onto the lower DSR plate for the 25x1 mm geometry test conditions. For the smaller, 8x2 mm geometry, samples were divided into two halves and placed on both the lower and upper DSR plates. In both cases, plates were heated to 100 ^{0}C for a specified time depending on the used DSR geometry. Table 5-1 shows the different geometries used and their corresponding testing conditions. Weights used in this table were calculated based on assuming that the sample volume is similar to a thin disc of binder with diameter equal to the plate diameter and a height equal to the gap depth. Times and temperatures used were found, after trials, to be sufficient for a sample to flow across the entire surface of the plate. Test geometries and modes will be discussed later in this chapter.

Testing Condition	**25x1 mm geometry**		**8x2 mm geometry**	
	B1	**B2/B3**	**B1**	**B2/B3**
Testing mode	Constant strain rate mode Creep mode @ T ≥ 25 ^{0}C		Creep mode @ T < 25 ^{0}C	
Sample weight (g)	0.505	0.6	0.104	0.15
Heating time (sec)	360	480	240	300

Table 5-1: DSR testing programme

Sample on lower plate



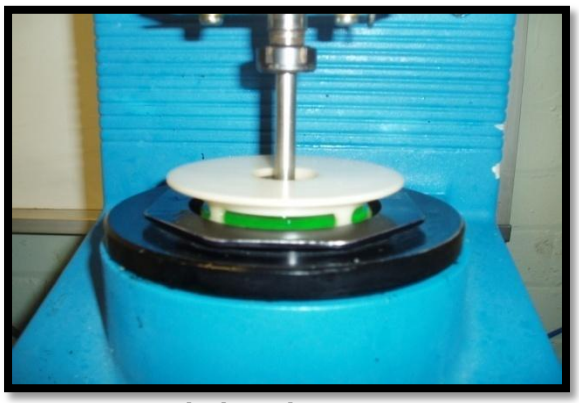
Plates in oven

Sample flow across plate

Lower plate positioning

Upper plate lowering

DSR ready to start



Fluid circulation

Small geometry

Figure 5-4: Specimen preparation procedure.

At the end of the heating process, samples flowed over the plates and were ready to be tested. The lower DSR plate was carried using a special holder and put in its place inside the DSR machine. Then it was tighten up using a hexagonal metal disc. The upper platen was screwed into its position after being protected with a plastic cap. The upper plate was then lowered and samples were sandwiched between the two plates. Then the system was flooded with a circulating fluid, kept at test temperature, for 10 minutes before commencing the test. This duration was necessary for samples to reach the desired test temperature gradually. Fig 5-4 shows pictures of specimen preparation procedure.

5.7.3 Testing geometries and modes

Two DSR testing geometries and two modes were used in this study. DSR geometries are related to both plate diameter and gap height. It is not uncommon to use plates of 8 or 25 mm diameters with 2 and 1 mm gaps respectively. The selection of the testing geometry is based on the operational conditions. It is recommended to use geometry of 25x1 mm for testing temperatures starting from 25 ^{0}C (Artamendi 2003). This recommendation conflicts with Airey and Hunter's study (2003). They suggested using this geometry for temperatures starting from 20 ^{0}C. Consequently, in this study, the larger geometry, 25x1 mm, was used at temperatures starting from 25 ^{0}C upwards while the smaller geometry, 8x2 mm, was used for tests undertaken at temperatures lower than 20 ^{0}C. At 20 ^{0}C, both geometries were tried. It was found that results from tests undertaken using the smaller geometry were in the same shape and behaviour of the rest results. Results from tests undertaken using the larger geometry had small shift which may be considered insignificant in some cases. However, for accuracy reasons, it was decided to use results from the small geometry only at 20 ^{0}C.

The DSR modes used in this study were the constant strain rate and creep modes. The constant strain rate mode was used to study the steady state permanent deformation of binders at high temperatures only ($T \geq 50$ ^{0}C), while creep mode was used at temperatures lower than 50 ^{0}C.

In the constant strain rate mode, a sinusoidal shear strain rate range was imposed upon the sample and the shear stresses required to produce the target deformation were recorded. Binders were subjected to a frequency range between 0.01 and 1 1/s over a temperature range between 50 and 70 ^{0}C. These conditions were selected based on the DSR machine compliance limits and the achievability of the steady state conditions for binders. At lower testing temperatures, DSR was found to reach its maximum loading capacity before the specimen reaches its steady state behaviour. Thus, creep mode was used at these lower temperatures.

In the creep mode, constant creep loads, ranged between 1 and 10 kPa, were applied and held on samples for 100 seconds and the resulting strains were recorded. Creep mode was used over a temperature range between 0 and 40 ^{0}C. The outcomes from creep mode, beyond this testing range, were found to be inconsistent.

5.7.4 Constant strain rate mode results

Data obtained from the DSR constant strain rate mode tests was used to establish stress strain relationships for all tested binders. Fig 5-5 shows the stress strain relationship for binder B1 tested at 60 ^{0}C.

It can be seen that, at each applied strain rate, the stress increased with strain up to a point, beyond which it levelled out before decreasing. It was noticed that the stress strain behaviour went through four stages. The first is characterised by the linear elastic stage in which binders behaved elastically and their stress strain relationship was linear. This stage was very short and happened at small strain values. In the second stage, non linear behaviour was observed up to a peak at which the material began to flow. The flow stage, the third stage, was very noticeable at lower strain rates. Beyond the flow region, the stress began to decrease with increased strain up to failure. Binder B1 was found to behave as explained at all test temperatures. Mastics B2 and B3 were found to have the same shape of behaviour regardless of the test temperature.

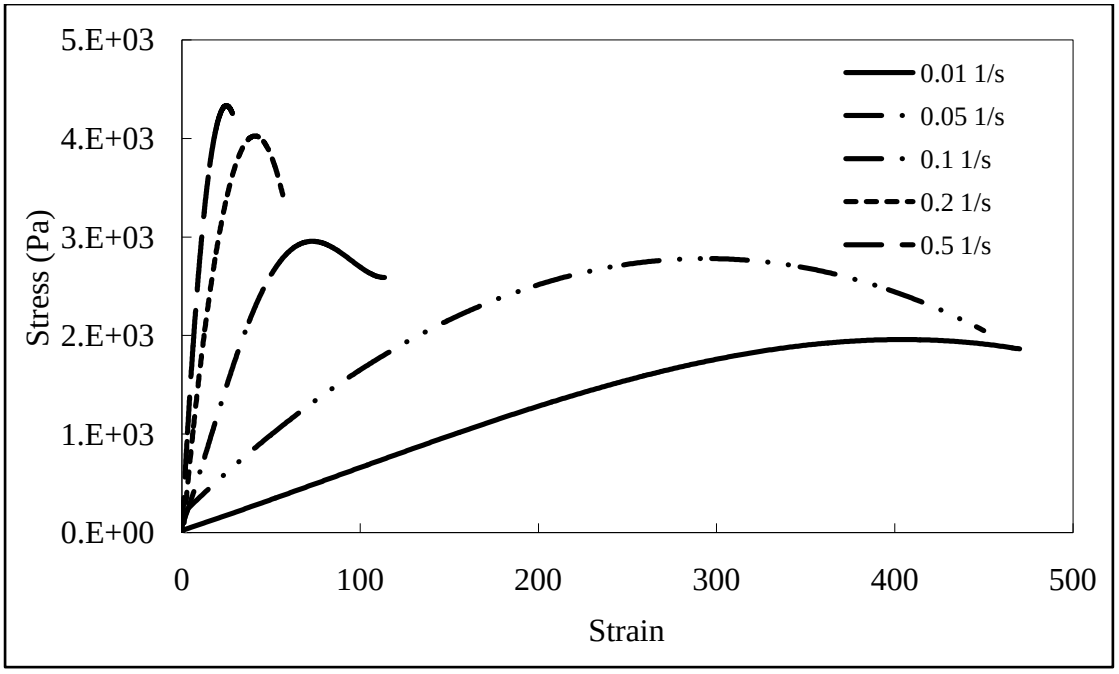


Figure 5-5: Stress strain relationship for binder B1 at 60 ^{0}C (Constant strain rate mode).

To find the steady state stress, a method proposed by Ward (1971) was adopted. In this method, the maximum observed stress, at each strain rate, was defined as the steady state stress at that particular strain rate.

The steady state stresses, found from the stress strain relationships, were plotted against their corresponding strain rates to produce a steady state permanent deformation curve for each binder over a range of temperatures between 50 and 70 ^{0}C. Figs 5-6 & 5-7 show these curves for binders B1 and B2 and B3 respectively. The MCM was fitted to the experimental results with which it was found to be in good agreement.

The steady state deformation curves of the mastics show that, although B2 and B3 are not identical binders, they had identical behaviour at 60 and 70 ^{0}C, whereas at 50 ^{0}C, the strain rates for B2 were higher than B3 although B2 contained more fines. The reason for this may be attributed to the fact that the two fines are different. B3 contained a blend of fines from limestone and IBAA, whereas B2 contained limestone

fines only. It has been reported that adding IBAA to limestone in a bituminous mixture improved its stiffness (Garrick and Chan 1993, Vassiliadou and Amirkhanian 1999, Zhang et al 1999, Ogunro et al 2004, Hassan and Khalid 2007). This may be due to the IBAA absorbing the more mobile components in bitumen. These components may react chemically with some elements in IBAA resulting in an enhanced IBAA toughness. Moreover, the non absorbed bitumen components would have more asphaltenes in the film coating the aggregates. Asphaltenes are known for their stiffening effect in bitumen. As a result, IBAA may have affected the overall deformation resistance of the mastic. The improved resistance was observed only at 50 ^{0}C. At higher temperatures, IBAA content was found to have insignificant effect and the mastics were dependent mainly on the filler/binder ratio.

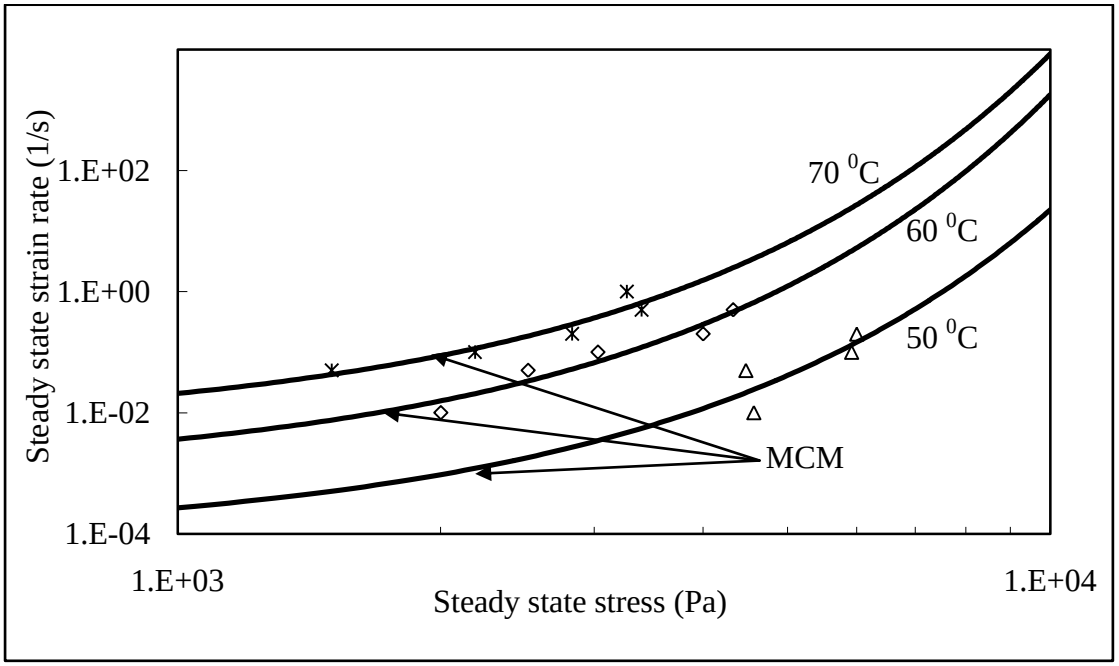


Figure 5-6: Steady state permanent deformation behaviour for binder B1 (Constant strain rate mode).

The constant strain rate mode could not be used at temperatures below 50 ^{0}C as the DSR reached its maximum loading limit before samples reached their steady state condition. This temperature is the lowest temperature above the bitumen's softening point which was reported in Chapter 3 as 43 ^{0}C. As a result, it was decided to use the

creep mode at temperatures lower than the bitumen's softening point to allow steady state attainment for the tested binders.

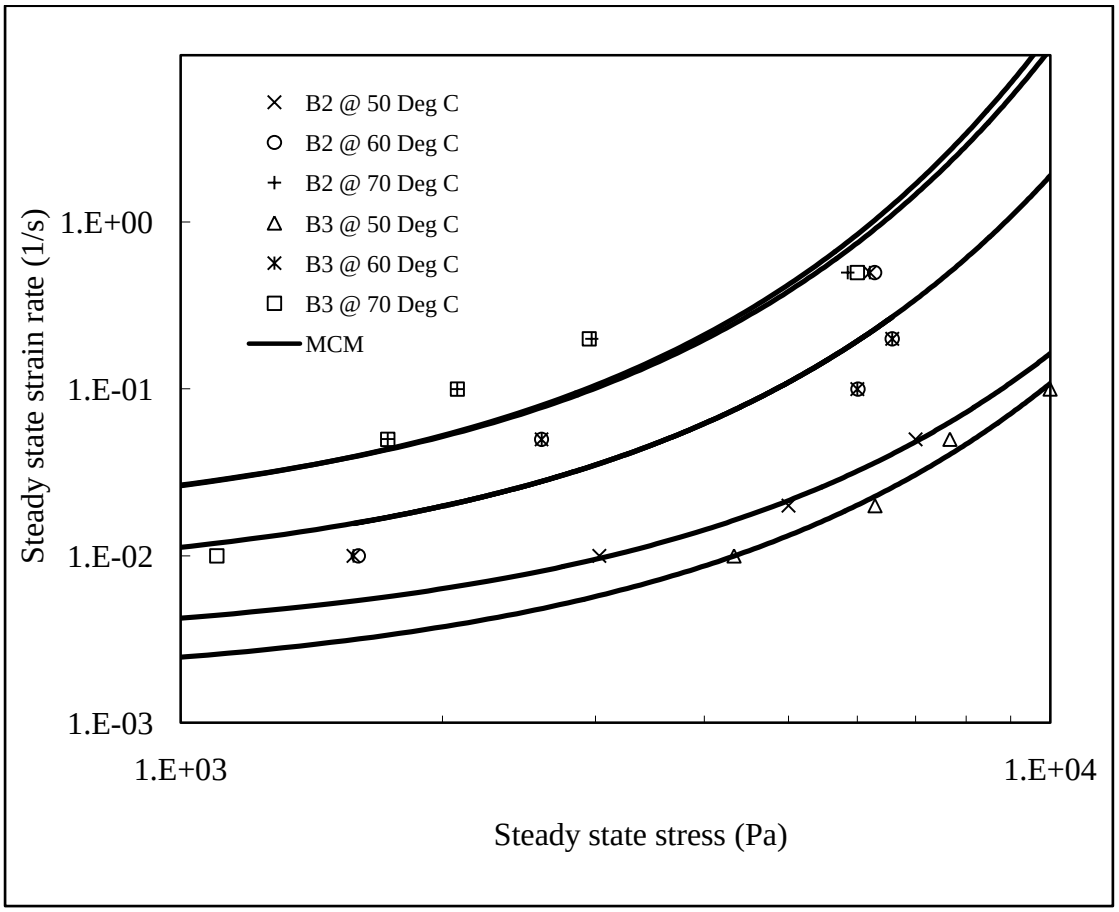


Figure 5-7: Steady state permanent deformation behaviour for mastics B2 and B3 (Constant strain rate mode).

5.7.5 Creep mode results

In creep mode, samples were subjected to a constant creep stress through a constant applied torque. This creep stress was held on samples for 100 sec. This duration was found to be sufficient for samples to reach their steady state. Data obtained from the creep mode tests was used to produce a relationship between shear strains and elapsed time after the application of the creep stresses. Fig 5-8 shows typical creep test results for mastic B2 from which it can be seen that a creep curve can be divided into two regions. In the first region, primary creep, the material behaved elastically. In the second region, the strain rate was approximately constant. This region was called the

secondary creep region and its strain rate was adopted as the steady state strain rate corresponding to a particular value of applied shear stress. A third region, not visible in Fig 5-8 but appeared only in some tests, exhibited tertiary creep in which the strain rate increased as the specimen became progressively damaged. Binder B2 was found to behave as explained at all creep stresses. Binders B1 and B3 were found to have the same behavioural shape regardless of stress and temperature used in the test.

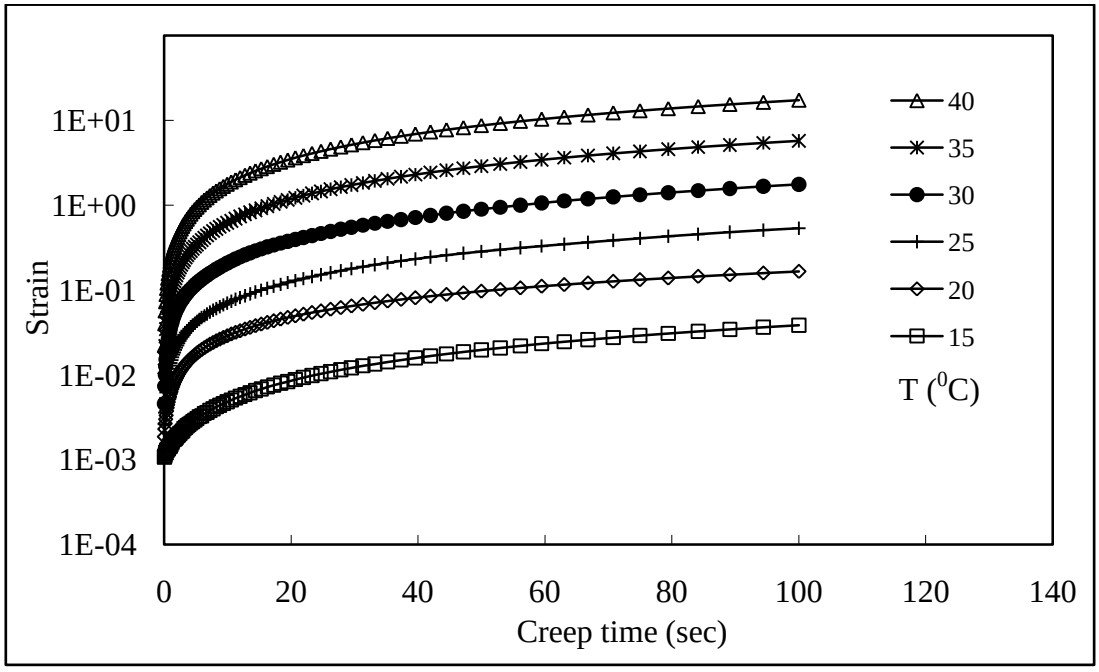


Figure 5-8: Creep behaviour for mastic B2 under creep stress of 5 kPa.

The steady state strain rates, found from the creep results, were plotted against their corresponding stresses to produce a steady state permanent deformation curve for each binder over a range of temperatures between 0 and 40 ^{0}C. Figs 5-9 & 5-10 show the steady state permanent deformation curves for binders B1, and B2 and B3 respectively. The MCM was fitted to the experimental results and it was found to produce a good fit.

Creep mode tests at temperatures above 40 ^{0}C were attempted aiming to find temperatures at which results from constant strain rate and creep tests overlap. It was found that creep results at temperatures above 40 ^{0}C did not fit to the MCM and they

had different behaviour shapes compared with those obtained from tests conducted at temperatures up to 40 ^{0}C.

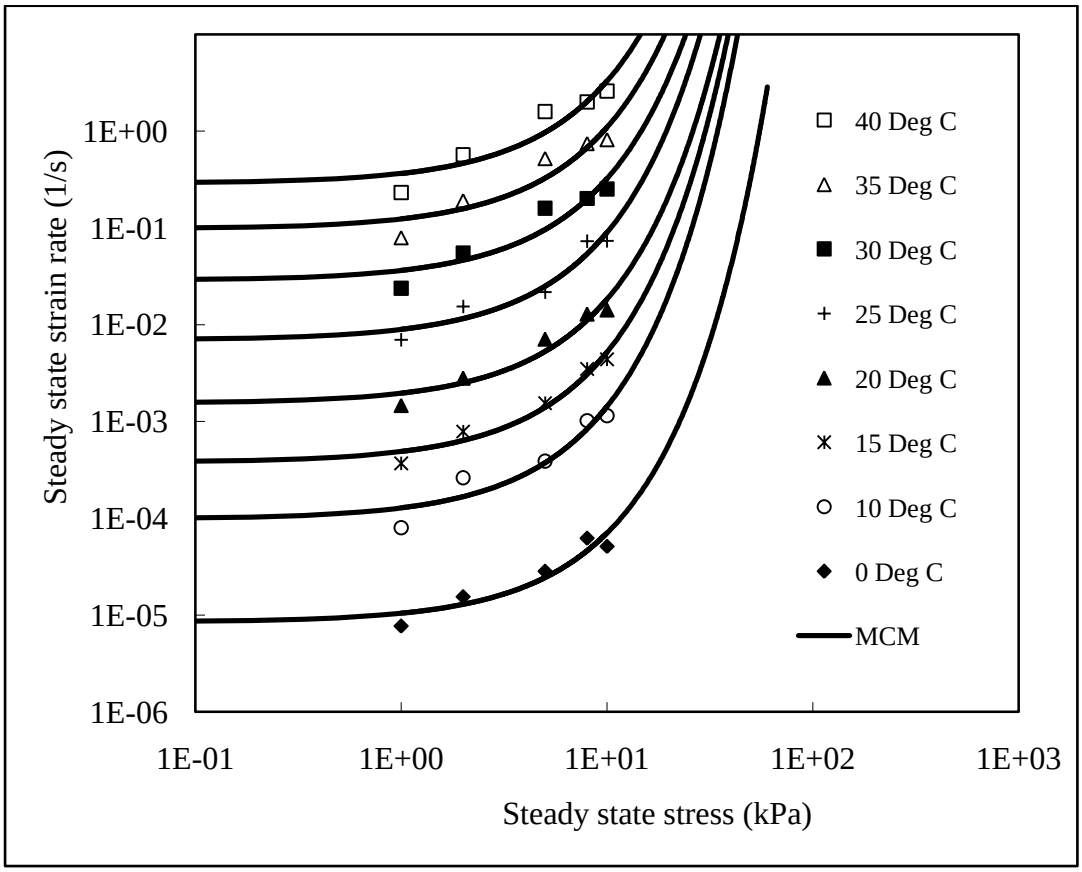


Figure 5-9: Steady state permanent deformation behaviour for binder B1 (Creep mode).

In summary, it was obvious that DSR creep mode was suitable to study binder's steady state permanent deformation behaviour at temperatures lower than the softening point of the pure bitumen used. On the other hand, the constant strain rate mode was suitable to do the same job at temperatures higher than the softening point of the pure bitumen used. Moreover, the results had not the same shape of behaviour, however captured by the MCM.

The steady state permanent deformation behaviour of binders determined from the DSR creep mode was found to follow a similar trend in behaviour as their respective mixtures, as will be explained in Chapters 6 and 7. Consequently, it can be concluded

that using the DSR creep mode was suitable to study binders' deformation behaviour. On the other hand, using the DSR constant strain rate mode needs further investigation to find out if mixtures' deformation behaviour at temperatures as high as 50 ^{0}C or even higher will exhibit same shape of behaviour as their respective binders at the same temperatures. This could not be achieved in this study because of lack of suitable facilities to study mixtures' deformation behaviour at such high temperatures without samples handling problems.

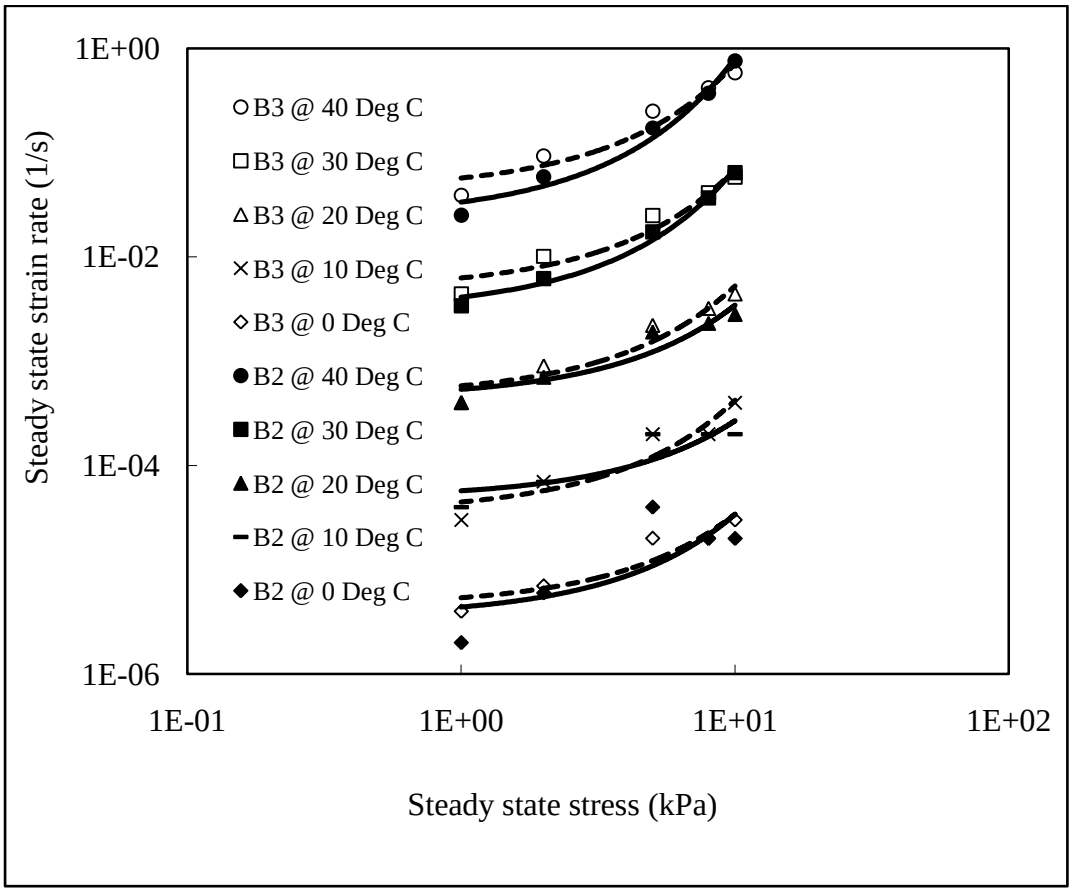


Figure 5-10: Steady state permanent deformation behaviour for mastics B2 and B3 (Creep mode).

5.7.6 Effect of IBAA on binder's deformation behaviour

Figs 5-9 and 5-10 show that B1 has higher strain rates, at the same temperature, compared with B2 and B3 which was expected for a pure binder. At temperatures below 20 ^{0}C and generally low stress levels, B3 had lower strain rates than B2.

However, at the same temperature range and higher creep stress levels, B3 had higher strain rates than B2. At temperatures between 20 and 40 ^{0}C, the situation is stress level dependent; B3 had higher strain rates than B2 at low stresses whereas B2 had higher strain rates at the high stresses.

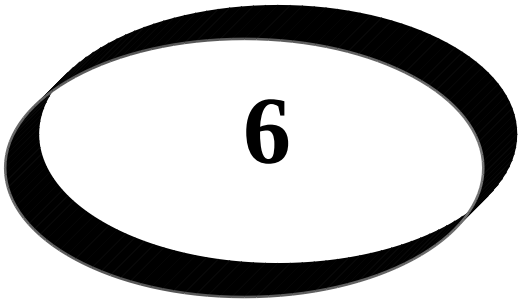
Based on these results, it can be stated that the pure binder's steady state permanent deformation performance was temperature and stress level dependent. However, it was softer than the two mastics in creep test mode. Regarding mastics, the steady state permanent deformation behaviour was also found to be dependent on temperature and stress level. At low temperatures and stress levels, the IBAA mastic, B3, exhibited higher deformation resistance than B2. This means that IBAA had an effect on the mastic's deformation resistance. The same effect was noticed at higher temperatures, i.e. between 20 and 40 ^{0}C, and high stress levels. However, at any other combination of temperature and stress level, i.e. high temperature and low stress level or high stress level and low temperature, IBAA was found to have no effect on the mastic's deformation resistance. In summary, IBAA improved the mastic's deformation resistance only under certain temperature and stress level conditions.

5.8 CONCLUDING REMARKS

The following conclusions can be made based on the presented results.

- The DSR creep mode can be used to predict binder steady state permanent deformation behaviour at temperatures up to 40 ^{0}C. The constant strain rate mode can be used for temperatures from 50 ^{0}C upwards.
- In the constant strain rate mode, the steady state stresses increased with strain rate and decreased with temperature.
- In the creep mode, the pure binder had higher strain rates than the mastics, at the same temperature.
- The strain rate for the pure bitumen was found to be more susceptible to a change in stress than the two mastics.

- Over a wide range of temperatures and stress levels, B3 has strain rates lower than B2. This means that the IBAA, in B3, has a stiffening effect on the binder leading to improved permanent deformation behaviour.
- IBAA was found to have no effect on binders' permanent deformation behaviour at temperatures > 50 ^{0}C.
- Machine compliance limits DSR coverage as DSR frequency mode could not be undertaken at low temperatures. Thus, other tools should be used in addition to allow a wider temperature range in studying steady state permanent deformation of pure binders and mastics.



PERMANENT DEFORMATION BEHAVIOUR – UNIAXIAL TESTS

6.1 INTRODUCTION

Permanent deformation or rutting is one of the most important failure modes in asphalt pavements (Barksdale 1967) that affects pavement life. Permanent deformation in asphalt mixtures can be defined as the irrecoverable cumulative deformation that occurs mainly at relatively high temperatures in the wheel paths as a result of repeated traffic loading. The permanent deformation results in depressions on the pavement surface along the wheel paths relative to other points on the surface. The depressions are as a result of downwards and lateral movement of the asphalt mixture. The downward movement is mainly due to compaction while the lateral movement occurs as a result of shear failure.

Permanent deformation is caused, mainly, by the accumulation of permanent deformation in all or some of the layers in the pavement structure which is considered to be the main component of rutting in flexible pavement. This is because of the increase in tyre pressures and axle loads, which causes high stresses in near surface asphalt layers. These stresses caused the majority of permanent deformation to be recorded in the top 75 to 100 mm of the asphalt layers (Brown and Cross 1992). Moreover, it was reported (European Commission 1999) that permanent deformation originated in bituminous layers is the most common form of pavement deterioration on European roads. Permanent deformation may also initiate other form of pavement distress such as cracking. Thus the study of permanent deformation properties of asphalt mixtures has become a focus of research, which aims to mitigate the problem of rutting in flexible pavements.

The aim of this chapter was to contribute towards understanding permanent deformation in bituminous mixtures containing IBAA. The mechanisms, factors affecting permanent deformation and methods of its evaluation in asphalt mixtures were reviewed. Special emphasis was given to the steady state permanent deformation behaviour of the IBAA bituminous mixtures, under uniaxial load testing conditions, and to the relation between this behaviour and that of their respective binders. The effect of IBAA content level on this behaviour was highlighted.

6.2 MECHANISMS OF PERMANENT DEFORMATION OF BITUMINOUS MIXTURES

The mechanism of permanent deformation in asphalt mixtures is dependent on the volumetric composition of the mix and the properties of its material components. The volumetric composition includes the volumes of aggregate, binder and air. In the past, it was assumed that permanent deformation of asphalt mixtures is developed by displacement of the mixture at constant volume (Dawley et al 1990). This mechanism is called shear deformation and is associated with lateral movement of material at constant volume. It was reported as the primary permanent deformation mechanism in asphalt (Hofstra and Klomp 1972, Eisenmann and Helmer 1987). Shear deformation mechanism takes place because of the viscous behaviour of bitumen (Deshpande

1997) and occurs throughout the pavement's life. Shear deformation of the asphaltic mixtures depends on the properties of constituent materials, their composition, stress state and environmental conditions.

In a different way, densification was reported as the main permanent deformation mechanism in asphalt. Densification, which is associated with a decrease in volume and an increase in density, takes place when the aggregate skeleton becomes more closely packed. It was found that the majority of densification of asphalt by traffic occurs relatively early in the pavement's life (Newcomb et al 1997).

Recent research (Sousa et al 1993, Florea 1994, Erkens, 2002, Tashman 2003, Rismantojo 2004, Masad et al 2005, Saadeh 2005, Collop et al 2006, Gibson 2006) has suggested that asphalt mixtures dilate after a densification period. This dilation is a phenomenon of volume increase that occurs in a particular media when loaded beyond the elastic limit (Song and Pellinen 2007). Dilation is usually expressed in terms of dilation gradient. The dilation gradient is the ratio of volumetric strain to shear strain. This mechanism has been noticed, in this research, to be the permanent deformation mechanism in IBAA bituminous mixtures. However, a short time densification mechanism was noticed before the onset of dilation as will be shown later in this chapter.

6.3 FACTORS AFFECTING PERMANENT DEFORMATION OF BITUMINOUS MIXTURES

The permanent deformation properties of asphalt have been found to be highly influenced by aggregate properties, binder properties, volumetric composition of mixtures and lab and field conditions.

6.3.1 Aggregate properties

Aggregate is a major portion of an asphaltic mixture and it is liable for the strength and toughness of the material. The physical properties of aggregates extensively affect the performance of asphalt pavement in service. The main aggregate properties which affects permanent deformation resistance of bituminous mixtures include surface texture, angularity, gradation, and filler.

The surface texture of the aggregate plays an extremely important role in the permanent deformation of bituminous mixtures. The type of aggregate surface texture depends on characteristics of the parent rock as well as the extent to which forces acting on the particle surface have smoothened or roughened the surface (Su 1996). Rough surface texture is known to increase the permanent deformation resistance especially at high temperatures. This is because it allows better bond between aggregate and binder. Angularity, or particle shape, is also important. It has been reported that angular materials increases permanent deformation resistance compared with rounded materials. (Uge and van de Loo 1974). This is because rough faces on the aggregate particles allow higher friction strength to be developed under loads which try to force particles to slide over each other. This was shown by Kalcheff and Tunnicliff (1982) who investigated the effects of crushed stone aggregate size and shape on asphalt mixture properties. They concluded that mixtures containing crushed coarse and fine aggregates with or without high proportions of mineral filler should be more resistant to permanent deformation resulting from repeated traffic loading. The same fact has been reported in a literature review study by Sanders and Dukatz (1992). Kobayashi et al (1997) conducted an extensive study of the effect of fine aggregate shapes on characteristics of asphalt mixtures using image processing method. They concluded that the use of angular fine aggregate can enhance performance of pavements with regard to rutting resistance. Moreover, the effect of aggregate angularity on plastic deformation of asphalt was investigated (Perdomo et al 1999). It was found that replacing rounded, smooth, sand-sized aggregates with rough, angular, porous particles, while keeping the other aggregates and total gradation unchanged, caused mixes containing 40% natural sand to have significantly lower resistance to plastic deformation than those mixes prepared without using natural sand (i.e. using angular and rough particles).

