The influence of hydrated lime on moisture susceptibility of asphalt mixtures

by

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ABSTRACT

Moisture damage is one of the major causes of premature failure of road pavements especially the pavements made using the flexible design concept. This failure occurs as a result of cohesion and adhesion loss between the binder and binder-aggregate interfaces respectively. One of the many ways of mitigating moisture damage effect is the use of anti-stripping agents like liquid polymers, Portland cement, hydrated lime, fly ash, flue dust, etc. Hydrated lime has been found to be one of the most efficient anti-stripping agents among all common additives to asphalt mixtures.

Although the majority of research on the use of hydrated lime in asphalt mixtures has been carried out in the USA, the beneficial effects of hydrated lime have also been reported worldwide especially in Europe. In the UK, the use of hydrated lime only started in the early 2000's and still needs a lot of research in terms of selection of aggregates which can be improved with the use of hydrated lime. Most of the aggregates used in the UK for asphalt mixtures are of good field performance and it is difficult to find aggregates with poor quality. That is the reason why for this research four aggregate types which are commonly used in the UK for flexible road payments are selected. One type of bitumen having penetration grade of 40/60 has been selected for the research. The four aggregate types include granite, limestone, basalt and greywacke.

This research focuses on an in-depth investigation of hydrated lime performance against moisture damage in bitumen mastics and asphalts mixtures as a whole. The full project has been broken down into three parts. The first part is a component level study, the second is mixture level study and the third is the study of practical adhesion. In the component level study, the effect of hydrated lime is quantified in terms of adhesion properties between a range of aggregates and binder combinations. For the component level testing, the effect of hydrated lime is quantified by adding it to the bitumen to make a mastic. The test techniques which are used for the component level assessment includes rolling bottle test and surface energy measurement. The second part focuses on the performance of hydrated lime as a whole inside asphalt mixture as filler replacement. The idea behind this methodology is to evaluate the real effect of hydrated lime in the mixture because if hydrated lime is used as additional filler in the mixture it will alter the mixture volumetric rather than simply affecting the mechanical response of the mixture through the properties of the hydrated lime. SATS test has been used

to quantify the effect of hydrated lime against moisture damage at mixture level. The third part of the research deals with the measurement of practical adhesion with and without moisture conditioning with the help of pull-off and peel-off test techniques called PATTI and Peel test.

The effect of hydrated lime either in the mastic or in the mixture has been found to be aggregate type dependent. Granite aggregates showed a good improvement in the performance against moisture damage resistance with the use of hydrated lime. Limestone aggregate didn't respond to the addition of hydrated lime in the light of most of the techniques considered in this research and the same applied to the basalt aggregates. Greywacke, on the other hand, responded well and showed an improved moisture damage performance with the use of hydrated lime. Another good thing observed in the results was the consistency between the results among the different test techniques. The results obtained in each technique are in line with each other and give the same conclusion for most of the combinations studied in this research.

To conclude, the effect of hydrated lime highly depends on the type of aggregate, its origin and its mineral composition. The aggregates used in this study were either of moderate or good field performance. Although a good improvement in the moisture damage performance of some combinations was clearly observed, it is highly recommended to incorporate the aggregates having bad field performance to see how hydrated lime improves their performance.

Keywords: Moisture Damage, Hydrated lime, Surface Energy, Adhesion, Asphalt Mixture, Rolling Bottles Test (RBT), Saturation Ageing Tensile Stiffness (SATS) Test, PATTI test, Peel test.

DEDICATION

To the prophet Muhammad (Peace and blessing be upon him), my parents, my wife's parents, my wife, my children, my teachers and my friends

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Chapter 1: Introduction

1.1 Background

The majority of roads in the world today are made using the flexible design concept, as the U.S. Department of Transport reported, 94% of U.S. paved roads are flexible in terms of design considerations (FHWA 2012). Asphalt mixture loses its strength and stiffness as a result of moisture damage (Ahmad and Airey 2010a; Ahmad et al. 2011). This moisture damage propagates further through two main mechanisms i.e. loss of adhesion between aggregates and bitumen called stripping and, secondly, loss of cohesion within the bitumen itself called softening (Ahmad and Airey 2010c; Ahmad and Airey 2010d; Bhasin 2007; Li 1997; Little and Bhasin 2006). The reason behind an early road failure may not be directly moisture, but it may indirectly affect the performance as the severity of already existing distresses like potholes, cracking, rutting and patches may increase in the presence of moisture (Bhasin 2007; Miller and Bellinger 2003). The total road length in the UK has been estimated to be about 246 thousand miles (Department of Transport 2015). For the year 2016/2017, local authorities in England and Wales spent a budget of £1.66 billion for road maintenance for this huge infrastructure, which was about 10% more compared to the previous years (ALARM 2013).

One of the most popular, useful and cost-effective ways used by transportation agencies to mitigate moisture damage is through the use of various additives and modifiers. These additives or modifiers can be found naturally or can be manufactured by chemical processing and can be added to either bitumen or aggregates through different techniques.

Anti-stripping agents like liquid polymers, portland cement, hydrated lime, fly ash and flue dust are usually used for moisture damage mitigation and among all, hydrated lime is the most common (Xiao et al. 2010). Hydrated lime has been known as an additive to asphalt mixture and gained a strong interest in the 1970s in the USA due to a drop in the quality of the bitumen and the petroleum crisis (Hicks 1991; Kandhal and Rickards 2001). Due to its good performance against distress from to moisture damage and frost, hydrated lime is now estimated to be used in about 10% of the asphalt mixtures produced in USA (Hicks and Scholz 2003).

The addition of hydrated lime (HL) to hot mix asphalt (HMA) has been reported to improve field performance as it resists moisture damage, reduces oxidative ageing, improves the

mechanical properties, decreases the rate and level of microcrack induced damage, and improves resistance to fatigue and rutting (Little and Petersen 2005). For optimum mixture performance, an addition of 1% - 2% HL by weight has been recommended depending on the distress type (Aragão et al. 2010; Gorkem and Sengoz 2009; Nejad et al. 2013).

Although the majority of research on the use of HL in asphalt mixtures has been carried out in the USA, the beneficial effects of HL have been reported worldwide especially in Europe (Caro et al. 2008; Lesuesur 2010; Voskuilen and Verhoef 2003). In UK, the use of HL has only started in the early 2000s and it still needs a lot of research in terms of selection of aggregates which can be improved with the addition of HL. Most of the aggregates used in UK for asphalt mixtures are of good quality and it is difficult to find aggregates of poor quality. That is the reason why for this research four aggregate types which are commonly used in UK for flexible road payments were selected for the moisture damage assessment with the addition of hydrated lime.

1.2 Problem statement

The use of hydrated lime (HL) in asphalt mixtures has been found to be very beneficial for moisture related damages. This statement is claimed to be true for both siliceous aggregates and limestone aggregates (Abo-Qudais and Al-Shweily 2007; Bagampadde et al. 2004; Blazek et al. 2000). The question is, whether HL only improves the performance of poor performing aggregates or it can be used to enhance the performance of good performing aggregates as well. In UK the use of HL has only started in the early 2000's (Caro et al. 2008) and there is a huge potential of research in this area. In one of the studies on asphalt mixture made by Airey et al. (2008), there was a significant improvement recorded with the addition of HL in one of the acidic aggregate. To go further and make a comprehensive understanding of the performance of different aggregate types commonly used in UK a study was needed. That is the reason why this study focuses on an in-depth investigation to understand the phenomena associated with the use of hydrated lime. Four commonly used aggregates have been selected along with one bitumen type for this study. It was not possible to get an extremely poor performing aggregate in UK so all the aggregates are of either moderate or good performance.

The effect of hydrated lime can be quantified by either adding it to the bitumen to make a mastic or by adding it as a filler replacement in mixtures. Hydrated lime can behave very differently with different aggregate types so its effect can be quantified with the help of various

techniques at the component level, at mixture level and also by using some practical adhesion measurement techniques. Based on the collective results from all the test techniques recommendations can be made if HL is beneficial for a certain type of aggregate bitumen combination and hence an optimum combination of aggregate, bitumen and filler can be proposed to get the best field performance against moisture related damages.

1.3 Aim and objectives

The overall aim of the study is to evaluate the effect of hydrated lime addition on moisture damage performance of asphalt mixtures using different sources of aggregates in the UK. The following is the list of objectives to achieve the specific aim.

1. Moisture damage assessment in the presence of HL on a component level using surface free energy technique and rolling bottles test.

2. Evaluation of HL performance at mixture level for aggregate types used and their combinations using SATS technique.

3. Study of adhesion under dry and wet conditions using practical adhesion measurement techniques i.e. (PATTI test and Peel test)

4. Comparison between all the studied test techniques to reach a conclusion if HL is beneficial for a specific aggregate type and suggestions for HL content giving better performance.

1.4 Methodology

Task 1

To measure the cohesive and adhesive bond strength in two materials, surface free energy measurement has proven to be a good tool. Moisture damage effects on the bond strength of aggregate-bitumen system can effectively be studied using this thermodynamic technique. Dynamic Vapour Sorption (DVS) technique has been used to calculate the surface energy of the aggregates. Whereas the dynamic contact angle (DCA) technique (Wilhelmy plate device) is employed for surface free energy assessment of bitumen. Many empirical testing techniques

on loose coated aggregates are available which give an indication of the adhesion potential between aggregate and bitumen. Among all the available techniques the rolling bottle test (RBT) is one of the most reliable techniques. So the idea was to employ this technique and compare it with the surface energy results to get a better understanding of the adhesion properties between aggregate and bitumen under moisture conditions (Objective 1).

Task 2

To evaluate the mixture behaviour as a whole against moisture damage, modified saturation ageing tensile stiffness (SATS) protocols are used. Different combinations of aggregate, bitumen and filler are tested to get an in-depth understanding of the influence of aggregate and filler type on the moisture damage performance of asphalt mixtures and also to figure out the effect of hydrated lime with the commonly used aggregates in the UK (Objective 2).

Task 3

Various techniques are used by researchers to get an idea about adhesion between two materials. These techniques can also be considered to measure bitumen-aggregate adhesion effectively. Two pull off test techniques, namely the Peel test (ASTM D1876, D3330) and the Pneumatic Adhesion Tensile Testing Instrument (PATTI) test, have been used to get adhesive properties of the two materials with and without water conditioning (Objective 3).

Task 4

A comparison between all the studies test techniques has been made for each aggregate type separately so as to check if the results from all the techniques are giving the same conclusion. Based on the results for all the combinations for each aggregate type, a HL content having better performance has been identified.

1.5 Thesis outline

This thesis has been divided into eight chapters. A brief summary of what each chapter includes has been discussed as follows;

Chapter 1

This chapter gives a brief background of the moisture damage problem in the asphalt mixture and addresses the mitigation measures to safeguard the road structure against premature failure. It also includes the problem statement, research objectives and also briefly discusses the methodology to achieve these objectives. In the end, thesis outline has been summarised.

Chapter 2

This chapter summarises the relevant literature review on sources of moisture in the road pavements and its effect on the performance. Details of different antistripping agents from the previous literature have been summarised and the beneficial effects of hydrated lime addition are highlighted. This chapter also talks about techniques for moisture damage assessment at component and mixture levels and also includes some of the techniques to access the bond strength between the bitumen/mastic and the aggregates. Theory of surface free energy has been discussed in detail to demonstrate how it can be used effectively for the moisture damage assessment in the asphalt mixture.

Chapter 3

This chapter covers the details of the material to be used for the study along with their sources and properties. It summarises the experimental programme for the study along with brief procedure of the selected test techniques to be used for this study.

Chapter 4

Results from the component level testing have been summarised in this chapter. These include rolling bottle test (RBT) results and surface free energy (SFE) results. Image analysis has been used to access the coating of the bitumen/mastic after specified periods of time and comparisons have been made between different combinations. Using the surface free energy of the aggregates and the bitumen/mastics, values for work of adhesion and work of de-bonding have been calculated to check the bond strength and the moisture susceptibility of the aggregates and bitumen/mastic combinations.

Chapter 5

To quantify the performance of aggregates and bitumen at the mixture level and to evaluate the effect of HL replacement of the mineral filler of the respective aggregates, the SATS test has been used and the results are presented for the four types of aggregates with three different filler combinations. The performance of the combinations with and without HL have been assessed based on the retained stiffness value after SATS conditioning.

Chapter 6

Two techniques have been used to evaluate the adhesion between different aggregates and bitumen/mastic combinations. These include Pneumatic Adhesion Tensile Testing Instrument (PATTI) test and Peel test. Again various combinations have been used and the testing is carried out after 0 days and 7 days' water conditioning. At the end again a comparative study has been carried out to quantify the beneficial effect of HL in the mastics.

Chapter 7

Comparison between all the test techniques has been made to check if all the techniques are on relevant and are consistent with each other.

Chapter 8

This chapter summarises the conclusions from the research study along with some recommendations for the work to be carried out in future.

Chapter 2: Literature Review

2.1 Introduction

Asphalt mixtures are widely used as a road pavement material across the globe. A durable mixture is required to sustain heavy loads form the anticipated traffic and also to withstand extreme weather conditions. The road pavements are designed to last for a certain period of time but sometimes premature failure occurs due to different distress types, which the pavement undergoes during its lifespan. One of the major distress among all is moisture damage.

Loss of strength, stiffness and durability of asphalt mixture in the presence of water is known as moisture damage. Performance of the asphalt mixtures is affected by the moisture damage as a result of cohesion and adhesion loss between binder itself and binder-aggregate interfaces respectively. Thus, in both cases damage to asphalt mixture occurs, giving a reduced stiffness moduli (Airey et al. 2008; Apeagyei et al. 2013; Collop et al. 2007; Qiao et al. 2013). As soon as the water comes in contact with the asphalt mixture it can enter into bitumen-aggregate interface through diffusion mechanism across bitumen film, can seep through the voids and cracks in the bitumen film if any or can enter directly through the partially coated aggregate surface (Stuart 1990). Another thing which could happen with the interaction of water with the asphalt mixture is the softening of bitumen film without a deboning between aggregate and bitumen. This loss in stiffness can be reversible with the removal of the moisture but there is one issue which is the application of load under this low stiffness. When the load is applied under low stiffness conditions as discussed earlier pavement deteriorate at an accelerated rate and the mechanism can become irreversible (Santucci 2002). Moisture can also indirectly damage the road pavements due to the fact that existing distresses including cracking, rutting and potholes deteriorate the pavement at an accelerated rate in its presence (Grenfell et al. 2014).

Asphalt mixture is made up of mainly two constituents i.e. bitumen and aggregates. So the properties of these constituents including aggregate mineralogy, aggregate surface texture, aggregate water absorption, bitumen chemistry and the compatibility of the two materials in terms of surface energy is of prime importance (Abo-Qudais and Al-Shweily 2007; Grenfell et al. 2014). Other factors such as void connectivity in mixture, volumetric properties of mixture

and ambient conditions also play an important role in deciding the susceptibility of mixture against moisture (Grenfell et al. 2012; Khan et al. 2013)

Now for the solution to this problem of moisture damage, various methods have been proposed. One of the most popular, useful and cost-effective way used by transportation agencies to mitigate the moisture damage is through the use of various additives and modifiers. These additives are either produced chemically or can be found naturally. Among all the additives and modifiers which are available today HL is one of the most popular in the market and is widely used against moisture damage.

To get an insight of the phenomenon associated with the moisture damage and how HL can be used to tackle the moisture damage problem, relevant literature has been summarised in this chapter. This includes literature about the sources of moisture, factors affecting moisture susceptibility, detailed mechanism of moisture damage, factors affecting moisture sensitivity, moisture mitigation measures, HL properties, effect of HL on mixture properties, effect of HL on bitumen and aggregates individually and ways to add hydrated lime with its optimum content.

2.2 Sources of moisture

Moisture sources in asphalt pavements may be broadly classified into two types; internal source and external source (Figure 2:1). As far as internal source is concerned, moisture may remain in asphalt mixture in case of failure to use fully dried aggregates (Santucci 2002). During manufacturing of asphalt mixture this residual moisture may come into contact with hot bitumen hence converting to steam and resulting in bitumen foaming (Read and Whiteoak 2003).

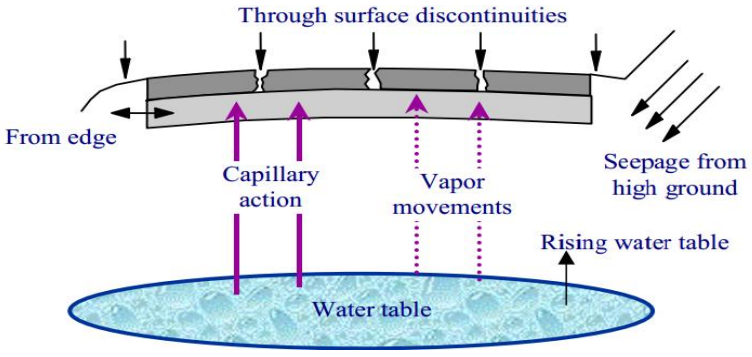


Figure 2:1: Sources of moisture in road pavements (NCHRP 2004)

External sources of moisture in asphalt pavement may include upward movement of water through capillary action or vapours in case of a high water table, lateral movement of water through pavement edges and surface infiltration due to rain fall as shown in the Figure 2:1 About 40% of the rainfall has been reported to enter into the pavement in some studies (AASHTO 1993; FHWA 1994)

2.3 Moisture damage types

There are two main types of moisture damage. First is the damage caused by the moisture directly and second is the indirect damage. The damages caused by the moisture directly includes stripping, ravelling, corrugation and water pumping. Among all of them stripping is most common which is defined as the adhesive failure between bitumen and aggregates due to the entrance of water either through bitumen or through aggregates.



Fatigue Cracking



Stripping



Pothole



Rutting

Figure 2:2: Common types of pavement distress

The strength of pavement is significantly reduced after the development of this distress. If stripping occurs in isolated spots throughout the pavement it can quickly convert into potholes. Indirect distresses are the one which are not directly caused by moisture damage but their severity is increased in the presence of moisture. These include Potholes, fatigue cracking and rutting. Some of the common failure types of asphalt pavement distresses are shown in Figure 2:2.

2.4 Mechanisms associated with moisture damage

Moisture damage is not a single phenomenon, there are several phenomena which are associated with it. As soon as the water comes in contact with asphalt mixture, mixture deterioration can start with one or more phenomena including detachment, displacement, spontaneous emulsion, pore pressure, and hydraulic scour (Grenfell et al. 2014). Each of the phenomenon listed above has been explained in details in the coming sections.

2.4.1 Detachment

Separation of bitumen film from the surface of aggregates by a thin water film without breaking the bitumen film is called detachment (Caro et al. 2008). This phenomenon can be explained with reference to different aspects. First of all, both aggregates and bitumen are different in nature, aggregates are hydrophilic and the bitumen is hydrophobic. So the attraction between the bitumen and aggregates is weaker than the attraction between water and aggregates and as soon as the water comes in contact with the aggregate it separates the two surfaces and phenomenon of detachment occurs. Secondly, the bitumen is mostly low polar material so the bond between the aggregates and bitumen is of weak nonpolar nature. But if we look at the nature of water, it is highly polar in nature and can form a strong bond with the aggregates in comparison to bitumen and results in detachment of the bitumen layer. Research has shown that the surface energy of water is less than the surface free energy of the bitumen and hence bitumen's wettability over aggregates is less compared to water (Road Research Laboratory 1962). Also when water reaches to bitumen and aggregate interface it can reduce the free energy of the system by replacing the bitumen layer on the surface of the aggregate leaving a more stable thermodynamic conditions.

2.4.2 Displacement

Displacement is the next stage to detachment and can be defined as the deterioration to the detached bitumen film. When water reaches the interface between bitumen and aggregate it detaches the bitumen film and the bond between the aggregates and bitumen film weakens. The application of load to this weak bond breaks the bitumen film and hence displacement occurs. There is another aspect in which the phenomenon of displacement can be explained. When water reaches the bitumen aggregates interface its PH value changes due to the mineral composition of aggregates, which changes the type of polar groups adsorbed and as a result aggregate and bitumen surfaces are coated with a double negative charge. The system becomes unstable and needs more water and which eventually breaks the weak bitumen layer and results in displacement (Hicks et al. 2003).

2.4.3 Emulsification

The degradation of bitumen film in the presence of water with the reduction of its hardness is called Emulsification. This process of emulsification accelerates in the presence of clay partials in the aggregates or additives in asphalt played as emulsifiers. This is a very slow process and occurs only if the mixture is submerged in water for a very long time. The rate of emulsification also depends on the nature of the bitumen and the additive types used in the mixture (Fromm 1974). In contrast to detachment and displacement, the failure type caused by the spontaneous emulsification is cohesive rather than adhesive. The reason behind this cohesive failure is the change in physico-chemical properties of bitumen which cuts down its cohesive strength. The process of spontaneous emulsification is difficult to detect in the field as the loss of bituminous coating can't be observed (St Martin et al. 2003). Apeagyei et al. (2014) found that this process is reversible and 80% of the stiffness can be recovered if water is fully drained out of the system. Also, the cohesive failure shows a more durable asphalt mixture to moisture exposure in comparison to adhesive failure.

2.4.4 Pore Pressure

In the presence of moisture, pressure can build up in the pores of asphalt pavements with the application of traffic load. This pressure continuously changes from compression to tension

and vice versa with the application of moving load and could lead eventually in de-bonding of the bitumen from the surface of aggregates in the form of revelling. Same effect is produced with the freezing of water in the pores where with the expansion of water serious damages occurs in the asphalt pavement (Asphalt Institute 2007)

A properly designed asphalt pavement tends to strain harden under the anticipated repeated loading and hence locks the aggregate matrix caused by densification. In this way, it will be difficult for the water to enter into the asphalt mixture and the chances of developing pour water pressure will be reduced (Bhairampally et al. 2000). On contrary to that if the pavement is not designed properly, microcracks will be developed with the moving load and will result in a progressive adhesion and/or cohesion loss. In the presence of water, this failure will be accelerated with the development of pore pressure in the microcracks.

2.4.5 Hydraulic scour

The washing out of the exposed pavement layer from the surface with the water flow is called hydraulic scour (Kringos and Scarpas 2005).When pavement saturation level is high and water remain trapped for a longer period time, stripping occurs. Fromm (1974) has suggested Osmosis and pull-back as two possible mechanisms of hydraulic sour. If the diffusion of water through asphalt mixture is considerable then there are chances that the pavement will hold a lot of water which could result in hydraulic scour (Cheng et al. 2001). In addition, the presence of salts and salt solutions in aggregate pores causes an osmotic pressure gradient to suck water through the bitumen film. As a result of this water in the asphalt mixture, it loses its cohesive and adhesive strength and will eventually lead to complete stripping of the bitumen from the surface of aggregates due to the phenomenon of hydraulic scour. This damage type is referred as mechanical which occurs after detachment / spontaneous emulsification. The amount of damage is dependent on the quantity of water held by the asphalt mixture.

2.5 Factors affecting moisture susceptibility

There are various factors affecting the moisture susceptibility of the asphalt mixture. These include;

- Aggregate properties
- Properties of Bitumen

- Interaction between bitumen and aggregates
- Volumetric properties of mixture
- Filler type

2.5.1 Aggregate properties

Asphalt mixture is composed of about 95% aggregate content that is the reason why aggregate properties are very important. Bitumen content in the asphalt mixtures is influenced by the properties of aggregates such as surface texture, shape and gradation. Bitumen content is directly related to the film thickness on the surface of aggregates. A thicker film is more resistant to moisture in comparison to thinner film (Santucci 2002).

Mineralogical properties of the aggregates are very important for a durable and moisture-resistant asphalt mixture. Their properties depend on their source and the source of rock from which they are produced. Different types of aggregates are used for the construction of asphalt pavements in the world. Aggregates may range from acidic to basic nature depending on their source (Asphalt Institute 2007). Now if we look at the nature of bitumen it is mostly acidic in nature. So the aggregate having basic nature is expected to make a stronger bond with the acidic natured bitumen. That is the reason why moisture susceptibility of the asphalt mixture containing acidic aggregates has been found to be higher than in the mixture containing basic aggregates (Collop et al. 2007). Experiments performed on asphalt mixtures with acidic aggregates have shown a declining trend in relative stiffness with increase in retained saturation level. On the other hand, asphalt mixture made with basic aggregates has been found to be very stable under the same conditions as shown in Figure 2:3 (Khan et al. 2013). Based on these facts it can be concluded that aggregate having basic nature performs better as compared to the acidic aggregate in a moisture susceptible environment.

Another factor which is worth considering while deciding the aggregate for asphalt mixture is the presence of clay particles. It can either be in the fine or can be in the form of a coating on the coarse aggregates. When water comes in contact with the clay particles they expand and weakens the bond between the aggregates and bitumen.

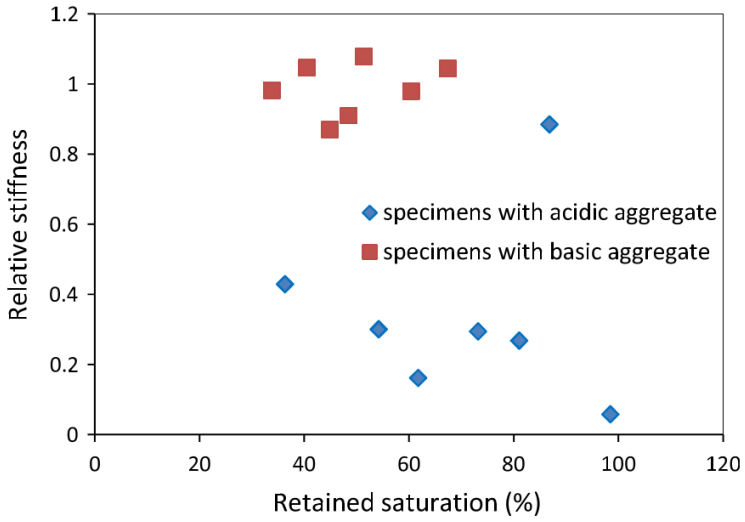


Figure 2:3: Comparisons between acidic and basic aggregate types after SATS conditioning

2.5.2 Properties of Bitumen

If we look at the chemistry of the bitumen, they consist of mostly hydrocarbons. They are non-polar materials (though a small percentage of hydrocarbons are also polar) but contain a certain amount of polar organic compounds. These polar organic compounds can have an acidic or basic nature. The examples of an acidic compound in bitumen are carboxylic acids. The acidic value of bitumen normally ranges from 1.5-5 mg KOH/g while the basic value ranges from 0-1 mg KOH/g (Asphalt Institute 2007). This is an indication that bitumen is mostly acidic in nature.

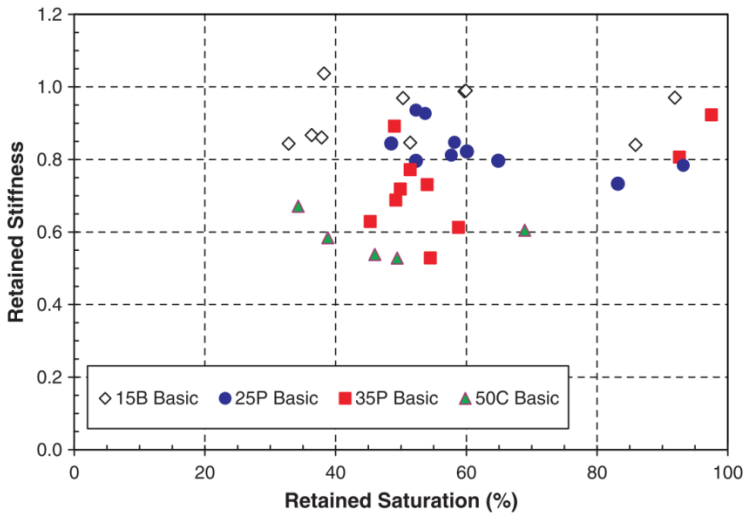


Figure 2:4:: SATS performance of different penetration grades, Airey et al. (2008)

Bitumen properties also play an important role in moisture damage resistance of asphalt mixture. Softer binders are found to be more moisture sensitive as compared to stiffer binders (Figure 2:4). The source of bitumen and how it is manufactured in the refinery may also influence moisture sensitivity (Ahmad 2011)

2.5.3 Interaction between bitumen and aggregates

As bitumen is mostly acidic in nature, it adheres well to the aggregates having basic characteristics like limestone. The calcium carbonate in the limestone reacts with the carboxylic acid in bitumen and forms a strong bond. On the other hand, as siliceous aggregates are acidic in nature, no chemical bond is formed between the bitumen and aggregate. Water, being polar in nature, can easily replace the bitumen from the aggregate surface.

The above explanation, however, is not always true as the material surface properties may be different than its bulk chemistry (Kim, 2009). Also, because of the complex chemistry of the bitumen, it is not possible to get similar results with even similar types of binders. In order to fully characterize the moisture susceptibility of a certain material, a lot of surface energy testing in conjunction with mechanical moisture sensitivity assessment is usually required. It is also important to study the chemistry of the materials in detail.

2.5.4 Mixture Volumetric properties

Volumetric properties have also been found to affect the potential for moisture resistance of asphalt mixtures. Referring to Figure 2:5, it can be seen that percentage saturation and void connectivity has been found to increase with void content and hence resulting in a decreased relative stiffness irrespective of the aggregate source.

2.5.5 Filler Type

Filler type has been claimed to have a pronounced effect on moisture damage performance of asphalt mixtures. Airey et al. (2008) in a study evaluated the effect of substituting 2% limestone (LS) filler with hydrated lime (HL) on moisture damage and found a significant improvement in mixtures containing acidic aggregates. On the other hand, substituting limestone filler with granite filler resulted in lowering retained stiffness values (Figure 2:6).

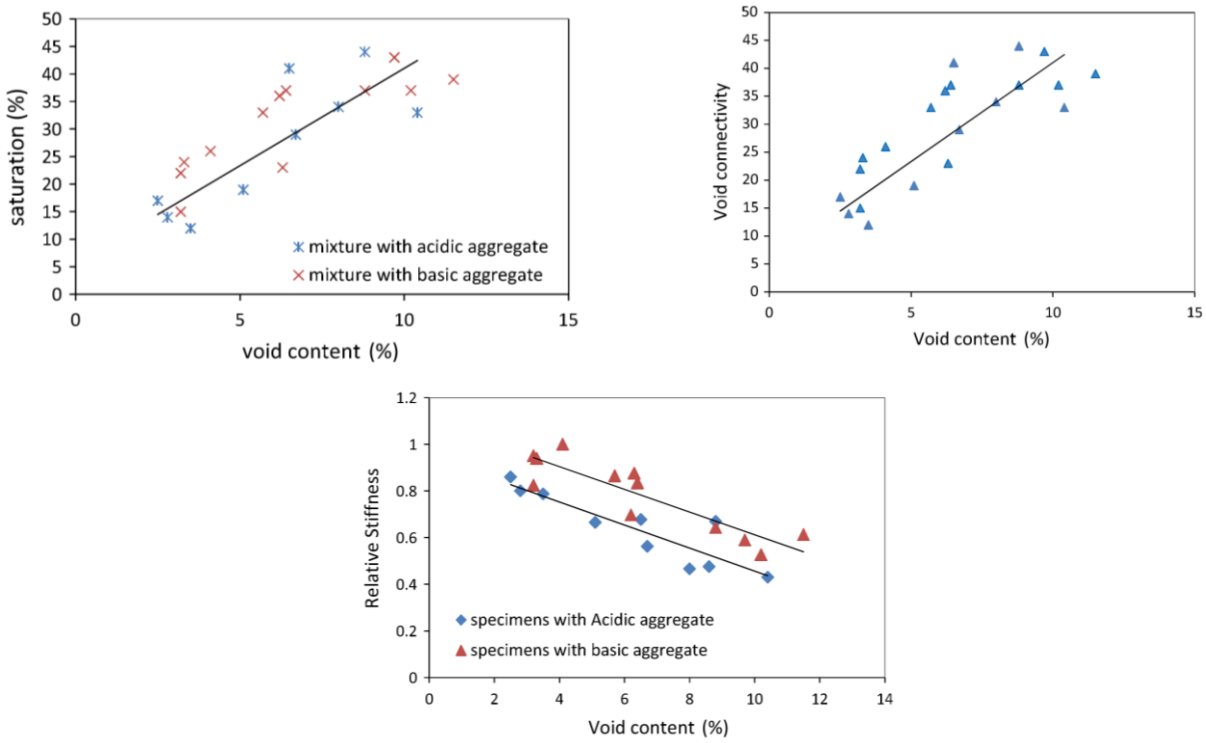


Figure 2:5: Effect of void content on saturation, void connectivity and relative stiffness (Khan et al. 2013) Filler Type

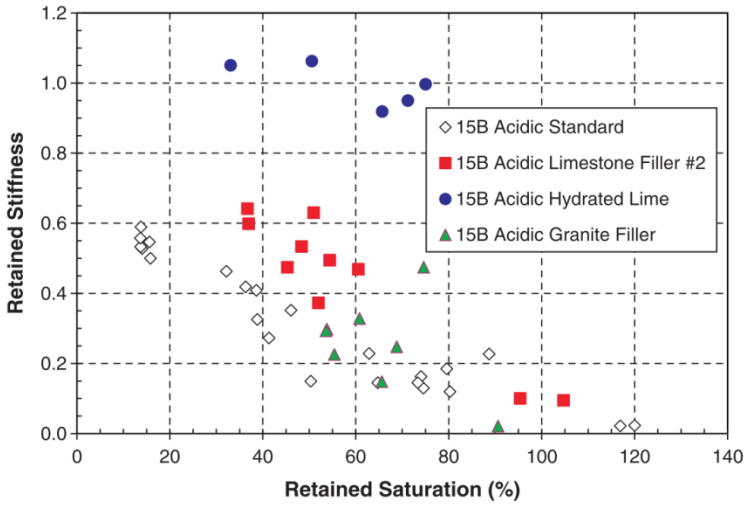


Figure 2:6: Effect of filler type and source on SATS moisture damage, Airey et al. (2008)

Since filler type can affect the properties of asphalt mixtures substantially a detailed analysis is needed to be carried out to get a better understanding of this fact using a variety of filler types.

2.6 Hydrated lime and asphalt mixture

Hydrated lime (HL) has been used as an additive in asphalt concrete for years (Kennedy 1984; Lesuesur 2010). HL started catching the interest in 1970 in the United States with a decline in the bitumen quality due to petroleum crises in 1973. At that time failures due to moisture and frost were among the most pressing failure types in the road pavements. Various efforts were made to tackle this issue and HL was found to be a most effective solution against these challenging damages (Hicks 1991). As a result of giving such a strong performance, HL was reported to be used in 10% of the asphalt mixtures produced in the US (Hicks and Scholz 2003).

Extensive research on the use of HL has been carried out in the last 40 years in the US and now it is referred as something more than an additive against moisture damage (Lesuesur 2010; Little and Petersen 2005). HL has proved to be a multifunctional additive which improves functional related properties of asphalt mixtures. It is considered as an active filler which improves moisture damage resistance, reduces chemical ageing in bitumen, and stiffens the mastic more than a normal mineral filler, hence results in no evidence of a negative effect on resistance to thermal cracking (Lesuesur 2010; Little and Petersen 2005; Sebaaly et al. 2006).

The beneficial effects of HL addition have been reported in the other parts of the world especially in Europe but the research in this field is not that developed in comparison to US (Lesuesur 2010). For example, a 20-25% increment in the durability of the pavement has been recorded by the French northern motorway company with the use of HL in the asphalt mixture (Raynaud 2009). Similarly, in Netherland, about 70% of the highways are consists of porous asphalt mixture having HL (Voskuilen and Verhoef 2003).