Gradation refers to the particle size distribution of aggregates inside bituminous mixtures. Excess using of certain size fractions may lead to acceleration of rutting occurrence. Several authors have studied the effect of aggregate gradation on resistance to permanent deformation. Dukatz (1989) showed that gradation is a main factor in permanent deformation resistance. Oliver et al (1997) carried out both field and laboratory study on several mixtures and concluded that, among other factors, aggregate gradation has a significant influence on rutting resistance. Carpenter and Enockson (1999) studied 32 overlay projects in USA and concluded that majority of the rutting problem can be attributed to aggregate gradation. Recently, Tarefder et al (2003) found that aggregate gradation comes in the third place, after binder content and temperature, amongst the most significant factors which affect rutting potential of asphalt mixtures.

Although the clear importance of aggregate gradation effect on permanent deformation resistance of asphalt, no consistent pattern was found in the literature for this effect. Brown et al (1984) concluded that continuously graded mixtures had more resistance to permanent deformation than gap graded mixtures. However, gap graded mixtures with a high volume fraction of coarse aggregate (more than 70% in their study) had high deformation resistance due to their high aggregate interlocking. Davis (1988) found that using large maximum aggregate size in low air void content asphalt pavements led to improved deformation resistance. Moreover, asphalt mixtures composed of different gradations but of similar mineralogical composition exhibited significantly different permanent deformation behaviour as found by Kandhal and Mallick (2001). On the other hand, Brown and Cross (1992) concluded that aggregate properties have little effect on permanent deformation when voids are less than 2.5% based on their study, which involved laboratory field collected samples. Even when percentage of voids is greater than 2.5%, Brown and Cross argued that it is the fine aggregate angularity and not the gradation that has a significant influence on permanent deformation resistance. Barksdale also (1967) studied rutting of paving materials and found that permanent deformation in dense graded asphalt concrete was not sensitive to gradation of aggregates. Consequently, researchers appear to have come to different conclusions with regard to the effect of aggregate gradation on resistance to permanent deformation of asphalt mixtures. However, most researchers

seem to agree that aggregate gradation has a slight influence on permanent deformation resistance.

Fillers are usually defined as material passing the 0.063 mm sieve. Fillers are added to paving mixtures to increase stability and to improve the bond between the mastic and the aggregates. Part of the filler is embedded in the bitumen forming mastic and increasing binder stiffness. The second part of the filler fills the voids in the aggregate matrix providing contact points between large aggregate particles which result in increasing density and strength of the compacted mixtures. Few researchers studied the effect of filler filling the aggregate voids on asphalt mixtures' permanent deformation resistance. The creep stiffness of dense asphalt mixtures was studied by Bolk et al (1982) using two different types of filler: limestone powder and fly ash. They concluded that filler type notably affected the creep stiffness, and consequently permanent deformation resistance, of the tested mixtures. Al Suhaibani et al (1992) studied three filler types: limestone dust, hydrated lime, and Portland cement. The effect of filler type and content on rutting potential of asphalt mixtures was investigated using a wheel tracking test. They concluded that rutting in asphalt mixtures is highly dependent on the softening point of the mastic, which, in turn, depends on the type and proportion of filler. Moreover, it was found that mixes made with limestone dust exhibited lower rut depth than those containing either Portland cement or hydrated lime. A similar research (Kavussi and Hicks 1997) studied the effect of using different fillers, e.g. limestone, quartz, fly ash, and kaolin, on the physical properties of asphalt mixtures. The authors concluded that filler type and amount have a considerable effect on the flexural properties of a mix. As a result of an increase in filler content, there was an increase in flexural stiffness. The mentioned studies emphasised the important effect for filler type on permanent deformation resistance of asphalt mixtures.

On the other hand, many researches studied the effect of filler embedded in binder on the permanent deformation resistance of asphalt mixtures. Heukelom and Wijga (1973) suggested that the stiffness of the mastic and asphalt mixtures is influenced by the stiffness of the bitumen and the content of filler and bitumen. Increasing filler

content led to an increase in binder viscosity and consequently in mixture stiffness. However, there is a limit to the amount of filler that can be added to mixtures as high proportion of filler requires more binder to cover the extra surface area, which may create problems in achieving the required compaction, and may aggravate ageing and cracking. In another study, Anderson et al (1992) observed that the relative stiffening effect of fillers depends on the type of binder. They concluded that fillers are expected to improve permanent deformation resistance of asphalt as a result of increasing moduli and this effect is expected to be bitumen-filler specific. However, the authors did not specify whether this effect also depends on the proportion of the filler in the mix. Kavussi and Hicks (1997) found that viscosity of filler-bitumen mastic is directly related to the particle size of the filler. Although the importance of filler effect on permanent deformation of asphalt mixtures is clearly highlighted in the literature, it is worth mentioning that different filler types have different effects on bitumen performance. Moreover, great attention should be drawn to the effects of filler on binder content, mixture workability, susceptibility to cracking, and moisture susceptibility of mixtures as these are highly influenced by filler type.

Thus the available literature on aggregate properties' effect on the permanent deformation resistance of asphalt mixtures shows the positive effect of both rough surface texture and angularity. Based on this fact and on the IBAA physical properties presented in Chapter 2, it was predicted that IBAA could have positive effect on permanent deformation resistance of bituminous mixtures. This effect was tested and will be shown in this chapter. On the other hand, literature shows mixed opinions with regard to aggregate gradation and filler effects. However, these two factors were, approximately, constants in this research and, consequently, did not contribute into permanent deformation behaviour of bituminous mixtures.

6.3.2 Binder properties

Bituminous binders are visco-elastic materials whose resistance to deformation under load is very sensitive to loading time and temperature. This behaviour depends on bitumen grade, stiffness and content. The effect of aggregate bitumen interaction plays another important role in the contribution of binder towards permanent

deformation of asphalt mixtures. This includes the effect of filler on binder properties which was discussed in the previous section.

Grade and stiffness of bitumen were found to appreciably affect the viscous deformation of bitumen. Hofstra and Klomp (1972) compared permanent deformation resistance in sections of asphalt made with various grades of bitumen. They found that the higher the bitumen grade the higher the rut depth was. Similar findings were concluded in a study by Mahboub and Little (1988) in which they concluded that less viscous bitumen resulted in less stiff bituminous mixture and, therefore, increased their susceptibility to rutting. Moreover, Heukelom and Wijga (1973) found that an increase in the stiffness led to a decrease in the amount of viscous deformation in the bitumen relative to the delayed-elastic and elastic deformation thereby leading to increased resistance against permanent deformation. De Visscher et al (2006) found that binder type had a significant effect on repeated load triaxial tests conducted on laboratory and field cores. Recent researche showed that modification of the bitumen can enhance the bitumen stiffness and the deformation resistance of the asphalt mixture. For example, Ghile (2006) studied dense asphalt mixtures composed of unmodified and modified bitumen. The study found that the modified bitumen not only improved the stiffness of the bitumen at high temperatures but also enhanced the resistance against permanent deformation of the dense mixtures under unconfined repeated load compression testing conditions. It worth noting that Tarefder et al (2003) concluded that binder grade is the most significant factor that affects rutting potential of asphalt mixtures.

Binder content is a main mixture design parameter. It is known that the amount of binder in the mixture affects the durability and performance of the mixture. Most of the studies on the effect of binder on permanent deformation resistance of mixtures found in the literature focused on the effect of binder type (or grade) rather than the binder content. However, studies that considered the effect of binder content found that it has important influence on resistance to permanent deformation of mixtures. Tests conducted on asphalt specimens having different binder contents and compacted to different void levels indicated that an increase in binder content results in a

decrease in resistance to permanent deformation (Asphalt research programme 1994). The same conclusion was presented by Coree and Button (1997). They tested bituminous mixtures made with 38 mm nominal maximum size aggregate, with three levels of binder contents. Results showed that an increase in binder content leads to an increase in rutting. In Saudi Arabia, a study on severe rutting found in a test road, concluded that using high binder content was the main cause of this problem (Lee and Al-Dhalaan 1989).

Based on these findings and on the mix design results presented in Chapter 3, it can be predicted that using IBAA, which required higher binder contents, may result in decreasing the permanent deformation resistance bituminous mixtures. This prediction is contrary to that prediction which was built on aggregate properties effects. Therefore, it is very difficult, when it comes to IBAA, to depend on mixtures' properties to predict the IBAA effect on rutting resistance of bituminous mixtures.

Studies on identifying the main controlling factor, between aggregate and bitumen, in permanent deformation of asphalt mixtures are not uncommon. However, this kind of investigation is beyond the scope of this research. Gibb (1996) conducted compressive cyclic uniaxial tests on two types of asphalt mixtures, dense bitumen macadam and hot rolled asphalt, over a range of temperatures using two bitumen grades. He found that at low binder stiffness conditions (specified temperatures and bitumen grade), the aggregate structure was more important than the binder in providing resistance to permanent deformation. On the other hand, at high binder stiffness conditions, it was the contrary. Thus, the study concluded that, up to a certain level of binder stiffness, the deformation resistance of the mixtures is dominated by the binder. Below that level, the binder is not able to contribute significantly to deformation resistance, and the role of the aggregate structure is dominant. However, no clear information was given about this critical point. In another study, Pellinen and Witczak (2002) found the aggregate influence to be more dominant than the influence of the binder on the modulus at high temperatures (> room temperature) and the binder influence to be more dominant over the aggregate influence at low temperatures (< room temperature).

Based on the above mentioned studies, the importance of bitumen role in permanent deformation resistance can not be neglected. It can be understood, that at high temperatures, the binder viscosity is high and aggregate interlock is more important in providing resistance to deformation. Under these conditions, high bitumen content reduces aggregate interlock and, therefore, the resistance to deformation is reduced. Low bitumen content will result in more aggregate interlock and, therefore, the resistance to permanent deformation is increased. At lower temperatures, medium bitumen content will give the maximum contribution of both aggregate interlock and binder viscosity to resistance to deformation.

6.3.3 Volumetric composition

The volumetric composition of asphalt mixtures is one of the important factors which affect mixture properties. The volumetric composition can be expressed in terms of voids. Voids in the pavement are controlled by bitumen content and compaction. Ford (1988) studied the effect of air voids on pavement performance. He measured voids in cores taken from the field and correlated it to measured rutting. The author concluded that air voids have effect on rutting with correlation coefficient of 0.674, and that rut depth increases with decreasing air voids. This study also showed that pavements with air void content of 2.5 or less had excessive rutting. Other studies (Brown and Cross 1992, May and Witczak 1992, Mallick et al 1995, Roberts et al 1996) suggested the minimum air voids to be 3% to minimise rutting. These recommendations were met in this research as can be seen in Chapter 3.

The effect of voids in mineral aggregate (VMA) was also investigated in several occasions. Cooper et al (1985) concluded that good resistance to permanent deformation requires low voids in the mineral aggregate. However, they cautioned that very low voids in mineral aggregate value may not allow sufficient voids in the aggregate for enough binder to ensure satisfactory compaction and to prevent overfilling that may occur as a result of extension of the volume of bitumen at high temperatures. This is because, at low voids in mineral aggregate, the mastic is forced between coarse aggregate particles reducing the number of contact points and, therefore, resistance to permanent deformation is reduced. Asphalt Institute (1997)

presented a table for the minimum percent voids in mineral aggregate to be used in hot mix design methods, see Table 6-1. The VMA recommendations were met in this research as can be seen in Chapter 3.

Design air voids (%) / **Nominal Maximum size (mm)**	**Minimum VMA (%)**		
	3.0	**4.0**	**5.0**
1.18	21.5	22.5	23.5
2.36	19.0	20.0	21.0
4.75	16.0	17.0	18.0
9.5	14.0	15.0	16.0
12.5	13.0	14.0	15.0
19.0	12.0	13.0	14.0
25.0	11.0	12.0	13.0
37.5	10.0	11.0	12.0
50.0	9.5	10.5	11.5
63.0	9.0	10.0	11.0

Table 6-1: Minimum percent voids in mineral aggregate (VMA) (Asphalt Institute 1997)

6.3.4 Laboratory and field conditions

There are some laboratory and field conditions that affect the permanent deformation behaviour of asphalt mixtures in a minor way compared to the above mentioned factors. Compaction effort during construction and, thereafter, during pavement service can affect the rutting resistance of asphalt. Moreover, temperature has an effect on bitumen properties and, consequently, mixture properties.

The goal of compacting asphalt pavement is to achieve an optimum air void content, to provide a smooth riding surface and to increase the bearing capacity of the material under construction. Linden and Van der Heide (1987) investigated the influence of compaction in asphalt mixtures. They concluded that the degree of compaction was a governing parameter in asphalt mixtures especially at low bitumen content. However, there were no direct research on the effect of laboratory or field compaction methods and efforts on the permanent deformation of asphalt mixtures. Therefore, this area, although beyond the scope of this study, needs more investigation.

As bitumen is a temperature dependent material, properties of bituminous mixtures are highly influenced by temperature. Thus, temperature is considered to have a large effect on permanent deformation of asphalt mixtures. Tarefder et al (2003) produced temperature effect as the second most significant influence on rutting potential of asphalt mixtures. Hofstra and Klomp (1972) found that increasing wheel track test temperature from 20 to 60 ^{0}C resulted in 250-350 times increase in rut depth. Linden and Van der Heide (1987) reported a significant increase in rutting in Europe during the very hot summers of 1975 and 1976. Similar findings reported for USA by Mahboub and Little (1988). Hence, it is desirable to test bituminous mixtures under relatively high test temperatures noting that low test temperatures, although should be included, could not be used on their own. Consequently, a relatively wide range of temperature, 5 to 40 ^{0}C, is considered in this research.

6.4 SSPD OF BITUMINOUS MIXTURES

Recent interest in studying steady state permanent deformation behaviour experimentally and theoretically has shown the need for investigating this behaviour for bituminous mixtures containing secondary materials, e.g. IBAA in this research. The term steady state permanent deformation will be used, in this study, as SSPD for brevity reasons. Recent studies on SSPD aimed to contribute towards understanding of the viscous behaviour of bituminous materials. Firstly, the SSPD behaviour of bitumen was investigated. This was followed by a few studies on the SSPD of idealised bituminous mixtures. Then, several realistic asphalt mixtures were studied. The SSPD behaviour of bitumen was discussed in Chapter 5. In this chapter, the SSPD behaviour for idealised and realistic mixtures is reviewed. The following sections in this chapter aim to study the SSPD behaviour of bituminous mixtures containing IBAA. This should enhance understanding of permanent deformation behaviour of asphalt. Moreover, it highlights an important mechanical property for bituminous mixtures containing secondary aggregate.

6.4.1 SSPD of idealised mixtures

Investigating the SSPD behaviour of idealised mixtures was an important step that followed the binder studies before moving onto investigating the behaviour of realistic mixtures. Idealised mixtures are bituminous mixtures consisting of bitumen and a very simplified aggregate grading (mainly sand with high bitumen content).

A number of publications (Deshpande 1997, Deshpande and Cebon 1999a, 1999b, 2000, 2004) investigated the SSPD behaviour of idealised asphaltic mixtures with various volume fractions ranging between 40 and 85% sand. Mixtures included a range of different types of sands in terms of sizes and shapes. The mixtures were made using the same bitumen as used by Cheung (1995) with the aim of relating mixtures' behaviour to their respective binders'. Two types of test were conducted on the produced specimens: compressive static uniaxial and triaxial creep and constant strain rate tests. A range of deviatoric stress levels, stress ratios (mean stress/deviatoric stress), strain rates and temperatures were used in the tests. It was

found that the SSPD behaviour of the idealised mixtures and the temperature dependency were of the same form as that of the respective bitumens with the aggregates acting as a stiffener (see Fig 6-1). Therefore, to describe the SSPD behaviour of the idealised mixtures, the model developed by Cheung (1995) for bitumen, and discussed in Chapter 5, was modified by introducing a stiffening factor S without affecting the general shape for the MCM. The new equation will be discussed later in this chapter.

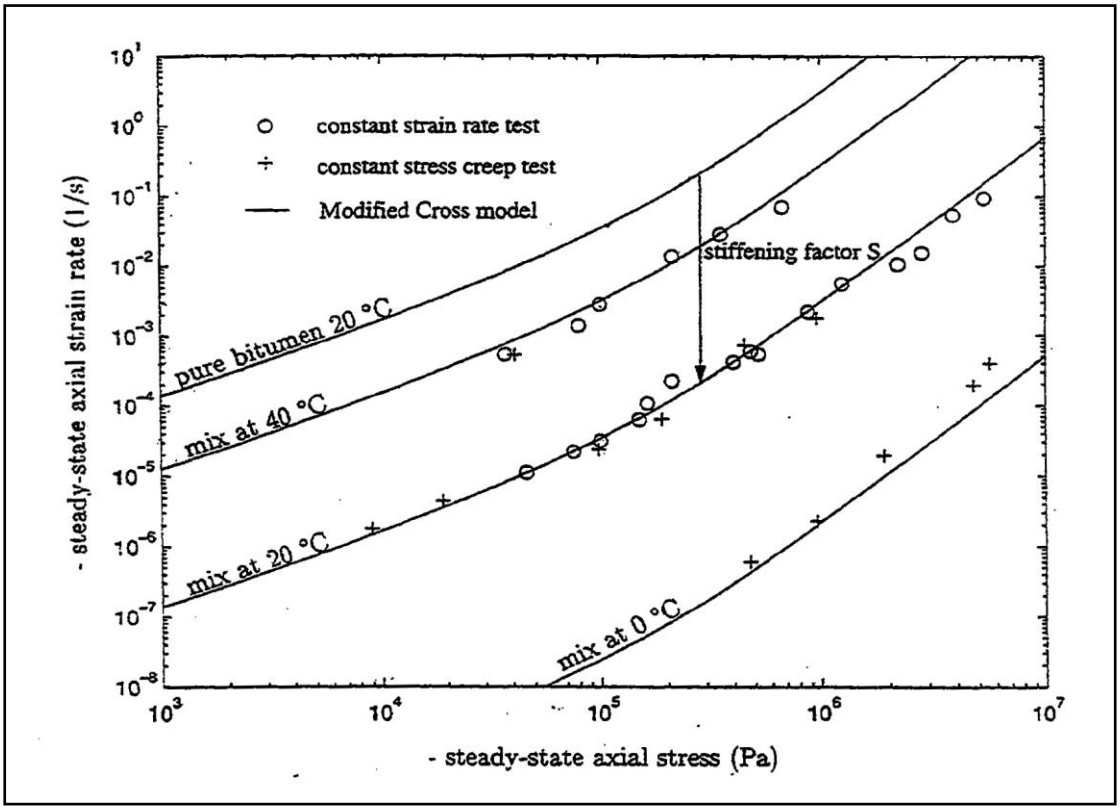


Figure 6-1: Steady state permanent deformation behaviour of an idealised mixture, with 64% by volume sand and 50 Pen pure bitumen (Deshpandee 1997).

The stiffening factor was found to depend on the volume fraction of aggregates in the idealised mixture, temperature, air void content and stress ratio, but was independent of aggregate size. It was found that the stiffening factor was the same in the linear and non-linear viscous regimes of behaviour.

The radial strain of the samples was also measured in the uniaxial and triaxial tests. It was found that the volumetric strain varies linearly with shear strain. The slope of the relation, known as the dilation ratio, was found to be independent of temperature, stress ratio and deviatoric stress, strain rate and type of test.

Other publications (Collop and Khanzada 1999, Khanzada 2000) investigated the relationship between the rutting performance of the idealised mixtures, measured by wheel tracking tests, and the mechanical properties of two idealised mixtures, measured by simplified uniaxial quasi-static and uniaxial repeated load tests. One idealised mixture comprised a single size sand (between 1.18 mm and 2.36 mm) and the other comprised two different sizes of sand (between 1.18-2.36 mm and 150-300 μm). Both mixtures were made with a 50 Pen bitumen. Uniaxial constant stress creep and constant strain rate tests were performed over a range of stresses and strain rates at 20 ^{0}C and 30 ^{0}C. Results showed that the SSPD behaviour of the idealised mixtures mirrored that of their respective bitumens with the stiffening effect of the aggregates. At high stress levels (>500 kPa), the SSPD behaviour was non-linear, following power law creep with a creep exponent of approximately 2.4, and at low stress levels (<70 kPa), the behaviour was linear with a power exponent of approximately 1. Between 70 kPa and 500 kPa, there was a transition from non-linear behaviour to linear viscous behaviour.

The deformation behaviour of the idealised mixtures under uniaxial repeated loading was also investigated over a range of temperatures and stress levels between 50 kPa and 100 kPa. The variation of the axial strain with the number of cycles was found to be similar, in shape, to that variation against time from static creep tests. Results showed that the SSPD behaviour of the mixtures under repeated load followed a power law creep function with creep exponent values varying between 1.1 and 1.5. In addition, laboratory wheel tracking tests were undertaken on the idealised mixtures over a range of temperatures and stress levels between 500 kPa and 1500 kPa. Rut depth against the number of load passes was plotted for each test condition. Similar to the patterns from static uniaxial and repeated load tests, after an initial period where the rutting rate decreased, the rut depth increased linearly in proportion to the number

of cumulative load passes. The constant rutting rate was used to characterise the SSP rutting rate of the mixtures. It was found that, over the range of stress levels applied in the tests, the rutting rate varied non-linearly with stress level. The non-linearity followed the power law creep function with the creep exponent values from 1.9 to 2.4 which are similar to the creep exponent of static creep tests.

6.4.2 SSPD of realistic mixtures

Very few studies were conducted on SSPD behaviour of bituminous mixtures. Khanzada (2000) investigated the SSPD behaviour of two realistic asphaltic mixtures, Hot Rolled Asphalt (HRA) and Dense Bitumen Macadam (DBM). Compressive static uniaxial and triaxial tests, uniaxial repeated load tests and laboratory scale wheel tracking tests were carried out on the mixtures. Khanzada designed both realistic mixtures with a binder content of 7% and target air void content of 7% with intention of making the specimens close to the idealised mixtures he had used as discussed in the previous section.

To study uniaxial SSPD behaviour, uniaxial tests under constant stresses and constant strain rates were carried out at 20 ^{0}C. The steady state strain rate from the uniaxial creep test results and the steady state stress from constant strain rate tests were used to characterise the SSPD behaviour of the mixtures. The experimental results showed that the uniaxial SSPD behaviour of the HRA and DBM mixtures had the same form as that of pure bitumen and the idealised mixtures with linear behaviour at low stress levels and non-linear power law creep function behaviour at high stress levels. However, the mixtures were stiffer than the idealised mixtures which had the same bitumen and different volume fractions of aggregate. At stress levels greater than 700 kPa, viscous deformation of the mixtures was non-linear with creep exponent of 2.4. The DBM was found to be stiffer than the HRA with the same bitumen. Comparison of the uniaxial test results on the DBM made using 100 Pen grade bitumen with the results of the mixtures made using 50 Pen bitumen showed that the form of the SSPD behaviour of the two mixtures was similar. However, the mixture using 100 Pen bitumen reduced the stiffening effect by approximately 6 times.

To investigate the triaxial SSPD behaviour of the realistic mixtures, triaxial creep tests were carried out on the HRA using 50 Pen bitumen and the DBM using 100 Pen bitumen, over a range of deviatoric stresses and stress ratios at 40 ^{0}C. It was found that the triaxial SSPD behaviour of the mixtures had the same form as that of the pure bitumen with a stiffening effect due to presence of aggregates and confining stresses. Similar to the idealised mixtures, both the HRA and DBM mixtures were observed to dilate under triaxial stress states. The volumetric strain for both mixtures was found to vary linearly with the shear strain. The dilation ratio was observed to be independent of the stress ratio and deviator stress; however, it was dependent on the volume fraction of aggregates. The values of dilation ratio for the HRA and DBM were observed to be higher than those for the idealised mixtures at corresponding stress ratio. This was attributed to the aggregate interlock resistance.

The SSPD behaviour of realistic mixtures under repeated loading was also covered in the same study. Uniaxial repeated load tests were conducted over a range of stress levels between 50 and 100 kPa at 40 ^{0}C. The creep exponents for the mixtures were found to be between 0.8 and 0.9. The laboratory wheel tracking tests were carried out over a range of temperatures and stress levels, and the steady state rutting rate was used to characterise the permanent deformation behaviour of the mixtures.

Taherkahni (2005) and Ossa et al (2006) studied the SSP uniaxial deformation behaviour of HRA and DBM mixtures. Constant stress creep tests over a range of stress levels and temperatures, and constant strain rate tests over a range of strain rates and temperatures were conducted on the mixtures. The SSPD behaviour of the mixtures was found to be well captured by the MCM. The creep exponent of the HRA and DBM mixtures was found to be 2.9 and 3.9 respectively. The axial strain level, at which the mixtures reach the steady-state strain rate, was found to be generally higher for the HRA and decreased with increasing temperature. Moreover, it was found to be independent of applied axial stress level. In addition, both mixtures were found to dilate under constant stress creep and constant strain rate deformation tests.

The SSP triaxial deformation behaviour of the HRA and DBM mixtures was also studied in the same research. Static triaxial tests over a range of deviatoric stresses and stress ratios were conducted on the mixtures at 35 ^{0}C. Results showed that variation of the axial and radial strain with time was observed to be qualitatively similar to that under uniaxial stress states. The triaxial SSPD behaviour of the mixtures was found to be well captured by the MCM with the addition of a stiffening factor which was found to be a function of the applied stress ratio. The confining stress was found to have a stiffening effect on the mixtures, where, at the same temperature and deviator stress for a mixture, the steady state strain rate was found to decrease with increasing stress ratio. The stiffening effect of the confining stress on the DBM mixture was found to be higher than that on the HRA. Both the HRA and DBM mixtures were observed to dilate under triaxial stress states. The dilation ratio was found to be independent of stress ratio and depends only on the type of mixture.

6.5 UNIAXIAL TESTS ON IBAA BITUMINOUS MIXTURES

6.5.1 Permanent deformation tests

The overall objective of material testing should be to reproduce as closely as in situ pavement conditions, including the general stress state, temperature, moisture, and general condition of the material. Types of test presently used to characterise the permanent deformation response of pavement materials include fundamental, empirical and simulative tests. These tests are reviewed by Brown et al (2001). The summary of this categorisation is as follow:

6.5.1.1 Fundamental tests

- Uniaxial and triaxial tests: unconfined (uniaxial) and confined (triaxial) cylindrical specimens in creep, repeated loading, and strength tests.
- Additional shear tests - shear loading tests:
    - Superpave Shear Tester - Shear Dynamic Modulus.
    - Quasi-Direct Shear (Field Shear Test).

- Superpave Shear Tester - Repeated Shear at Constant Height.
- Direct Shear Test.

- Diametral tests: cylindrical specimens in creep or repeated loading test, strength test.

6.5.1.2 Empirical tests

- Marshall Test.
- Hveem Test.
- Corps of Engineering Gyratory Testing Machine.
- Lateral Pressure Indicator.

6.5.1.3 Simulative tests

- Asphalt Pavement Analyzer.
- Hamburg Wheel-Tracking Device.
- French Rutting Tester (LCPC Wheel Tracker).
- Purdue University Laboratory Wheel Tracking Device.
- Model Mobile Load Simulator.
- Dry Wheel Tracker (Wessex Engineering).
- Rotary Loaded Wheel Tester (Rutmeter.)

The following sections present two types of the uniaxial tests which were conducted on the bituminous mixtures containing IBAA. The tests are constant strain rate and constant stress tests.

6.5.2 Specimen manufacture

Cylindrical samples of 67 mm diameter and 134 mm high were manufactured and used in uniaxial tests. The same procedure presented in Chapter 3 was used to manufacture bituminous slabs of 100 mm thickness. These slabs were cut into two halves. Each half was then turned on its side and cored to produce cylinders of 67 mm diameter and 150 mm high. Cylinders were trimmed at both ends to produce

specimens of 134 mm high. Each half produced three cylinders. Fig 6-2 shows a summary for this process.

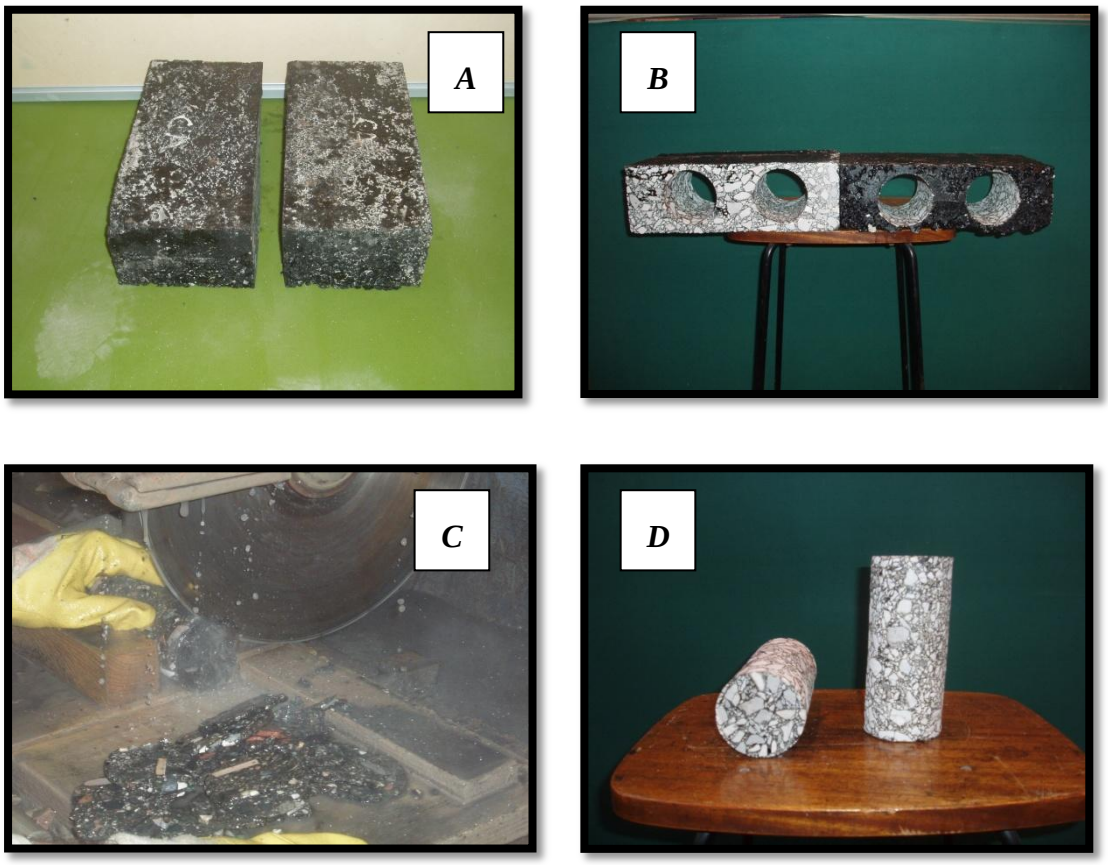


Figure 6-2: Specimens manufacture: A) Slab into 2 halves – B) Halves after coring – C) Cylinders trimming – D) The final product 134 x 67 mm.

Specimen dimensions were selected to give a height to diameter ratio of 2:1. This ratio was necessary to overcome the end effects and to minimise the reduction in the axial deformation (Blab et al 2006). The usual recommended dimensions for this ratio are 200 x 100 mm. These dimensions were not achievable in the University of Liverpool laboratory. This is attributed to the maximum allowed slab height which can be achieved using the available roller compactor. This height is limited to 100 mm. Therefore, it was decided to produce slabs of 100 mm thickness and cut them into halves of 100 x 305 x 152.5 before coring them from their side. The effect of coring from the slab side on the volumetric properties of the produced cores compared to those produced by coring from the top of the slab was investigated. The CDM and

VIM for the produced cylinders were compared with those produced for mix design purposes. The difference was found to be insignificant for all tested mixtures as shown in Table 6-2.

Mix Reference	CDM (g/cm^3)		VIM (%)	
	65 x 100 mm cores	143 x 67 mm cores	65 x 100 mm cores	143 x 67 mm cores
OA	2.380	2.372	7.31	7.35
AA	2.293	2.202	7.42	7.5
BA	2.177	2.019	11.01	11.04
CA	2.063	2.009	14.04	14.1

Table 6-2: Effect of coring direction on specimens' volumetric parameters

6.5.3 Test equipment

The equipment arrangement, shown in Fig 6-3, for the uniaxial tests comprises two main components. The first component is a 10 tonne hydraulic load cell mounted on an Avery Denison compression loading frame. This frame is motor controlled which is itself controlled by a microprocessor system within the machine. The microprocessor system monitors the crosshead displacement and the applied force on the specimen, as measured by the load cell, and sends this information to a controlling computer for subsequent data processing. The adjustable crosshead within the frame can be traversed up or down by means of recirculation ball screws. A gearbox connected to the motor system drives these ball screws. A strain gauge is attached to the crosshead and used to monitor the load. The position of the cross-head is also monitored by an encoder system. The drive system comprises quadrant servo motor driving through the gearbox to the ball screws and hence moves the cross-head with speeds ranging between 0.5 and 500 mm/min to a maximum travel length of 1000

mm. The second component is a temperature controlled cabinet (-20 to 50 ^{0}C) which is used to manage the test temperature. The cabinet is mounted on the loading frame.