An improvement in the permanent deformation resistance in asphalt mixtures having multi-modal pavement distresses has been observed with the addition of HL and it is recommended to consider fundamental material properties and other performance characteristics together to make an optimised scenario (Aragão et al. 2010). The stiffness of the asphalt mixture also increases with the addition of HL, making it more moisture damage resistant (Zou et al. 2013).

Dony et al. (2012) evaluated the effects of moisture damage, freeze-thaw and de-icing salts on asphalt mixtures using HL as an additive and found an increased resistance of the asphalt mixture against all three environment damages.

Table 2:1: Hydrated Lime and Effect of de-icing salt

Aggregates	G1 6/10 Rhyolite		G2 6/10 River Gravel		G3 6/10 Micro-diorite	
Filler in mastic	G1	hydrated lime	G2	hydrated lime	limestone	hydrated lime
Coverage in water	65%	70%	<20%	40%	> 80%	> 80%
Coverage in salted water	50%	70%	<20%	<20%	>80%	> 80%

Results are shown in Table 2:1 in terms of remaining bitumen coverage on aggregates after the test and it can be clearly seen that HL has been found to be very efficient against the adverse effects of de-icing salt.

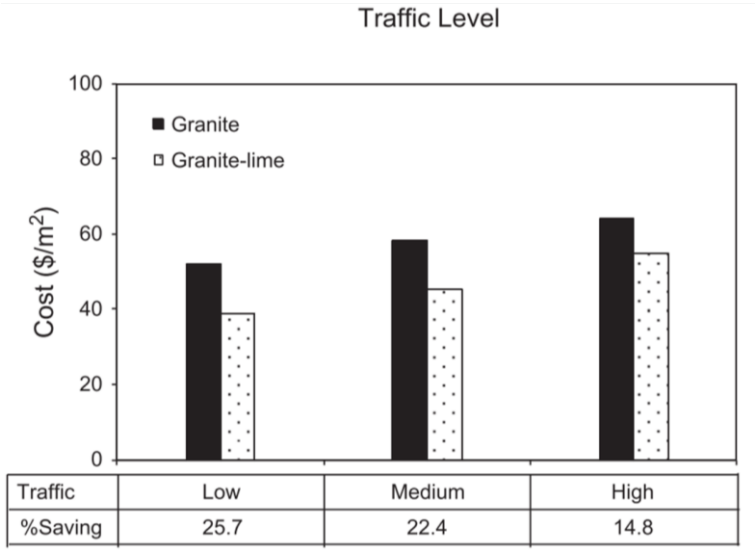


Figure 2:7: Cost comparisons of untreated and lime-treated mixtures based on ER

Along with all the beneficial effects of HL discussed above on bitumen, aggregate and asphalt mixture as a whole, it has been found to save 15-26% of the overall cost (Figure 2:7) of the road pavements in comparison with the untreated pavements (Zou et al. 2013). This cost saving has been estimated based on an Energy Ratio (ER) criteria which is treated as a measure of fracture performance of asphalt pavements. Additional thickness and cost of the untreated mixture were determined to get the same level of performance (ER) in comparison with HL treated mixture.

2.7 Hydrated lime properties

Hydrated lime consists of mainly Calcium hydrooxide, $Ca(OH)_2$. Calcium hydrooxide is obtained by the hydration of Calcium oxide, CaO which is obtained by heating the highly pure limestone in dedicated kilns at 900^{o}C (Boynton 1980)

Table 2:2: Grade of hydrated lime in accordance with EN 459-1

Grade	CaO + MgO (Percentage Weight)	Available lime (Percentage Weight)
CL90 S	≥ 90	≥ 80
CL80 S	≥ 80	≥ 65
CL70 S	≥ 70	≥ 55

As far as application to asphalt mixture is concerned, highly pure hydrated lime is used. This hydrated lime type is called CL90S according to EN 459-1 or referred as Type -1 according to ASTM C1097 and AASHTO M303. There are some other grades also depending on its purity and are used in different applications. Table 2:2 shows some details about the common grades used in the industry according to EN 459-1.

Hydrated lime comes in the form of a white powder having a particle density of around 2.2 Mg/m^3 and apparent density in a range of 0.3 to 0.8 Mg/m3, which is calculated in accordance with EN 459-2 or ASTM C110.

2.8 Hydrated Lime as a filler

Being its mineral origin, HL is often compared to the other types of mineral fillers in the asphalt industry. European standards for asphalt mixture from EN 13108-1 till EN13108-7 clearly categorises HL as a filler and hence it can be evaluated against the standards on aggregates for the construction of asphalt pavements as detailed in EN 13043. This standard also describes the combined use HL and other fillers. This standard mainly talks about the properties of the fillers which contributes to stiffening effect in the bitumen by measuring voids in dry compacted fillers and softening point using Delta ring and ball.

The Rigden voids in the dry compacted fillers are calculated by first calculating the density of fully dried compacted specimen and then dividing it by the particle density of the filler. The Rigden voids vary between 28% to 45% (Grabowski et al. 2009; Vansteenkiste and Vanelstraete 2008) for different mineral filler with a usual range of 30%-34% for commonly used filler such as limestone. HL, on the other hand, is different in terms of Rigden voids and have a higher range of 60-70% (Lesuesur 2010).

The softening point of the bitumen is measured with the help of delta ring and ball method. With the addition of HL to the bitumen, it increases its delta ring and ball. As a result when HL is used in the form of mixed filler, i.e. 15% and 30% by the weight of the filler, it increases the delta ring and ball value by 2-10^{o}C and 8-20^{o}C respectively (Grabowski et al. 2009).

Table 2:3: Comparison of some commonly used properties of mineral filler and hydrated lime (Lesuesur 2010)

Property	Method	Unit	Hydrated Lime	Mineral Filler
Particle density	EN 1097-7	Mg/m^3	2.2	2.6-2.9
Voids in dry compacted filler	EN 1097-4	%	60-70	28-45
Mass in kerosene	EN 1097-3	Mg/m^3	0.3	0.5-0.9
Blaine Specific surface	EN 196-6	cm^2/g	>10,000	7000
Specific surface area	BET (N_2 dsorption)	cm^2/g	150,000-200,000	14,000-95,000

A comparison of some properties of mineral fillers and hydrated lime is presented in Table 2:3. The properties presented in Table 2:3 are just a guide as these may vary significantly depending on the source of the mineral filler. Filler from the sources such as fly ash has a very different range of the properties discussed in the above table so one must not confuse it with the mineral filler.

2.9 Hydrated lime against Moisture and Frost damage resistance

Damage due to moisture and freeze-thaw cycles are common phenomena associated with the road pavement made with asphalt mixtures. It eventually results in the loss of aggregates form the mixture as illustrated in Figure 2:8. When water comes in contact with the asphalt mixture, the bond bitumen and aggregate weakens which gradually results in the separation of aggregates from the mixture.



Figure 2:8: Aggregate stripping due to moisture damage

The separation of aggregates on the surface of the asphalt layer is called stripping or revelling (Caro et al. 2008). In some cases, the separation of aggregates also takes place from the bottom layer due to the generation of water pressure induced by traffic and is called Flushing (Kandhal and Rickards 2001). These damages if untreated can transform into potholes. Also with the freeze-thaw cycles in the cold regions, these damages can go worse at an accelerated pace.

HL has been found very efficient against damage caused by moisture by many studies (Izzo and Tahmoressi 1999; Kim et al. 2008; Lesueur and Little 1999; McCann and Sebaaly 2006).

There are various methods available for the moisture damage assessment and will be explained in detail in the coming sections of this chapter.

Mohammad et al. (2008) in a study to evaluate the effect of HL against moisture damage using Hamburg Wheel tracking test, found a good reduction in the rut depth with the addition of 1.5% HL replacement with the original mineral filler. Similarly, in another study, Hamburg Wheel tracking test was used for moisture damage assessment and there was a huge decrement observed in the rut depth when 2% HL was used in comparison to 0.5% Liquid antistrip LAS2 (Maldonaldo 2008).

There are various studies which assess the effect HL using Lottman test. Some are listed here. In a study, Kim et al. (2008) found improved retained strength in the mixture having 1% HL. In another study made by Maldonaldo (2008), more than 100% increment in the retained strength was observed with the addition of 2% HL. Similarly, a significant improvement in the retained strength was observed with the addition of 1.5% HL in the form dry lime on damp aggregates or lime slurry (McCann and Sebaaly 2006).

Kim et al. (2008) also studied the effect of 1% HL addition using repeated Lottman test and found a massive increase from 11% retained strength (for the base material without HL) to 49% and 40% for dry lime on damp aggregates and lime slurry respectively, after 6 cycles. Another assessment of HL addition in bitumen mastic was undertaken using repeated Lottman test and a clear increment in indirect tensile strength was noted after 6, 10 and 12 cycles (Huang et al. 2005). There are various other evidence from the past research available stating the beneficial effect of HL in the form of dry HL application to the dry aggregate, dry HL to damp aggregates, mixing lime slurry to dry aggregates or adding HL to bitumen in the form of bitumen mastic. Also, Asphalt Task Force (2010) summarises a good number of research works which states the effect of HL addition to asphalt mixture.

2.10 Improvement in Mechanical properties with the use of HL

Hydrated lime has been reported to enhance the mechanical properties of asphalt mixtures. These improvements are usually assessed with some mechanical tests before and after some conditioning protocols. It has been found that hydrated-lime-treated mixtures have higher quality and modulus than unmodified blends.

This section will look at mastics rheology, that is a combination of just bitumen and filler, demonstrating the effect of HL. Also, this segment will review the mechanical properties of hydrated-lime modified asphalt mixtures, and specifically modulus, strength, resistance to rutting, fatigue and thermal cracking.

2.10.1 Mastic

So as to better comprehend the properties of asphalt mixtures, numerous researchers have utilized intermediate materials, for example, mastics, i.e. mixes of just bitumen and filler, as a model framework. The thought behind this examination is that the material sticking together the aggregates inside the mixture isn't the bitumen only but is the bitumen mixed with the finest components of the mineral called ‘filler’.

On examining mastics, it was clear that a mastic made with hydrated lime is very different in behaviour than a mastic made with typical mineral filler. It has been found that when mixed with the bitumen hydrated lime has a higher stiffening impact than typical mineral fillers.

Many studies in the past affirmed that properties, like viscosity and stiffness, are increased when hydrated lime is utilized rather than normal mineral filler (Kim and Little 2004; Kim et al. 2003). Most of the bitumen types show a higher stiffening impact with hydrated lime than ordinary mineral fillers. As a general guideline, and taking a normal asphalt mixture with 5% mineral filler and 5% bitumen, the substitution of 1% and 2% mineral filler by 1% and 2% hydrated lime separately would be equal to utilizing a bitumen with a R&B softening temperature higher by 2.5°C and 8°C respectively.

The stiffening of the bitumen with the addition of HL is highly dependent on the temperature. In fact, the low temperature examines demonstrate that hydrated lime is like other mineral fillers in terms of stiffening impact at low temperature (Lackner et al. 2005). Wortelboer et al. (1996) made study to observes the stiffening effect of different mastic at different temperature and found that the mastic with HL behaves very similar to the other mastic below the room temperature but give higher stiffness at an elevated temperature compared to the mastic without HL filler (Figure 2:9)

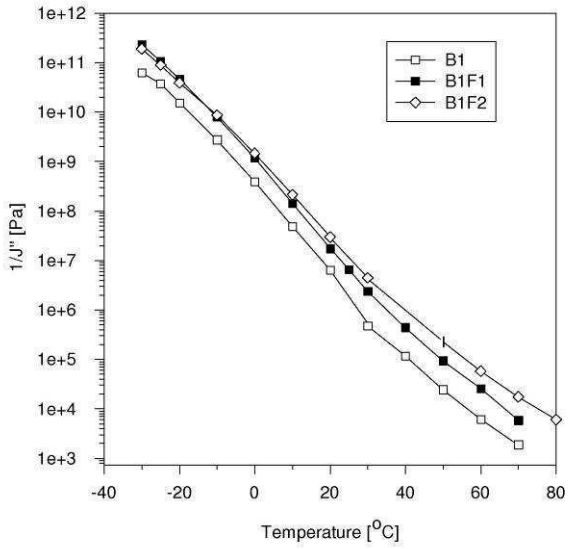


Figure 2:9: Temperature-dependent stiffening effect of hydrated lime in comparison to limestone filler: inverse of the imaginary compliance (1/J") at 10 rad/s versus temperature for a reference straight-run 70/100 bitumen (B1) and the same bitumen with 50wt.% of limestone filler (B1F1) or mixed limestone filler containing 40% hydrated lime (B1F2) (Wortelboer et al. 1996)

Other studies also confirm that the stiffening impact of hydrated lime is highly dependent on the temperature, with a similar behaviour like mineral filler underneath room temperature yet more successful above (Hopman et al. 1999; Khattak and Kyatham 2008; Lesueur and Little 1999).

2.10.2 Modulus increment

Modulus is a key mechanical property of materials. It is the proportion of the stress applied to the material and the subsequent strain. Modulus is called Young or tension modulus when it is measured in tension and is referred as Coulomb or shear modulus when measured in shear. The mechanical properties of an asphalt mixture depend on temperature and loading time, because of its viscoelastic conduct (L. Francken Ed. 1998). In this way, the modulus is dependent on temperature and time (or frequency) and is expressed in the form of a complex number called complex modulus.

For the design of pavement layers, the modulus is of prime significance, as the distribution of stress in each layer of pavement is linked to the modulus. For a given load and thickness, higher modulus implies lower stresses in the layer.

Despite the fact that the modulus is an intrinsic property, and should be independent of the test set-up, but little contrasts have been observed in the literature when the modulus is measured in compression, flexion, tension or indirect tension. Testing geometry (test sample shape and measurements) and the type of signals (force or deformation controlled, sinusoidal, amplitude) also found to affect the results. So it is very important to mention the details of measuring conditions while presenting the results of asphalt mixture modulus.

A limited set of studies shows how hydrated lime influences the modulus of asphalt mixtures. The most intensive investigation is carried by Professor M. W. Witczak and J. Bari at Arizona State University (Bari and Witczak 2005; Witczak and Bari 2004). They quantified the dynamic modulus in compression-tension between - 10°C and 54.4°C and frequencies running from 0.1 to 25 Hz for 17 different mixtures. The blends were made with 4.2-5.2% of 4 distinct binders and contained 0, 1, 1.5, 2, 2.5 or 3% hydrated lime as a filler substitute. Hydrated lime increased the modulus of asphalt mixtures by 8% up to 65% over the range of mixtures and hydrated lime concentrations at all temperatures and frequencies, with an overall average increase of 25%. In another examination by F. Thiago S. Aragao and collaborators with the University of Nebraska, the optimum percentage of 1.5% was concluded out of 0.5, 1.0, 1.5, 2.0 and 3.0% (Aragão et al. 2008). One of the weak point of the study was that there was no reference material was used in comparison to hydrated lime for the investigation. In another examination, M. Ghouse Baig and H. I. Abdul Wahhab at King Fahd University (Saudi Arabia) found 4% hydrated lime content as optimum in a comparative study made with 1, 2, 3, 4 and 5.5% HL in the mixture (Baig and Wahhab 1998).

To find out the effect of HL addition on the modulus Asphalt Task Force (2010) summarised some of the research work conducted in the past. Out of 71 mixtures containing HL, 42 were found to have an improved modulus with the addition of HL. So not all the mixtures will show an improved performance in terms of modulus with the addition of HL but the improvement was highly dependent on the different type of mixture and hence their constituents.

Another examination by M. Stroup-Gardiner and J. A. Epps with the University of Nevada analysed different ways of adding hydrated lime to a mixture (Stroup-Gardiner and Epps 1990). They found that the modulus could be affected by the way HL is added to the mixture.

2.10.3 Strength improvement

Strength is an engineering mechanical property of materials. It is a measure of maximum stress which is required to break a material. For the asphalt mixtures, strength is typically measured either in the form of compression or indirect tension at ambient temperature. Although Modulus and strength are of different nature, as one is intrinsic property (modulus) and the other is non-intrinsic and depends on the sample shape and dimensions. Generally, it is simpler to quantify strength than the modulus, thus in materials engineering strength dominate the modulus.

As there exists a relationship between the modulus and strength so there will be a similar effect on modulus and strength with the variation in the mix variables. That is the reason why the strength is at the maximum value at optimum bitumen content and increase with the increase in the bitumen modulus and decrease with the air voids content (L. Francken Ed. 1998).

A large number of studies uses strength value for the assessment of HL addition against moisture damage resistance. The strength is measured before and after some conditioning protocols and retained value is calculated and then compared to the mixtures without HL (Ameri and Esfahani 2008; Kok and Yilmaz 2009; Maldonaldo 2008).

2.10.4 Rutting resistance

Rutting has been seen on asphalt mixtures since the very start, however, turned out to be of significant importance after World War 2 with the increase in the traffic loading (Sousa et al. 1991). Rutting happens as a result of excessive traffic loading when the applied load is more than the permissible limits of the asphalt mixture and results in a permanent plastic deformation as shown in the Figure 2:10.

This rutting potential is in a direct relation to low-speed loads and high temperatures. In any case, rutting is a complex phenomenon as the asphalt mixture fails in a visco-plastic manner under the anticipated conditions.

With reference to the mix design, rutting potential could be more with the factors such as excessive bitumen content, presence of round shaped aggregates, excessive sand content, or

high deformability of the binder used (Sousa et al. 1991). Hence, the factors which favours the stiffening of the mixture also improve resistance to rutting for the same mix design.



Figure 2:10: Rutting in an asphalt mixture

A large number of researchers have reported the beneficial effects of HL against rutting resistance. Some of them include (Baig and Wahhab 1998; Little and Petersen 2005; Pilat et al. 2000).

2.10.5 Resistance to Fatigue Cracking

Fatigue cracking happens in road pavements made up using asphalt pavement as a result of progressive traffic load and hence creating the cracks which start from the bottom of the layer to the top (Figure 2:11)

The factors favouring the fatigue include, inadequate layer thickness and bad adhesive bonding between the successive pavement layers as both are responsible for high flexural stress at the bottom of the asphaltic layer. As far as mix design is concerned, resistance to fatigue can be enhanced with a high bitumen content or by using high-performance binder grades (L. Francken Ed. 1998).