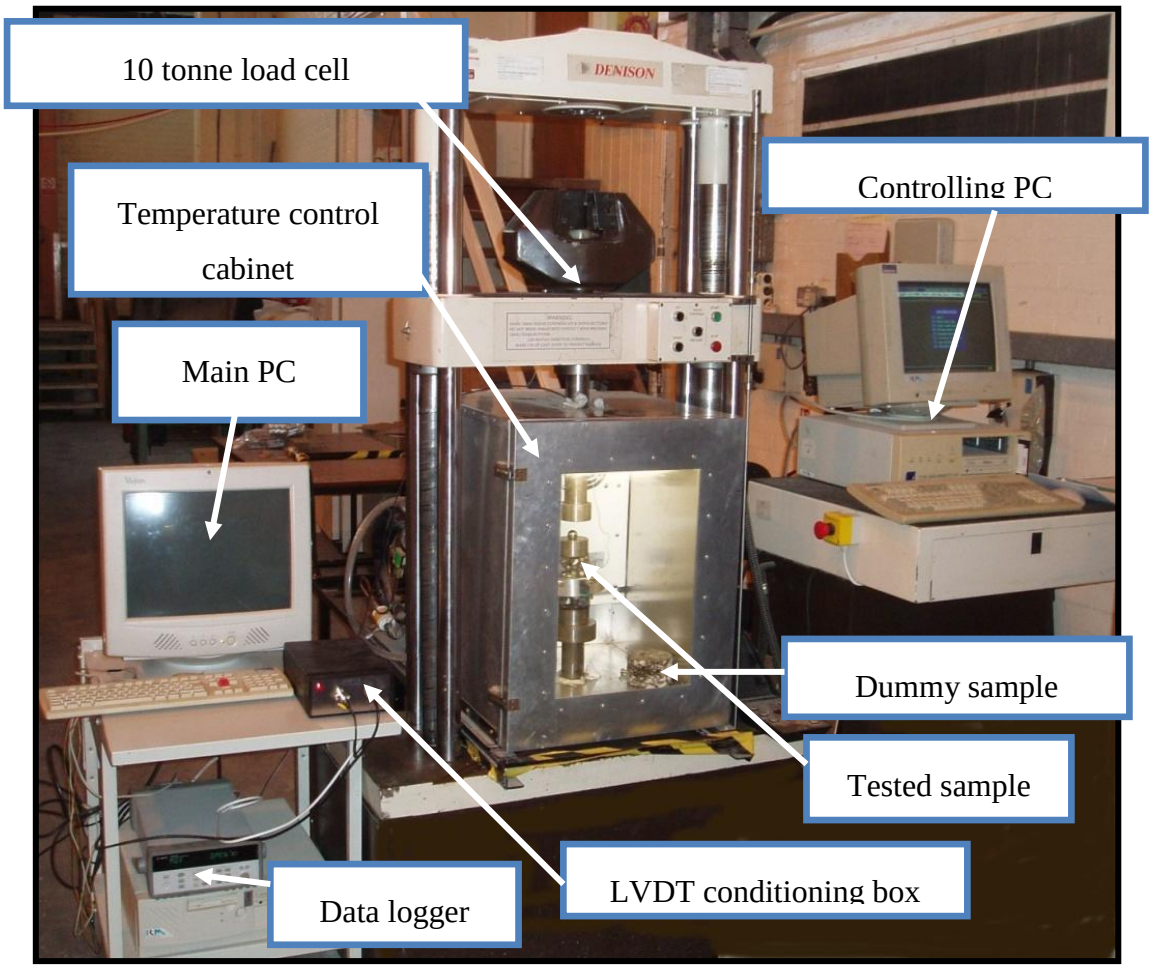


Figure 6-3: Uniaxial test arrangements.

The sample is placed between two steel platens and surrounded by a radial collar to measure the radial deformation via a Linear Variable Deferential Transducer (LVDT). The collar, shown in Fig 6-4A, is made from Aluminium with internal diameter of 67 mm. It has been provided with two rubber pads which are controlled by two screws. A spring with appropriate stiffness is used at one end to facilitate collar movement. The opposite end is opened and provided with a LVDT tunnel. Two metal bars were attached to the open end to be used in keeping the collar in position using a rubber band. The LVDT is an inductive sensing device that produces an AC output voltage proportional to the mechanical displacement of a small iron core. The LVDTs used

were of spring return type version RDB D6/05000A with ±5 mm range which can work effectively in a temperature range between -20 and 125 ^{0}C. The LVDT is connected to conditioning box which converts the LVDT response to deformation. The conditioning box and the controlling computer are connected to a main computer via a data logger. The main computer links the axial and radial deformations to their corresponding applied stress.

6.5.4 Specimen instrumentation

After manufacturing, samples were kept at a temperature of 5 ^{0}C. Prior to testing, sample height and diameter were measured accurately. The mid-height was marked to help with collar alignment. To ensure uniformity of temperature, samples were conditioned in the temperature control cabinet, at the testing temperature, for 2 to 6 hrs preceding the test. This duration was found to be adequate for internal sample temperature to reach testing temperature. The temperature inside a cored dummy sample was measured hourly until it had reached the desired test temperature. The collar, Fig 6-4A, was then held in place, at the middle third and as close as possible to the mid-height of the sample, by gluing its pads to the sample. A calibrated LVDT, Fig 6-4B, was fitted into the collar. A rubber band with a proper stiffness was used to help in holding the collar in position. Care was taken to ensure that the rubber band can keep the collar in position without giving any constrains to the specimen or causing depressions at the points of contact. The horizontal alignment of the collar was checked before the sample was turned on its side and left for at least 10 min to give enough time for glue hardening, Fig 6-4C.

Specimens were then placed vertically on the lower steel platen after smearing it with silicon grease as a lubricant. This lubricant was applied too to the top platen and the upper surface of the sample; to ensure uniform stress distribution and to minimise friction and thus, prevent sample bulging. This lubricant was noticed to be effective since no traces of sample sticking to the platens were observed after testing.

A steel bearing ball was used above the upper platen to eliminate any load eccentricity. Then, the loading arm was brought down, Fig 6-4D, applying a small pre-load to take out any relaxation in the system. Specimens were then allowed to deform under a uniaxial compression stress.

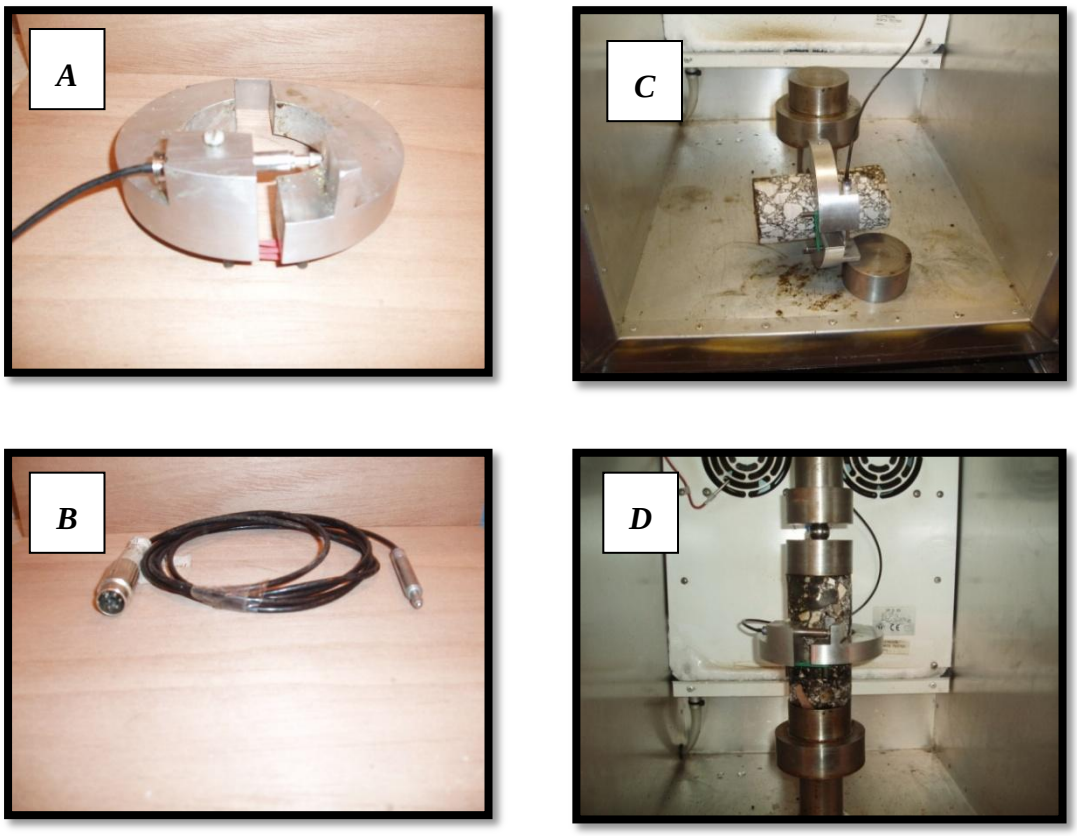


Figure 6-4: Sample instrumentation in a uniaxial test.

6.5.5 Strains measurement system

Measuring specimen's axial strain usually takes place by either recording the crosshead displacement (Deshpande 1997, Khanzada 2000) or via LVDTs attached parallel to the vertical axis of the specimen (Garba 2002, Kaloush 2002, Gibson 2006). The former method has one problem which is related to the crosshead deformation. When the machine applies a load the crosshead deforms as well as the sample. The recorded displacement is the cumulative of crosshead and sample displacements. However, crosshead deformation could be neglected if it is too small

compared with sample deformation (Erkens 2002) and this was the case in this study. The latter method, Fig 6-5, measures the deformation between the two platens. This method seems to be more accurate than measuring the crosshead displacement. However, this method has some limitations. To attach LVDTs to a mixture, it is necessary to use a supporting system with pips penetrating the sample, which may lead to unforeseen effects on the deformation of the specimen. Moreover, this system needs the specimen's deformation to be limited to the LVDT measuring range. In this study, preliminary tests showed that the expected axial deformation may exceed 10 mm. Therefore, it was decided to use the crosshead displacement method to measure the deformation of the samples.

Figure 6-5: Axial strain LVDTs (Gibson 2002).

With regard to radial strain measurement, all methods found in the literature are based on the same idea. The sample is surrounded by a tool which deforms by the same amount of the sample deformation. This deformation expresses the radial strain. This tool had two main shapes. The first shape has been used at TU Delft on several occasions (Erkens 2002, Muraya 2007) to measure the radial strain of asphalt mixtures. In this case, the radial deformations were measured by two separate systems, a string and a circumferential kit as shown in Fig 6-6. The first system consisted of a string wrapped around the specimen and passing over two potentiometers. This system has a relatively large range. The second system is a chain of very smooth rollers in combination with an extensometer; this system is very

sensitive but has a limited range. This method, although found effective in some cases, has some limitations. The large range of the string caused it to miss the initial part of the deformations while the chain had a limited range which is not suitable for large deformations. Moreover, in order to combine the data from both systems to an overall radial deformation signal, the systems must measure the same behaviour. Because they cannot physically be at the same height, this requires uniform deformation which cannot be guaranteed for mixtures made from secondary aggregates. The second shape, which is using a collar and a LVDT, was presented for the first time by Clyton et al (1989) and has been used successfully at Cambridge and Nottingham Universities (Deshpande 1997, Taherkhani 2005). The collar and LVDT shape, which has been discussed earlier in this chapter, was found to be simple, easy to use and effective at different temperatures and loading ranges.

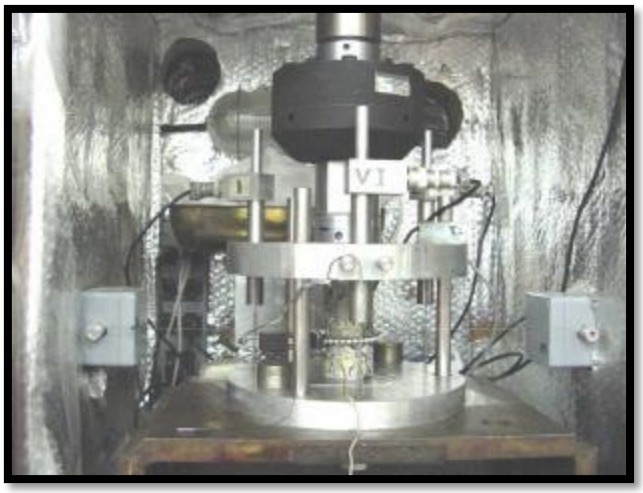


Figure 6-6: Radial strain measuring system used at TU Delft (Muraya 2007).

6.5.6 Uniaxial constant strain rate test (UCSRT)

In the uniaxial constant strain rate tests (UCSRT), specimens were tested at temperatures of 20 and 40 ^{0}C over a wide range of strain rates. Table 6-3 shows the used testing programme.

After preloading, the compression machine was used to apply immediate constant strain rate loads up to failure. Failure was considered to occur when the stress/strain

curve passes its peak value and, therefore, strain increases and stress decreases. This phenomenon indicates that samples start to flow. This flow stage is preceding the steady state stage. The steady state stage is the target for this study. Consequently, it was decided to stop the test a few second after the flow stage occurred. For each strain rate, the stress-strain relationship was captured and recorded by a computer.

Mix reference	**Test type**	**Test temp. (^{0}C)**	**Strain rate (1/S)**	**Creep stress (kPa)**
OA AA BA CA	UCSRT	20	0.00005, 0.0001, 0.0005, 0.001	N/A
		40	0.00005, 0.0001, 0.0005, 0.001	N/A
	UCST	5	N/A	500, 1000, 2000, 3000
		20	N/A	500, 1000, 2000, 3000

Table 6-3: Uniaxial compression testing programme

6.5.7 Uniaxial constant stress test (UCST)

In the uniaxial constant stress tests (UCST), specimens were tested at 5 and 20 ^{0}C under a range of creep stresses as shown in Table 6-3. This test is a creep test in which the compression machine was allowed to apply a constant creep stress over sample in two stages. In the first stage, a rapidly increased load was applied within a very short time. Time varies depending on the required creep stress. At the end of the first stage the machine reached the required creep stress. In the second stage, this stress was held over samples for 1800 sec. This creep time was found, in preliminary

tests, to be adequate for mixtures to reach steady state conditions. In each test, the axial and radial strains were recorded over creep time.

6.6 UNIAXIAL TEST RESULTS

6.6.1 UCSRT results

For each strain rate, the stress-strain relationship was captured and recorded. For all mixtures, the relationship between stress and strain, both axial and radial, had the same behavioural trend. This relationship consisted of four stages. In the first stage, the stress-strain relationship resembled a linear elastic behaviour. This stage proceeded over a narrow range of strains and was followed by a second stage in which the relationship exhibited an inelastic increase in the load, up to a peak value, which represents the steady state stress (Ward 1971), as explained in Chapter 5. This was followed by a descending softening part up to failure as shown in Fig 6-7.

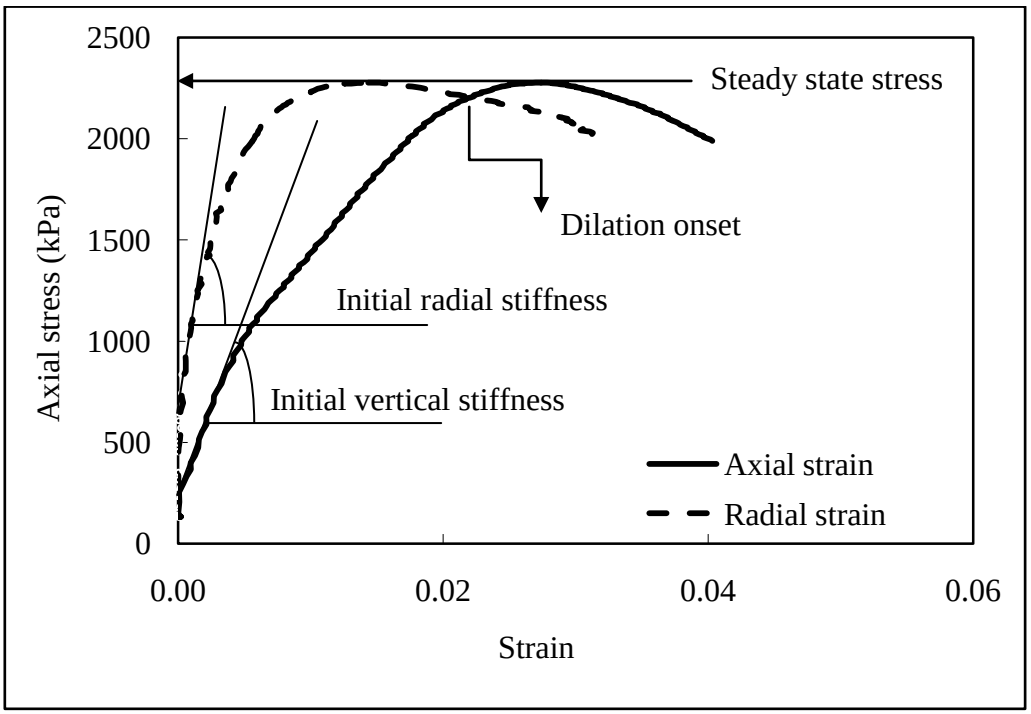


Figure 6-7: Typical UCSRT result (Mix AA at 0.001 1/s strain rate and 20 ^{0}C).

For all mixtures, it was noticed, at any stress value, that the axial strain was higher than the radial strain. In addition the radial strain reached the steady state stage before the axial strain and starts to decrease while the axial strain is continue to increase up to a point after which the axial strain reaches the steady state stage and levels before starting to decrease. This was not the case at the early beginnings of the tests as both strains were almost identical. This may be attributed to the occurrence of pre-deformation compaction. The samples could have undergone a small amount of compaction, only in the vertical direction, before commencement of deformation. This compaction has been accompanied by a decrease in volume (Erkens et al 2002, Collop et al 2006). This decrease has been followed by a volume increase when the internal cracks start to open in a phenomenon known as dilation (Erkens 2002). The point of intersection of the two curves, in Fig 6-7, was found to present the point at which the radial strain is equal to the axial strain. This point is assumed to be the point of initiation of dilation or dilation onset point. Therefore, it is clear that the onset of dilation led to accelerating the strain increase and, consequently, failure. Dilation behaviour of the mixtures will be further discussed in a later section.

The assumption of specifying the dilation onset point is based on the typical volumetric strain against time, as in Fig 6-8, which always obtained from a typical triaxial test. However, in this case the horizontal stresses were used as zero. Fig 6-8 shows that, under triaxial loading conditions, the volumetric strain decrease before commencing an increase which is similar to dilation phenomenon. This leads to occurrence of a point at which the volumetric strain is zero. As the volumetric strain equals the sum of the radial strain and the axial strain therefore, when the volumetric strain tends to zero the axial strain must have the same value of the radial strain. Consequently, the point at which axial strain equals the radial strain can be assumed as dilation onset point.

The stress-strain relationship curves were used to determine the initial vertical and radial stiffness of the mixtures. These values were determined through measuring the slope of the elastic region of the curve. These stiffness values were compared to the stiffness values measured in the indirect tensile stiffness test and presented earlier in

Chapter 3. A good agreement was found between the two test results as shown in Table 6-4.

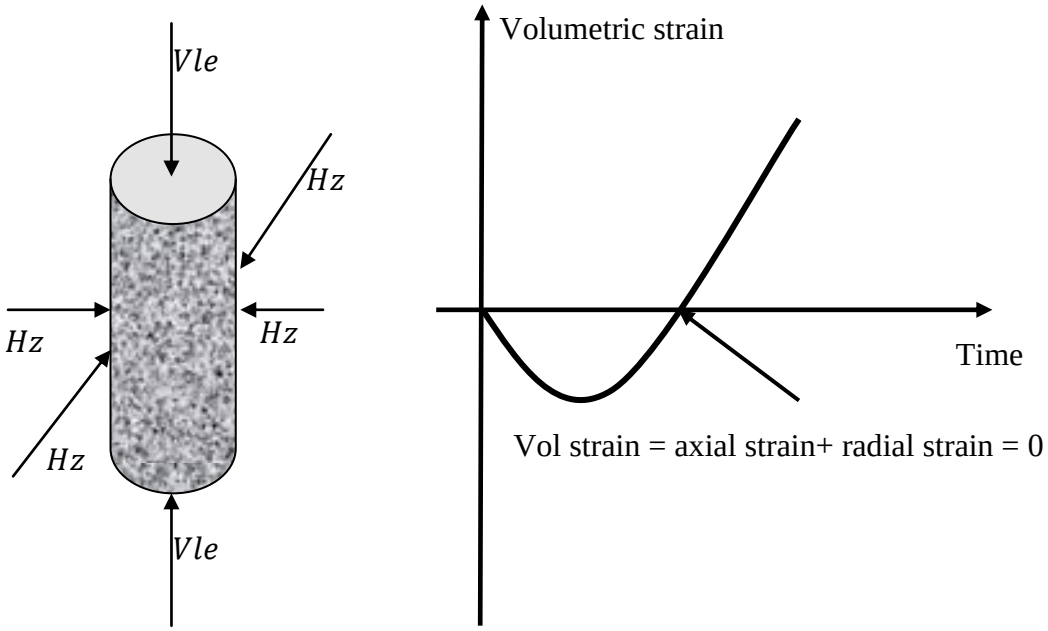


Figure 6-8: Typical triaxial stress conditions.

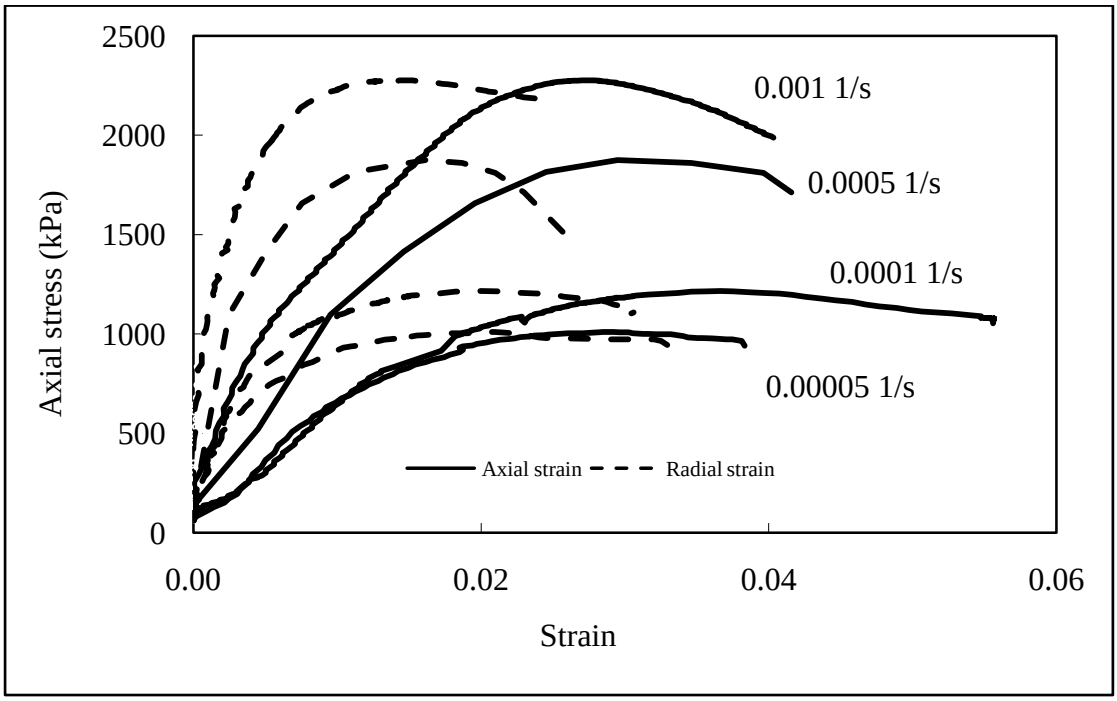


Figure 6-9: Effect of strain rate on UCSRT results (Mix AA at 20 ^{0}C).

Mix	OA	AA	BA	CA
Stiffness from NAT (MPa)	1480	1805	2138	1700
Stiffness from UCSRT (MPa)	1450	1790	2110	1680

Table 6-4: Stiffness of the produced mixtures

The loading rate and test temperature were found to have a significant effect on the steady state stresses. It was noticed that, regardless of the type of mixture, i.e. IBAA content level, increasing strain rate resulted in increasing the steady state stress as shown in Fig 6-9. At the same time, this strain rate increase led to a shorter flow region. This means that the mixture tends to be more brittle at higher strain rates. With regard to temperature effect, increasing test temperature led to a decrease in the steady state stress as shown in Fig 6-10. This observation was noticed on all mixtures. Therefore, it is concluded that IBAA content level did not affect the mixtures' behaviour trends in the UCSRT.

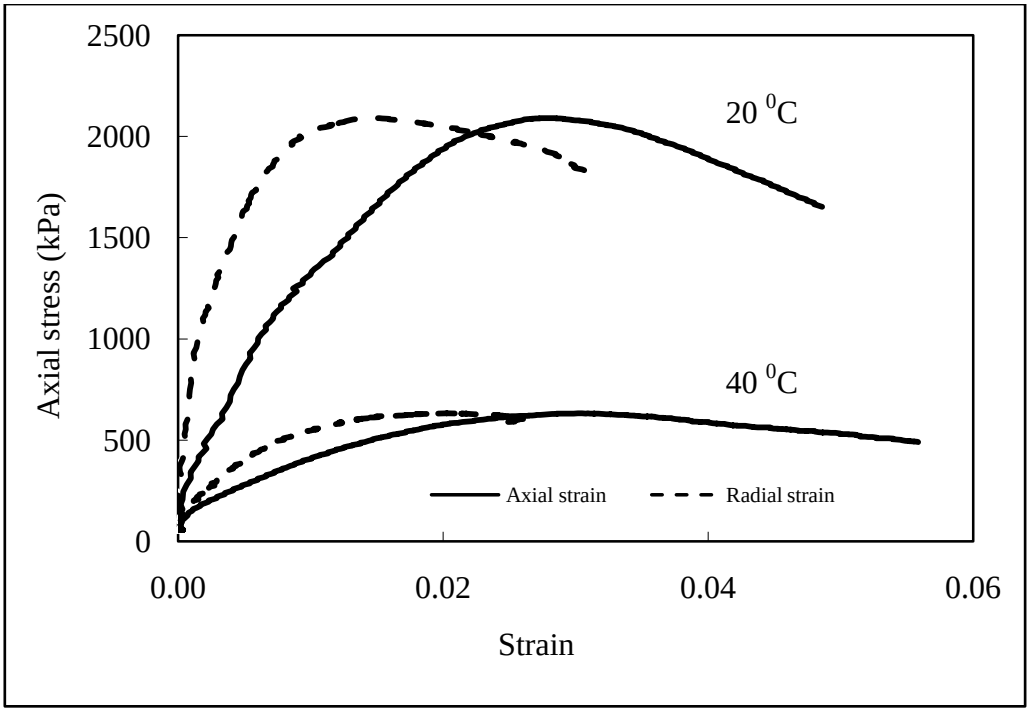


Figure 6-10: Effect of temperature on UCSRT results (Mix OA at 0.001 1/s).

6.6.2 UCST results

For all mixtures, axial and radial strains over time were recorded under a range of creep stresses. Fig 6-11 shows typical creep test results, from which it can be seen that a creep curve can be divided into three regions. In the first region, primary creep, the material underwent deformation at a high strain rate. In the second region, the strain rate was predominantly constant. This region was called the secondary creep region and its strain rate was adopted as the steady state strain rate corresponding to a particular value of applied compressive stress, as explained in Chapter 5. A third region, appeared only in a few tests, exhibited tertiary creep in which the strain rate increased significantly as the specimen became progressively damaged.

In UCST, the radial deformation behaviour, seen in Fig 6-12, was similar to that noticed in the UCSRT results. At the beginning of the test, the axial strain value was higher than that of the radial strain up to a point after which the radial strain values were higher than the axial strain values. This point represents the onset of dilation. This observation is similar to the findings of the UCSRT results and can be attributed to the same reasons discussed earlier.

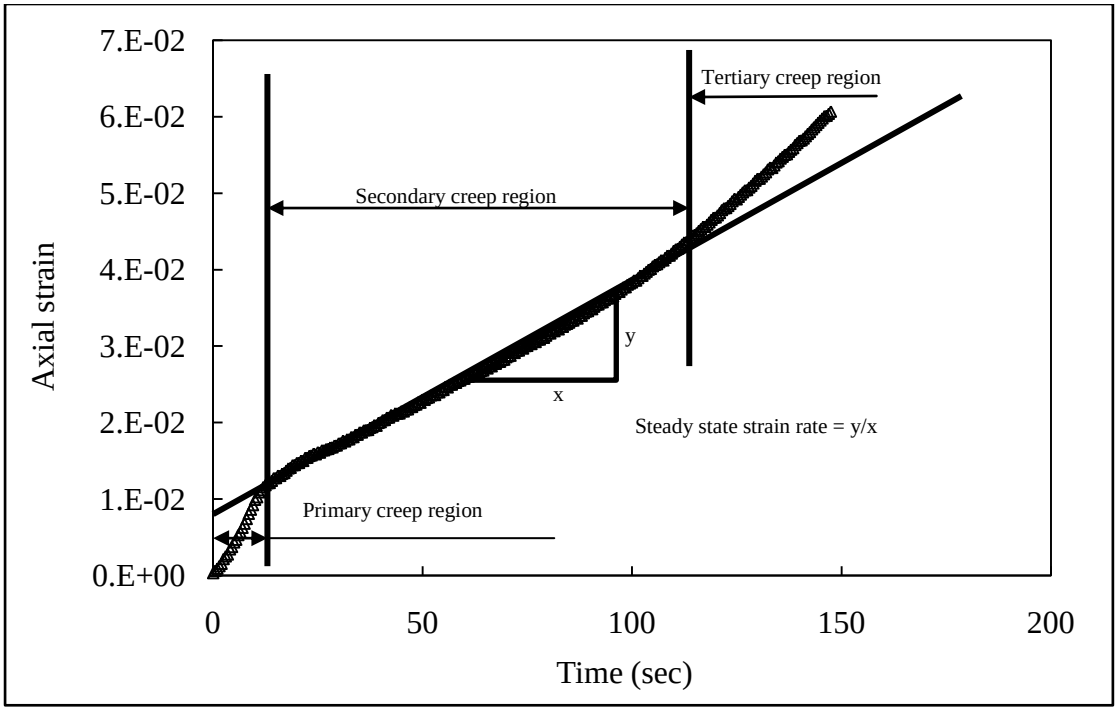


Figure 6-11: Typical UCST result (Mix BA – 2000 kPa – 5 ^{0}C).

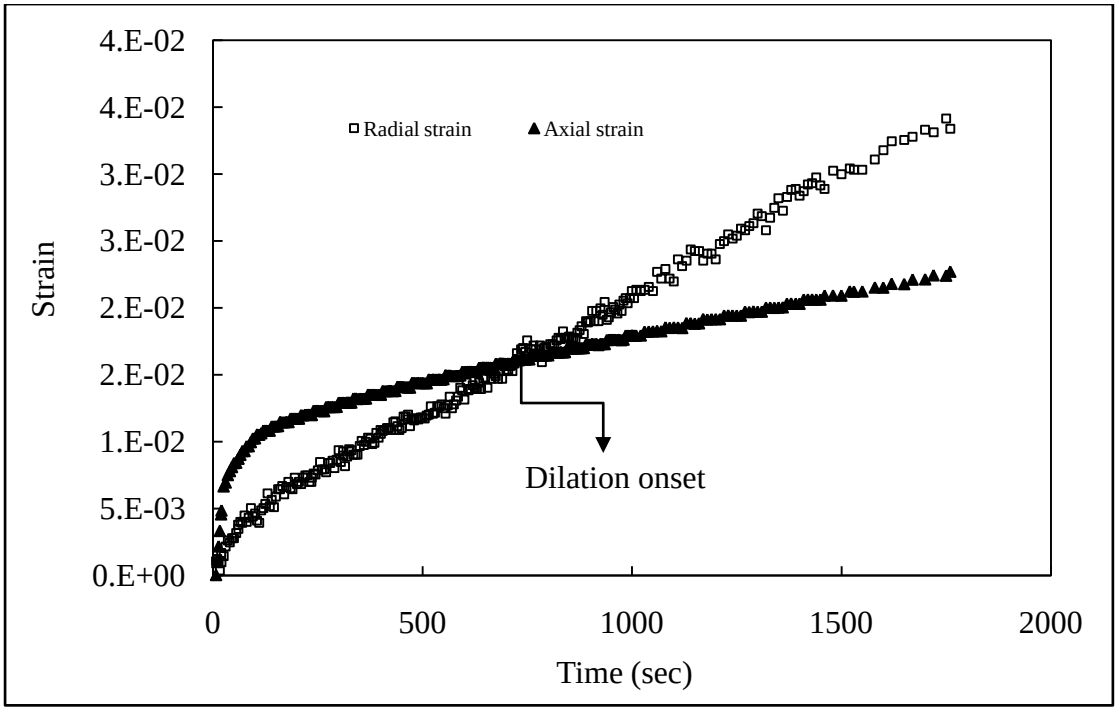


Figure 6-12: Radial and axial strain from UCST (Mix BA – 2000 kPa – 5 ^{0}C).

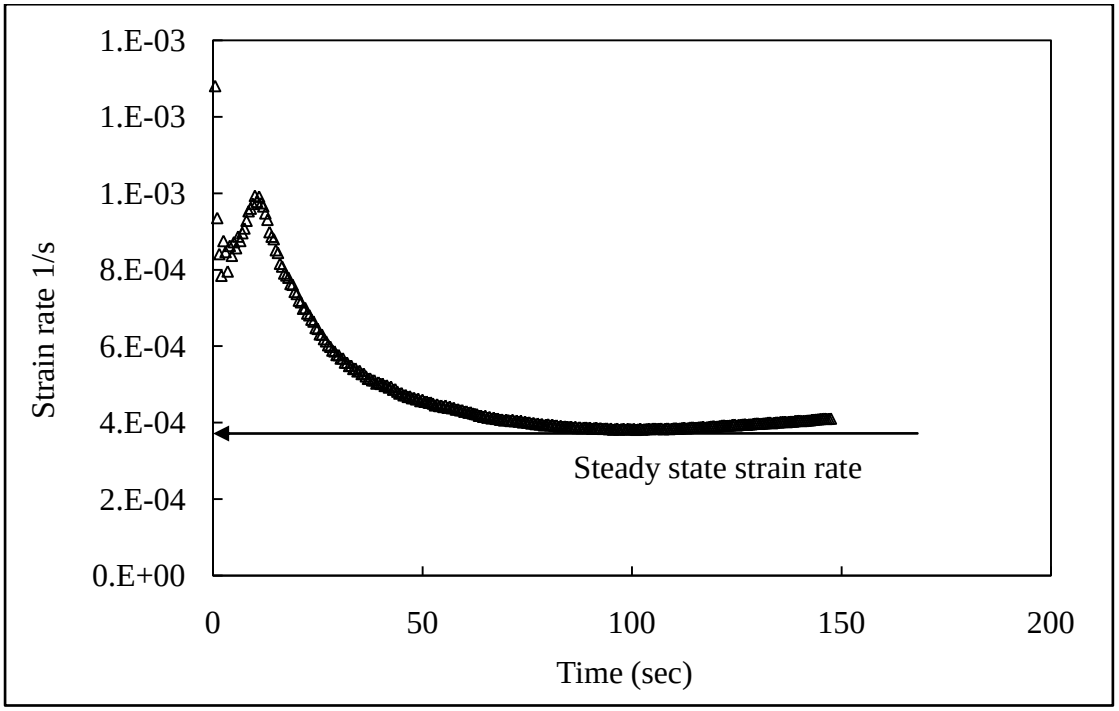


Figure 6-13: Variation of strain rate against time in UCST (Mix BA – 2000 kPa – 5 ^{0}C).

Fig 6-13 shows the variation of strain rate against time in a UCST. From this figure, it is noticed that the strain rate starts to increase with time in the primary creep region. Then it decreases dramatically until it levels out. This levelling out is considered to be the steady state strain rate. When sample starts to flow, the strain rate starts to increase at a very small rate up to failure. This figure is used to find the steady state strain rate for the mixtures. The steady state strain rate results obtained from Fig 6-13 and Fig 6-11, for each mixture and testing condition, were averaged and the resulting value was used as the final steady state strain rate.

The effects of test temperature and creep load on the resulting steady state strain rate were also investigated in the UCSTs. It was found that decreasing test temperature led to a longer time before reaching the tertiary creep region and to a smaller steady state strain rate, under the same creep stress, as shown in Fig 6-14. On the other hand, Fig 6-15 shows that, at the same test temperature, increasing the creep stress led to a reduction in time needed to reach the tertiary creep region and to an increase in the steady state strain rate.

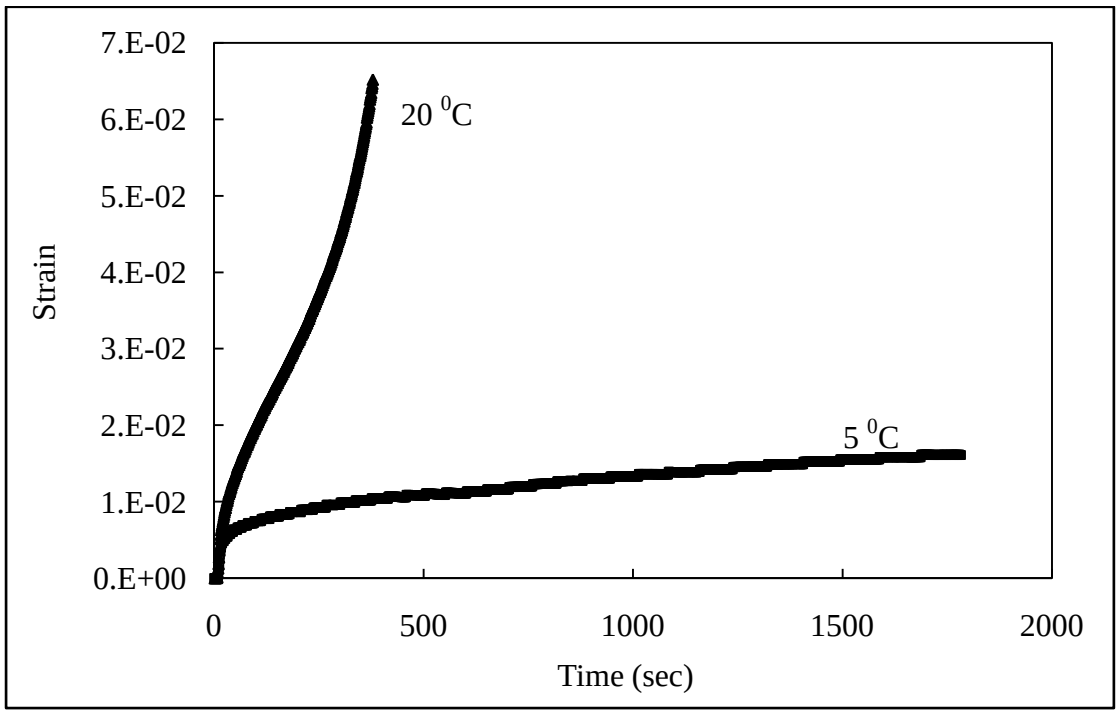


Figure 6-14: Effect of temperature on UCST results (Mix AA – 1000 kPa).

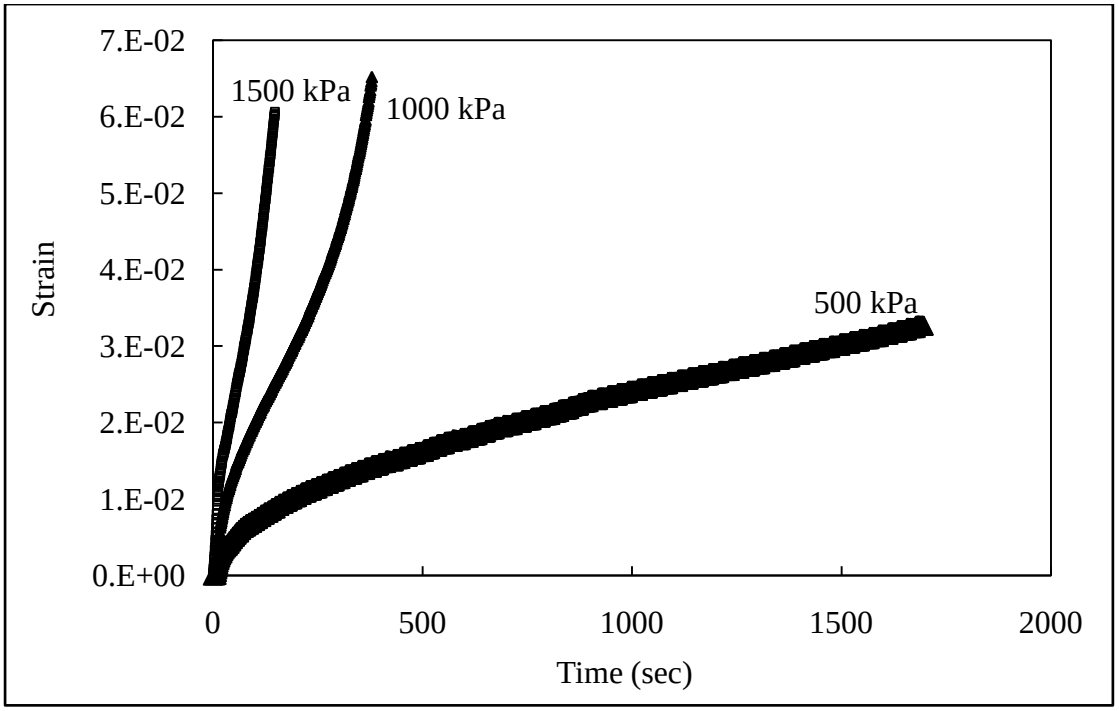


Figure 6-15: Effect of creep stress on UCST results (Mix AA – 20 ^{0}C).

6.6.3 Volumetric behaviour

Studying the volumetric behaviour of the mixtures is important in understanding their deformation characteristics. To do so, the radial deformation of the specimens was measured during the UCSRTs and UCSTs as explained earlier.

Fig 6-16 shows the variation of volumetric strain, calculated using Eq 6-1, with the axial strain under selected testing conditions. It is obvious that, initially, the volumetric strain decreased due to densification up to a point, after which the volumetric strain increased almost linearly with the axial strain. The trough point is referred to as the point of dilation onset, as shown earlier in Fig 6-8. This dilation had a constant value called dilation gradient. Dilation gradient is shown in Fig 6-17 in which the radial against axial strain was plotted. From this figure, it was noticed that initially the radial strain did not change significantly up to a level after which it increased almost linearly with the axial strain. The slope of this linear part is the dilation gradient. Similar result can be shown using a plot of volumetric strain against

the absolute value of the shear strain, which was calculated using Eq 6-2, as shown in Fig 6-18. The linear relationship from Fig 6-18 can be expressed as in Eq 6-3.

$$\varepsilon_v = 2\varepsilon_r + \varepsilon_a \tag{6-1}$$

$$\varepsilon_s = \varepsilon_a - \frac{\varepsilon_v}{3} = \frac{2}{3}(\varepsilon_a - \varepsilon_r) \tag{6-2}$$

$$\varepsilon_v = Dg\,|\varepsilon_s| \tag{6-3}$$

where: ε_v is the volumetric strain; ε_r is the radial strain; ε_a is the axial strain; ε_s is the shear strain; and Dg is the dilation gradient.

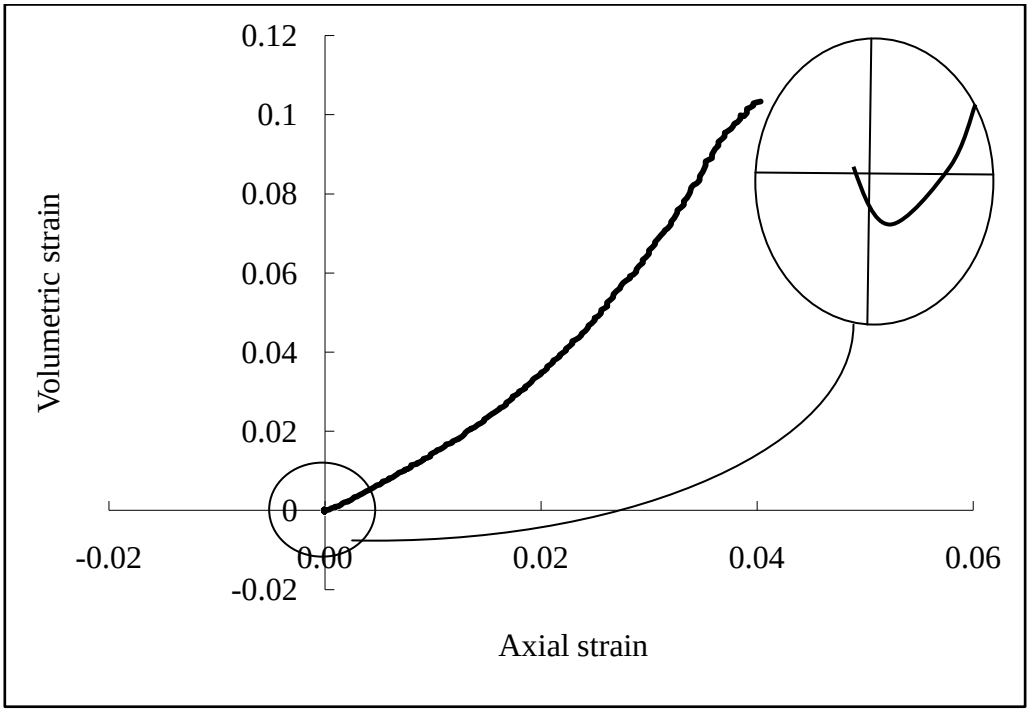


Figure 6-16: Variation of volumetric against axial strain in UCSRT (Mix AA – 0.001 1/s – 20 ^{0}C).

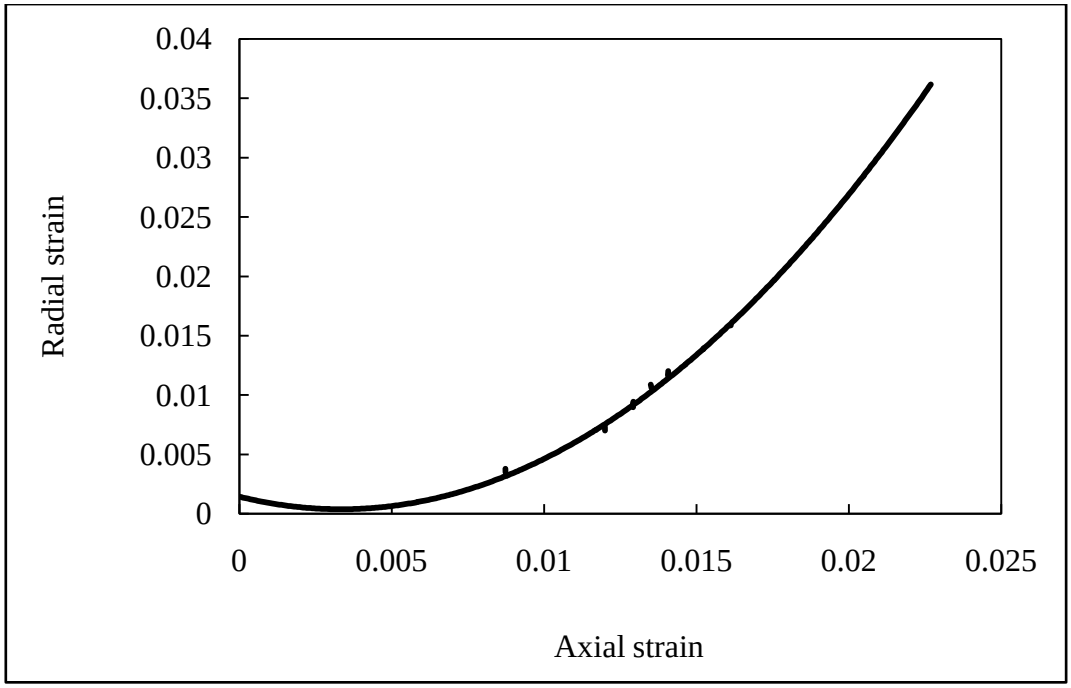


Figure 6-17: Variation of radial against axial strain in UCST (Mix BA – 2000 kPa – 5 ^{0}C).

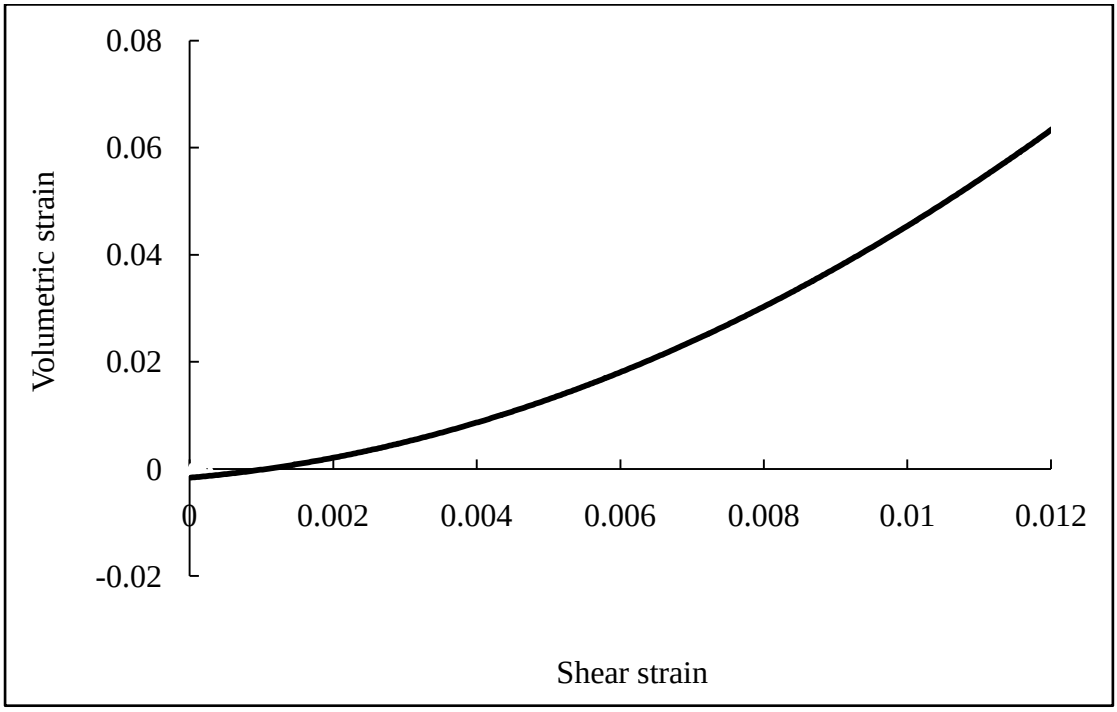


Figure 6-18: Variation of volumetric against shear strain in UCST (Mix BA – 2000 kPa – 5 ^{0}C).

6.6.4 Dilation of mixtures

The dilation gradient from each test was calculated using Eq 6-3. The average value for each mixture at the same temperature was calculated and presented in Fig 6-19. The dilation gradient values were found to be independent of the applied stress for all mixtures. This observation is consistent with results obtained for idealised asphaltic mixtures (Deshpande 1997, Khanzada 2000) and for typical UK dense bitumen macadam and hot rolled asphalt materials (Taherkhani 2005). Dilation gradients at 5 ^{0}C were observed to be very close to the values recorded at 20 ^{0}C. The same observation was reported for typical UK dense bitumen macadams (Taherkhani 2005). The reason beyond this may be attributed to the high dilation potential of the tested mixtures. It is known that mixtures with high binder content and soft binder grade have increased dilation. The mixtures tested in this study are such types of mixture as shown in Chapter 3. This increased dilation can be seen from the recorded values for dilation gradient, shown in Fig 6-19. These values are considered high compared with those published by Taherkhani (2005). This increase may be attributed to the type and contents of binder used in this study. These increased values may diminish the difference in dilation gradients at 5 and 20 ^{0}C. However, the mixtures exhibited higher dilation gradients at 40 ^{0}C. This shows, as expected, that more dilation occurred at higher temperatures.

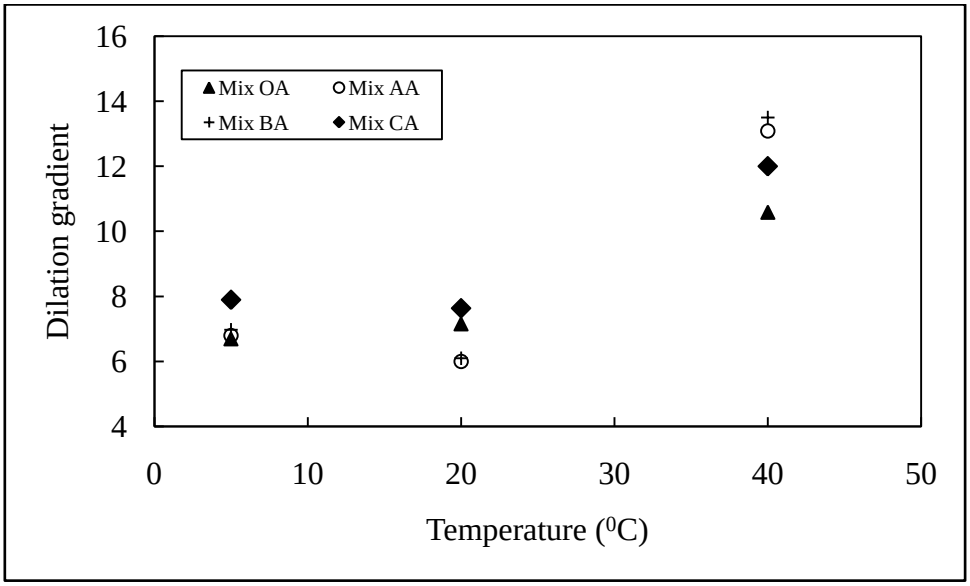


Figure 6-19: Dilation gradient values for mixtures under uniaxial testing conditions.

6.6.5 Poisson's ratio

Poisson's ratio is an important parameter in flexible pavement design procedures and in constitutive models for asphalt. This parameter is defined as the negative ratio of the radial strain to the axial strain in the elastic region of behaviour. Poisson's ratio is temperature dependent and it ranges between 0.1 and 0.45 for asphaltic materials (Read 1996).

Poisson's ratio can be determined from UCSTs on asphalt mixtures by plotting the ratio of radial strain to axial strain as a function of time. Fig 6-20 shows an example for this relation at specific testing conditions. From this figure it can be seen that initially there is an instant value for the radial/axial strain ratio. This value is the Poisson's ratio. Results for the other mixtures showed that Poisson's ratio ranged between 0.2 and 0.4 which is within the accepted range as it is known that Poisson's ratio has no meaning if values were over 0.5 (Collop et al 2006). Results also showed that Poisson's ratio increased with temperature as shown in Fig 6-21.

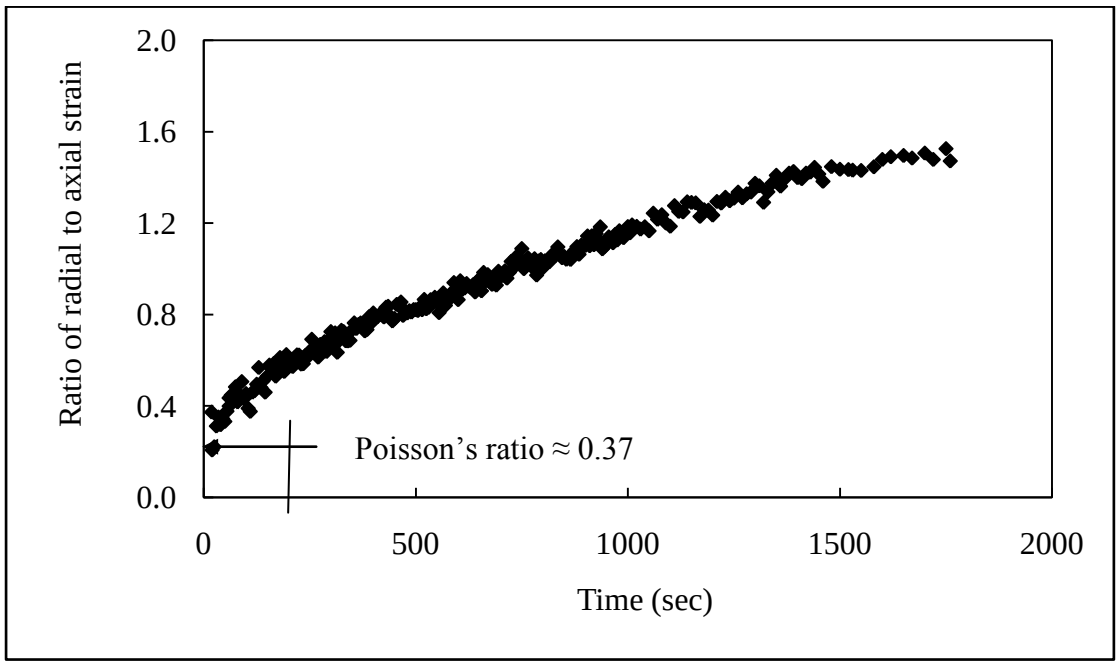


Figure 6-20: Variation of radial to axial strain ratio against time in UCST (Mix BA – 2000 kPa – 5 ^{0}C).

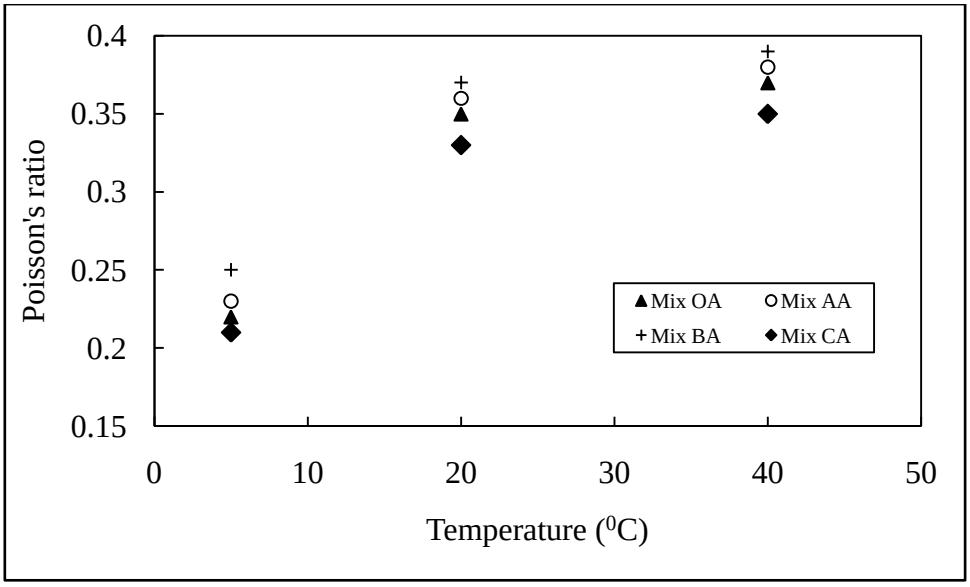


Figure 6-21: Effect of temperature on Poisson's ratio results in UCST.

6.6.6 Steady state permanent deformation behaviour

Steady state stresses were plotted against their corresponding steady state strain rates, obtained from UCSRTs and UCSTs, to produce steady state permanent deformation curves for the mixtures at each testing temperature. Steady state stresses were determined from the UCSRTs as explained in section 6.6.1 corresponding to the applied strain rates while the steady state strain rates were determined from the UCSTs as explained in section 6.6.2 corresponding to the applied creep stresses. A double logarithmic scale was used to present the results.

The experimental results were found to be adequately captured by a Modified Cross Model (MCM), which was originally developed by Cross (1965) and then modified by Cheung (1995) and Deshpande (1997). The MCM, expressed in Eq 6-4, was used successfully, in numerous studies, to predict bituminous mixtures' permanent deformation characteristics (Deshpande and Cebon 2000, Khanzada 2000, Tahekhani 2005). These bituminous mixtures included idealised mixtures with different aggregate fractions and typical UK dense bitumen macadams and hot rolled asphalts.

$$\sigma = \frac{S\ \sigma_0\ \dot{\varepsilon}}{\dot{\varepsilon_p}} \left\{ \frac{1}{1+\left(\frac{S\ \dot{\varepsilon}}{\dot{\varepsilon_p}}\right)^m} \right\} \qquad (6\text{-}4)$$

where: σ is the uniaxial stress; $\dot{\varepsilon}$ is the uniaxial strain rate; S is the stiffening factor; and σ_0, m, and $\dot{\varepsilon_p}$ are material constants for the mixtures. More details on these parameters will be discussed later in this section.

Figs 6-22 to 6-25 show results for these steady state behaviour curves for mixes OA, AA, BA, and CA respectively together with the relevant binder results. The MCM predicts that the steady state deformation behaviour of bituminous mixtures containing IBAA is linear at low stress levels, while at high stress levels the mixtures exhibit non linear power law viscous behaviour. This behaviour is similar to that of idealised asphalt mixtures (Deshpande 1997, Khanzada 2000) and of typical dense bitumen macadams and hot rolled asphalts (Taherkhani 2005).

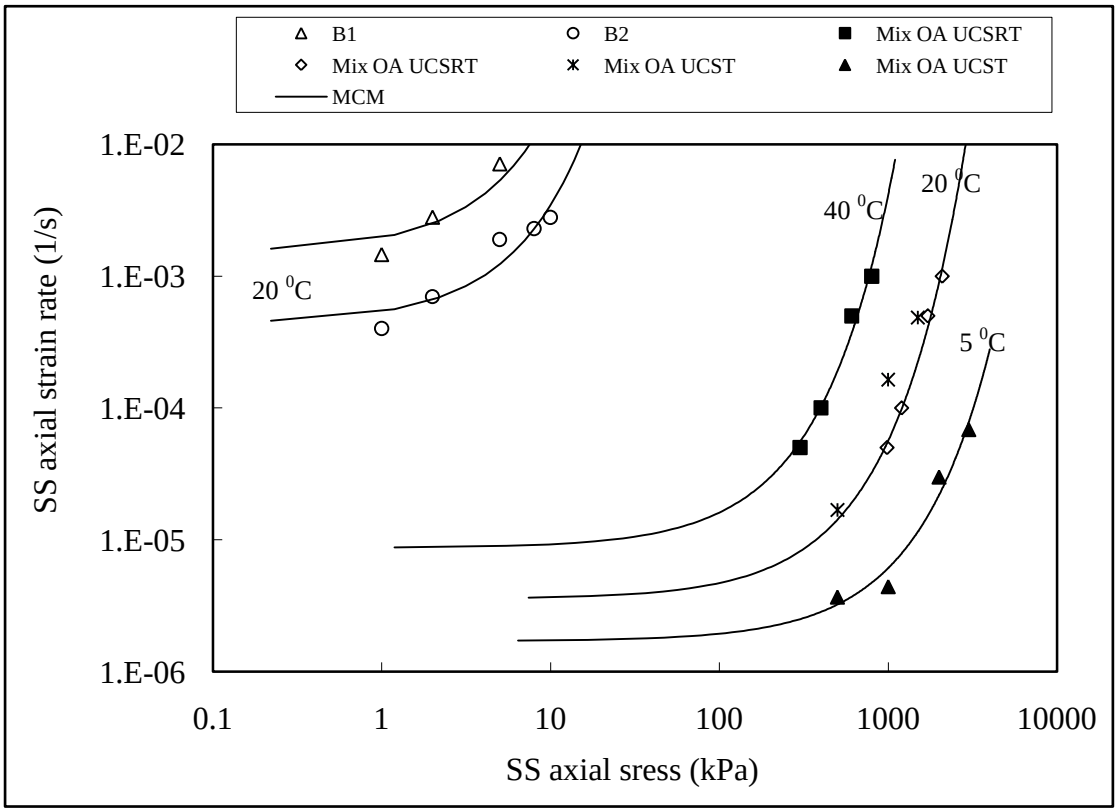


Figure 6-22: Steady state permanent deformation behaviour of mix OA.

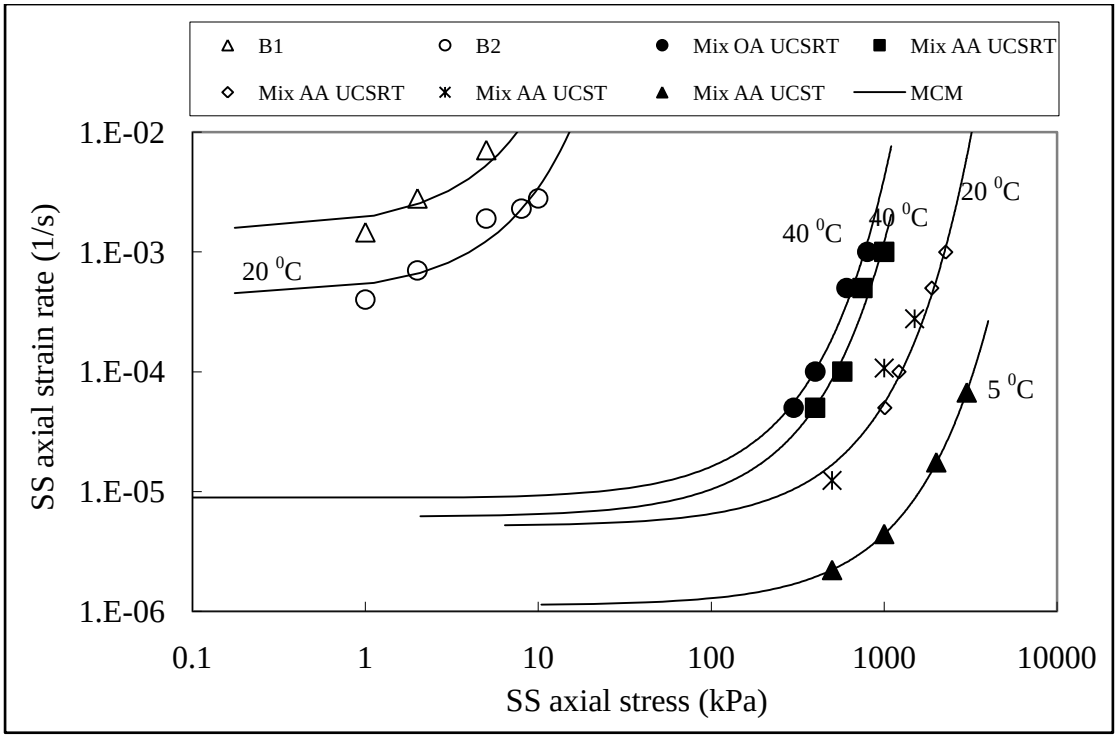


Figure 6-23: Steady state permanent deformation behaviour of mix AA.

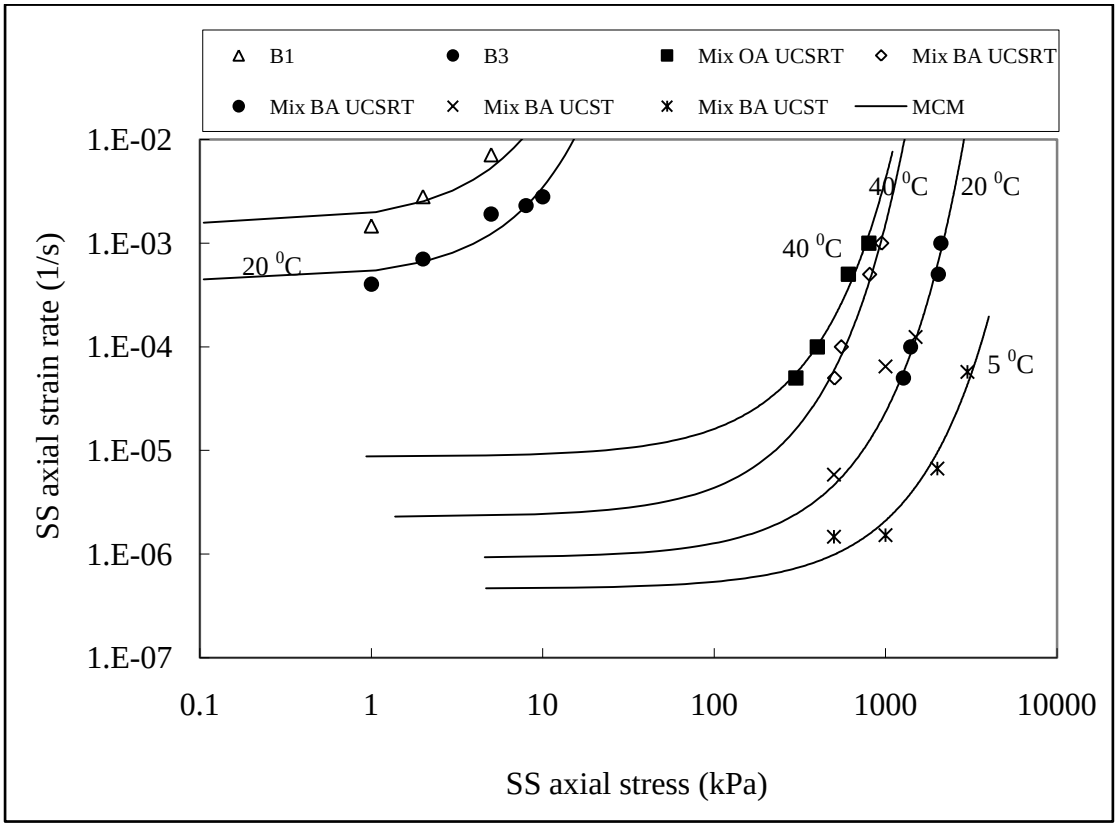


Figure 6-24: Steady state permanent deformation behaviour of mix BA.