The main failure mode which is used in the design of the pavement layer is the Fatigue cracking. Hence the pavement layers are designed thick enough so that they can withstand the fatigue cracking till the end of their design life which may range from 10 to 40 years (FEHRL 2004).



Figure 2:11: Fatigue cracking in an asphalt mixture

A limited number of studies are made to quantify the effect of HL against fatigue resistance but still, evidences are there that HL increases the resistance of asphalt mixture against fatigue cracking (Baig and Wahhab 1998; Little and Petersen 2005; Mohammad et al. 2000).

2.10.6 Resistance to Thermal Cracking

This cracking is generally observed in the cold regions of the world. The low temperature which is imposed on the bitumen mixture in these areas makes it brittle. With this low-temperature thermal shrinkage occurs and results in the stresses which can be higher than the material resistance and hence can generate big cracks as shown in the Figure 2:12.

Same as fatigue cracking, for the mix design consideration, resistance to thermal cracking can be enhanced with a high bitumen content or by using high-performance binder grades (Monismith et al. 1994). A very limited number of studies are published to evaluate the effect of HL addition on thermal cracking. In a study conducted by McCann and Sebaaly (2006), there was no significant difference was noted on thermal cracking on the HL modified mixtures and the unmodified mixtures.



Figure 2:12: Transverse thermal cracking

Resistance to thermal cracking somewhat linked with mastic toughness and in another study, it was found that HL toughens the mastic to the same extent as other mineral fillers do if used in the same volumetric proportions (Lesueur and Little 1999). Based on this fact it can be concluded that the performance of the HL modified mixture may not be any different than the one without as far as resistance to thermal cracking is concerned.

2.11 Effect of HL on the aggregates when used in asphalt mixture

A pronounced effect of HL addition to the aggregates has been observed. This effect could be in the form of surface modification and/or clay flocculation. Both of these phenomena are explained in the light of previous research as follows;

2.11.1 Surface Modification of Aggregates

Hydrated lime has been reported to modify the surface properties of aggregates (Lesueur et al. 2013). Hydrated lime has been found more effective in the asphalt mixtures having siliceous aggregate than limestone aggregates (Bagampadde et al. 2004; Grönniger et al. 2010; Hicks 1991).

Cationic and anionic surfactants are naturally present in bitumen and with limestone aggregate both the surfactants made a strong bond but only cationic surfactants make a strong bond with siliceous aggregates (Curtis et al. 1993). Hence, anionic surfactants are easily displaced by water due to a weak bond between aggregate and bitumen. Addition of HL results in the

precipitation of calcium ions on to the surface of aggregate making it more favourable for bitumen (Ishai and Craus 1977).

When HL is present in the mastic, calcium ions accumulate at the aggregate surface and react with the acids from the bitumen to make a water-insoluble salt (Figure 2:13 A and B). With the formation of this insoluble salt on the surface of aggregate, surface roughness increases which favour aggregate-bitumen adhesion (Blazek et al. 2000).

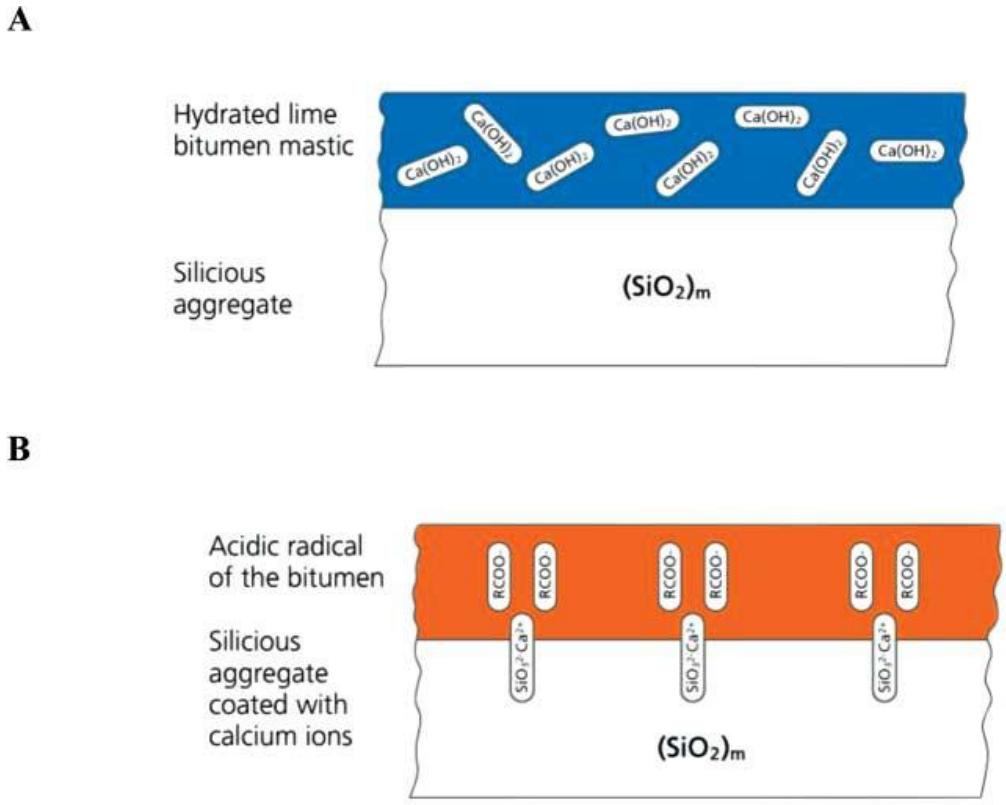


Figure 2:13: Effect on Aggregates surface with HL (Ishai and Craus 1977)

Being an active filler hydrated lime react strongly with the aggregates. This statement can be justified by the fact found in the previous research that upon bitumen extraction a part of HL was not recovered and the percentage was variable depending on the aggregate type. When HL was used with limestone filler the percentage of unrecovered HL was 10% while for basalt filler about 40% of HL as found missing (Schiffner 2003).

Surface modification is not the only phenomenon associated with the addition of HL as this unique filler has also been reported to improve the adhesion properties of even limestone aggregates and bitumen (Huang et al. 2005; Mohammad et al. 2008).

To evaluate the effect of HL on aggregates Nejad et al. (2013) used the surface free energy approach and claimed a reduction of acidic component of the surface free energy (SFE), promotion of the basic component of SFE in aggregates (Table 2:4), and a reduction in the total SFE of aggregates. As a result of this reduction in the total surface energy of aggregates, the surface energy of the asphalt-aggregate-water system as a whole decreased, giving a more stable thermodynamic condition.

Table 2:4: SFE Components of Aggregate with and without HL treatment

Types of aggregates	Standard deviation of total SFE	Total SFE, Γ (J/m^2)	Nonpolar component, Γ^{LW} (J/m^2)	Polar component, Γ^{AB} (J/m^2)	Acidic component, Γ^{+} (J/m^2)	Basic component, Γ^{-} (J/m^2)
Limestone	0.0021	0.21086	0.06123	0.14963	0.48421	0.01156
Limestone + HL	0.0046	0.20971	0.06279	0.14692	0.45236	0.01193
Granite	0.0027	0.39918	0.04430	0.35488	0.67898	0.04637
Granite + HL	0.0041	0.39508	0.04638	0.34870	0.62831	0.04838

The results shown in Table 2:4 clearly demonstrate that there is hardly a difference in the total surface energy of aggregates with and without HL addition. Also, work of adhesion and de-bonding is not calculated which can give a real insight of the moisture damage mechanism. Furthermore, no consideration of specific surface area (SSA) is considered in the for energy ratio calculation so more investigation is required to support the claimed argument.

2.11.2 Clay flocculation

Hydrated lime has been found very efficient against moisture damage for the aggregates having clay. That is the reason why in some states of USA like California, Nevada, Colorado and Utah where a large amount of clay has been reported in the aggregates HL is used for the construction of asphalt mixture to be used in road pavements (Aschenbrener 1995). Clay may occur naturally inside the rock and be liberated as a result of crushing. HL flocculates the clay particles, preventing them from building a water-displaceable barrier between bitumen and aggregate (Little 1995).

2.12 Effect of HL on bitumen when used in asphalt mixture

Hydrated lime has been also reported to modify the properties of bitumen by reacting chemically and changing its properties and/or physically by changing its porosity. Both of these interactions have been discussed in details in the coming section.

2.12.1 Chemical Interaction

The phenomenon of ageing in the bitumen is very complicated and a lot of research has been conducted in this area (Petersen and Glaser 2011). It was first discovered in 1970 in a research conducted in Utah state that HL can slow down the ageing of the bitumen. Bitumen was extracted from the field specimens and the viscosity of the specimens having HL was compared to the one without HL and it was found that the bitumen extracted from the specimen having HL was low in viscosity than the one without (Chachas 1971). This finding is in line with the recent research where it is now a well-known fact that HL decreases the ageing rate due to a slower increase in the viscosity with the time (Lesuesur 2010; Little and Petersen 2005). The reason why HL slows down the ageing rate lies in slowing down the rate at which asphaltene content produces (Verhasselt and Puiatti 2004; Wisneski et al. 1996)

Another factor behind slow ageing for HL modified bitumen is the slow rate of formation of carbonyl content which is directly correlated with viscosity (Verhasselt and Puiatti 2004). This effect was only observed at a temperature of 88^{o}C or higher and was not observed at 60^{o}C or below. The HL treated bitumen even after removal of HL from them gives a reduced ageing effect (Petersen et al. 1987; Plancher et al. 1976). It is important to note that all of the above-mentioned facts were only observed with the HL treated bitumen and none of them was found in the bitumen treated with limestone filler (Huang et al. 2002; Petersen et al. 1987)

Previous studies demonstrate that HL chemically reacts with the bitumen. This behaviour of HL, when added to the bitumen, was first discovered in a study conducted by western research institute (WRI) in 1976 when four bitumen of different chemical composition were selected for the study. 1:1:600 solution of bitumen, HL and benzene was prepared for all types of bitumen and the solution was left for 24 hours to react between all the three components. With the help of centrifugal solvent extraction, they found that about 4-6% of bitumen by weight was adsorbed on HL (Plancher et al. 1976). Later, in another study conducted in Netherlands, it was also found that hydrated lime is more efficient than the limestone filler as far as bitumen filler interaction is concerned. This study found that the adsorption of bitumen from several solvents (ethyl chloride, n-heptane, toluene, tetrahydrofuran) on active limestone filler containing 25% by weight HL was 1.4 to 2.1 times higher than the one without HL for different bitumen sources (Hopman et al. 1999)

Some of the results from a study made for WRI are presented in Table 2:5. The concentration of different functional groups in different types of bitumen is presented with and without HL treatment. It is clear from the Table 2:5 that the concentration of carboxylic acid, dicarboxylic

anhydride, and 2-quinolones have reduced in the HL treated bitumen. The ketones concentration was increased and there was no significant change recorded in the concentration of sulphoxides (Plancher et al. 1976)

From the research presented above, it can be deduced that HL clearly reacts with some components of bitumen which includes Acids, anhydrides and 2-quinolones. A similar conclusion was drawn in a more recent study by (Petersen et al. 1987) where 150 grams of bitumen from various sources and origin was agitated for 6h at a temperature of 150^{o}C with various concentration of HL or hydrated dolomitic lime. The lime was then solvent extracted and the ageing was quantified at 60^{o}C using the ageing index. The results were compared between the base bitumen (untreated) and the lime-treated bitumen (various concentrations) and are presented in the Figure 2:14, where beneficial effect of HL and hydrated dolomitic lime are obvious in most of the combinations in the form of a lower ageing index.

Table 2:5: Functional groups concentration in various types of bitumen before and after HL treatment (Plancher et al. 1976)

Bitumen		Ketones mol/l	Carboxylic acids mol/l	Dicarboxylic anhydrides mol/l	2-Quinolones mol/l	Sulphoxide mol/l
B-2959	Untreated	0.015	trace	0.001	0.003	0.015
	Treated	0.039	bld	bld	bld	0.013
B-3036	Untreated	0.021	trace	0.001	0.001	0.022
	Treated	0.039	bld	bld	bld	0.019
B-3051	Untreated	0.017	0.014	0.003	0.009	0.010
	Treated	0.039	bld	0.001	0.004	0.008
B-3602	Untreated	0.045	0.060*	bld	0.011	0.015
	Treated	0.100	0.014*	0.007	0.006	0.015

Note: bld = below level of detection; ∗ present as carboxylate salts.

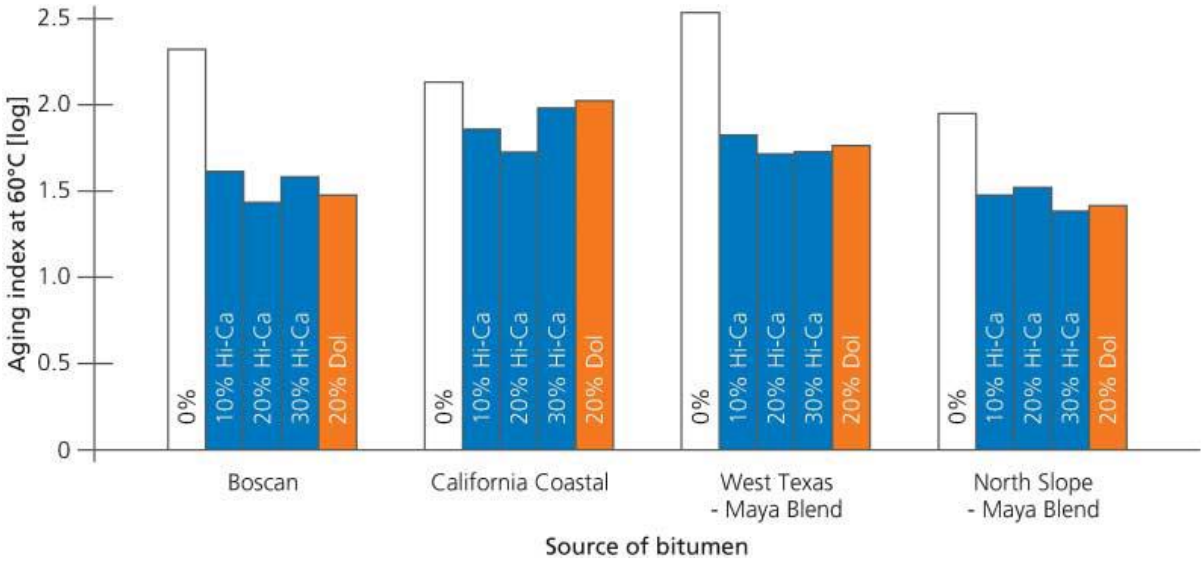


Figure 2:14: Ageing index at 60ºC for various sources of bitumen with different concentration of HL (Hi-Ca) and hydrated dolomitic lime (Dol) (Petersen et al. 1987)

Table 2:6: Function groups concentration for various combination of bitumen having HL and hydrated dolomitic lime bitumen before and after TFAAT ageing (Plancher et al. 1976)

Lime Treatment		Ketones mol/l	Carboxylic acids mol/l	Dicarboxylic anhydrides mol/l	2-Quinolones mol/l	Sulphoxide mol/l
None	Unaged	0.00	0.015	0.000	0.017	0.02
	Aged	0.28	0.015	0.007	0.017	0.35
10 wt.% high calcium	Unaged	0.03	0.005	0.000	0.016	0.03
	Aged	0.24	0.004	0.005	0.016	0.32
20 wt.% high calcium	Unaged	0.03	0.003	0.000	0.014	0.03
	Aged	0.22	< 0.002	0.006	0.017	0.34
30 wt.% high calcium	Unaged	0.03	< 0.002	0.000	0.013	0.03
	Aged	0.21	< 0.002	0.006	0.014	0.32
20 wt.% Dolomitic	Unaged	0.03	0.005	0.000	0.013	0.03
	Aged	0.22	0.005	0.006	0.014	0.34

Similarly, Infrared spectroscopy was used to calculate the concentration of different functional groups present in the bitumen and results were again compared between the base bitumen (untreated) and the lime-treated bitumen (various concentrations) and presented in Table 2:6.

By looking at the results presented in Table 2:6 it is clear that the in the presence of HL the reduction in the concentration of Ketone, anhydrides and above all carboxylic acid was observed which are produced as a result of ageing. In another research conducted by Little and Petersen (2005) it was stated that HL reacts chemically with the acids present in the bitumen and neutralises their bad effects and hence slowing down the ageing phenomenon.

An acid-base reaction is not the only phenomenon which comes under chemical interaction of bitumen and HL. HL also acts as an inhibitor of the oxidation catalysts naturally present in the bitumen (Petersen et al. 1987; Plancher et al. 1976).
The interaction between the bitumen and HL is highly influenced by the bitumen chemistry and hence its source or origin (Petersen et al. 1987; Plancher et al. 1976; Verhasselt and Puiatti 2004). This interaction between bitumen and HL can be categorized into two consequences.

1. The neutralized polar molecules by the HL are adsorbed strongly on the surface of HL and prevents them to react further after chemical ageing (Petersen et al. 1987; Plancher et al. 1976). This slows down the ageing kinetics as these polar molecules are more prone to ageing.
2. The neutralized polar molecules are also prevented to diffuse at the bitumen-aggregate interface. So only non-acidic surfactants of bitumen can move to bitumen-aggregate interface (Bagampadde et al. 2004). These surfactants are normally amine based and are not easily displaced by water (Curtis et al. 1993; Stuart 1990). So there will be improved resistance to moisture damage.

As a conclusion, it can be stated that as a result of the chemical interaction between the bitumen's acidic molarities and HL an improved ageing resistance and improve adhesion can be achieved.

2.12.2 Physical Interaction

As discussed already that HL has more voids (65%) in the dry compacted state (Rigden Voids) in comparison to the other mineral fillers (30%-34%). The reason for this high porosity lies in the high porosity of HL particles. For the mineral fillers, the porosity comes from the voids

between the particles whereas in the case of HL porosity is the sum of voids between the particles and the voids within the HL particles, hence a greater value is achieved (Figure 2:15).

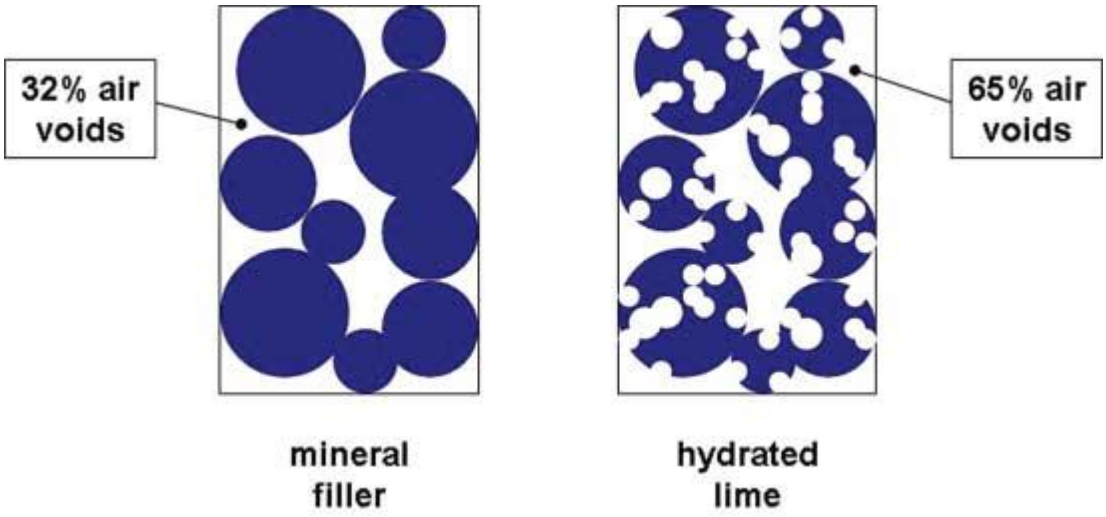


Figure 2:15: Comparison of Rigden voids for mineral filler and Hydrated lime (Lesueur et al. 2013)

This high porosity of HL increases the overall stiffness of the mixture and makes it stronger against rutting, fatigue and thermal cracking. This increased stiffness of the asphalt mixture also makes it more moisture damage resistant (Zou et al. 2013).

2.13 Moisture damage assessment

Due to ageing and moisture exposure, the durability of asphalt pavements gradually decreases and chances of premature pavement failure increase. In order to evaluate the effect of ageing and moisture damage many laboratory procedures have been devised. An extensive literature review on moisture sensitivity test methods for bituminous pavement materials has been carried out by Airey and Choi (2002). These procedures have been broadly classified into two categories i.e. tests on loose coated aggregates and tests on compacted asphalt mixture. There are some other techniques also available which have been found very useful in understanding moisture susceptibility of asphalt mixtures. These include Surface free energy (SFE) approach, peel test and PATTI test. Surface energy approach involved the calculation of surface energy of the aggregates and bitumen. Using the surface energy of these two components, work of

adhesion (under dry conditions) and work of de-bonding (under wet conditions) is calculated with the help of thermodynamic equations. This work of adhesion and work of de-bonding is referred as intrinsic adhesion measurement. On the other hand, Peel and PATTI test are used for the practical adhesion measurement and can be used for the dry and water conditioned samples.

2.13.1 Tests on loose coated aggregates

Test procedures for loose coated aggregates consist of first coating the aggregates with the bitumen and then immersing them in water or a chemical solvent for a specified period of time at room or a specified temperature. The immersion of the loose coated aggregates can be either in static, shaking or rolling conditions. After a specified period of time aggregates are removed from the water and assessed for remaining coating of bitumen by either visual inspection or with the help of some image analysis techniques.

These test methods are considered as easy to perform and test results are in the form of a comparative analysis and which do not give an insight into the moisture damage mechanism taking place within the samples. So it is a good idea to take advantage of some other testing techniques to get more confidence in the test results. A summary of these tests has been presented in Table 2:7.

Table 2:7: Moisture Damage Tests for Loose Aggregates

Test Method	Reference	Extra Features
Static immersion test	AASHTO T182, ASTM D1664	16-18 hours in water, results are visually assessed for retained bitumen coating.
Total Water immersion test	Shell Bitumen (1990)	48 hours in water, results are visually assessed for retained bitumen coating.

Dynamic Immersion Test	Whiteoak D (1991)	16-18 hours in water with mechanical agitation, results are visually assessed for retained bitumen coating.
Chemical immersion test	Whiteoak D (1991)	The sample is immersed in Solution containing sodium carbonate, results are visually assessed for retained bitumen coating.
Rolling Bottles Test method	BS EN 12697	Up to 72 hours in water with mechanical agitation by glass road in rolling bottles, results are visually assessed for retained bitumen coating after 6, 24, 48 and 72 hours.
Boiling water test	ASTM D3625	10 minutes in boiling water, results are visually assessed for retained bitumen coating.
Ancona stripping test (AST)	Bocci and Colagrande S. (1993)	45 minutes in boiling water, results are visually assessed for retained bitumen coating.
Boiling water Stripping test	Choquet and Verhasselt (1993)	10 minute in boiling Water followed by a chemical attack, Stripping Ratio is calculated with a reference calibration curve.
Ultrasound method	Vuorinen and Valtonen (1999)	Immersion in water with the ultrasonic attack, results are visually assessed for retained bitumen coating.

Net adsorption test	Curtis et al. (1993)	Shaking aggregates in a solution containing a mixture of bitumen and toluene followed by addition of water and another soaking for 8 hours, the amount of adsorbed bitumen on the aggregate surface and desorbed from the coated aggregate is measured in a spectrophotometer.
Modified net adsorption test	Walsh et al. (1996)	Follow the same test procedure as Net adsorption test, the initial adsorption value is calculated by providing an indicator to assess the affinity and resistance to stripping of the aggregate-bitumen system

It is difficult to select one or two procedures out of all listed in Table 2:7. By looking at the research conducted previously it can be found that Jorgensen (2002) ran a round robin test on boiling water and Rolling bottle test and concluded that boiling water test was able to identify the combinations of binder and aggregates having bad performance but the Rolling bottles test can rank these combination, which is a plus point of rolling bottles test.

In another study Liu et al. (2013) made a comparative study considering five empirical tests on loose mixtures for performance evaluation including static immersion test, rolling bottle test (RBT), boiling water test (BWT), total water immersion test and the ultrasonic method. Surface free energy (SFE) tests on aggregate and bitumen were also performed to confirm the performance with these empirical methods. RBT and BWT were found to be more sensitive procedures among the five empirical procedures in predicting moisture damage performance. Mixture ranking given by RBT was found to be in agreement with the results of SFE testing.

In a most recent study made by Porot et al. (2016) round robin test on the Rolling bottle test has been conducted to check its reliability and the repeatability of this most commonly used test to check the affinity between bitumen and aggregate. The test was conducted in 8 different laboratories across the globe in accordance with EN 12697-11 method A. It was concluded that the repeatability if the test across different laboratories is poor and one of the reason could be

visual assessment. On the other hand, the reproducibility and repeatability of the test within the same laboratory was observed to be good.

Based on all the previous research work, RBT can be said to be one of the most efficient empirical procedure for the assessment of affinity between the bitumen and the aggregate but there are some weak points as well. First weak point is the visual assessment and the second is the poor repeatability among different laboratories. If some arrangements are made to replace the visual assessment method with some sort of digitized assessment the first issue can be resolved. Now for the second problem, as far as someone is testing in one lab under same conditions following same procedure the results will be repeatable and reproducible and hence different combinations can be compared. Also, it will be a good idea to use some other techniques, such as SFE technique, SATS test, Peel test or some pull off techniques to get more confidence in the results obtained from RBT.

2.13.2 Tests on compacted asphalt mixture

The test method on the compacted asphalt mixture usually involves the measurement of some mechanical properties of the mixture before and after moisture conditioning. These properties may include parameters such as indirect tensile strength or stiffness which are used to assess the damage after moisture conditioning (Airey et al. 2008). The compacted asphalt mixture can be prepared either in the laboratory or can be cored out in the field. For the assessment of the indirect tensile stiffness and strength, Nottingham Asphalt test or Instron can be used. Similarly, there is another parameter which can be calculated for the assessment of permanent deformation under repeated load, called rutting. Rutting resistance under moist conditions can be quantified with the help of Hamburg wheel tracking device. The test methods which are commonly used for the assessment of moisture damage resistance of compacted asphalt mixtures are listed in Table 2:8.

Table 2:8: Moisture Damage Tests for Compacted Mixtures

Test Method	Reference	Thermal cycling	Performance tests
Freeze-thaw pedestal test	Kennedy et al. (1982)	23^{o} C for 3 days followed by -12^{o} C for 15 hrs, 23^{o} C for 45 mins & 49^{o} C for 9 hrs	Cracking of the specimen over a fulcrum after a number of thermal cycles.
Immersion compression test	AASHTO T165, ASTM D1075	49^{o} C for 4 days or 60^{o} C for 24 hours, 23^{o} C for 4hours	Compressive strength
Marshall stability test	AASHTO T245	Vacuum treatment under water @ 0^{o} C to 1^{o} C, 60^{o} C for 24 hrs	Marshall stability
Lottman procedure	Lottman (1982)	Distilled water at partial vacuum of 600 mm Hg for 30 mins, atmospheric pressure for 30 mins, -18^{o} C to -12^{o} C for 15 hrs, 60^{o} C for 24 hrs	Indirect tensile strength and stiffness
Modified Lottman procedure	AASHTO T283	Distilled water at partial vacuum of 508 mm Hg until 60% to 80% saturation, -18^{o} C to -12^{o} C for 15 hrs, 60^{o} C for 24 hrs	Indirect tensile strength and stiffness

Immersion wheel tracking test	Mathews and Colwill (1962)	Submerged in water at 40^{o}C	Wheel tracking at 25 cycles/min
Environmental conditioning system (ECS)	Terrel and Al-Swailmi (1994)	Water at partial vacuum of 254 mm Hg or 508 mm Hg for 30 mins, 3 hot cycles at 60^{o} C for 6 hrs, one Freeze at -18^{o} C for 6 hrs	Resilient modulus and permeability at 25^{o} C
Saturation ageing Tensile stiffness (SATS) Test	Grenfell et al. (2012)	Distilled water at partial pressure of 0.5 MPa, 85^{o} C for 24 hrs	Indirect tensile strength and stiffness

Out of all the test methods mentioned in the Table 2:8, Lottman's test (AASHTO T283) and Hamburg Wheel Tracking test (HWTD) have been used extensively in the past for the evaluation of moisture damage resistance of the compacted mixtures (Hu et al. 2017; Huang et al. 2005; Kim et al. 2008; Maldonaldo 2008; McCann and Sebaaly 2006; Mohammad et al. 2008).(Hu et al. 2017; Huang et al. 2005; Kim et al. 2008; Maldonaldo 2008; McCann and Sebaaly 2006; Mohammad et al. 2008)

A test method was developed by the Nottingham Transportation Engineering Centre (NTEC) known as the Saturation Ageing Tensile Stiffness (SATS) Test, to measure the moisture sensitivity of asphalt mixtures (Collop et al. 2004). Airey et al. (2006) used SATS and AASHTO T283 to test high modulus base (HMB) material. The relative performance of the two tests was compared in terms of measured retained stiffness modulus of the specimens. The values obtained from AASHTO T283 were approximately double than SATS for same saturation level. This gives an indication that SATS was more severe on the samples. The drop in the stiffness modulus for samples tested with SATS was due to the combined effect of moisture and air (ageing). The values obtained from SATS testing were comparable to the

stiffness modulus values observed on a trial site where HMB material was placed. This gives an indication that SATS probably better simulates the field conditions as compared to AASHTO T283, at least for the set of mixtures that were analysed.
SATS measures the combined effects of ageing and moisture using the following steps:

- Initial saturation of specimen
- Ageing and saturation in a high temperature and pressure environment for extended period
- Calculation of retained saturation
- Calculation of retained stiffness modulus as:

$$\text{Retained stiffness modulus} = \frac{\text{Stiffness modulus after test}}{\text{Stiffness modulus before test}} \quad (2.1)$$

The application of the SATS test was limited to basic aggregates and hard binders initially. Later it was optimised for a universal asphalt mix application (Grenfell et al. 2012).

2.14 Concept of adhesion and surface energy

In an asphalt mixture, the primary function of the bitumen is to provide an adhesive layer to bind aggregate particles together. A strong adhesive bond between the aggregate and the bitumen is very important in order to ensure good performance of a pavement.
The bond of bitumen with some aggregates is not strong enough even when they are dry and clean. However, all the aggregates are easily wetted by water, and its presence results in stripping (Read and Whiteoak 2003). The reasons for this are the surface/physico-chemical characteristics of both the bitumen and the aggregates and the individual chemical nature of these materials. Different types of aggregates have different chemical characteristics which influence their affinity for bitumen. Aggregates with high silicon oxide content, e.g. quartz and granite (i.e. acidic rocks) are classified as ‘hydrophilic’ (water-loving) and are generally more difficult to coat with bitumen than basic rocks such as limestone.
Aggregates being high surface energy materials possess unbalanced surface charges (Bhasin 2007). If the aggregate surface is coated with a liquid of opposite charge its surface energy demands are satisfied and the result is a strong adhesive bond. When two different liquids are present, e.g. bitumen and water, the liquid that will best satisfy the energy requirement will

adhere more strongly to the aggregate. The stripping of bitumen, from the surface of aggregate in the presence of water, is due to the fact that water, being polar in nature, better satisfies the surface energy demands of aggregates which results in replacement of the bitumen from the aggregate surface (Read and Whiteoak 2003). The bitumen, on the other hand, is non-polar in nature and hardly makes any chemical bond with the aggregates, except for having a small/weak acidic character because of the presence of carboxylic acid which makes it possible to bond well with the basic aggregates like limestone.

As the chemical nature of two bitumen can vary considerably (Read and Whiteoak 2003), so adhesion characteristics can be very different even if tested with same aggregates. In the same manner, different aggregates can have very different surface characteristics depending on their origin even if the mineralogy is same. So, it is very important to study the surface characteristics of the two materials along with their interfacial characteristic (adhesion) in order to evaluate the moisture damage resistance of the resultant asphalt mixture and/or for the recommendation of any material treatments or modifications. Some results from the past studies of surface energy of few bitumen and aggregate types have been presented in the Table 2:9 and Table 2:10 for a general understanding.

Table 2:9: Components of surface energy for different bitumen types from previous studies

Bitumen Type	Surface energy components (mJ/m^2)			Total surface energy, γ (mJ/m^2)	Reference
	γ^{LW}	γ^{+}	γ^{-}		
60/70 Pen	13.69	1.45	0.65	15.63	Nejad et al. (2013)
PG 64	13.08	2.55	1.79	17.36	Kakar et al. (2016)
40/60 Pen	30.60	0.00	0.00	30.60	Liu et al. (2013)

Table 2:10: Components of surface energy for different bitumen types from previous studies

Aggregate Type	Surface energy components (mJ/m^2)			Total surface energy, γ (mJ/m^2)	Reference
	γ^{LW}	γ^{+}	γ^{-}		
Limestone	61.23	484.21	11.56	210.86	Nejad et al. (2013)
Granite	44.30	678.98	46.37	399.18	
Limestone	58.01	1.76	401.07	111.15	Kakar et al. (2016)
Limestone	75.30	108.90	49.70	222.40	Liu et al. (2013)
Granite	69.10	17.30	568.30	267.50	

According to thermodynamics, adhesive and cohesive bonds are directly related to surface energy characteristics of bitumen and aggregate. Adhesive bond parameters for aggregates and binder under dry and wet conditions can be studied using their surface energy for different combinations of bitumen, aggregate and filler (Cheng et al. 2002). Using surface energy measurements, we may also understand the mechanisms that affect adhesive or cohesive bonds.

2.14.1 Surface Energy Theory

The amount of work required to create a unit surface area of a material in vacuum is called surface free energy. The presence of free energy on the material surface is the result of the differences in the intermolecular forces acting on the surface molecules as compared to forces acting on the molecules which are in the bulk of the material. The molecules at the surface of a material are attracted towards the bulk of the material. This can be explained by the example of a water droplet. A falling water droplet from a tap take a spherical shape (minimum surface area shape) as the molecules from the surface are attracted inside the bulk of the liquid until the minimum possible surface area is achieved. The molecules at the surface thus have more energy due to the difference in the intermolecular forces between the surface and the bulk of the material. This excessive energy of the surface molecule is referred as surface free energy.

Work is required to be done to bring these molecules to the surface and increase the surface area of the material.

The term ‘free energy’ is used as it is related to the Gibbs free energy or work done, but not the total energy or enthalpy of the system (Bhasin 2007). In case of liquids, the term ‘surface free energy’ or ‘surface energy’ is replaced by ‘surface tension’. In case of solids, the reduction in surface area is not evident because of the limited mobility of the solid molecules, but the intermolecular forces still exist.

According to the Good, Van Oss and Chaudhury approach, the forces that are responsible for surface energy are divided into two components namely, non-polar (dispersive) component and polar component. The dispersive forces are termed as Lifshitz-van der Waals forces and the polar forces are divided into Lewis acid and Lewis base types of forces (Van Oss et al. 1988). The surface energy, γ, can be given by the following equation:

$$\gamma = \gamma^{LW} + \gamma^{AB} \tag{2.2}$$

Where;

γ^{LW} = Lifshitz–van der Waals component of the surface energy

γ^{AB} = Acid-base component.

A molecule is formed as a result of chemical bonding of atoms. In polar molecules, the arrangement of atoms is such that one end of the molecule possesses a positive charge while the other end has a negative charge. This is because of uneven distribution of electrons in the orbits or shells which makes them unstable and provides them with a tendency to interact with other polar molecules. While in nonpolar molecules, the electrons are distributed more symmetrically and thus all the charges cancel out each other. Lifshitz-van der Waals forces are considered to be the non-polar or dispersive forces. These are believed to consist of three main components; London dispersion forces, Debye induction forces and Keesom orientation forces (Rieke 1997). All these forces represent different types of dipole/induced dipole interactions between neighbouring molecules and electronic shells. These forces between molecules in a material are viewed as residual electrostatic forces.