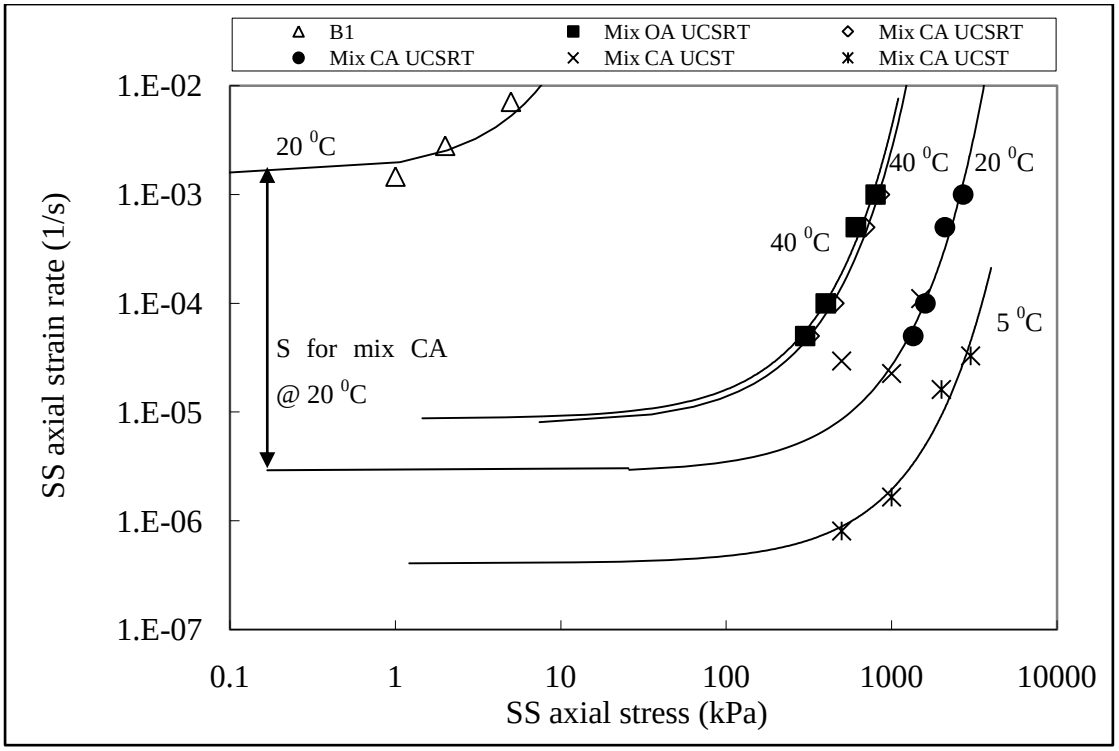


Figure 6-25: Steady state permanent deformation behaviour of mix CA.

The steady state results, under uniaxial testing conditions, show a very good agreement with binder modelling data presented in Chapter 5 and included in the four preceding figures. In these figures B1, B2 and B3 are the binders used in Chapter 5 to study steady state permanent deformation of binders. The mixtures' curves showed a shift from that of their respective binders tested at 20 ^{0}C. This shift is attributed to the stiffening effect of the aggregate matrix and the ageing of bitumen during mixing. The latter is due to the thin films of bitumen subjected to high temperatures during mixing, which harden the binder. The aggregate matrix stiffening effect can be expressed as the stiffening factor, S, which can be determined experimentally, as shown in Fig 6-25, and used for modelling purposes via the MCM as shown in Eq 6-4.

This stiffening factor was found to increase with IBAA content up to 60%, at all test temperatures. At 40 ^{0}C, adding 30% IBAA led to 150% increase in S, compared to the control mix, while adding 60% IBAA increased S by 4 times. At 20 ^{0}C, 30% IBAA led to 140% increase in S while 60% IBAA led to 400% increase. The case at 5 ^{0}C was similar; 30% IBAA doubled S and 60% IBAA increased it by 5 times. For 80%

IBAA, S was at its highest value, 6600, at 5 ^{0}C and decreased with temperature to 500 and 250 at 20 and 40 ^{0}C respectively. This may be attributed to the high binder content used in mix CA. A significant amount of this binder content was absorbed by the IBAA resulting in high voids content, low adhesion properties and a thin bitumen film. Increasing the temperature led to softening the bitumen film and, consequently, a reduction in stiffness. Table 6-4 shows a summary of the obtained stiffening factor values.

Mix	Temperature (^{0}C)	Stiffening factor
OA	5	1000
	20	250
	40	222
AA	5	2000
	20	350
	40	330
BA	5	5000
	20	1000
	40	900
CA	5	6600
	20	500
	40	250

Table 6-4: Stiffening factor values

6.6.7 MCM parameters

In this section, the determination of the MCM parameters is discussed. The exponent m, can be determined using a power law exponent called n which represents the exponent of the non-linear viscous part of the model. The relationship between m and n is presented in Eq 6-5.

$$m = 1 - \frac{1}{n} \tag{6-5}$$

With regard to $\dot{\varepsilon}_p$, it can be determined using Eq 6-6. This equation is based on using the Williams–Landel-Ferry equation (Williams et al 1955) and was presented by Cheung (1995) on the basis of the tested materials being temperature dependant. As the mixtures tested in this study are temperature dependant, thus it was decided to use Eq 6-6 as a tool to find $\dot{\varepsilon}_p$ for the tested mixtures. With regard to σ_0, it can be calculated from the MCM itself using the known steady state stress and strain rate values. Table 6-5 presents a summary for the MCM parameters for all tested mixtures.

$$\dot{\varepsilon_p} = \dot{\varepsilon_{pc}} \ exp\left(\frac{2.303\ c_1^s\ (T-T_s)}{c_2^s + (T-T_s)}\right) \tag{6-6}$$

where: $\dot{\varepsilon_{pc}}$ is the reference strain rate at T = 5 ^{0}C; c_1^s is a bitumen universal constant = 8.86; c_2^s is a second bitumen universal constant = 101.6; and T_s is a reference temperature = 30 ^{0}C (Cheung 1995).

Parameter	Mix OA	Mix AA	Mix BA	Mix CA
$\dot{\varepsilon}_p$ (1/s)	1.42E0-7	8.51E-08	5.68E-08	3.11E-08
m	0.755	0.729	0.814	0.777
σ_0 (kPa)	59.98	36.76	103.43	60.59

Table 6-5: MCM parameters under uniaxial testing conditions

6.7 EFFECT OF IBAA ON MIXTURE DEFORMATION BEHAVIOUR

Results of UCSRT showed that increasing IBAA content level up to 60 % led to an increase in the steady state stress, at the same strain rate. However, using 80% IBAA content level led to a reduction in the steady state stress compared to mixtures with 60% IBAA. This behaviour was temperature independent.

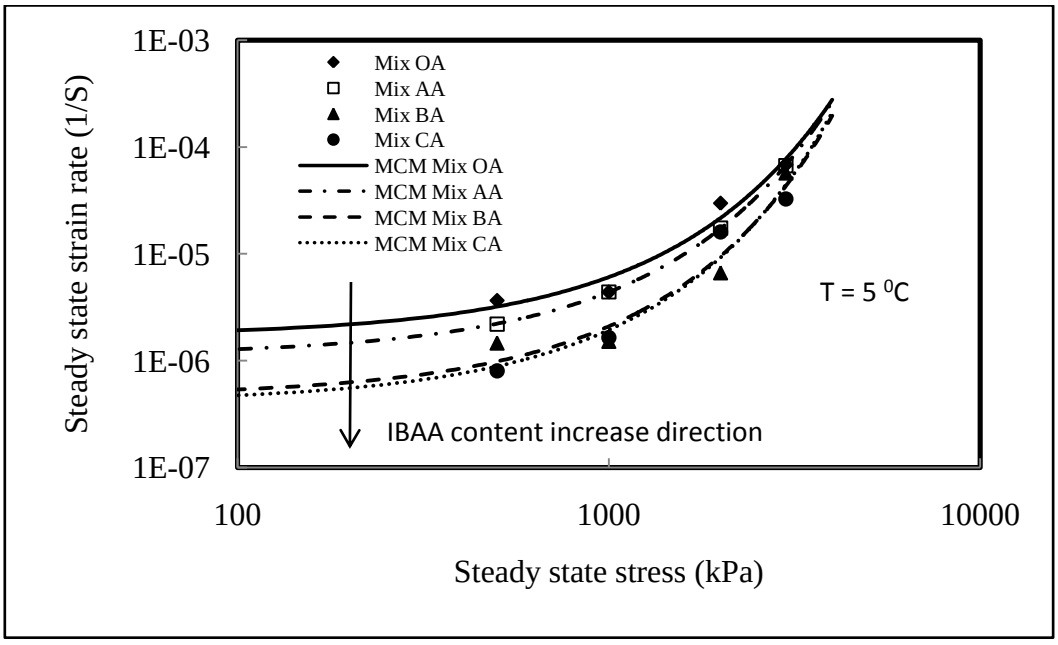


Figure 6-26: Effect of IBAA content level on mixtures' steady state permanent deformation behaviour at 5 ^{0}C.

In the UCSTs, similar behaviour to that recorded in the UCSRTs was noticed at 20 ^{0}C, with regard to a decrease in the steady state strain rate. At 5 ^{0}C, it was noticed that increasing IBAA content level led to a general decrease in the steady state strain rate, at the same creep stress.

Figs 6-26 to 6-28 show the steady state deformation behaviour at 5, 20 and 40 ^{0}C respectively for the tested mixtures. From these figures, it is clear that mixes AA and BA exhibited an increase in their steady state permanent deformation resistance compared to mix OA, the control mix, regardless of the test type or temperature.

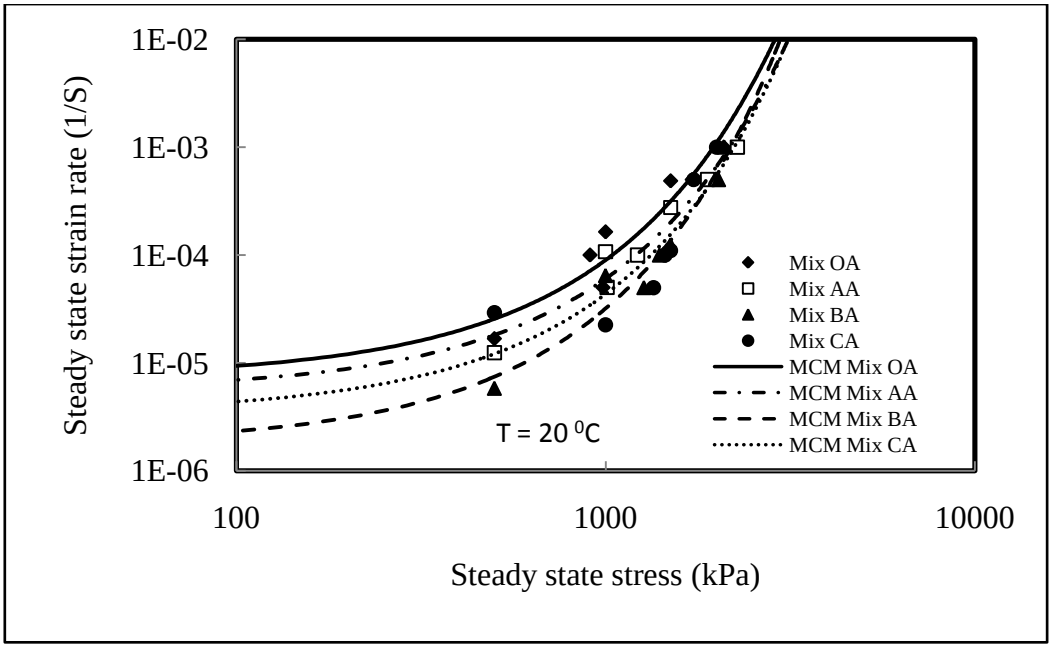


Figure 6-27: Effect of IBAA content level on mixtures' steady state permanent deformation behaviour at 20 ^{0}C.

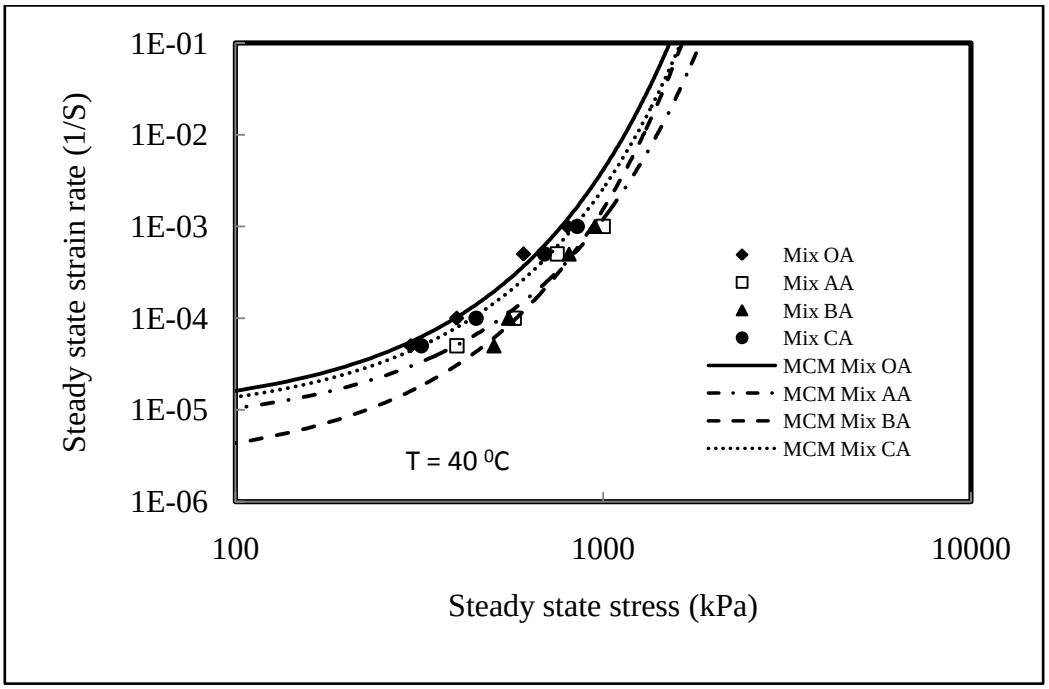


Figure 6-28: Effect of IBAA content level on mixtures' steady state permanent deformation behaviour at 40 ^{0}C.

This means that IBAA, up to 60% content level, had a noticeable positive effect on the mixtures' deformation resistance. Mix CA, on the other hand, although showed an improved deformation resistance compared to mix OA, was less resistant compared to mix BA at 20 ^{0}C and mix AA at 40 ^{0}C. This behaviour may be attributed to the same reasons mentioned in section 6.6.6.

6.8 CONCLUDING REMARKS

The general findings from the results discussed in the preceding sections can be summarised as follows.

The monotonic uniaxial constant strain rate test is useful to study the steady state permanent deformation behaviour of bituminous mixtures containing IBAA at high temperatures. At low temperatures, the uniaxial creep test is more desirable. Both tests can be used at 20 ^{0}C. Both tests can be useful in determining the dilation onset point in addition to the steady state stresses and steady state strain rates.

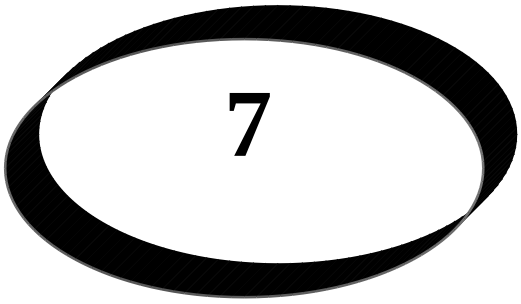
For all mixtures, the slope of the linear region of the volumetric against shear strain plot, referred to as the dilation gradient, was found to be independent of stress level and dependent on IBAA content and test temperature. It was found that dilation gradient values at 5 and 20 ^{0}C are very close but increased with temperature.

Poisson's ratio of the IBAA bituminous mixtures was determined using the plot of radial to axial strain ratio as a function of elapsed time from the creep test. Poisson's ratio for the different mixtures was found to be within a range of 0.2 to 0.4.

The steady state deformation behaviour of the IBAA bituminous mixtures was found to be well captured by the Modified Cross Model. The Modified Cross Model predicted that the steady state deformation of bituminous mixtures containing IBAA is linear at low stress levels while at high stress levels the mixtures exhibit non linear power law viscous behaviour. The IBAA mixtures' deformation behaviour was similar to their respective binders with a stiffening factor resulting from the effect of

aggregate matrix and ageing of bitumen during mixing. This stiffening factor was found to be dependent on temperature and IBAA content level.

It was shown that IBAA content has a significant effect on mixtures' permanent deformation behaviour. At low temperatures, the higher the IBAA content the higher the deformation resistance was of the bituminous mixture. At high temperatures, the same effect was noticed except for mix CA.



PERMANENT DEFORMATION BEHAVIOUR – TRIAXIAL TESTS

7.1 INTRODUCTION

In a real pavement, asphaltic mixtures are under three dimensional states of stress. The state of triaxial stress in the asphaltic mixtures within the upper layers has a significant effect on the deformation behaviour of the pavement. In the majority of research carried out on the deformation behaviour of asphaltic mixtures, uniaxial tests, usually in tension or compression, have been performed. Nevertheless, triaxial testing has long been used for understanding the general behaviour of asphaltic mixtures.

In the literature, a few researchers have studied the steady state permanent deformation behaviour under triaxial loading conditions. The key publications found in this area will be discussed here. The steady state permanent deformation of idealised asphaltic mixtures under triaxial conditions was investigated by Deshpandee et al (1999), Collop et al (2001) and Ossa (2004). They found that the steady state permanent deformation behaviour of the idealised mixtures with more than 64% volume fraction of aggregate strongly depends on the deviatoric and confining stresses. They conducted monotonic triaxial creep tests with different stress ratios. The stress ratio was defined as the mean stress divided by the deviatoric stress. This mean stress was calculated as the average value of the principal stresses. Results showed that the steady state axial creep behaviour of the mixtures was found to have the same form as that of the pure bitumen. The aggregate was found to have a stiffening effect which increased with increasing the volume fraction of aggregate.

Taherkhani (2005) studied the steady state permanent deformation behaviour of typical realistic mixtures under triaxial testing conditions. Two typical UK hot rolled asphalt (HRA) and dense bitumen macadam (DBM) mixtures were studied. The static triaxial creep tests were performed at 35 ^{0}C over a range of deviatoric stresses and stress ratios. Results showed that the triaxial steady state permanent deformation behaviour of the HRA and the DBM mixtures was found to be similar to the uniaxial steady state permanent deformation behaviour with non-linear power law viscous behaviour over the range of utilized stress levels, and was well captured by the modified Cross model (MCM). Moreover, it was found that both mixtures were observed to dilate under triaxial stress with the volumetric strain varying linearly with the shear strain. The slope, defined as the dilation ratio, was found to be independent of the stress ratio. These findings are similar to the uniaxial loading condition tests' which were presented in Chapter 6. These findings do not show significant differences between uniaxial and triaxial testing conditions and consequently do not promote testing bituminous mixtures under both loading conditions. However, it is noticed that in the reviewed literature the creep triaxial tests were used at high temperatures while constant strain rate tests were used under uniaxial loading conditions at the same high temperature which may cause credibility problems considering the comparison between the results of the two types of tests. Moreover,

the narrow source of references in this area and the complex composition and the difficult to predict behaviour of IBAA merit investigating the steady state deformation behaviour of IBAA bituminous mixtures under triaxial loading conditions.

In this chapter triaxial constant strain rate tests were undertaken at 40 ^{0}C using different confining pressures and four types of mixtures. The aim of this chapter is to study the effect of confining pressure on the steady state permanent deformation of the IBAA bituminous mixtures. Moreover, the applicability of the MCM was investigated. Firstly, the experimental work for development of the test is explained followed by discussion of the test results.

7.2 TRIAXIAL TESTS

This section presents the triaxial tests conducted on bituminous mixtures containing IBAA. The test used is constant strain rate test under confinement pressure. The tests were undertaken at temperature of 40 ^{0}C.

7.2.1 Specimen manufacture

The same sample geometry discussed in Chapter 6 was adopted for the triaxial tests on IBAA bituminous mixtures.

7.2.2 Test equipment

In the triaxial compression tests, a 14 kN loading frame was used. Fig 7-1 shows the equipment arrangement which comprises a standard triaxial cell of 150 mm diameter, housed inside a -5 to +50 ^{0}C temperature controlled cabinet. The loading frame is a CRT-NU14 – Universal servo pneumatic testing system. This machine is a development of the Nottingham Asphalt Tester (NAT). The CRT-NU14 uses a high precision pneumatic servo valve in conjunction with a specially developed low friction actuator, and sophisticated data acquisition which gives performance that is equal to many servo hydraulic systems. The system comprises rigid stainless steel test

frame with adjustable height cross-head, pneumatic actuator with low friction seal and pneumatic servo-valve, integral displacement transducer (14 kN maximum static load at 7 bar), load transducer (±20 kN capacity), and acquisition and control system.

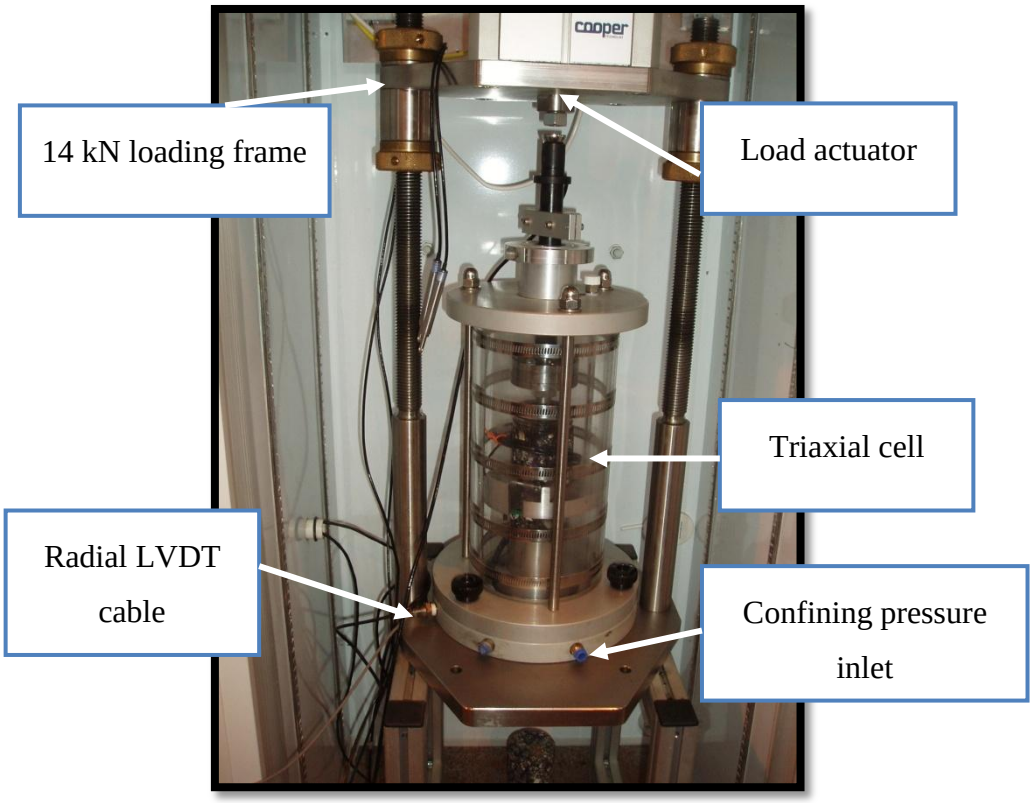


Figure 7-1: Triaxial test equipment.

Fig 7-2 shows the cell used in this research with a maximum confining pressure capacity of 280 kPa. This cell is a standard type, which is commonly used in soil mechanics. The cell had been designed for testing specimens of 100 mm diameter and 200 mm height. Some extra parts were designed to make it suitable for the specimen in this study of 134 mm in height and 67 mm in diameter. The cell consists of an aluminium base, cell chamber, submersible piston, valves and some fittings for connecting to the pressure gauge, air pressure supply and the LVDT for measuring radial strain.

A confining pressure was applied to the specimens using an air compressor connected to the triaxial cell. The same instrumentation system used to measure the radial

deformation in the uniaxial tests was used in the triaxial tests as well. The collar with the LVDT was housed inside the triaxial cell. An extension cable was used to acquire the LVDT displacement. This cable was connected to the conditioning box and then to the data logger which was connected to a PC. This PC was used to acquire the axial displacement and the applied load from another computer connected directly to the CRT-NU14. The axial strain was recorded using the load line displacement as using vertical LVDTs was not allowed by this machines' software.

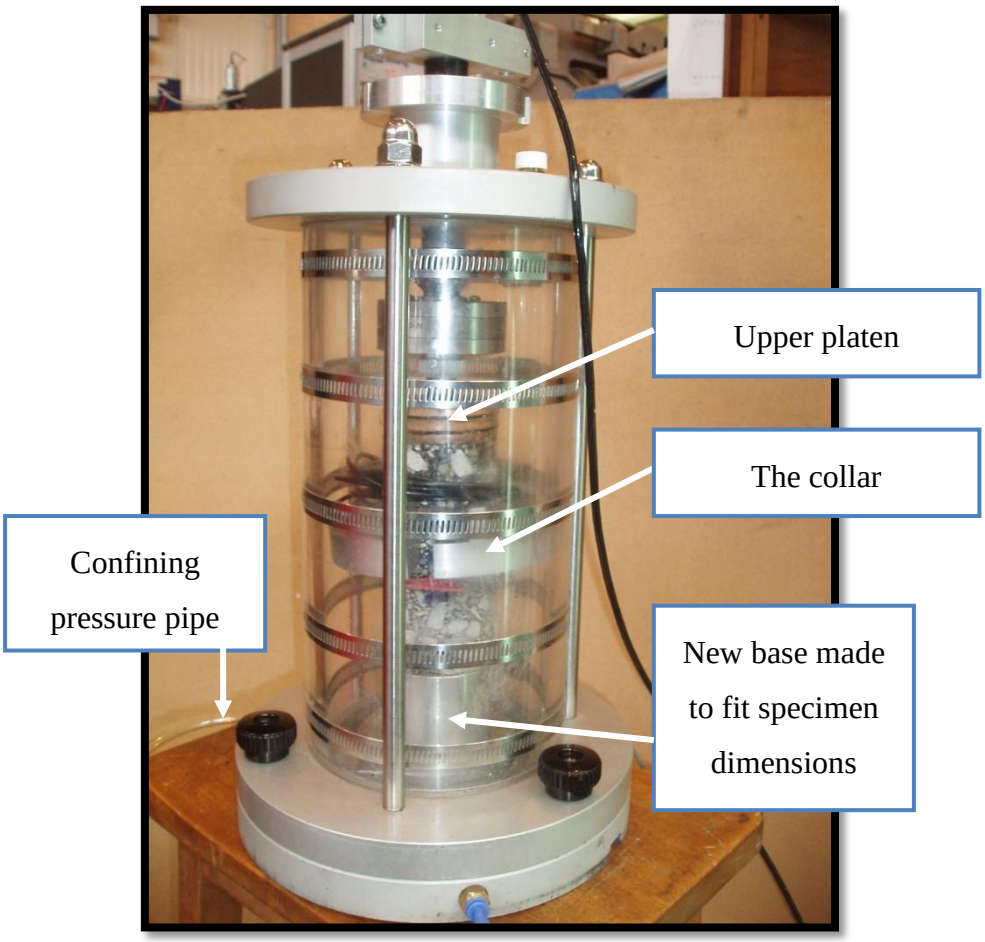


Figure 7-2: The triaxial cell.

7.2.3 Specimen instrumentation

After manufacturing, samples were kept at a temperature of 5 ^{0}C. To ensure uniformity of temperature, samples were conditioned inside the temperature control cabinet, at the testing temperature, for 2 to 6 hrs preceding the test. This duration was

found to be adequate for internal sample temperature to reach testing temperature. The temperature inside a cored dummy sample was measured hourly until it had reached the desired test temperature.

Prior to testing, samples height and diameter were measured accurately. The mid-height was marked to help with collar alignment. The collar was then held in place using the same steps used in the uniaxial tests. Specimens were then placed vertically on the cell's base, as seen in Fig 7-3, after smearing it with silicon grease as a lubricant. The LVDT cable was connected to the cable extension. Then the top of the sample was smeared with silicon grease before being covered with the upper platen. The triaxial cell was then put on. The whole cell was housed inside the temperature controlled cabinet.

Figure 7-3: Sample instrumentation in the triaxial cell.

7.2.4 Test procedure

Firstly, the triaxial cell left for 30 to 45 minutes inside the temperature controlled cabinet before commencing testing. This time was allowed to ensure that specimens were restored to the test temperature as all the specimen instrumentation steps had to

be undertaken outside the temperature controlled cabinet. The time allowed was varied based on the difference between the test temperature and the laboratory temperature. A dummy sample was used to control the time needed to restore test temperature.

Before testing, samples were subjected to a preloading to take out any relaxation in the system. Then, the confining pressure was applied gradually. When the confining pressure reached the desired level, the specimens were allowed to deform under constant strain rate up to failure. Both axial and radial displacements against the applied load were measured and recorded.

The triaxial tests were conducted at 40 ^{0}C. This temperature was chosen as a high temperature which causes high permanent deformation. Higher temperatures were not used to avoid problems associated with handling the specimen as the softening point of the binder was measured as 43 ^{0}C. Trial tests at higher temperatures showed significant problems associated with specimen handling. Moreover, test duration was very short which led to spurious results. Trial tests at lower temperatures showed no differences in the results at different confining pressures. This may be attributed to the limited range of the confining pressure of the used machine. These confining pressures range between 0 and 200 kPa. However, high confining pressures may be considered as very extreme conditions. The tested mixtures were designed for binder course layers in asphalt pavements which mean that the confining pressure caused by the overburden pressure may be limited to maximum of 100 kPa. This is assuming that the surface layer thickness is 25 - 50 mm. The confining pressure, which equals the overburden pressure (assuming an axle load of 10 tonnes) multiplied by the layer thickness, results as a maximum of 100 kPa. Therefore, the confining pressures used in this study were 50, 100 and 150 kPa.

The test type was chosen as triaxial constant strain rate test. The reason for this was to use equal basis in comparing uniaxial to triaxial results. The uniaxial tests showed that uniaxial constant strain rate is the appropriate test type at high temperatures. The

strain rates used in the triaxial tests were 0.0001, 0.0002 and 0.0005 1/s. Higher strain rates were not allowed due to machine limitations.

7.3 TRIAXIAL TEST RESULTS

7.3.1 Stress-strain relationship

The stress-strain relationship was recorded, under triaxial loading conditions, for the mixtures over a range of strain rates and confining pressures. The stress used is the vertical stress calculated according to Eq 7-1 and presented in Fig 7-4. The resulting relationship showed the same shape of curve for all mixtures. At the same strain rate, the higher the confining pressure the higher the steady state stress was. This result was attributed to the increase in sample resistance due to confinement. Moreover, at high strain levels, samples exhibited lower strain at higher confining pressures, as expected. That was not the case at the early stages of the tests. This may be attributed to factors such as platen friction and particle reorientation. Fig 7-5 shows a typical triaxial result. The steady state stresses corresponding to the applied strain rates were determined using the same method as in the uniaxial constant strain rate tests (UCSRT).

$$\sigma_v = \frac{Q}{a} + \sigma_a \quad (7\text{-}1)$$

where: σ_v is the vertical stress; Q is the applied vertical force; a is the cross sectional area of the specimen; and σ_a is the confining stress as shown in Fig 7-4.

Fig 7-6 shows the stress-strain relationship under specified testing conditions. Similar to UCSRT, it was noticed that the radial strain reached the steady state stage before the axial strain and starts to decrease while the axial strain is continue to increase up to a point after which the axial strain reaches the steady state stage and levels before starting to decrease. Consequently, it is concluded that IBAA bituminous mixtures did not exhibit a significant change in their permanent deformation behaviour trends due to applying confining pressures. This does not mean that there was no effect of the confining pressure. This effect will be discussed in the following sections.

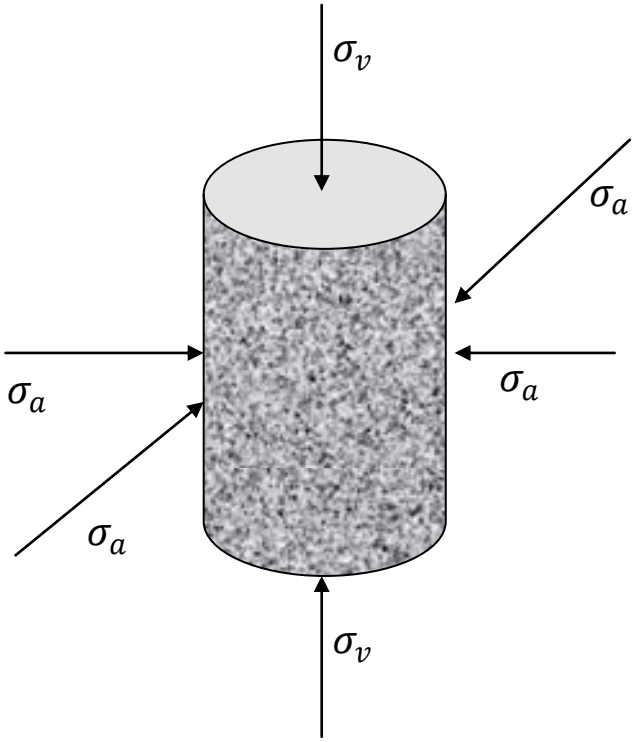


Figure 7-4: Triaxial applied stresses.