As water and aggregates are polar substances while bitumen is a non-polar material. Thus, water has a greater attraction for aggregate surfaces than bitumen. Also, most liquids spread on a high energy surface like aggregates.

In polar components, the Acid-Base interactions include electron acceptor-electron donor interactions & hydrogen bonding. According to acid-base theory (Van Oss et al. 1988), the acid-base component can be given as:

$$\gamma^{AB} = 2\sqrt{\gamma^{+}\gamma^{-}} \tag{2.3}$$

Where;

γ^{+} = Lewis acid component of surface interaction

γ^{-} = Lewis base component of surface interaction

Surface free energy dimension corresponds to that of force per unit area i.e. mJ/m^2 or erg/cm^2. In pavement engineering, we are concerned about the surface energies of bitumen and aggregate, and the effect of these on bitumen-aggregate adhesion in dry and wet conditions.

2.14.2 General Principles

Several theories have been presented in the past in order to describe the adhesion forces between two materials. The thermodynamic theory is the most widely used theory to quantify adhesion between the two materials (Hefer 2005). The intermolecular forces at the interface of materials are related to the surface energies of the material. As the thermodynamic theory is a study of energy changes, the surface energy of a material is explained through Gibbs free energy. Gibbs free energy at constant temperature and pressure is given by the following equation:

$$\Delta G = \Delta H + T\Delta S \tag{2.4}$$

where;

ΔG = Gibbs free energy

ΔH = Enthalpy of the system

T = Temperature

ΔS = Entropy

Enthalpy is the heat content of a system. The tendency of a system is to convert potential energy into work and heat. The amount of heat given off or absorbed during this process is called enthalpy and the increase in the randomness of the system is called entropy. Entropy is also considered as the amount of heat loss when work is done.

Gibbs free energy can be described as the excess energy of the system associated with the surface and interface and is also referred to as free energy of adhesion or cohesion (Hefer 2005). As mentioned earlier, the surface energy is described as the reversible work required to create a unit surface area of the material. In a reversible process, the maximum useful work that can be obtained from a system is equal to the change in Gibbs free energy with a minus sign. So, the work of adhesion can be given by the following equation.

$$W^a = -\Delta G^a \tag{2.5}$$

In general, for stability, a system can move towards a reduction of energy. When two materials are bonded strongly with each other they are in a stable condition and have a lower free energy and more energy is required to separate them (Barton 1997). In most of the industrial processes, mixtures of materials are used (as a bitumen-aggregate combination) rather than the single compounds. At the interface between two different materials, the adhesive properties are very important for the existence of that interface.

Adhesion between a solid and liquid can be given by Dupre's equation;

$$W_{SL} = \gamma_{SA} + \gamma_{LA} - \gamma_{SL} \tag{2.6}$$

Where S, L and A represents solid, liquid, and air respectively.

When two different types of materials/mediums are involved then surface energy can be referred as the Work of Adhesion while for identical or the same material the energy becomes the Work of Cohesion.

The work of cohesion for a material can be given by the following relation:

$$\gamma_1 = \frac{1}{2} W_{11} \tag{2.7}$$

$$W_{11} = 2\gamma_1 \tag{2.8}$$

where;

γ_1 = Surface Energy of the material

W_{11} = Work of Cohesion

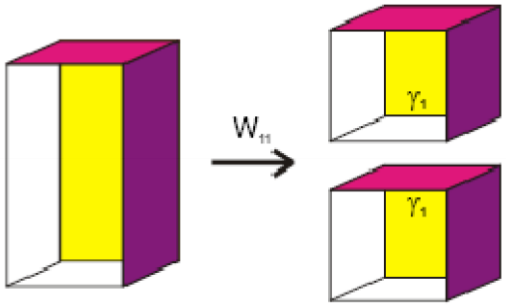


Figure 2.16: Work of Cohesion (Hansen 2004)

When two different materials are separated, the energy required to increase the interfacial area between the two materials is given by:

$$\gamma_{12} = \gamma_1 + \gamma_2 - W_{12} \tag{2.9}$$

The work of adhesion, W_{12}, can be given by the following relationship:

$$W_{12} = \gamma_1 + \gamma_2 - \gamma_{12} \qquad \text{(Dupre's equation)} \tag{2.10}$$

where;

γ_1 = surface energy of material 1

γ_2 = surface energy of material 2

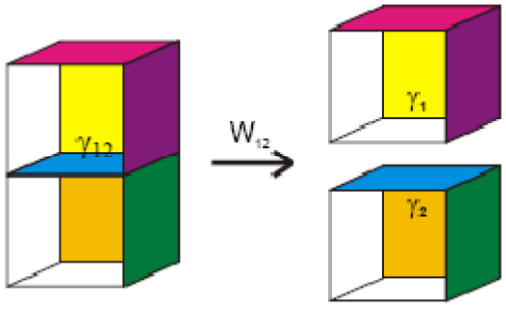


Figure 2.17: Work of Adhesion (Hansen 2004)

2.14.3 Bitumen-Aggregate Adhesion and Moisture Susceptibility

Moisture damage depends on the interaction of surface energy components of aggregate, bitumen, and water. The adhesive and cohesive bond strength of a bitumen aggregate system both in presence of water and without the presence of water on the interface can be calculated using the surface energy concept.

As mentioned earlier, according to Van Oss-Chaudhury-Good theory the surface free energy of a liquid or solid is composed of the Lifshitz van der Waals (LW) and Lewis acid/base (AB) forces and is given as:

$$\gamma = \gamma^{LW} + \gamma^{AB} \tag{2.11}$$

The acid/base components are further separated into Lewis acid, γ^+, and Lewis base, γ^-, components.

$$\gamma^{AB} = 2\sqrt{\gamma^+\gamma^-} \tag{2.12}$$

The bond strength of a material (binder or aggregate; in our case) is the combination of these Lifshitz-van der Waals (non-polar) and Lewis acid/base forces and the total bond strength can be given as:

$$\Delta G^c = -2\gamma = \Delta G^{cLW} + \Delta G^{cAB} \tag{2.13}$$

where;

ΔG^c = total bond strength, Gibbs free energy of the material

ΔG^{cLW} = Non-polar, Lifshitz-van der Waals component

ΔG^{cAB} = Polar, Acid-Base component

The Gibbs free energy of adhesion can be defined in the same manner for both the two-component and three-component systems.

$$\Delta G_{12}^a = \gamma_{12} - \gamma_1 - \gamma_2 = \Delta G_{12}^{ad} + \Delta G_{12}^{ap} \tag{2.14}$$

or

$$\Delta G_{12}^a = \gamma_{12} - \gamma_1 - \gamma_2 = \Delta G_{12}^{aLW} + \Delta G_{12}^{aAB} \tag{2.15}$$

Using the same assumption of the additivity of surface energy components (forces) proposed by Fowkes and then by Owens and Wendt, the Berthelet geometric means for the determination of work of adhesion for dispersive and polar interactions can be expressed as:

$$W_{12} = -\Delta G_{12}^{a} = 2\left(\gamma_1^d \gamma_2^d\right)^{\frac{1}{2}} + 2\left(\gamma_1^p \gamma_2^p\right)^{\frac{1}{2}} \tag{2.16}$$

The Berthelot geometric mean rule can also be used to calculate the Lifshitz-van der Waals component of surface free energy in terms of work of adhesion or free energy of adhesion as follows (Van Oss et al., 1988):

$$W_{12} = -\Delta G_{12}^{aLW} = 2\sqrt{\gamma_1^{LW} \gamma_2^{LW}} \tag{2.17}$$

However, the acid-base component cannot be treated in the same fashion as these interactions are specific and are only possible between interaction partners with complimentary acid-base properties. The relationship for the work of adhesion or free energy of adhesion for the AB component of surface free energy due to the complementarity of the acid-base interaction is therefore determined as (Van Oss et al. 1988):

$$W_{12}^{AB} = -\Delta G_{12}^{aAB} = 2\sqrt{\gamma_1^{+} \gamma_2^{-}} + 2\sqrt{\gamma_1^{-} \gamma_2^{+}} \tag{2.18}$$

The work of adhesion between two materials based on the three-component (acid-base) theory is therefore determined by the following equation:

$$W_{12} = 2\sqrt{\gamma_1^{LW} \gamma_2^{LW}} + 2\sqrt{\gamma_1^{+} \gamma_2^{-}} + 2\sqrt{\gamma_1^{-} \gamma_2^{+}} \tag{2.19}$$

The full version of the Young-Dupré equation when considering the adhesion between two materials, such as a liquid and a solid, is therefore given as:

$$\gamma_{Li}(1 + \cos\theta_i) = 2\sqrt{\gamma_S^{LW} \gamma_{Li}^{LW}} + 2\sqrt{\gamma_S^{-} \gamma_{Li}^{+}} + 2\sqrt{\gamma_S^{+} \gamma_{Li}^{-}} \tag{2.20}$$

Where γ_{Li} is the surface energy of the i^{th} liquid.

The work of adhesion between two materials, such as bitumen and aggregate can then be calculated by performing surface free energy calculations on both materials and then using

equation 2.19. If the value of adhesion is positive, it means that the two phases of the material tend to bind together with a lower magnitude dictating the likely mode of failure.

It is also very useful to know the surface energy of adhesion in the presence of water when considering moisture damage. For the general case, the free energy of adhesion for two different materials in contact with a third medium, ΔG^{a}_{132}, can be explained by the following equation:

$$W_{132} = -\Delta G^{a}_{132} = \gamma_{13} + \gamma_{23} - \gamma_{12} \tag{2.21}$$

Using the process proposed by Good and van Oss (1991); Van Oss et al. (1988), equation 2.22 can be used to calculate the adhesion between bitumen and aggregate in the presence of water, where subscribes 1, 2, and 3 represent bitumen, aggregate, and water, respectively.

$$\begin{aligned} W_{132} = {} & 2\gamma_3^{LW} + 2\sqrt{\gamma_1^{LW}\gamma_2^{LW}} - 2\sqrt{\gamma_1^{LW}\gamma_3^{LW}} - 2\sqrt{\gamma_2^{LW}\gamma_3^{LW}} \\ & + 4\sqrt{\gamma_3^{+}\gamma_3^{-}} - 2\sqrt{\gamma_3^{+}}(\sqrt{\gamma_1^{-}} + \sqrt{\gamma_2^{-}}) - 2\sqrt{\gamma_3^{-}}(\sqrt{\gamma_1^{+}} + \sqrt{\gamma_2^{+}}) \\ & + 2\sqrt{\gamma_1^{+}\gamma_2^{-}} + 2\sqrt{\gamma_1^{-}\gamma_2^{+}} \end{aligned} \tag{2.22}$$

The two bond energy parameters in the dry condition (equation 2.19) and for the bitumen-aggregate system in water (equation 2.22) are used to predict the moisture sensitivity of asphalt mixtures.

2.14.4 Intrinsic adhesion measurements

For the intrinsic adhesion measurement between the bitumen and aggregate, the surface energy of these two components (i.e. binder and aggregate) is calculated with the help of different techniques. Surface free energy is then used to calculate the work of adhesion in the dry and wet conditions with the help of the equations discussed in the previous section.

For the measurement of the surface free energy of the aggregates, dynamic vapours sorption (DVS) can be used. DVS machine measure any change in mass of the test sample as it absorbs the vapours of the water or any probe liquid in an inert carrier gas. Microbalance measures any change in the mass of the sample showing its adsorption or desorption characteristics (DVS-

Advantage 2007). Using adsorption isotherm, the surface energy of the aggregate sample is calculated which is based on the concentration of vapours adsorbed or desorbed by aggregates. The further details of the method and the test procedure are mentioned in Chapter 3.

Now for the bitumen, the surface free energy can be calculated with the help of dynamic contact angle measurement using Wilhelmy plate device. This device is capable of measuring the contact angle indirectly by immersing a glass plate of known dimensions into a liquid of known surface energy and calculates contact angle from the measured force (Adamson and Gast 1997). Again all the details of the method for measurement of the surface free energy of bitumen are presented in Chapter 3.

2.14.5 Practical work of adhesion

As discussed earlier, the destructive laboratory tests are used to define the term 'Practical Adhesion'. These tests may include tensile 'pull-off' or 'peel-off' tests. The fundamental adhesion at the interface and the response of the adhesive substrate, glue and their boundary region are well explained by this kind of adhesion phenomenon. There can be different failure approaches in the destructive laboratory tests namely adhesive, cohesive or mixture of the two (Packham 1992)

Peel test is one of the widely used test method for adhesion assessment of flexible laminates using tension testing machine. The unbound end of the specimen is clamped in the tension testing machine and a pull-off force is applied at a constant rate. Results are recorded automatically as a function of pull-off load versus distance peeled. It is a very quick way to understand the adhesion potential of two materials.

Techniques are also available for the evaluation of practical adhesion using pull-off methods. The Pneumatic Adhesion Tensile Testing Instrument (PATTI) is one of the test method to evaluate the bitumen-aggregate bond strength. The PATTI is comprised of a portable pneumatic adhesion tester, pressure hose, piston, reaction plate and a metal pull-off stub. For testing the piston is placed over the pull-out stub and the reaction plate is screwed onto it. Then, a pressure hose is used to introduce compressed air to the piston. During the test, a pulling force is applied to the specimen by the metal stub. The portable device displays the results in terms

of the pull-off tensile strength which is a measure of the cohesive or adhesive strength depending upon the type of failure.

Chapter 3: MATERIALS AND METHODS

3.1 Introduction:

This chapter describes in detail the materials used in this study. The details about some basic characteristics of these materials have been summarised. This chapter also includes the details of the testing programme for this study which has been divided into three further categories namely; component level testing, mixture level testing and adhesion testing. Each test technique used has been explained in details along with sample preparation and testing procedure. At the end, a summary is presented.

3.1.1 Aggregate

Four aggregate sources have been selected for this research including limestones, granite, basalt and greywacke. The basic properties of the aggregates used have been presented in Table 3:1

Table 3:1: Physical characteristics of crushed aggregate

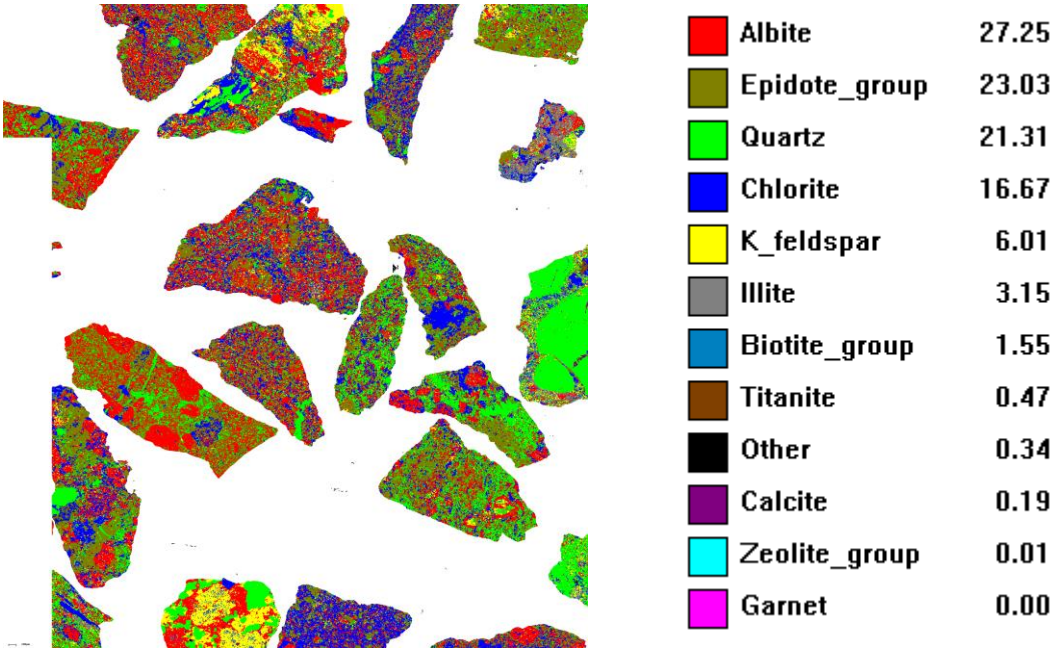
Aggregate Type	Source	Particle density (Mg/m^3)	Water absorption (%)
Granite	Bardon Hill	2.77	0.40
Limestone	Tunstead Quarry	2.70	1.20
Basalt	Duntilland Quarry	2.96	2.41
Greywacke	Haughmond Hill	2.80	0.90

To get an idea about the mineralogy of the different aggregates used in this study results from mineral liberation analyser (MLA) are presented in Figure 3:1 for the three aggregate types i.e. granite, limestone and basalt. The results from the greywacke were not available, so a discussion about it mineralogy, in general, has been included.