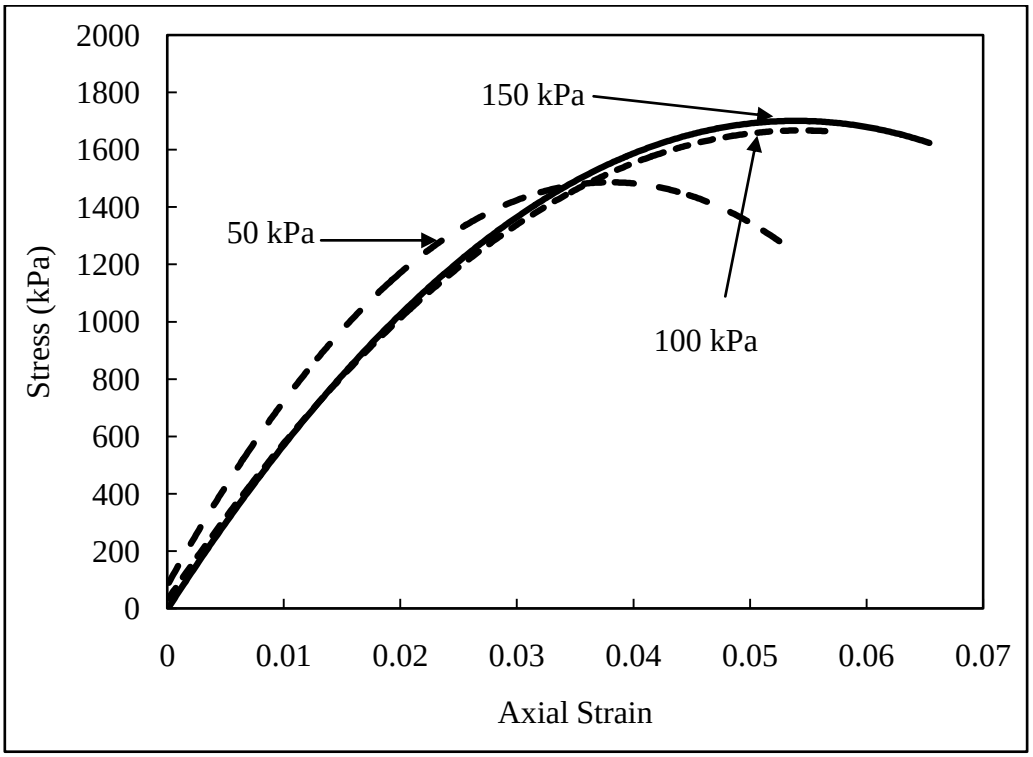


FIGURE 7-5: Typical triaxial test results (Mix BA at 0.0002 1/s and 40 ^{0}C).

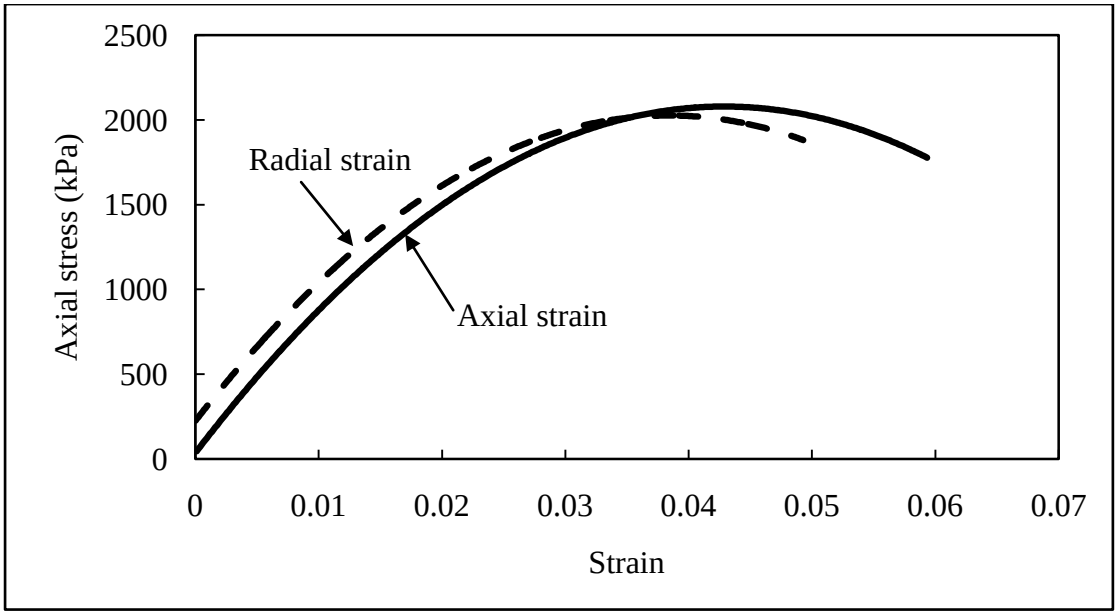


FIGURE 7-6: Axial and radial strains from triaxial test results (Mix BA at 0.0005 1/s strain rate, 100 kPa confining pressure and 40 ^{0}C).

7.3.2 Volumetric behaviour

Fig 7-7 shows the variation of volumetric strain with the shear strain from a triaxial test under selected testing conditions; similar results were obtained from uniaxial tests as shown in Chapter 6. It is obvious that, initially, the volumetric strain decreased due to densification up to a point, after which the volumetric strain increased almost linearly with the axial strain due to dilation with a linear slope equals dilation gradient.

The dilation gradient was found to be independent of the applied confinement for all tested mixtures. However, it increased under triaxial loading conditions compared with its value under uniaxial testing conditions. Similar results were obtained for idealised asphaltic mixtures (Khanzada 2000) and for typical UK dense bitumen macadam and hot rolled asphalt materials (Taherkhani 2005) which support the feasibility of replacing conventional aggregate with IBAA in bituminous mixtures.

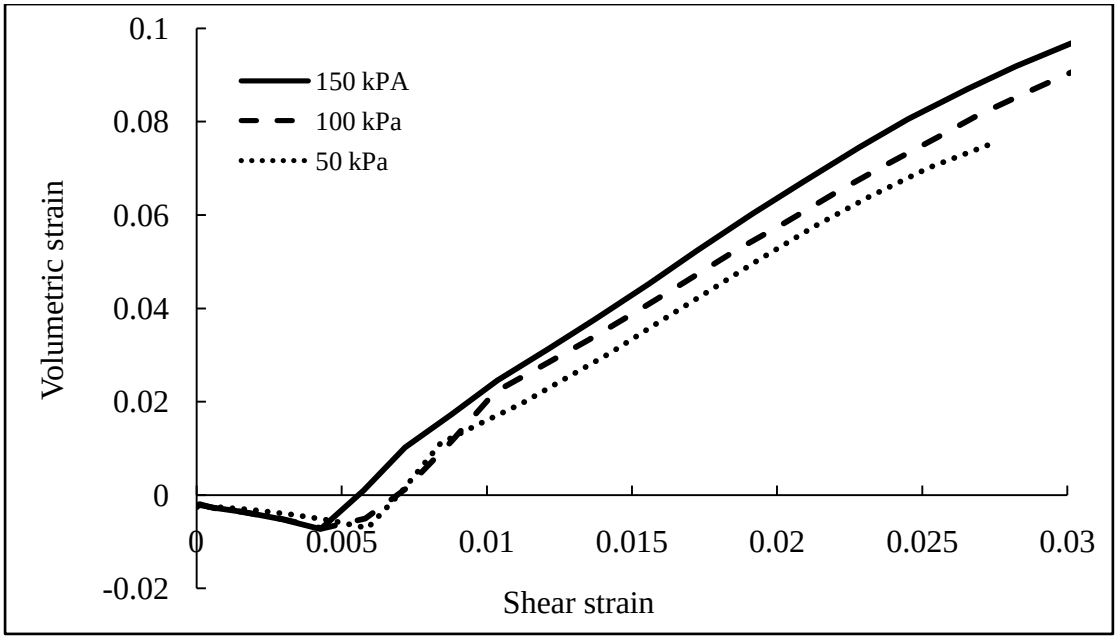


FIGURE 7-7: Variation of volumetric against shear strain in triaxial tests (mix Mix BA at 0.0005 1/s strain rate and 40 ^{0}C).

Dilation gradients, calculated as explained in Chapter 6, under triaxial testing conditions did not show significant deviation from those found from the uniaxial tests. They showed a minor increse within a range of 5%. This shows that testing IBAA bituminous mixtures under triaxial conditions did not have any additional merits, with regard to dilation behaviour, over uniaxial tests.

7.3.3 Steady state permanent deformation

With respect to the steady state deformation under triaxial testing conditions, the same behaviour observed under uniaxial testing was noticed again as mentioned earlier in this chapter. Therefore, the MCM can be used to predict the steady state permanent deformation performance under triaxial conditions. The confinement resulted in higher deformation resistance for mixtures and, therefore, higher stiffening factor S. Fig 7-8 shows an example for triaxial results at 50 kPa confining pressure compared to the uniaxial results. Results at higher confinement pressures showed that the higher the confinement the higher the deformation resistance was. S values for mixes OA, AA, BA, and CA, at 50 kPa confining pressure, were 8000, 11000, 13500, and 10000 respectively. These values show increases in S by 36, 33, 15, and 40 times compared

with the uniaxial results. Using 100 kPa confining pressure increased S by 40% more while using 150 kPa led to additional increase by 20%. These results could be considered more representative of real road loading conditions as the triaxial loading condition is more simulative to the real conditions.

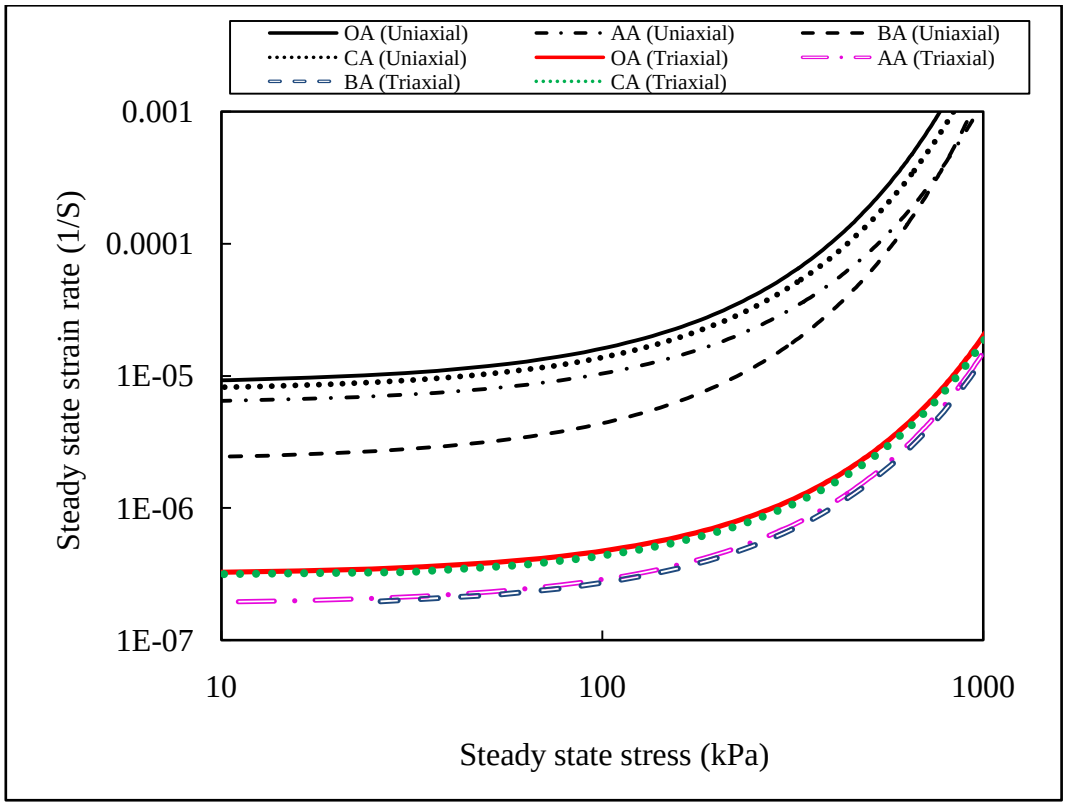


FIGURE 7-8: Steady state permanent deformation behaviour under triaxial testing conditions.

7.3.4 MCM parameters

The MCM parameters were determined using the same procedure explained in Chapter 6 and are presented in Table 7-1.

Parameter	Mix OA	Mix AA	Mix BA	Mix CA
$\dot{\varepsilon}_p$ (1/s)	2.288E-05	1.373E-05	9.153E-06	5.019E-06
m	0.852	0.858	0.859	0.850
σ_0 (kPa)	301.9	288.3	273.7	323.9

Table 7-1: MCM parameters under triaxial testing conditions

7.4 EFFECT OF IBAA ON MIXTURES' DEFORMATION BEHAVIOUR

From Fig 7-8, it can be seen that the qualitative permanent deformation behaviour of the IBAA bituminous mixtures under triaxial testing conditions was similar to that under uniaxial testing conditions at 40 ^{0}C. It is clear that mixes AA and BA exhibited an increase in their steady state permanent deformation resistance compared to mix OA, the control mix. This means that, up to 60% content level, IBAA had a noticeable positive effect on bituminous mixtures' deformation resistance. Mix CA, on the other hand, although showed an improved deformation resistance compared to mix OA, was less resistant compared to mix BA. This behaviour may be attributed to the same reasons related to binder content and discussed in Chapter 6. This trend of behaviour was independent on the confining pressure level.

7.5 CONCLUDING REMARKS

The general findings from the results discussed in the preceding sections can be summarised as follows:

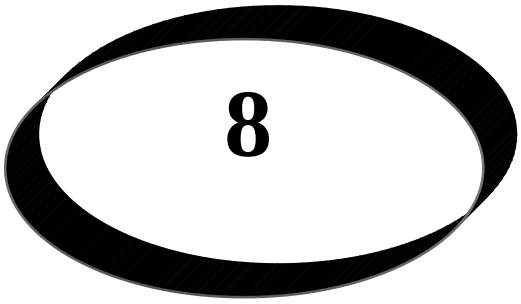
The monotonic triaxial constant strain rate test is useful to study the steady state permanent deformation behaviour of bituminous mixtures containing IBAA at high temperatures. At the same strain rate, the higher the confining pressure the higher the

steady state stress was. However, the qualitative resulted deformation behaviour was similar to that noticed using uniaxial testing conditions.

For all mixtures, the slope of the linear region of the volumetric against shear strain plot, referred to as the dilation gradient, was found to be independent of confining stress level and dependent on IBAA content. It was found that dilation gradient values did not change significantly comparing with those values obtained using the uniaxial tests.

Under triaxial testing conditions, the steady state deformation behaviour of the IBAA bituminous mixtures was found to be well captured by the Modified Cross Model. The Modified Cross Model predicted that the steady state deformation behaviour of bituminous mixtures containing IBAA is linear at low stress levels while at high stress levels the mixtures exhibit non linear power law viscous behaviour. The IBAA mixtures' deformation behaviour was similar to their respective binders' with a stiffening factor resulting from the effect of aggregate matrix and ageing of bitumen during mixing. This stiffening factor was found to be dependent on IBAA content level and the confining pressure.

It was shown that IBAA content has a significant effect on mixtures' permanent deformation behaviour. The higher the IBAA content the higher the deformation resistance was of the bituminous mixture except for mix CA.



FRACTURE AND CRACK PROPAGATION

8.1 INTRODUCTION

Bituminous mixtures are usually subjected to cracking as a consequence of the repeated application of traffic loads, thermal cycling or a combination of the two mechanisms. Cracking is considered as one of the major distress modes in asphalt pavement. Application of load traffic and temperature changes cause generation of tensile stresses at the bottom of the pavement layer. These tensile stresses remain under the tensile strength of the pavement layer for a number of load applications and thermal cycling after which cracks initiate at weak points of the pavement layer. These cracks, then, propagate towards asphalt layer surface until fracture occurs.

This chapter presents the fracture characteristics of bituminous mixtures containing IBAA. Linear Elastic Fracture Mechanics (LEFM) approach has been used to evaluate crack resistance of the tested mixtures aiming to investigate the effect of IBAA content on these properties. LEFM leads to the determination of the stress intensity

factor, also known as fracture toughness, fracture energy and Paris Law parameters. The crack propagations were monitored using a digital camera. A recent approach was adopted in interpreting crack growth. In addition, a novel approach was presented to obtain asphalt damage parameters using fracture tests.

8.2 CRACKING IN BITUMINOUS MIXTURES

Cracking in pavements has been shown to be a major source of distress afflicting asphalt in roads. Presence of cracks in pavement considerably affects the effective modulus of the bituminous mixtures which in turn lessens the influence of the temperature on pavement performance (Evdorides et al 2006). Moreover, these cracks lead to weakening the foundation of the pavement structure as a result of water penetration (Seong et al 2005).

Cracking consists of two phases: crack initiation and crack propagation. The former is generally described as the accumulation of micro cracks to form a macro crack due to the repeated load application and cyclic temperature variation. Further application of repeated load and temperature variations, leads to growth of macro cracks through the material in a phenomenon known as crack propagation.

Crack propagation can follow one or more of the three fracture modes illustrated in Fig 8-1 depending on the loading configuration (Artamendi and Khalid 2006). In mode I, opening or tensile mode, the crack tip is subjected to a normal stress and the crack surfaces move directly apart so that the displacements of the crack surfaces are perpendicular to the crack plane. In mode II, sliding mode, the crack tip is subjected to an in-plane shear stress and the crack surfaces slide over one another so that the displacements of the crack surfaces are in the crack plane and perpendicular to the crack front. Finally, in mode III, tearing mode, the crack tip is subjected to an anti-plane (out-of-plane) shear stress and the crack surfaces move parallel to the crack front and remain in the crack plane. A combination of any two of the three fracture modes constitutes a mixed mode of loading. For instance, a combination of mode I and mode II forms a mixed-mode I-II. Mode I loading is the most commonly

encountered and it is associated with a local displacement in which the crack surfaces move directly apart. In this chapter, only mode I is considered as the aim was to investigate the effect of IBAA content level on crack properties of bituminous mixtures so, there was no interest in studying the effect of loading modes on these properties.

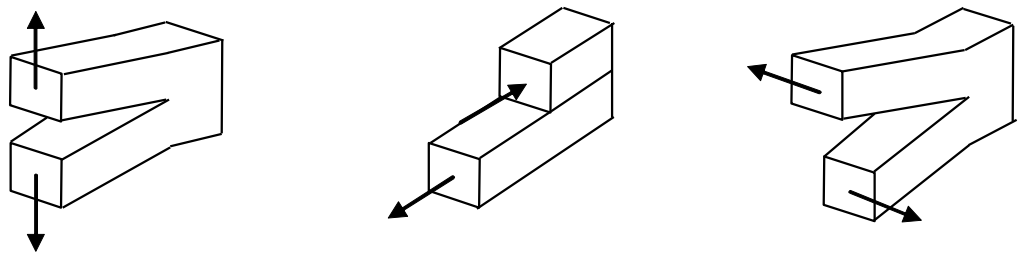


Figure 8-1: Modes of fracture in bituminous mixtures.

8.2.1 Factors affecting crack resistance

Cracking resistance of bituminous mixtures is their ability to withstand repeated loading without fracture. It is affected by a number of factors. The main factors include stresses induced in the pavement which in turn depends on traffic speeds and axle load (Collop and Cebon 1995). Mode of loading is also an important factor when characterising bituminous mixtures. Brown (1979) showed that there is a difference in crack resistance, in terms of failure mechanism, depending on mode of loading as the failure occurs by formation of cracks at specified points of high stress concentration. Then, these cracks propagate through the material until fracture occurs. This propagation depends on the stress intensity at the crack tip. More factors include test wave form and rest periods. Said (1988) concluded that the haversine wave shape is the closest form to the field conditions while inclusion of rest periods results in a general increase in crack propagation time (Bonnaure et al 1982, Francken and Clauwert 1987).

Mixture variables have, also, an effect on crack resistance of bituminous mixtures. These variables include mixture stiffness, bitumen properties and content, and air voids content. Mixture stiffness is considered as one of the most important factors which affect the crack resistance properties of bituminous mixtures. In stress controlled mode of loading, the higher the mixture stiffness the longer the time needed for failure. On the other hand, in strain controlled mode of loading there is a decrease in failure needed time (Pell 1973).

Bitumen properties have a noticable role in crack resistance properties of bituminous mixtures. In stress controlled mode of loading, higher bitumen stiffness leads to higher crack resistance (Copper and Pell 1974) while in strain controlled mode of loading the converse is true (Brown 1988). In addition. it was found that increasing bitumen content (Gibb 1996) and decreasing air voids content (Judycki 1991) led to an increase in crack resistance properties.

8.3 FRACTURE MECHANICS

8.3.1 Fracture mechanics and bituminous mixtures

Conventionally, fracture behaviour of bituminous mixtures has been characterised by comparing calculated stresses with the tensile strength of the material. This method assumes the tensile strength as the fracture criteria, accordingly the higher the tensile strength the higher the resistance to fracture. However, fracture mechanics theory establishes that high strength materials can be very susceptible to fracture in the presence of cracks. These cracks act as stress concentrators and have a fundamental effect on the fracture strength. If a crack is formed in a bituminous pavement layer, the pavement will fail at very much lower level of stress than it can sustain without the crack.

Fracture mechanics theory was first applied to bituminous mixtures to depict crack propagation in asphalt based on linear elastic analysis. Based on Linear Elastic Fracture Mechanics (LEFM), Irwin (1957) introduced the stress intensity factor, also

known as fracture toughness, to uniquely define the stress field at the crack tip. In pure tension loading, mode I, Paris and Erdogan (1963) found that the crack growth rate is a function of the stress intensity factor of that loading mode. The relationship between stress intensity factor and crack growth rate is commonly known as Paris Law.

Moavenzadeh (1967) and Bahgat and Herrin (1986) used fracture mechanics theory to evaluate brittle fracture in bituminous materials. These trials were continued by Majidzadeh et al (1969, 1971) who used fracture mechanics to analyse fatigue and fracture of asphalt mixtures. Aglan and Figueroa (1993) used the critical stress intensity factor as a failure criterion for bituminous mixtures. More recently, a new developed fracture toughness formula (Kim and El Hussein 1997) was used to reflect the non-linear load deflection behaviour prior to attainment of the peak load of quasi-brittle materials. This method was called the effective crack model and used as a direct method for determination of fracture toughness of asphalt beams experimentally.

Based on elastic-plastic fracture mechanics (EPFM), Abdulshafi and Majidzadeh (1985) applied the critical strain energy release rate, also known as critical J-Integral, to bituminous mixtures. Notched samples with various initial crack lengths were tested in load controlled mode to obtain load displacement relationship. The slope of this relationship was found to be the J-Integral value.

Dongre et al (1989) employed both the fracture toughness and the J-Integral as a fracture criterion of bituminous mixtures. They tested notched beams under three point bending at different temperatures and different notch to beam depth ratio. The study showed that the critical value of the J-Integral, as a fracture characterisation parameter, is sensitive to binder and mix properties while the fracture toughness, as a fracture parameter, is not sensitive to binder grade or source. This research showed, as well, the applicability of both, stress intensity factor and J-Integral, to asphalt fracture. However, no relationship between the two parameters was presented. More recently, the fracture toughness, fracture energy and the J-Integral have been used as a fracture

criterion for asphalt pavements by a number of researchers e.g. Hossain et al 1999, Mull et al (2002) and Li and Marasteanu (2004). Again, there were no trials to find the relationship between these parameters. This relationship was presented recently by Kuai et al (2009). They found that it is possible to obtain J-Integral in terms of stress intensity factor and, moreover, they demonstrated that J-Integral is applicable for linear elastic fracture mechanics.

The fracture mechanics theory has been applied not only to conventional bituminous mixtures but also to mixtures incorporated modified binders (Mull et al 2002, Othman et al 2005, Mull et al 2005, Othman 2006, Haryanto and Takahashi 2007). In these studies, it was found that the addition of either crumb rubber or other modifiers enhanced the asphalt mixture resistance to crack propagation. Despite all this research, the application of fracture mechanics theory to bituminous mixtures is still limited to laboratory investigations. Moreover, it was not applied to bituminous mixtures containing secondary or recycled materials.

8.3.2 Stress intensity factor criterion

Fracture mechanics theory has established that materials can be very susceptible to fracture in the presence of cracks. These cracks act as stress concentrators and have a significant impact on the fracture resistance of the material. Based on linear elastic analysis, Irwin (1970) obtained solutions for crack tip stress distribution associated to the three major modes of loading shown in Figure 8-1. Based on concepts of elastic theory (Timoshenko 1934), if a crack is subject to a tensile stress, the stresses at the crack tip, for the notations shown in Fig 8-2, are found to be as in Eq 8-1 and 8-2.

$$\sigma_x = \frac{K_1}{\sqrt{2\pi r}}\cos\frac{\theta}{2}\left(1-\sin\frac{\theta}{2}\,sin\frac{3\theta}{2}\right)$$

$$\sigma_y = \frac{K_1}{\sqrt{2\pi r}}\cos\frac{\theta}{2}\left(1+\sin\frac{\theta}{2}\,sin\frac{3\theta}{2}\right) \qquad (8\text{-}1)$$

$$\sigma_z = \nu\left(\sigma_x + \sigma_y\right) for\ plane\ strain\ \&\ \sigma_z = 0\ for\ plane\ stress$$

and,

$$\tau_{xy} = \frac{K_1}{\sqrt{2\pi r}} \cos\frac{\theta}{2}\left(\sin\frac{\theta}{2}\, cos\frac{3\theta}{2}\right)$$

$$\tau_{xz} = 0 \qquad (8\text{-}2)$$

$$\tau_{yz} = 0$$

where σ is the tensile stress; K_1 is the stress intensity factor; θ and r are the polar coordinates; ν is Poisson's ratio; and τ is the shear stress.

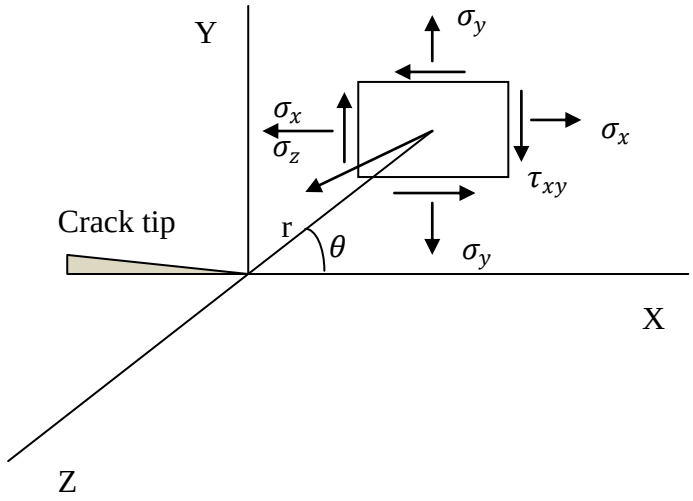


Figure 8-2: Distribution of stresses in neighbourhood of crack tip.

The stress intensity factor depends on the configuration of the cracked component, i.e. its geometry, and on the load application mode. Generally, stress intensity factor may be expressed as in Eq 8-3 (Callister 1997):

$$K_1 = Y\,\sigma\,\sqrt{\pi a} \qquad (8\text{-}3)$$

where: σ is the applied stress; Y is a dimensionless parameter that depends on the crack and specimen size and geometry, as well as on the manner of load application; and a is the notch or crack length.

The stress intensity factor,K_1, uniquely defines the stress field at the crack tip. Once it has been known, it is then possible to determine the maximum stress intensity factor that would cause failure. This critical value,K_{1c}, is known as the fracture toughness of the material. Fracture toughness is a material property which represents its resistance to brittle fracture when a crack is present. Fracture toughness can, therefore, be defined as a measure of a material's resistance to crack extension when the stress state near the crack tip is predominantly plane strain, plastic deformation is limited and opening mode monotonic loading is applied (Artamendi and Khalid 2007). Fracture toughness can be used to calculate the critical load, at which a construction with a certain crack length, fails. Moreover, it helps in calculating the critical crack length at which a construction fails under a specific axle load. However, with fracture toughness, it is not possible to predict how long a construction needs to fail (Hofman et al 2003).

If specimen thickness is large relative to the dimensions of the crack, a condition of plane strain dominates. Under this condition, the fracture toughness becomes independent of specimen thickness (Hertzberg 1996). In addition, linear elastic analysis assumes that stresses at the crack tip are infinite. In reality, however, stresses at the crack tip are finite due to blunting and inelastic deformation. Accordingly, a small region around the crack tip, known as crack inelastic zone, is formed in which the material behaves plastically rather than elastically. Thus, the elastic stress analysis becomes increasingly inaccurate as the inelastic region at the crack tip grows. However, for sufficiently large bodies, the inelastic zone might be small compared to the specimen geometry. It follows that, at large distances compared to the inelastic zone but still small compared to the specimen dimensions, the elastic stresses are dominant and linear elastic behaviour before failure prevails. This is commonly referred to as small scale yielding. So, as long as the condition of small scale yielding is satisfied, stress intensity factor characterises the crack tip stresses, strains and displacements even within the crack tip inelastic zone (Artamendi and Khalid 2006). Based on these conclusions, only the LEFM is adopted in this research.

8.3.3 Fracture energy

Fracture energy, G_f, can be utilized as a parameter to describe the fracture resistance of asphalt (Tan et al 1994) and is defined as the rate of energy dissipation which controls the failure mode from crack initiation to full depth crack propagation. Fracture energy is applicable to both linear elastic and elastic-plastic analysis, (Hossain et al 1999, Li and Marasteanu 2004). The fracture energy can be calculated by dividing the work of fracture, W, i.e. the area under the load versus load-line displacement curve, by the ligament area (A_{Lig}), i.e. the product of the ligament length and the specimen thickness (RILEM TC 50-FMC 1985); as shown in Eq 8-4.

$$G_f = {W}/{A_{Lig}} = {\int Pdu}/{A_{Lig}} \tag{8-4}$$

8.3.4 J-Integral

Asphalt is a viscoelastic material therefore, its fracture behaviour can be characterised by means of the parameter J-Integral, *J*. Rice (1968) developed a method to characterise fracture behaviour based on determination of an energy term that expresses the change in potential energy with changing crack length. It was shown that the change in potential energy could be released by a path-independent line integral, so called J-Integral, around the crack tip. To calculate *J*, Schapery (1984) integrated a non-linear viscoelastic constitutive equation and presented the result as shown in Eq 8-5.

$$J = \int_i \left(W_j\, dy - T_j \frac{\partial u}{\partial x}\, ds\right) \tag{8-5}$$

where: W_j is the strain energy density; T_j is the stress vector acting on the contour; u is the displacement vector; ds is the increment along contour i; and x and y are coordinates normal to the crack front.

For linear elastic conditions, J represents the energy made available at the crack tip or in other words the crack driving force. For elastic conditions, it corresponds to the energy release rate, G, which is expressed mathematically as in Eq 8-6. In this case, elastic conditions, it was found that $J = G$ (Anderson, 2005).

$$G = K_1^2 / E^* \tag{8-6}$$

where:

$$E^* = E \quad \text{(plane stress)} \tag{8-7}$$

$$E^* = E/(1 - \nu) \quad \text{(plane strain)} \tag{8-8}$$

where: ν is Poisson's ratio

On the other hand, for viscoelastic condition, J no longer represents the available energy at the crack tip because of energy dissipation. However, the correspondence principle of viscoelasticity, demonstrated by Schapery (1984), makes it possible to define a generalized time-dependent J-Integral by forming a J_e, which is a pseudo-elastic J-Integral, with the linear elastic case as shown in Eq 8-9 (Kuai et al 2009). This J_e is equal to the energy release rate G. Kaui et al (2009) showed that the viscoelastic problem can be converted to an elastic problem with the pseudo stress and strain parameters. Then the generalized J-Integral is given as in Eq 8-10.

$$J_e = \int_i \left(W^e \, dy - T_j \, \frac{\partial u^e}{\partial x} \, ds \right) \tag{8-9}$$

$$J = E_R \int_{t_0}^{t} \left(D(t-\tau) \frac{\partial J_e}{\partial \tau} d\tau \right) = \int_{t_0}^{t} \left(D(t-\tau) \frac{\partial K_1^2}{\partial \tau} d\tau \right) \tag{8-10}$$

where: W^e is the pseudo strain energy density; u^e is the pseudo displacement vector; E_R is a reference modulus; τ is the retardation time for the i^{th} element and $D(t)$ is the creep compliance.

8.4 CRACK PROPAGATION

In many practical applications, failure can occur if initially small cracks grow to critical length during in-service loading by mechanism such as fatigue. Linear Elastic Fracture Mechanics (LEFM) analysis has been used to characterise crack growth rate by means of the well known Paris Law (Paris and Erdogan 1963), in which the rate of crack propagation is a function of the stress intensity factor. This law is given by the following expression:

$$da/dN = A\ (\Delta K_1)^n \tag{8-11}$$

where: da/dN is the crack growth rate; ΔK_1 is mode I stress intensity factor range ($K_{max} - K_{min}$); A and n are constants depend on the material and on the test conditions; a is the crack length; and N is the number of load cycle applications.

In this research, samples were of semi circular bending (SCB) geometry and their ΔK_1 value can be calculated using Eq 8-12 (Lim et al 1993).

$$\Delta K_1 = (\Delta P/2rt)\ Y_1 \sqrt{\pi\ a} \tag{8-12}$$

where: ΔP is the applied load range ($P_{max} - P_{min}$); r is the specimen radius; t is the specimen thickness; and Y_1 is normalised mode I stress intensity factor which can be determined, for SCB samples with span to diameter ratio (s/r) of 0.8 as in the current study, using Eq 8-13 (Lim et al 1993):

$$Y_1 = 4.782 - 1.219\ (a_0/r) + 0.063 \exp(7.045\ (a_0/r)) \tag{8-13}$$

where: r is the sample radius and a_0 is the notch depth as shown in Fig 8-3.

The determination of the Paris Law constants, A and n in Eq 8-11, involves the calculation of the crack growth rate, da/dN, and the mode I stress intensity factor range, ΔK_1, for the corresponding crack length values. Different techniques have been used to determine crack growth rate. For instance, the British and US standards for

metallic materials (BSI 2002, ASTM 2005) suggest using either a secant method, in which da/dN is determined from the slope of a line joining two adjacent points, or an incremental polynomial approach, in which a second order polynomial is fitted to sets of successive data points. For bituminous materials, on the other hand, some researchers (Jacobs et al 1996, Roque et al 1999) have fitted a third-order polynomial to the crack growth phase which was then numerically differentiated. Other researchers (Artamendi and Khalid 2006, 2007) used successfully the secant method.

As the crack propagation of asphalt is normally accompanied with large scale yielding, not small scale yielding as assumed by LEFM, it has been shown, by Dowling and Begley (1976), Lambert et al (1988) and recently Kuai et al (2009), that J-Integral can be used instead of stress intensity factor, in accordance with Eq 8-14, to be more suitable to characterize conditions at the crack tip.

$$da/dN = A_j\ (\Delta J)^m \qquad (8\text{-}14)$$

where: A_J & m are constants; and ΔJ is the contour integral for cyclic loading which can be calculated by plugging Eq 8-6 into Eq 8-10 before solving the integration.