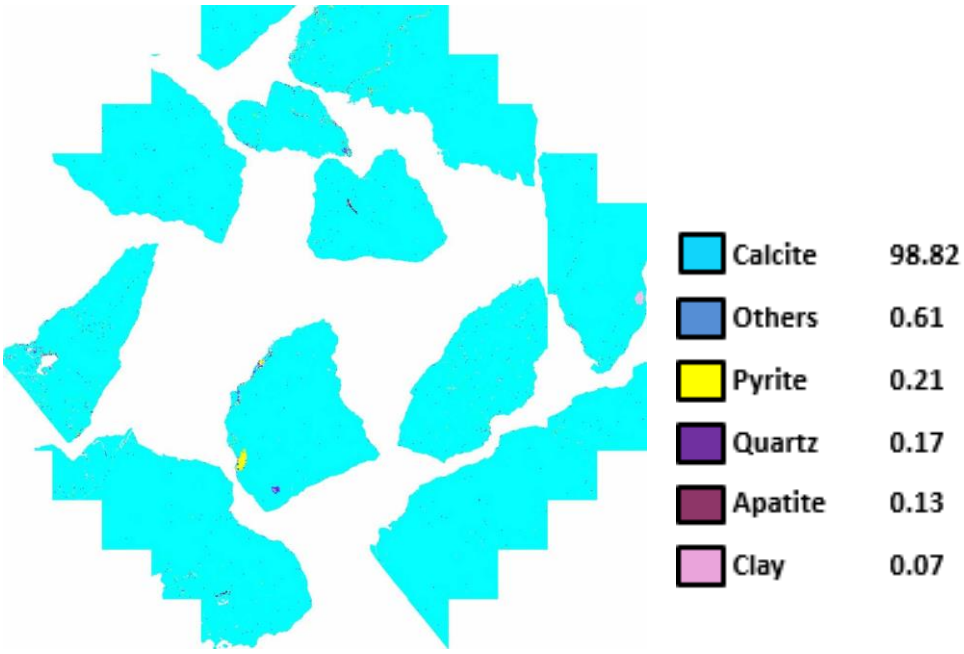
Granite is composed of more than ten different minerals with a major portion of Albite, Epidote, Quartz and Chlorite. On the other hand, the limestone has a very simple composition as about 98 % is Calcite. Basalt has also a complex mineralogical composition like granite and is composed of mainly Chlorite, Albite, Epidote and Calcite. The greywacke is a variety of

sandstone generally characterized by its hardness, dark colour, and poorly-sorted, angular grains of quartz, feldspar, and small rock fragments set in a compact, clay-fine matrix. (Dunford 2013)

Granite



Limestone



Basalt

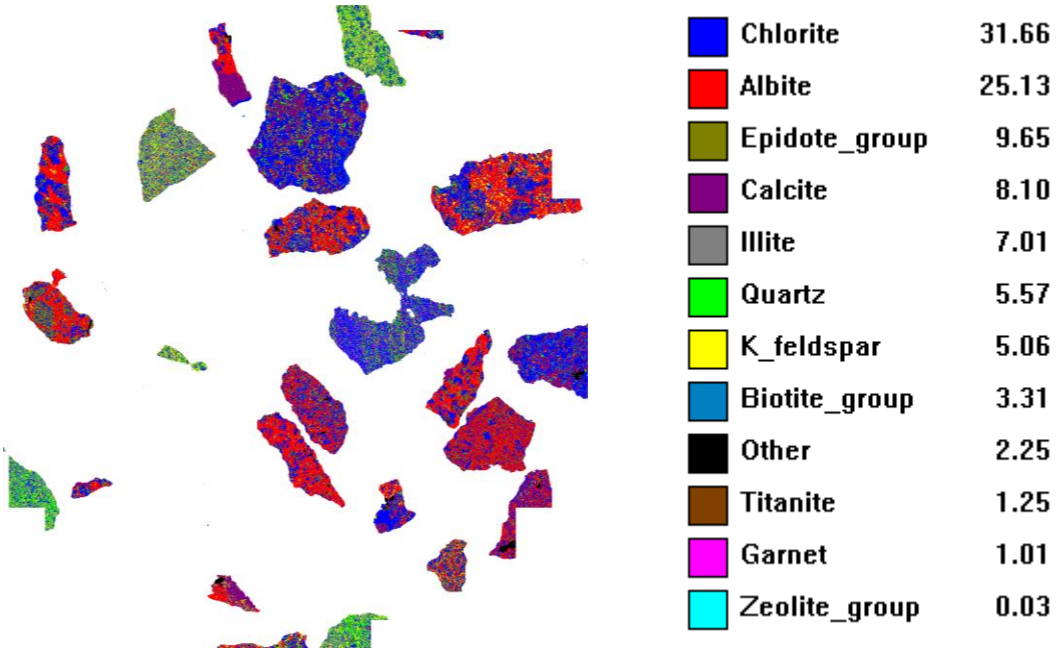


Figure 3:1: Mineral composition of granite*, limestone and Basalt* using MLA analysis

* Source: Zhang et al. (2015)

3.1.2 Filler

As filler type affects asphalt mixture properties significantly, five fillers have been identified to be used including limestone, granite, greywacke, basalt and hydrated lime (HL). As this study focuses on the effect of HL on the performance of asphalt mixture so four fillers i.e. limestone, granite, basalt and greywacke, are used with their parent aggregate type and HL are used as a replacement for certain percentages within the mastic or mixture as further explained in the Sections 3.1.3 and 3.1.4.

To qualify the effect of HL it can either be added in the bitumen to make a mastic or can be added to the aggregate to form a mixture. In this research both of these methods have been used. Addition of HL in the neat bitumen to make mastic can be justified by previous research as to better comprehend the properties of asphalt mixtures, numerous researchers have utilized intermediate materials, for example, mastics, i.e. mixes of just bitumen and filler, as a model framework. The thought behind this examination is that the material sticking together the aggregates inside the mixture isn't the bitumen only but is the bitumen mixed with the finest components of the mineral called ‘filler’ (Asphalt Task Force 2010). Now the addition of HL to the aggregates can be sub-divided into three categories (1) dry hydrated lime to the dry

aggregate (2) dry hydrated lime to the wet aggregates (3) HL slurry to the aggregates. Many studies show that method of HL addition to the aggregates do not affects the mixture's moisture sensitivity (McCann and Sebaaly 2006). In this research, for the mixture level testing, HL has been added as dry HL to the dry aggregates as this is the most convenient method which allows a good control of the HL dose.

3.1.3 Bitumen and Mastics

As discussed earlier, the testing programme has been divided into three types; the first type being component level testing, the second type is mixture level testing and third is adhesion testing. The detailed testing programme will be discussed in the Sections 3.3, 3.4 and 3.5 of this chapter. For the component level and adhesion testing, each aggregate type will be tested with four combinations of binders including one neat bitumen (40/60 pen) and three mastics. The properties of base bitumen used in this study has been shown in Table 3:2

Table 3:2: Properties of 40/60 bitumen used in the study

Property	Value	Standard
Specific Gravity	1.03	BS 2000-549 (2007)
Penetration Index at 25°C	45	BS EN 1426 (2000)
Softening Point (°C)	50	BS 2000-58 (2007)

In total 12 different types of mastics have been used in combination with four types of aggregates. The notation, composition and the type of the aggregate with which these mastics have been used for a range of testing techniques are presented in Table 3:3.

Table 3:3: Summary of mastics notation, composition and type of aggregate used in combination

Serial No.	Mastic Notation	Composition	Used in combination with Aggregate type
1	50% G	50% granite filler + 50% 40/60 pen neat bitumen	Granite
2	40% G + 10% HL	40% granite filler + 10% Hydrated lime + 50% 40/60 pen neat bitumen	Granite
3	30% G + 20% HL	30% granite filler + 20% Hydrated lime + 50% 40/60 pen neat bitumen	Granite
4	50% LS	50% limestone filler + 50% 40/60 pen neat bitumen	Limestone
5	40% LS + 10% HL	40% limestone filler + 10% Hydrated lime + 50% 40/60 pen neat bitumen	Limestone
6	30% LS + 20% HL	30% limestone filler + 20% Hydrated lime + 50% 40/60 pen neat bitumen	Limestone
7	50% B	50% basalt filler + 50% 40/60 pen neat bitumen	Basalt
8	40% B + 10% HL	40% basalt filler + 10% Hydrated lime + 50% 40/60 pen neat bitumen	Basalt
9	30% B + 20% HL	30% basalt filler + 20% Hydrated lime + 50% 40/60 pen neat bitumen	Basalt
10	50% GW	50% greywacke filler + 50% 40/60 pen neat bitumen	Greywacke
11	40% GW+ 10% HL	40% greywacke filler + 10% Hydrated lime + 50% 40/60 pen neat bitumen	Greywacke
12	30% GW+ 20% HL	30% greywacke filler + 20% Hydrated lime + 50% 40/60 pen neat bitumen	Greywacke

3.1.4 Asphalt Mixture Variables

For the mixture level testing, 14 mm close graded surface course in accordance with BS EN 13108-1 (2016) has been selected. The gradation curve has been plotted in Figure 3:2 for all the four aggregate types used with maximum and minimum limits as per BS 4987-1 (2005). Different variables for mixture level testing are as follows:

- Binder type (one): 40/60 pen
- Binder content (one): 5% by mass
- Air void content (range): 8-10% air voids
- Filler content (one, with three compositions): 5% by mass

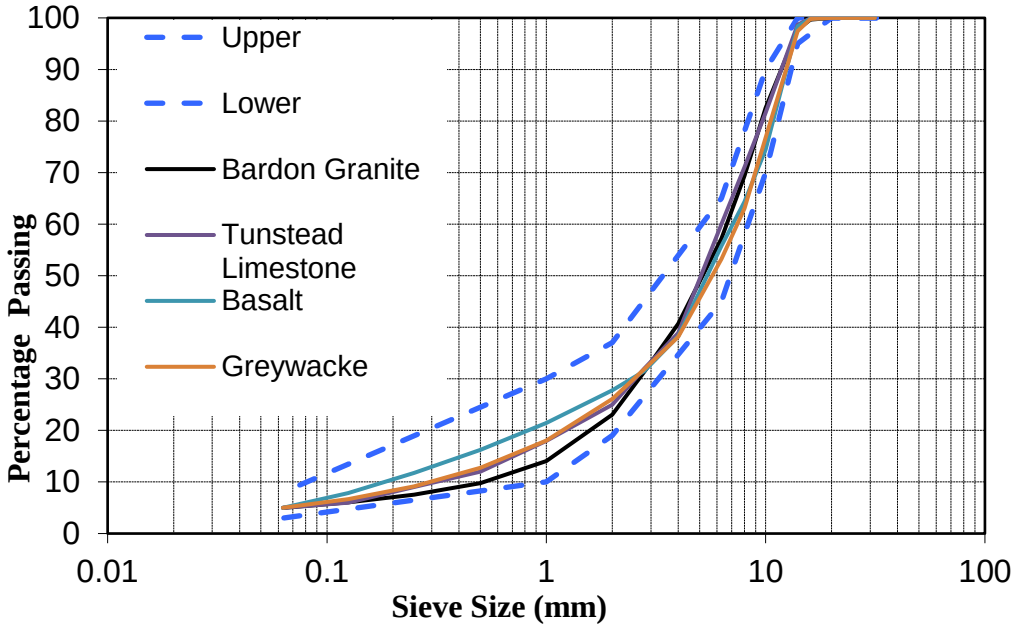


Figure 3:2: Gradation curve for granite, limestone, basalt and greywacke with upper and lower limits

As discussed earlier filler content has been fixed to 5% throughout this study and three different compositions have been used for each aggregate type. Composition 1 is composed of 0 percent HL with 5 percent respective filler depending upon the main aggregate source in the mixture. For example, if the main source is granite, composition 1 will be 0 percent HL plus 5 percent granite filler. Same principle will be used in case of limestone, basalt and greywacke aggregates. On the same lines, composition 2 will consist of 1 percent HL and 4 percent of the parent filler. And similarly, composition 3 will be made up of 2 percent HL and 3 percent of either granite, limestone, basalt or greywacke filler.

This means that HL will be used as a replacement for the main filler content and not as additional filler. The idea behind this methodology is to evaluate the real effect of HL in the mixture because if HL is used as additional filler in the mixture it will be the effect of a change in mixture volumetric rather than the effect of HL that will be more dominant.

3.2 Experimental programme

The experimental programme has been divided into three parts. The first part includes testing for moisture damage assessment on component level. The second part will cover assessment of moisture damage on mixture level and the third part is adhesion testing before and after moisture conditioning. A flow chart for the testing programme has been shown in Figure 3:3.

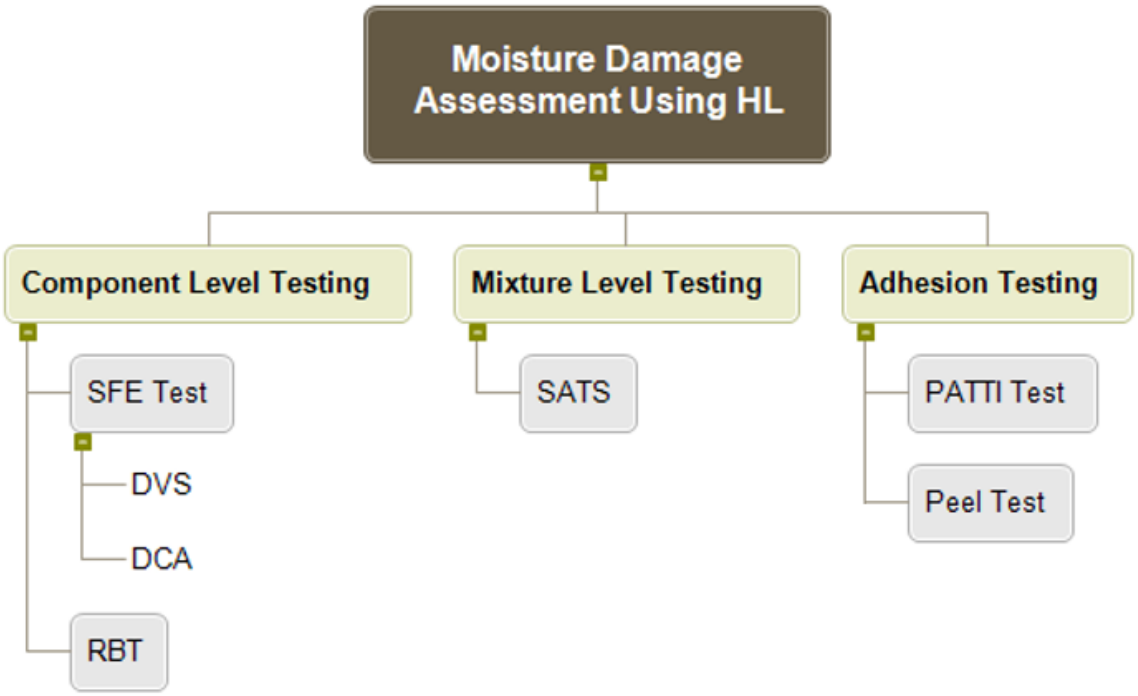


Figure 3:3: Testing Programme Flowchart

3.3 Component level Assessment

3.3.1 Rolling Bottle Test Method

The rolling bottle Test (RBT) has been conducted in accordance with BS EN 12697-11 (2012). It is a measure of affinity between aggregate and bitumen. This affinity is measured by visual inspection in terms of the degree of bitumen coating on loose bitumen coated aggregates after

the influence of mechanical stirring in water. Clean and fully dried aggregates particles are coated with approximately 0.1 mm thick layer of bitumen. These coated aggregates are then stored at room temperature for 12 to 64 hours before testing.

The steps involving the samples preparation for RBT test in accordance with BS EN 12697-11 have been summarised as follows;

1. Separate about 600 grams of aggregates passing 10 mm sieve and retaining on 6.3 mm sieve in accordance with BS EN 12697-2 (2012) to get a 6/10 mm fraction of the aggregates to be tested.
2. Now wash the aggregates to remove any dust or dirt.
3. Place the aggregates in a ventilated oven at 110 $\pm$ 5 ^{o}C temperature to get moisture free specimen.
4. A representative sample of bitumen or mastic should be selected in accordance with BS EN 58 (2012)
5. Now heat the aggregates and bitumen at mixing temperature BS EN 12697-35 (2016)
6. Mix the aggregates and binder intensively with the spatula to get a uniform coating of bitumen/mastic on the aggregate surface. Binder should completely coat the surface of aggregate. In case 100% coating was not achieved, discard the sample.
7. Spread in the mixed material immediately on a flat metal lid or on a silicon paper. Ensure that material should be loosely spread as individual particles.
8. Leave the mixed material on the metal lid or silicon paper for 12-64 hours at ambient temperature (20 $\pm$ 5 ^{o}C). Avoid direct sunlight exposure and also dust contamination.
9. Separate the coated material into three parts, 150 $\pm$ 2 grams each portion.
10. Mark the bottles with the sample name and number and fill each bottle with de-ionised or distilled water to about 50% of its volume. Water temperature should be 5 $\pm$ 2 ^{o}C.
11. Now with the moist fingers, transfer the separated amount of 150 $\pm$ 2 grams in each bottle and fill the rest of the bottle to the shoulder level with de-ionized or distilled water having a temperature of 5 $\pm$ 2 ^{o}C.
12. Place a glass rod in each bottle as shown in Figure 3:4 and seal the bottle with a screw cap.



Figure 3:4: Rolling bottle test assembly

The step by step procedure for RBT has been listed as follows:

1. Set the rotation speed of the machine to 60 min^{-1} $\pm$ 10%.
2. Place the bottles on the rolling bottles machine and start rolling procedure.
3. The test should be performed at room temperature between 15^{o}C and 25^{o}C and avoid direct sunlight exposure.
4. Stop the test after 6 hours $\pm$ 15 minutes.
5. Pour the water from the bottles into a beaker.
6. Empty the aggregate from the bottles into a test bowl having a white background and fill the bowl with fresh de-ionised or distilled water. This helps in making a clear observation of the retained bitumen coating on the surface of loose aggregates.
7. Estimate the bitumen coating very carefully and round it to a nearest 5%. A lamp can be used to facilitate a clear observation.
8. Discard the water from the test bowl and return the aggregates to the test bottles. Now refill the bottles from the original water from the beaker, put glass rod back and seal them with screw caps again and continue rolling.
9. Repeat the steps 5-8 after a total rolling time of 24, 48 and 72 hours and record the coverage of the bitumen on the aggregates at these points.

3.3.2 Surface Free Energy Approach

Surface free energy (SFE) is defined as the amount of energy/work required to create a unit surface area of a material in vacuum. Theory of thermodynamics says that bonds having either adhesive or cohesive nature are related to surface energy characteristics of bitumen and aggregate directly. Using values of surface energy of bitumen and aggregates, adhesion bond

parameters, with and without water presence, can be effectively studied for different bitumen, aggregate and filler combinations (Cheng et al. 2002).