In this chapter, Paris Law was used, using stress intensity factor, to investigate the effect of IBAA content level on crack propagation resistance of bituminous mixtures. Moreover, the applicability of Paris Law using J-Integral, as a recent technique, to IBAA bituminous mixture was examined.

8.5 SEMI CIRCULAR BENDING TEST

Several types of tests are available for the determination of material crack properties. These properties are usually evaluated using the overall response of a sample when subjected to a specific loading mode. The response of the material is controlled by geometry and material factors and it is clear that the geometry effects should be as limited as possible. Therefore, it is recommended that the stress fields applied to the specimen should be as simple as possible.

The most common types of crack propagation tests are: Thermal Stress Restrained Specimen (TSRST) (Monosmith et al 1965); Indirect Tension Test (ITT) (Hadipour and Anderson 1988); Direct Tension Test (DTT) (Bolzan and Huber 1993); and recently, the Semi Circular Bending (SCB) test (Molenear et al 2002).

Much interest has recently been witnessed in semi circular bending (SCB) test as a laboratory tool to study the resistance to crack propagation of asphalt mixtures. The principle of the semi circular bending test is shown in Fig 8-3. The test was originally used in rock mechanics to determine the characteristics of the material investigated and, then, adopted to be used in asphalt. European draft standards have been issued that adopts the SCB in a monotonic (CEN 2005) and cyclic (CEN 2006) modes to determine the fracture toughness of notched and un-notched specimens.

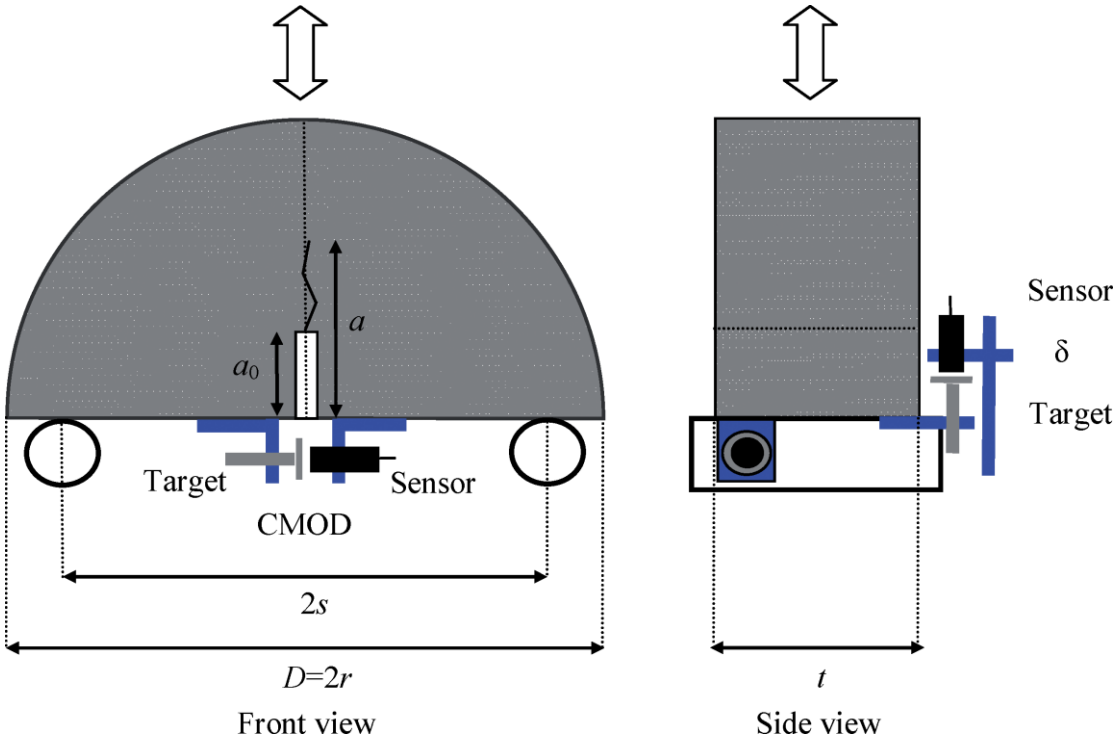


Figure 8-3: Schematic of semi circular bending test sample.

Use of SCB test in asphalt showed a number of advantages over other crack properties tests. One of such advantages is the fact that a clear crack develops in the SCB test without wedging near the loading strip (Molenaar et al 2002). This latter phenomenon

is quite often observed in the ITT as seen in Fig 8-4. This clear crack dominates the failure mode to be tension effect rather than any other effect. This gives relevant information on the tensile characteristics of the material tested. Moreover, the SCB is a simple, low cost test that easily can be performed on cored samples.

Figure 8-4: SCB test crack compared to ITT (Molenaar et al 2002).

8.5.1 Sample preparations

To prepare samples for SCB tests, see Fig 8-5, bituminous slabs were manufactured as detailed in Chapter 3. Cylindrical cores of 153 mm diameter, D, and 65 mm thickness, t, were cored. Each cylinder was then cut, perpendicular to the axis, in half to obtain semi circular samples. These were then notched at mid-point in the direction of the load using a diamond-tip saw tile cutter. Notch length, a_0, was 15 mm, which have a notch to radius ratio, a_0/r, of 0.2. Samples were then stored in an incubator at a temperature of 5 ^{0}C until test commencement. For cyclic SCB tests, sample faces were painted in white for photos capturing purposes as the used digital camera can detect black and white colours only. Then the transducer holders and targets were glued to their positions.

8.5.2 Testing programme

Four different mixtures were tested; a control mix and three IBAA mixtures. The tested mixtures had the same composition detailed in Chapter 3; OA, AA, BA, and CA. Two types of test were employed in this research. Firstly, monotonic SCB tests

were conducted with the aim of determining the fracture strength, stress intensity factor and fracture energy of the samples at a temperature of 5 ^{0}C. These tests were followed by cyclic SCB tests in which the mixtures were tested under a cyclic load of 1.5 kN under a haversine load frequency of 1 Hz and 5 ^{0}C temperature.

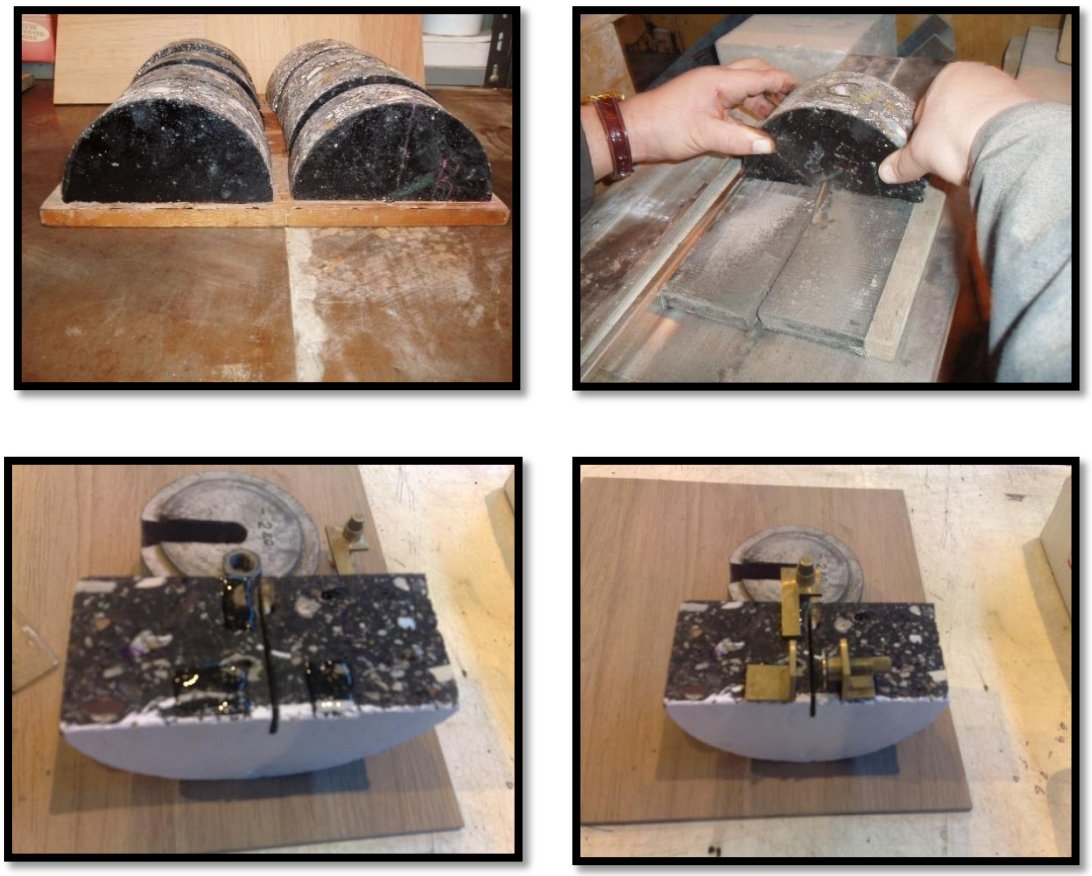


Figure 8-5: Sample preparations for SCB tests.

8.5.3 Fracture test machines

For monotonic SCB tests, Fig 8-6, the same machine which has been used to carry out the uniaxial tests, presented in Chapter 6, is used to perform these tests. The temperature controlled cabinet was used to keep the temperature at the desired level. Temperature monitoring was carried out by means of thermocouple situated just inside the crack tip. A dummy sample was used for this purpose. The applied load was displacement controlled, at 10 mm/min (Artamendi and Khalid 2007). The load-line displacement and the force were monitored and recorded as measured by the load

cell. The load was applied at the top of the specimen which was symmetrically supported by two rollers with a span, *2s*, of 122 mm.

Figure 8-6: Monotonic SCB test arrangements.

Cyclic SCB tests were carried out using the CRT-NU14 servo-pneumatic loading machine presented in Chapter 7, Fig 8-7. The machine applied a haversine cyclic load at selected constant amplitude of 1.5 kN and frequency of 1 Hz to the top of the SCB specimen, which was symmetrically supported by two rollers. The span, $2s$, was again 122 mm, which gave a span to diameter ratio of 0.8. The specimen and test frame were inside a temperature control cabinet with a double glazed door. The door contained a heating element to avoid condensation.

A load cell was used to measure the load amplitude, ΔP, the maximum load, P_{max}, and the minimum load, P_{min}, which is the contact load, during each loading cycle. The displacement measuring system consisted of two Kaman KD-2300 2S non-contact sensors. The sensors measured the crack mouth opening displacement (CMOD) and the vertical displacement, δ, at the mid-point of the specimen. These non-contact sensors use inductive, i.e. electromagnetic, technology to determine the position of a target relative to the sensor. They had a range of 0 to 5 mm and a resolution of 0.25 μm.

For this type of sensors, target material and dimensions are paramount for good performance. The main requirement for a target material is that it be conductive. In addition, non-magnetic materials are recommended. In this work, brass, i.e. non-magnetic, targets of 15 mm diameter and 0.8 mm thickness were used. Furthermore, holders glued to the specimens were used to position the sensors and targets, as seen in Fig 8-3. The two sensors measured both permanent and resilient displacements. The permanent, non-recoverable displacement was defined as that accumulated after each load cycle. The resilient displacement, on the other hand, was defined as the difference between the maximum and minimum displacements in one load cycle, i.e. peak-to-peak displacement (Artamendi and Khalid 2007).

Crack propagation was monitored by a Marlin F-080B digital camera fitted with a 35 mm lens and capable of operating at 20 fps. The camera was positioned on a tripod 0.5 m away from the specimen, as seen in Fig 8-7. A software program triggered the camera to take a frame as the vertical displacement, measured by the sensor, increased

by 0.1 mm. The same program copied, named and stored the photo files on the hard disc as they were taken. As the sensor range was 5 mm, the maximum number of photos taken during a single test was 50. It should be noted, however, that for some specimens failure occurred before this displacement level was reached.

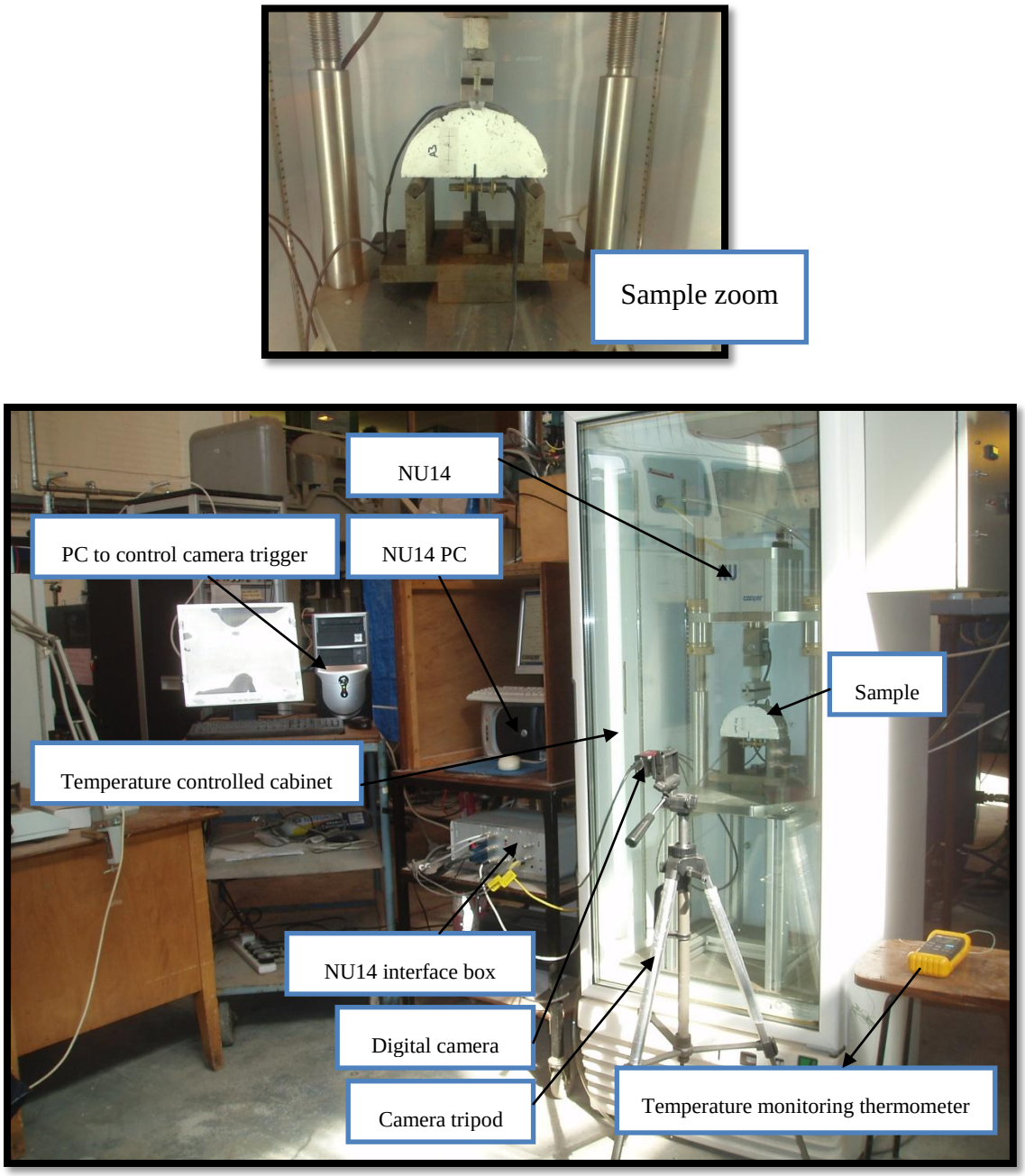


Figure 8-7: Cyclic SCB test arrangements.

An image analysis program was developed in-house to measure crack lengths. The procedure adopted in this software was based on the number of pixels from the start to the end of the crack following two methods: a straight line and an effective crack path, i.e. length along the actual crack path, as shown in Fig 8-8. To account for the position of the specimen relative to the camera, a 25 mm marker was stuck to the specimen. The specimens were painted in white using an emulsion-based paint to facilitate the location of the crack as explained earlier. The digital photographs were 1024×770 pixels in size.

Fig 8-8 shows a typical photograph of a cracked SCB specimen taken during a test. It can be seen that the crack started to propagate from the tip of the notch where the concentration of stresses was highest. The crack tended to propagate in the direction of the applied load, thus, perpendicular to the maximum principal tensile stress. Also shown on the figure are the two methods which can be used in analysis. The effective crack length method was adopted in this work to monitor crack growth due to its higher accuracy.

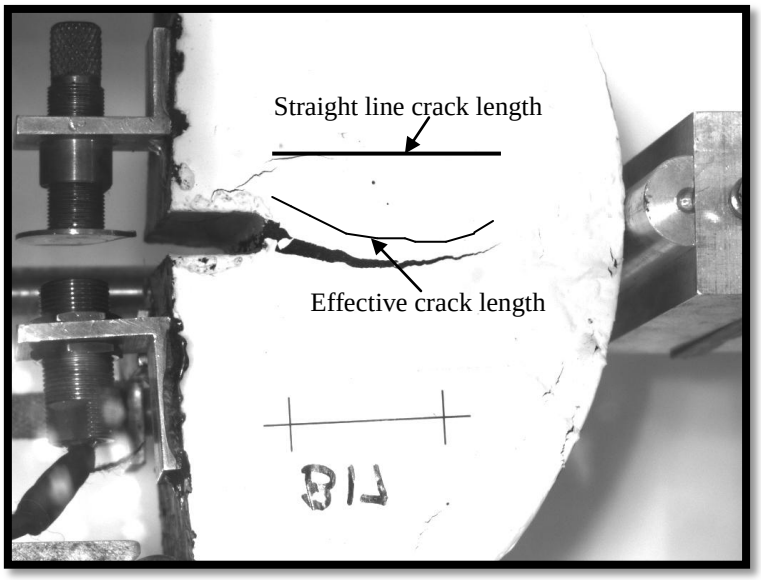


Figure 8-8: A typical cracked specimen during a SCB test.

8.6 SCB MONOTONIC MODE

The load deflection relationships of notched SCB samples corresponding to the tested four bituminous mixtures are presented in Fig 8-9. From this figure, it is noticed that the load deflection response was generally linear, independent of IBAA content level. This linear relationship turned into a non-linear response a short time before the maximum load was reached. This is because of formation of plastic zone ahead of the crack tip. This non-linear behaviour was dependant on the mix type, i.e. IBAA content level. Once the maximum load was reached, the crack propagated in a way dependent on mix type until the sample failed.

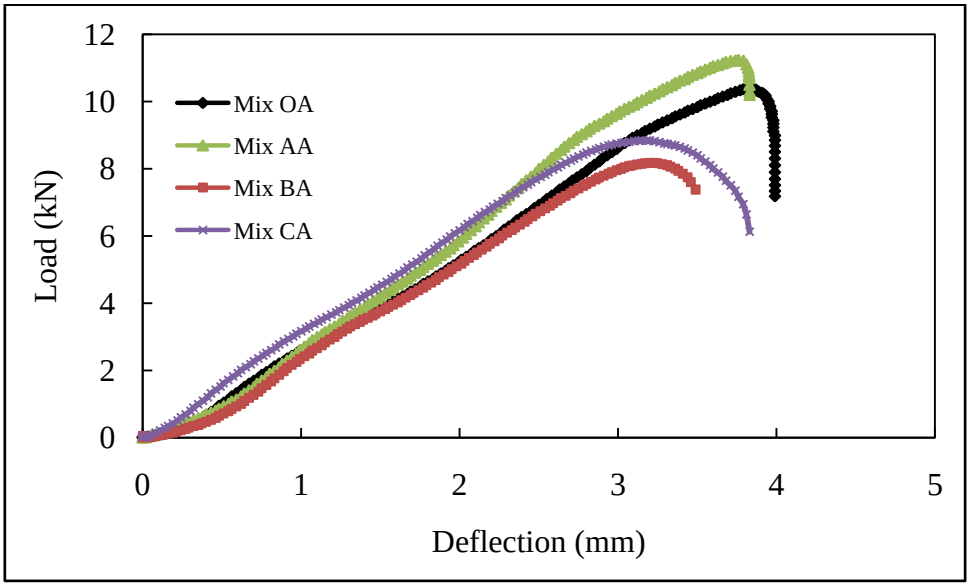


Figure 8-9: Load deflection curves of monotonic SCB tests at 5 ^{0}C.

It is obvious that the behaviour of mix AA, which contains 30% IBAA, is very similar in shape to the control mix, mix OA. This similarity is clear in the formation of the non-linear zone and in the crack propagation rate after the maximum load is reached. For both mixtures, the non-linear behaviour starts at relatively high deflection values. Moreover, after the maximum load was reached, cracks propagated very fast and failure occurred within such a short time that it may be considered as sudden failure. Taking into account the maximum load, which is linked to fracture strength, it is obvious that adding 30% IBAA to the mixture slightly enhanced its fracture strength. For mixtures BA and CA, which contain 60 and 80% IBAA respectively, the

formation of the non-linear zone was at a deflection values less than those of mixtures OA and AA. The non-linear behaviour starts at a relatively small deflection values. In addition, after the maximum load was reached, the crack propagated at a relatively slower rate compared to mixtures OA and AA as seen by the low decay of the curves. It is obvious that adding 60% IBAA modestly reduced the fracture strength of the bituminous mixtures. However, adding 80% IBAA resulted in a slight increase in this strength, compared with mix BA. This increase was not enough to reach the same strength achieved by either mix OA or mix AA.

Fracture toughness values have been calculated from load versus deflection curves, Fig 8-9. Critical load values, P_0, which are required to calculate the applied stress in Eq 8-3, were determined from these curves using a procedure given by BSI (2005), for metallic materials, and amended by Molenaar and Molenaar (2000) to consider the heterogeneity of asphalt. In this procedure, first, a straight line was fitted to the linear part of the load-deflection curve (line 1), as seen in Fig 8-10. Then, a second line with 10% reduced slope (line 2) was drawn from the origin. If the maximum load, falls between these two lines, then, P_0 is used as the maximum load. If not, P_0 is the intersection between line 2 and the load-deflection curve which was the case for the four mixtures in this work. Fig 8-10 shows a typical determination example for mix CA. The critical load values were converted into stresses before calculating the fracture toughness.

As the fracture energy appears to be a much better indicator for determining the resistance of the material to fracture than other indirect measures such as tensile strength (Wagenor et al 2005), it was adopted in this research. The fracture energy approach clearly distinguishes between the materials according to differences in mastic properties, whereas the indirect tensile strength was shown to greatly underestimate the tensile strength of highly ductile mixtures (Yin et al 2008). Fracture energy values were also calculated using Eq 8-4. The area under the load deflection curves was used to represent the fracture work where the ligament area was calculated as the product of sample thickness by the difference between sample radius and notch depth. Fracture energies were relatively small as expected because of the fact that at

low temperatures, the crack tended to propagate through both the aggregates and the mastic which results in not consuming too much energy for crack propagation (Wagoner et al 2005).

The fracture toughness and fracture energy values, for each mix, were compared to the control mix, OA, and the resulted ratios were presented in Table 8-1. Ratios, instead of values, were used to eradicate any difference in sample geometries and any overestimate occurred in fracture energy calculations. These overestimates are attributed to the fact that the calculation method included energy consumed in deformations at the supports and, to a lesser extent, at the point of load application, together with any viscous deformations, albeit very small, in the bulk material.

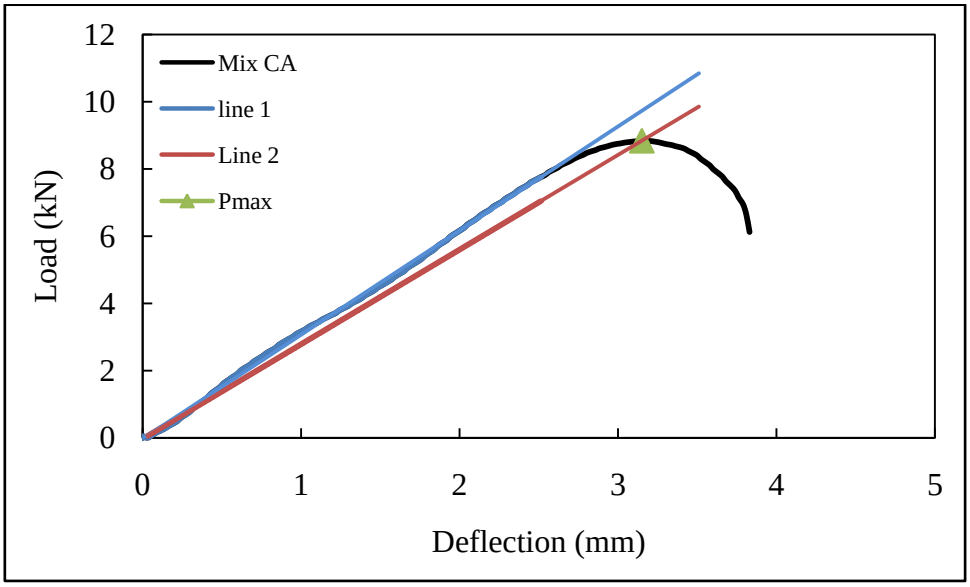


Figure 8-10: Determination of critical load value; Mix CA.

Material	Mix AA	Mix BA	Mix CA
Fracture toughness ratio	1.08	0.786	0.852
Fracture energy ratio	1.02	0.708	0.963

Table 8-1: Fracture toughness and fracture energy ratios

Generally, from the results shown in Fig 8-9 and Table 8-1, it can be said that adding 30% IBAA improved the fracture resistance of bituminous mixtures. This can be attributed to the increase in bitumen content and mixture stiffness which overcame the increase in voids content. Then adding more IBAA, i.e. 60%, resulted in a decrease in fracture strength due to the effect of the high voids content being larger than the effect of bitumen content and mixture stiffness increase. Adding 80% IBAA resulted in, for not entirely known reason, an increase in fracture strength, although lower than that of the control mix.

8.7 SCB CYCLIC MODE

8.7.1 CMOD and vertical displacement

In Figs 8-11 to 8-14, the permanent CMOD and vertical displacement were plotted against the corresponding number of cycles to failure. For mix OA, Fig 8-11, both values were, approximately, the same until the mid-way through the test, after which, the CMOD increased rapidly. For mix AA, the CMOD values were higher than those for the vertical displacement from the beginning of the test till the end, as seen in Fig 8-12. Mix BA, shown in Fig 8-13, exhibited a scenario close to that of mix AA. For mix CA, the vertical displacements were higher than the corresponding CMOD until the mid-way of the test after which the vice versa occurred.

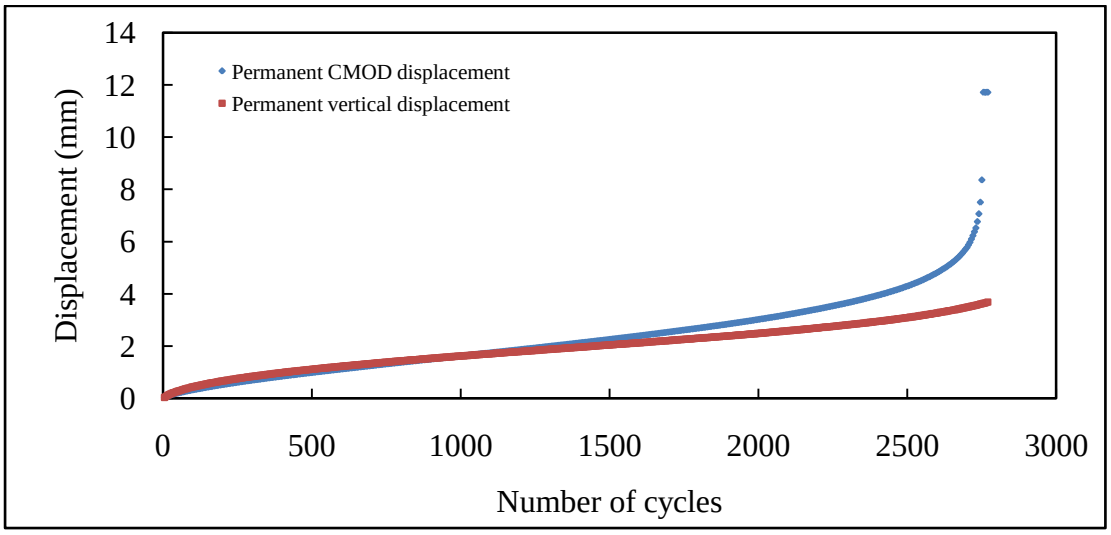


Figure 8-11: Permanent CMOD and vertical displacement for mix OA.

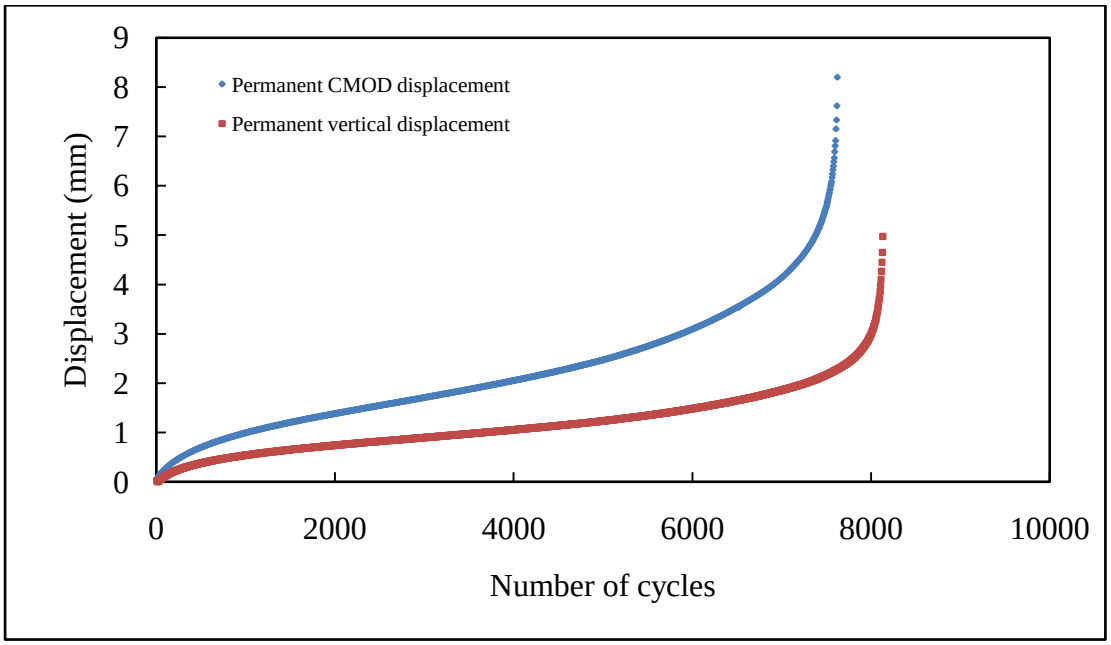


Figure 8-12: Permanent CMOD and vertical displacement for mix AA.

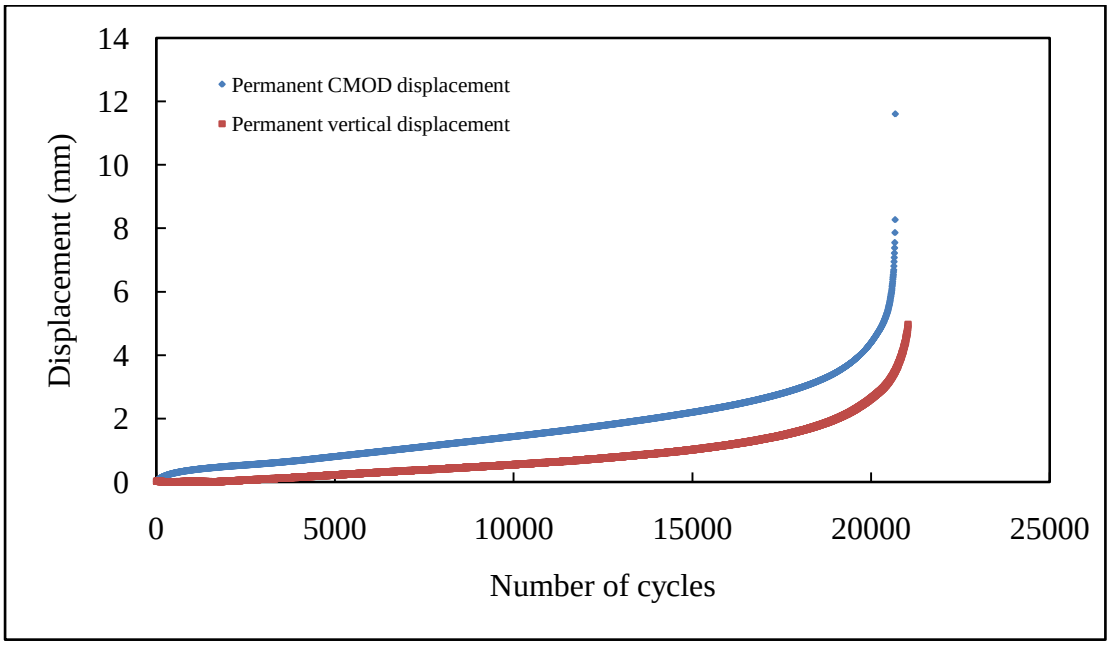


Figure 8-13: Permanent CMOD and vertical displacement for mix BA.

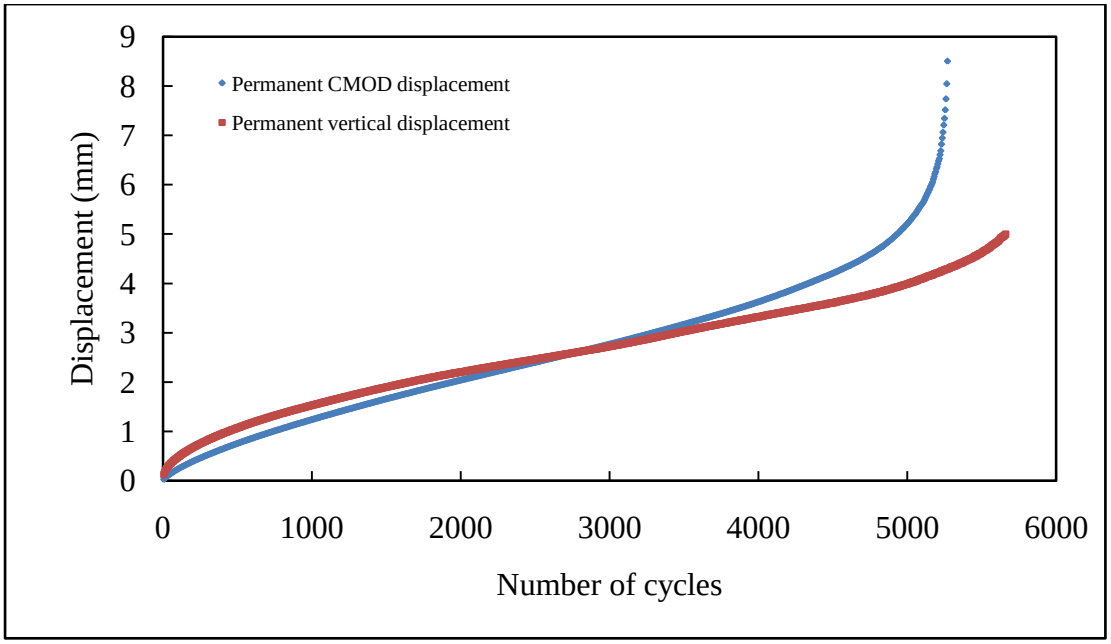


Figure 8-14: Permanent CMOD and vertical displacement for mix CA.

In Figs 8-15 to 8-18, the resilient CMOD and vertical displacement were plotted against the corresponding number of cycles to failure. For all mixes the resilient vertical displacements were higher than the corresponding CMOD values, however fairly stable, till close to failure. Then, the CMOD values increased dramatically till failure.

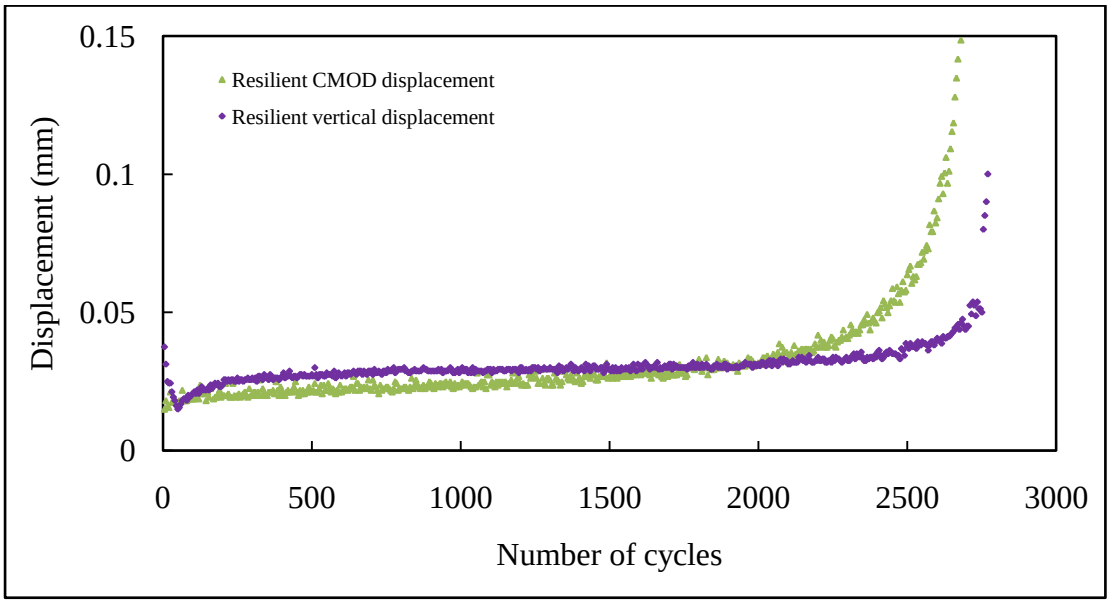


Figure 8-15: Resilient CMOD and vertical displacement for mix OA.

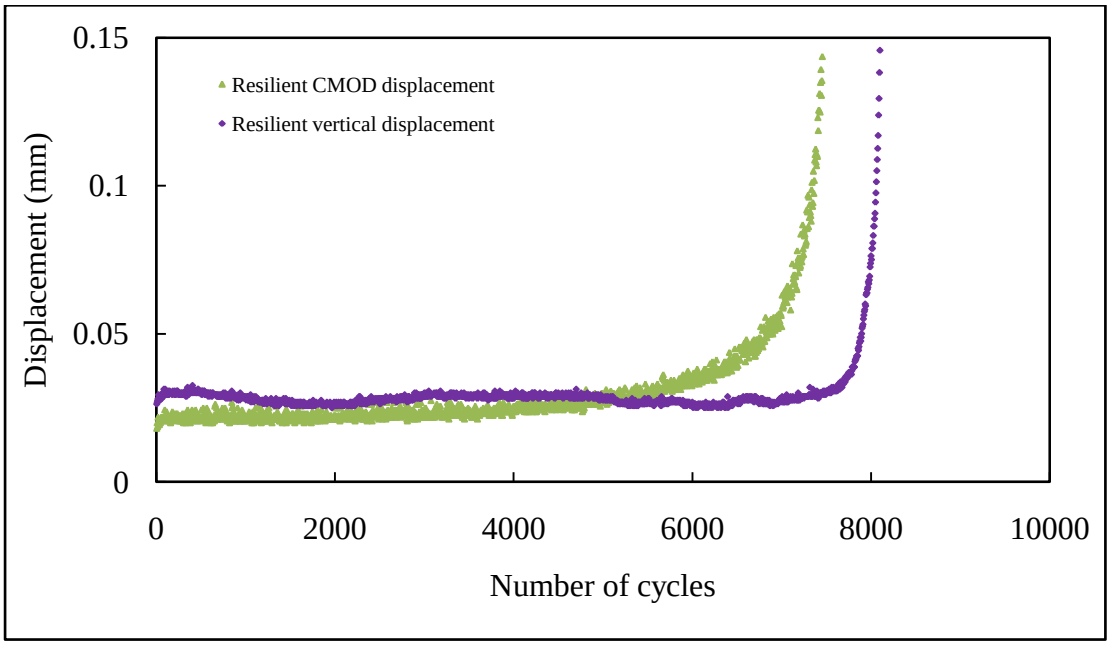


Figure 8-16: Resilient CMOD and vertical displacement for mix AA.

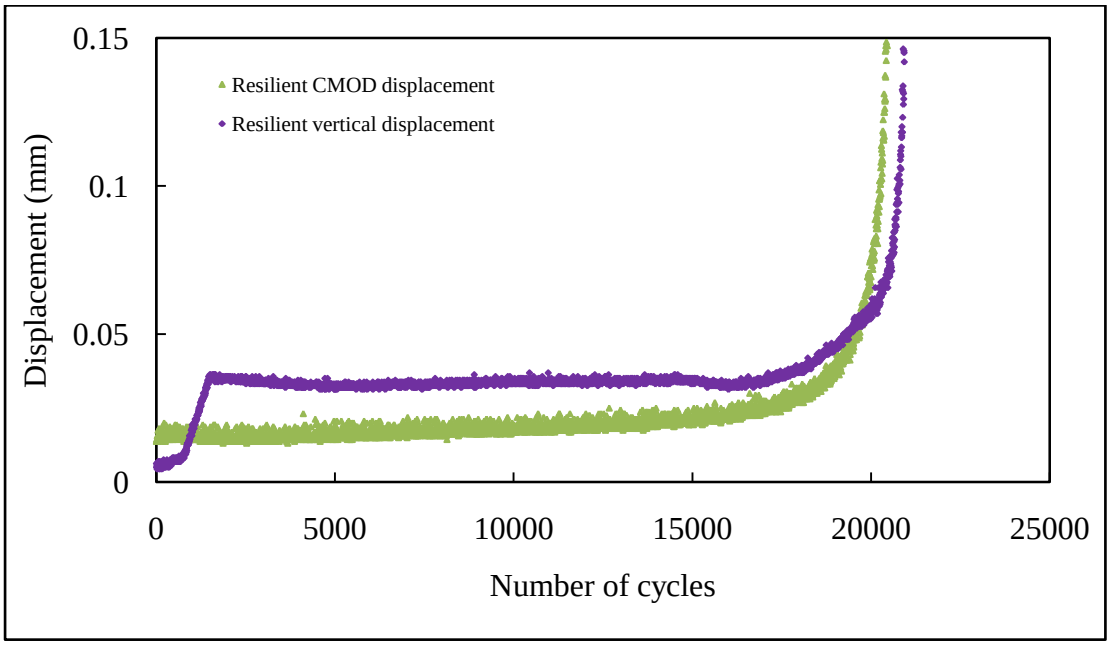


Figure 8-17: Resilient CMOD and vertical displacement for mix BA.

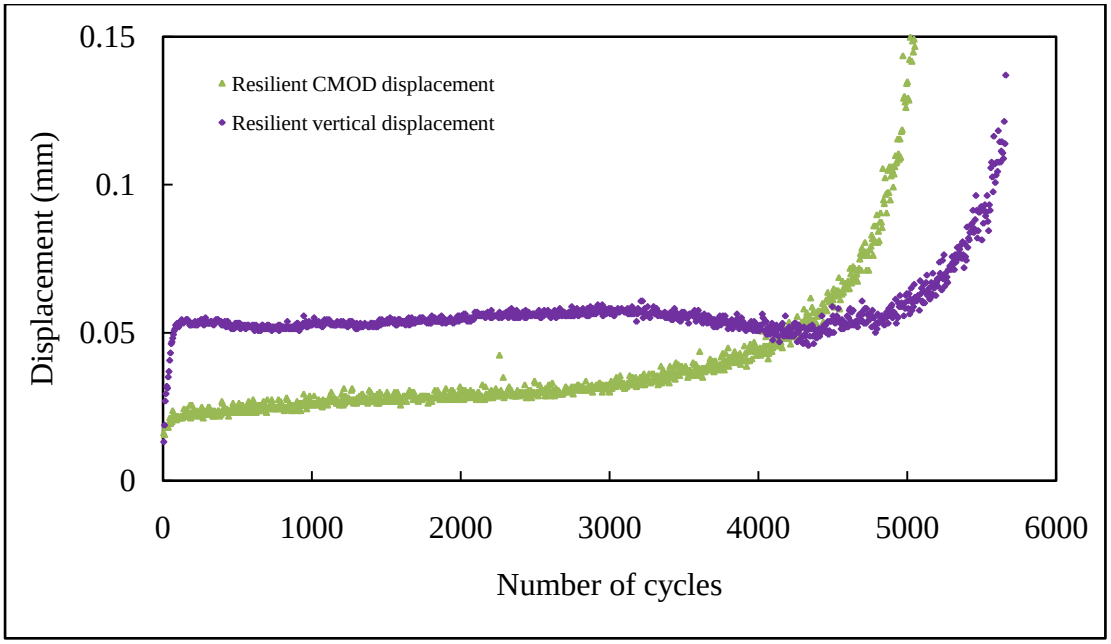


Figure 8-18: Resilient CMOD and vertical displacement for mix CA.

8.7.2 Crack length

Fig 8-19 shows the measured crack length against the number of cycles, for the four tested mixtures. It is indicative that each mix had a stable crack growth phase. This phase was relatively short for mix OA and increased with adding IBAA, for mixes AA and BA. Then it was shortened again for mix CA. Moreover, it is obvious that adding IBAA, up to 60% content level, led to a significant increase in the number of cycles up to failure. This means that adding IBAA led to an increase in the ductility of the mixture. This was not the case when 80% IBAA was used as mix CA exhibited a decrease in its ductility resulted in a reduction of the number of cycles to failure to be close to those of mix OA.

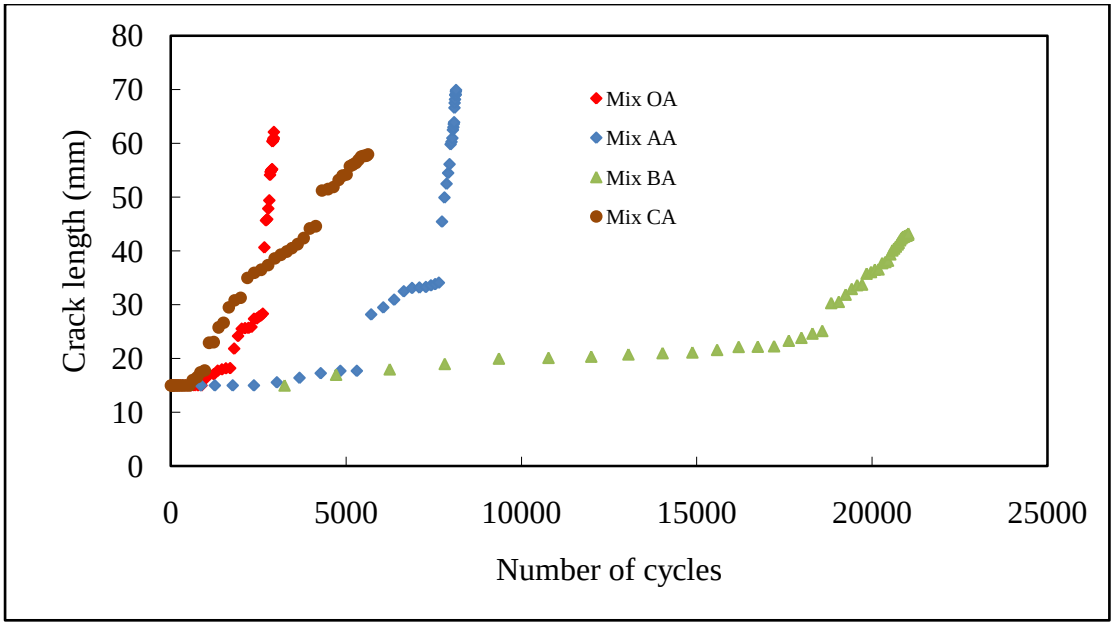


Figure 8-19: Crack length against number of cycles.

8.8 CRACK PROPAGATION MODELLING

8.8.1 Modelling using stress intensity factor

To study the effect of IBAA content level on Paris Law constants, the secant method (Wolfe 1959, Press et al 1992) was used to find out the Paris Law constants. In this method, a line is fitted to two adjacent points and the slope of the line is the crack growth rate, da/dN. Furthermore, the average crack length for the interval is used to calculate the corresponding ΔK_1 value, which is given by Eq 8-12. Although this method is very simple, it also exhibits the most scatter in da/dN values. Nevertheless, it is the accurate determination of the crack length and not the method to determine its rate that determines the scatter. The crack growth rate was plotted against ΔK_1 on a log-log scale and a straight line was fitted to the data to determine the Paris Law constants, A and n. Fig 8–20 show the relationship between the mode I stress intensity factor range, ΔK_1, and crack growth rate, da/dN, for the four mixtures used. From the relationships in these figures, the Paris Law constants, A and n, have been determined based on average values from duplicate tests and are presented in Table 8-2.

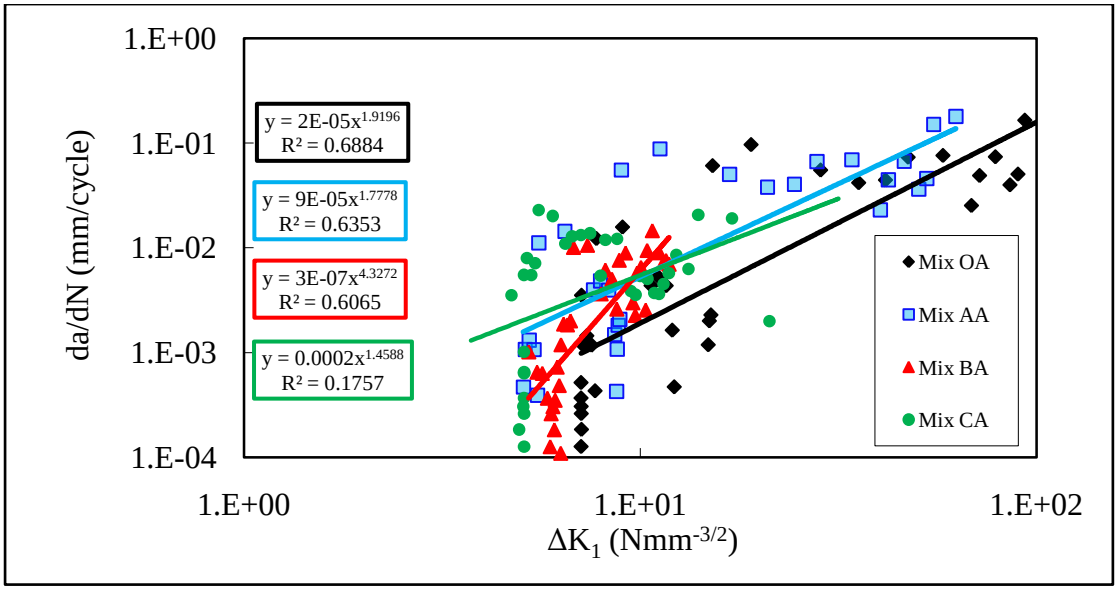


Figure 8-20: Relation between crack growth rate and stress intensity factor.

Mix	OA (no IBAA)	AA (30% IBAA)	BA (60% IBAA)	CA (80% IBAA)
A	2.0 E-5	9.0 E-5	3.0 E-7	2.0 E-3
n	1.9196	1.7778	4.3272	1.4588
R^2	0.6884	0.6353	0.6065	0.1757

Table 8-2: Paris Law constant using stress intensity factor

The observed scattering of the data might suggest that the difference in *n* values, which is known as the sensitivity of the stress intensity (Collop et al 2004) is insignificant for all mixtures expect for mix BA which had a relatively high *n* value. Although no Paris Law constants were found in the literature for IBAA bituminous mixtures, the *n* values, obtained in this study, are comparable with *n* values obtained for High Modulus Base mixtures obtained from Compact Tension tests conducted at 10–25 ^{0}C, which were reported as between 1.0 and 8.0 (Sewell et al 2000). Moreover, mixes OA, AA and BA show good correlation while mix CA shows a low fit correlation which shows the poor repeatability of this mix.

8.8.2 Modelling using J-Integral

Before finding Paris Law constants using J-Integral, few parameters were determined to be used in Eq 8-10. Firstly, the creep compliance for each mix was found using the results of uniaxial creep tests, undertaken at 5 ^{0}C, presented in Chapter 6. Then the modulus was calculated using the slope of the linear part in the load deflection curve obtained from the SCB monotonic tests. The energy Poison's ratios presented in Chapter 6 were used accompanied with modulus values to calculate K_1. Then, ΔJ was calculated in a way similar to the calculation procedure of ΔK_1. Results were presented in Fig 8-21 from which new Paris Law constants, A_J & m, were determined and presented in Table 8-3. These results did not show any significant change in the IBAA effect on crack propagation resistance of bituminous mixtures as all mixes kept, to a certain degree, the same behaviour trends they exhibited using the stress intensity factor. This may show the applicability of the J-Integral to IBAA bituminous mixtures and, moreover, support this recent technique which uses J-Integral instead of stress intensity factor in Paris Law. The advantage of using the J-Integral compared to the classical Paris law is that the J-Integral can take care of loading time and temperature dependencies of the bituminous mixtures as viscoelastic materials; however, were not covered in this study.

Comparing the Paris Law constants obtained using the stress intensity factor and J-integral, it is obvious that the using J-integral led to improved correlation for mix CA and worse correlation for mix AA. Mixes OA and BA did not show a significant change. These differences may be attributed to the personal interpretation for the crack growth path. Different interpretations lead to different correlations. Therefore, it is clear that there is a need to find out an accurate method to calculate crack lengths at different cycles.

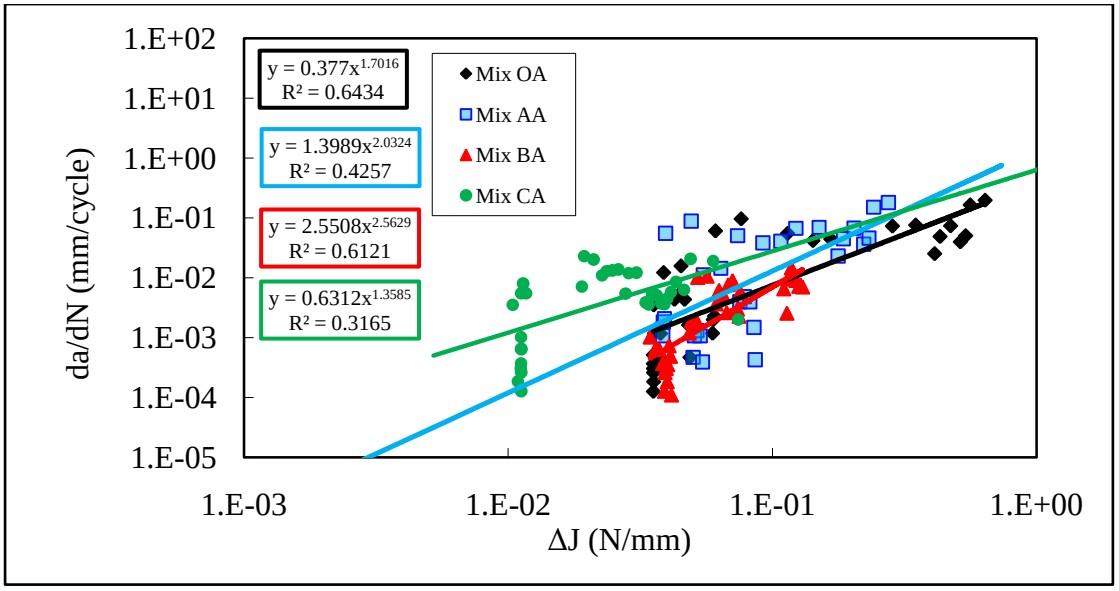


Figure 8- 21: Relation between crack growth rate and J-Integral.

Mix	OA (no IBAA)	AA (30% IBAA)	BA (60% IBAA)	CA (80% IBAA)
A_J	0.377	1.399	2.55	0.631
m	1.701	2.032	2.562	1.35
R^2	0.643	0.425	0.612	0.316

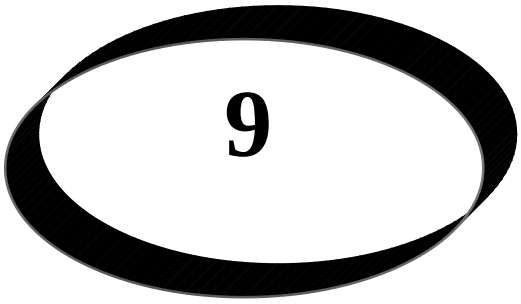
Table 8-3: Paris Law constant using J-Integral

8.9 CONCLUSIONS

The general findings from the preceding sections are:

- The cyclic semi-circular bending test, under monotonic and cyclic load conditions, adopted in this chapter was found suitable to study the resistance to crack growth of IBAA bituminous mixtures using linear fracture mechanics principles.
- Under monotonic load conditions, the load deflection response was generally linear, independent of IBAA content level.

- Under monotonic load conditions, adding 30% IBAA improved the fracture resistance of bituminous mixtures while adding 60% resulted in a decrease in fracture strength. Adding 80% IBAA resulted in a slight increase in fracture strength however lower than that of the control mix.
- In cyclic SCB tests, the crack length was measured using a digital camera and a simple technique which is called effective crack length.
- Under cyclic load conditions, adding IBAA, up to 60% content level, led to a significant increase in the number of cycles up to failure. This means that adding IBAA led to an increase in the ductility of the mixture. This was not the case when 80% IBAA was used as mix CA exhibited a decrease in its ductility resulted in decreasing the number of cycles up to failure to be close to those of mix OA.
- Paris Law was found suitable to characterise crack growth properties of IBAA bituminous mixtures using stress intensity factor or J-Integral.
- The difference in the sensitivity of the stress intensity was relatively insignificant for all mixtures expect for mix BA which had a relatively high n value.
- Paris Law constants were found to compile with corresponding results obtained for High Modulus Base mixtures.



CONCLUSIONS AND RECOMMENDATIONS

9.1 CONCLUSIONS

The main conclusion of this research is that IBAA can be incorporated, safely and successfully, in bituminous mixtures for flexible pavements. IBAA content level as high as 80%, by weight, was achieved. However, certain cautions were highlighted for such level. Using up to 60% IBAA in bituminous mixtures was found to be acceptable and improved most of the studied bituminous mixtures' properties. A summary for this research's findings is presented in Table 9-1. This table emphasises that using 30 and 60% IBAA led to a general improvement in bituminous mixtures' mechanical and environmental properties. The exceptions exhibited minor cutbacks and can be accepted. Using 80% IBAA, however led to similar improvements like the 30 and 60% IBAA, showed, for not entirely known reasons, hard to explain cutbacks in some properties comparing with 60% IBAA content.

Mix X compared to Mix Y **Mixture's properties**	**AA to OA**	**BA to OA**	**CA to OA**	**BA to AA**	**CA to BA**
Stiffness	Increased	Increased	Increased	Increased	decreased
Air voids	Increased	Increased	Increased	Increased	Increased
Optimum binder content	Increased	Increased	Increased	Increased	Increased
Water ingress resistance*	decreased	decreased	decreased	decreased	decreased
Ageing effect resistance	Increased	decreased	Increased	decreased	Increased
pH	N/A**	N/A	N/A	decreased	decreased
Leaching of Sulphate	N/A	N/A	N/A	Increased	Increased
Leaching of Chloride	N/A	N/A	N/A	Increased	Increased
Mastic resistance to permanent deformation	N/A	Increased	N/A	N/A	N/A
Resistance to permanent deformation at 5 ^{0}C	Increased	Increased	Increased	Increased	Increased
Resistance to permanent deformation at 20 ^{0}C	Increased	Increased	Increased	Increased	decreased
Resistance to permanent deformation at 40 ^{0}C	Increased	Increased	Increased	Increased	decreased
Fracture strength*	Increased	decreased	decreased	decreased	Increased
Crack growth resistance	Increased	Increased	Increased	Increased	decreased

*Insignificant changes ** N/A: not applicable

Table 9-1: Effect of IBAA content level on bituminous mixtures' properties

Generally, it can be said that adding IBAA to bituminous mixtures led to a general increase in the mixtures' resistance to permanent deformation and crack growth. Moreover, it is obvious that, in general, no risky environmental concerns were raised. The detailed conclusions related to the investigated mechanical and environmental properties of IBAA bituminous mixtures can be summarised in the following sections.

Against these positive effects of IBAA usage in bituminous mixtures there are two main negative points which should be taken into consideration. The first point is related to long term Sulphate leaching which needs to be accurately estimated and its effect on the surroundings should be studied before usage. The second point is an economic concern. Results of this research, supported by literature review, show that incorporating IBAA in bituminous mixtures led to an increase in binder demand. This increase results in an increase in mixture's cost. However, using IBAA led to a decrease in aggregates' cost and consequently mixture's cost. As a result, it is recommended to undertake a detailed comparison between any conventional bituminous mixture and IBAA's to find out the economic cost however, the environmental issues may be considered as well.

9.1.1 Mechanical properties of IBAA bituminous mixtures

From the surveyed literature, supported by the findings of this research, IBAA is considered as heterogeneous material which acts as lightweight aggregate. IBAA is considered as a suitable material to be used as natural aggregate replacement in asphalt.

From the results of mix design method adopted in this study, the following conclusions can be drawn:

- Mix design results show that mix AA, with 5.5% OBC and 7.4% voids content, is suitable for binder courses in flexible pavement according to the UK specifications, which allow up to 8% VIM.
- Using IBAA 60% in bituminous mixtures is not suitable for UK binder course in flexible pavements from voids viewpoint. However, it meets the other UK

requirements. Nonetheless, relatively high binder contents of 6.5% were required for adequate performance.

- The 80% IBAA mixture seemed to have a unique trend regarding the effect of binder on design parameters. Despite this, using 7.5% as optimum binder content seems to fulfil the mixture requirements.
- Addition of IBAA significantly improves the mixtures' ITSM values, excluding mix CA, at 80% IBAA.
- The control mix retained stiffness, after a water immersion regime, was found to be 70%. Using IBAA led to a reduction in the retained stiffness of the mixtures. However, this reduction was less than 10% compared with the control mix.
- IBAA mixtures' stiffness increased when subjected to an ageing protocol. This increase was significantly high at 60% IBAA, whereas the 0, 30 and 80% mixtures underwent insignificant changes to their stiffness values.

Rheology study of selected binders containing IBAA has shown that:

- The DSR creep mode can be used to predict binder steady state permanent deformation behaviour at temperatures up to 40 ^{0}C. The constant strain rate mode can be used for temperatures from 50 ^{0}C upwards.
- In the constant strain rate mode, the steady state stresses increased with strain rate and decreased with temperature.
- In the creep mode, the pure binder had higher strain rates than the mastics, at the same temperature.
- The strain rate for the pure bitumen was found to be more susceptible to a change in stress than the two mastics.
- Over a wide range of temperatures and stress levels, B3 has strain rates lower than B2. This means that the IBAA, in B3, has a stiffening effect on the binder leading to improved permanent deformation behaviour.
- IBAA was found to have no effect on binders' permanent deformation behaviour at temperatures > 50 ^{0}C.
- Machine compliance limits DSR coverage as DSR frequency mode could not be undertaken at low temperatures. Thus, other tools should be used in

addition to allow a wider temperature range in studying steady state permanent deformation of pure binders and mastics.

From studying the permanent deformation behaviour and crack resistance properties of IBAA bituminous mixtures, the following conclusions can be drawn:

- The monotonic uniaxial constant strain rate test is useful to study the steady state permanent deformation behaviour of bituminous mixtures containing IBAA at high temperatures. At low temperatures, the uniaxial creep test is more desirable. Both tests can be used at 20 ^{0}C. Both tests can be useful in determining the dilation onset point on top of the steady state stresses and steady state strain rates.
- For all mixtures, the slope of the linear region of the volumetric against shear strain plot, referred to as the dilation gradient, was found to be independent of stress level and dependent on IBAA content and test temperature. It was found that dilation gradient values at 5 and 20 ^{0}C are very close while these values increased at higher temperature.
- Poisson's ratio of the IBAA bituminous mixtures was determined using the plot of radial to axial strain ratio as a function of elapsed time in creep test. Poisson's ratio for the different mixtures was found to be within a range of 0.2 to 0.4.
- The steady state deformation behaviour of the IBAA bituminous mixtures was found to be well captured by the Modified Cross Model. The Modified Cross Model predicted that the steady state deformation behaviour of bituminous mixtures containing IBAA is linear at low stress levels while at high stress levels the mixtures exhibit non linear power law viscous behaviour. The IBAA mixtures' deformation behaviour was similar to their respective binders' with a stiffening factor resulting from the effect of aggregate matrix and ageing of bitumen during mixing. This stiffening factor was found to be dependent on temperature and IBAA content level.
- It was shown that IBAA content has a significant effect on mixtures' permanent deformation behaviour. At low temperatures, the higher the IBAA content the higher the deformation resistance was of the bituminous mixture. At high temperatures, the same effect was noticed except for mix CA.

- The monotonic triaxial constant strain rate test is useful to study the steady state permanent deformation behaviour of bituminous mixtures containing IBAA at high temperatures. At the same strain rate, the higher the confining pressure the higher the steady state stress was. However, the qualitative resulted deformation behaviour was similar to that noticed using uniaxial testing conditions.
- For all mixtures, the slope of the linear region of the volumetric against shear strain plot, referred to as the dilation gradient, was found to be independent of confining stress level and dependent on IBAA content. It was found that dilation gradient values did not change significantly comparing with those values obtained using the uniaxial tests.
- Under triaxial testing conditions, the steady state deformation behaviour of the IBAA bituminous mixtures was found to be well captured by the Modified Cross Model. The Modified Cross Model predicted that the steady state deformation behaviour of bituminous mixtures containing IBAA is linear at low stress levels while at high stress levels the mixtures exhibit non linear power law viscous behaviour. The IBAA mixtures' deformation behaviour was similar to their respective binders' with a stiffening factor resulting from the effect of aggregate matrix and ageing of bitumen during mixing. This stiffening factor was found to be dependent on IBAA content level and the confining pressure.
- It was shown that IBAA content has a significant effect on mixtures' permanent deformation behaviour. The higher the IBAA content the higher the deformation resistance was of the bituminous mixture except for mix CA.
- Under monotonic load conditions, adding 30% IBAA improved the fracture resistance of bituminous mixtures while adding 60% resulted in a decrease in fracture strength. Adding 80% IBAA resulted in a slight increase in fracture strength however lower than that of the control mix.
- Under cyclic load conditions, adding IBAA, up to 60% content level, led to a significant increase in the number of cycles up to failure. This means that adding IBAA led to an increase in the ductility of the mixture. This was not the case when 80% IBAA was used as mix CA exhibited a decrease in its ductility resulted in decreasing the number of cycles up to failure to be close to those of mix OA.

- Paris Law was found suitable to characterise crack growth properties of IBAA bituminous mixtures using stress intensity factor or J-Integral.
- The difference in the sensitivity of the stress intensity was relatively insignificant for all mixtures expect for mix BA which had a relatively high n value.
- Paris Law constants were found to compile with corresponding results obtained for High Modulus Base mixtures.

9.1.2 Environmental properties of IBAA bituminous mixtures

Work undertaken on the leaching potential of IBAA incorporated in bituminous mixtures draw the following conclusions:

- The pH and electrical conductivity values were found to decrease dramatically after coating IBAA with bitumen, whereas the sulphate was found to increase, with no known reason, in some mixtures.
- The time-dependent leaching behaviour showed that sulphate was found to be of the highest release from both the non bituminous blend and the bituminous mixture. Therefore, much attention has to be drawn to its effects on the environment if this high IBAA level bituminous mixture were to be adopted in any construction.
- The diffusion coefficient emphasised that the mass transfer of sulphate was relatively more mobile than the other detected constituents.
- A three-dimensional model can be used to predict the cumulative release from a binder course layer considering a worst case scenario.

9.2 RECOMMENDATIONS FOR FURTHER WORK

Visual observations during manufacture of IBAA bituminous mixtures showed clear coating problems. Some IBAA components e.g. ceramic and glass did not absorb bitumen during mixing. Furthermore, they did not accept well bitumen coating. This phenomenon had no significant effects on the overall performance of the developed mixtures. However, further work is needed to arrive at a mechanism which may improve the coating ability of IBAA aggregate.

Although the environmental study undertaken in this research concluded that IBAA in bituminous mixtures can be considered as safe for environment, concerns of sulphate leaching over a long time has been raised. This issue needs further investigations. Theoretical models of sulphate release from IBAA bituminous mixtures may help in understanding this behaviour.

Asphalt damage parameters may be obtained using fracture test outputs instead of traditional fatigue tests. However, this area needs more investigations. Undertaken fracture tests under different loading conditions, frequencies and temperatures may lead to better understanding of the applicability of fracture tests to study damage of asphalt.

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