3.3.2.1 Surface energy of aggregates

For the calculation of SFE of aggregates, the Dynamic vapours sorption (DVS) measurement technique has been used.

The DVS machine measures any change in mass of the test sample as it absorbs the vapours of the water or any probe liquid in an inert carrier gas. The sample is hung to the microbalance through a pad. An inert gas having the probe vapours is passed through the sample at a defined speed and temperature. Microbalance measures any change in the mass of the sample showing its adsorption or desorption characteristics (DVS-Advantage 2007). Using adsorption isotherm, the surface energy of the aggregate sample is calculated which is based on the concentration of vapours adsorbed or desorbed by aggregates.

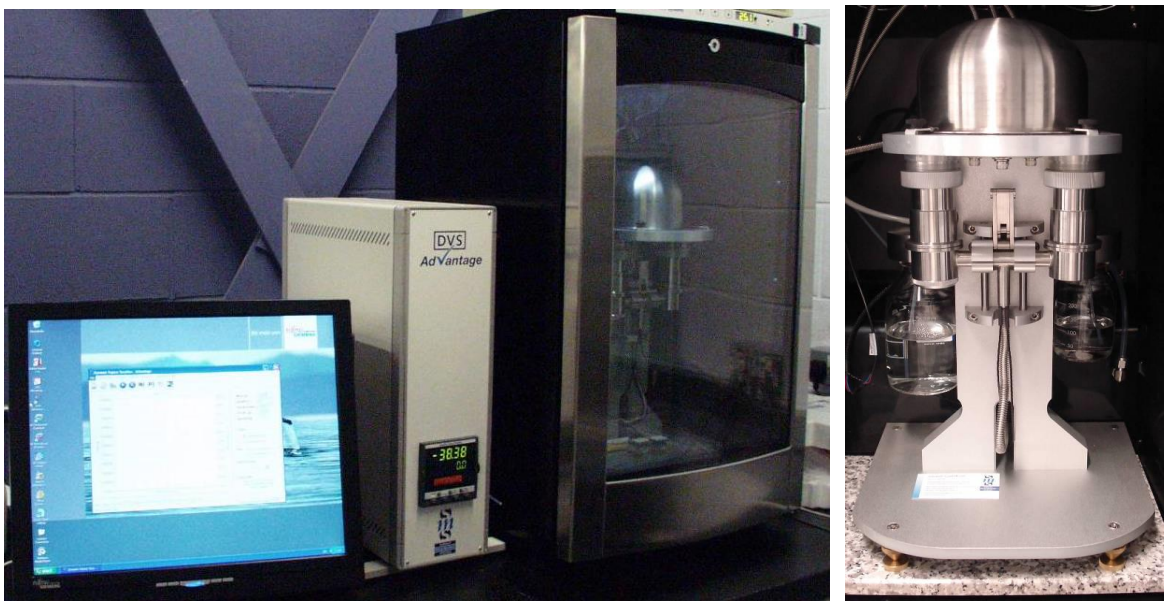


Figure 3:5: DVS Advantage System

Aggregates samples passing 5mm sieve and retaining on 2.36 mm sieve have been used in this particular research. Other fine fractions and fillers can also be tested with DVS advantage system. Aggregates are thoroughly cleaned and washed with water to remove any dirt or dust and are dried to constant mass. Sample is stored at room temperature in a glass vial with a cap on the top and are ready to be tested.

Dust free aggregates are once again dried at high temperature to remove any entrapped moisture in the sample chamber. Probe liquid vapours are introduced in the sorption chamber in a controlled and a gradual incremental manner to get required relative pressures. To obtain the adsorption isotherm, equilibrium mass of the vapours adsorbed into the said sample is recorded at each relative pressure. Once adsorption isotherm is calculated, it is then used to calculate the spreading pressure at equilibrium of the probe vapours on the aggregate samples. For at least three probe liquids, equilibrium spreading pressure is calculated and surface energy components of the aggregate sample are calculated with the help of equation of work of adhesion.

3.3.2.2 Surface energy of bitumen

Surface free energy of bitumen has been calculated by Dynamic contact angle (DCA) measurement using Wilhelmy Plate Device. When a liquid which comes in contact with a solid it either spreads on the surface due to attraction force or is repelled as a result of dispersion force. The contact angle 'θ' in Figure 3:6 is a thermodynamic quantity that describes this attraction or repulsion between solids and liquids at a point of contact.

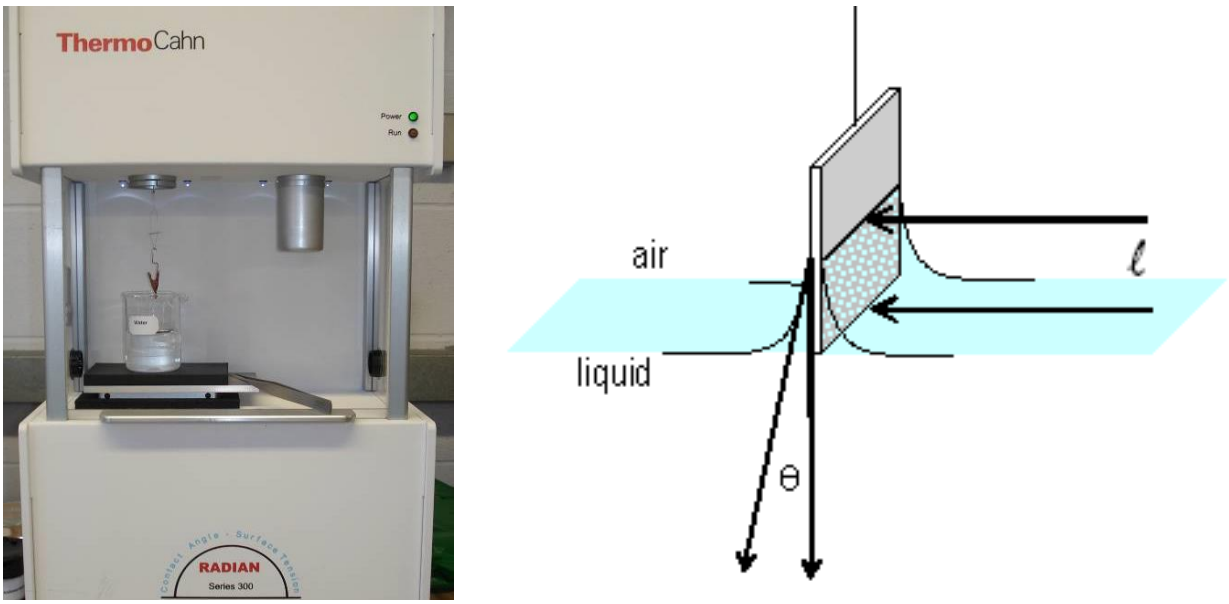


Figure 3:6: Contact Angle at Solid/Liquid/Vapour Contact Point

Wilhelmy plate method device is capable of measuring the contact angle indirectly by immersing a glass plate of known dimensions into a liquid of known surface energy and

calculates contact angle from the measured force (Adamson and Gast 1997). The device comprises of a microbalance of high sensitivity, movable stage which can be raised or lowered at the required speed to immerse or remove glass plate coated with bitumen to and from the probe liquid. This machine uses WinDCA software to record and analyse the results in the form of advancing and receding angle (Cahn WinDCA user manual 1999).

Steps involved in the sample preparation have been discussed as follows:

Microscope glass slides having dimensions 24 mm x 40 mm are used to coat the sample of bitumen or mastic to be tested.

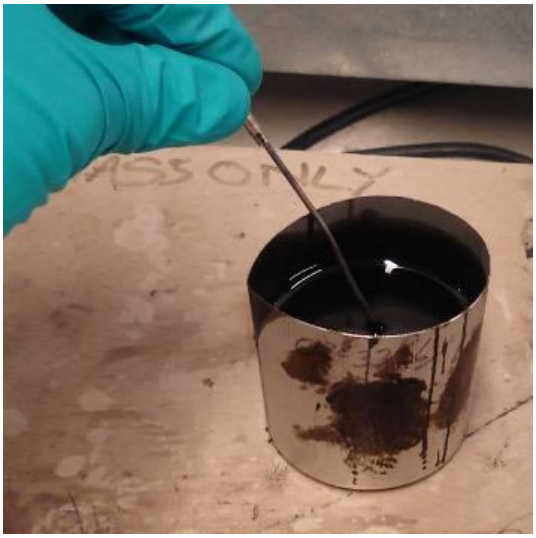
Bitumen or mastic sample is filled in a small tin having a mass of approximately 50 grams and placed in the oven for about 15 mins at mixing temperature. After that, it is removed from the oven and placed on the hot plate to maintain the required temperature. The sample is thoroughly mixed to get a uniform blend.

The glass slide is cleaned with acetone and then with distilled water. After that this glass slide is heated on a blue flame from both sides to remove any moisture or dust.

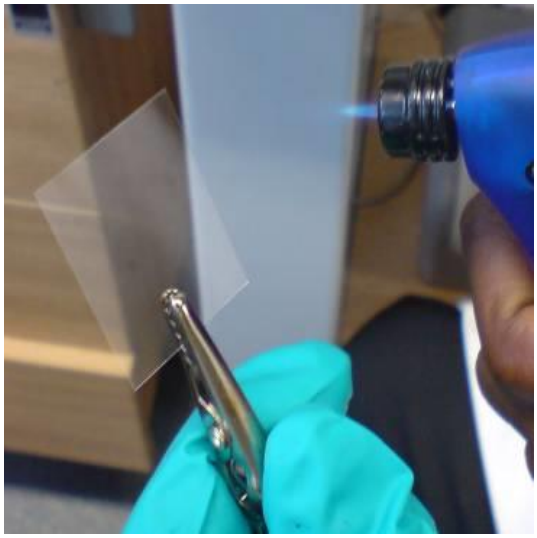
Once cleaned, the glass slide is dipped into the molten bitumen or mastic sample to get a 0.15mm coating and immediately inverted to allow the excessive sample to drain, leaving a uniform coated surface.

The prepared slides are placed in a slide holder and are kept in a desiccator for 24 hours before testing (Figure 3:7). For each probe liquid, 4 slides are prepared. The prepared sample after 24 hours of conditioning in desiccator are suspended on the microbalance of the DCA machine. The sample is immersed in the probe liquid at a certain speed and then recovered (removed) from it at the same speed. Both advancing and receding angles are measured from the force equilibrium conditions. The contact angle is calculated by the software accompanying the machine as discussed previously.

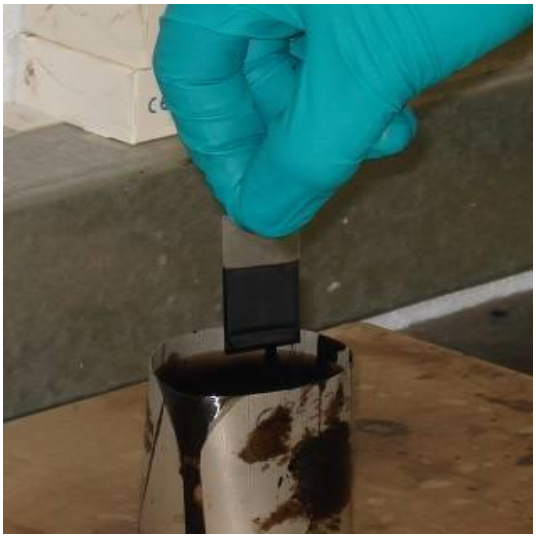
To get the surface energy of the bitumen or mastic, the contact angle with at least three probe liquids is required to solve the equations and hence determine all the components of surface energy.



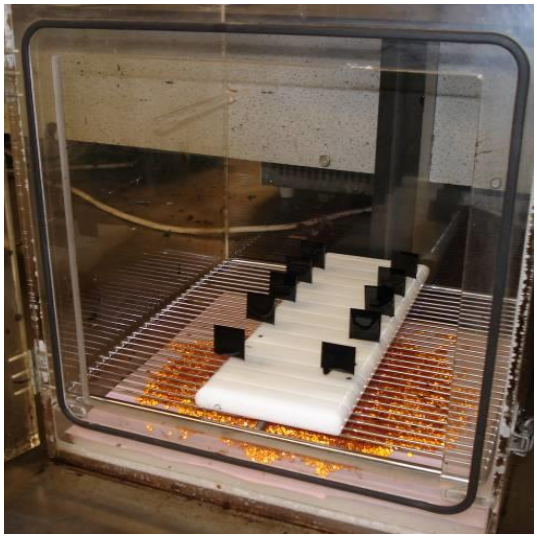
Binder in the tin



Passing glass side in blue flame



Dipping glass slide into binder



Sample kept in holder in a desiccator

Figure 3:7: Steps for sample preparation in the DCA test

3.4 Mixture level testing

3.4.1 Saturation ageing tensile stiffness (SATS) test

To study the moisture damage in the asphalt mixes the saturation ageing tensile stiffness (SATS) procedure was developed prior to mechanical testing (Collop et al. 2004). The application of the SATS test was limited to basic aggregates and hard binders initially. Later it

was optimised for a universal asphalt mix application (Grenfell et al. 2012). For this particular research modified SATS test protocols have been used.

SATS test setup is shown in Figure 3:8 All the main features have been discussed in details as follows:

- SATS test assembly consists of a strong vessel big enough to hold 5 identical asphalt cores having 100 mm diameter and 60 mm thickness.
- The vessel is capable of controlling test temperature and pressure.
- The cores are saturated with water before the test and are mounted on a porous tray as shown in Figure 3:8b.
- A known quantity of the distilled water is poured into the vessel so that the specimen at position P5 remain immersed during the conditioning period.
- Porous tray mounted with five specimens are placed in the vessel and 0.5 MPa pressure is applied at 85^{o}C temperature for 24 hours. This is followed by another 24 hours cooling period and after that vessel is de-pressurised and opened.
- Specimens are removed from the vessel and saturated surface dried (SSD) mass is noted.

a b c



Figure 3:8: SATS test configuration a) Test Setup b) Porous tray c) inside view of vessel

A summary of the step by step test procedure has been presented as follows:

- For each test run indirect tensile stiffness modulus (ITSM) for selected five unconditioned test specimens is calculated at 20^{o}C in accordance with BS EN 12697-26 (2012) using Nottingham Asphalt Tester (NAT). This stiffness is recorded as "initial stiffness".
- The dry mass of each specimen is calculated after testing for stiffness.
- After the calculation of dry weight, the samples are immersed in distilled water at a temperature of 20^{o}C and are vacuum saturated at 65kPa for about half an hour.
- The wet mass for each specimen is then measured and the initial saturation is calculated in terms of percentage saturation level.
- SATS vessel is filled with distilled water so that water can be maintained between level P4 and P5 after loading the samples in the vessel. Before placing the samples in the vessel, a temperature of 85^{o}C is maintained for about 2 hours.
- After maintaining the above mentioned temperature for two hours, samples mounted on the porous tray are introduced in the vessel and vessel is sealed from the top. Now the vessel is pressurised gradually to maintain a pressure of 0.5 MPa.
- Samples are kept in the vessel at 0.5 MPa pressure and 85^{o}C temperature for 24 hours.
- After 24 hours the temperature is dropped to 30^{o}C to let the sample to cool down for next 24 hours but the pressure is still maintained at 0.5 MPa.
- After a cooling period of 24 hours the vessel pressure is gradually released. Once at atmospheric pressure the vessel is opened and samples along with tray are removed from the vessel.
- Samples are surface dried and the SSD determined. Percentage saturation is calculated using SSD weight and referred to as "retained saturation"
- Samples are conditioned at 20^{o}C for at least four hours and are tested for Indirect tensile stiffness modulus (ITSM) again. This stiffness after SATS conditioning is referred to as "final stiffness".
- The ratio is of initial stiffness and final stiffness is calculated which is named as Retained stiffness modulus".

3.5 Adhesion Testing

3.5.1 Pneumatic Adhesion Tensile Testing Instrument (PATTI) test

PATTI test is used to evaluate the bond between aggregate and bitumen/mastic in terms of fracture or tensile strength. This could be the cohesive bond strength of adhesive depending on the type of failure. PATTI was developed by the National Institute of Standards and Technology (NIST). The full test setup is shown in Figure 3:9 PATTI is used to calculate the tensile strength which gives an indication of bond strength between aggregate and binder. The camera with the setup is used to capture the image of the failure surface.

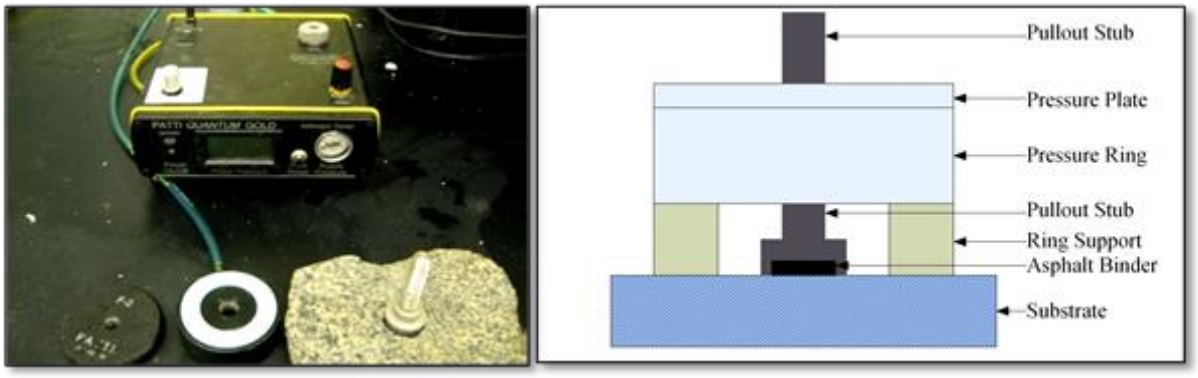


Figure 3:9: PATTI test setup and apparatus details

Aggregate substrate is prepared by wet cutting the boulders into required shape and size using a diamond-edged saw cutter. These aggregate substrates are then washed to remove any dirt or dust followed by drying to a constant mass.

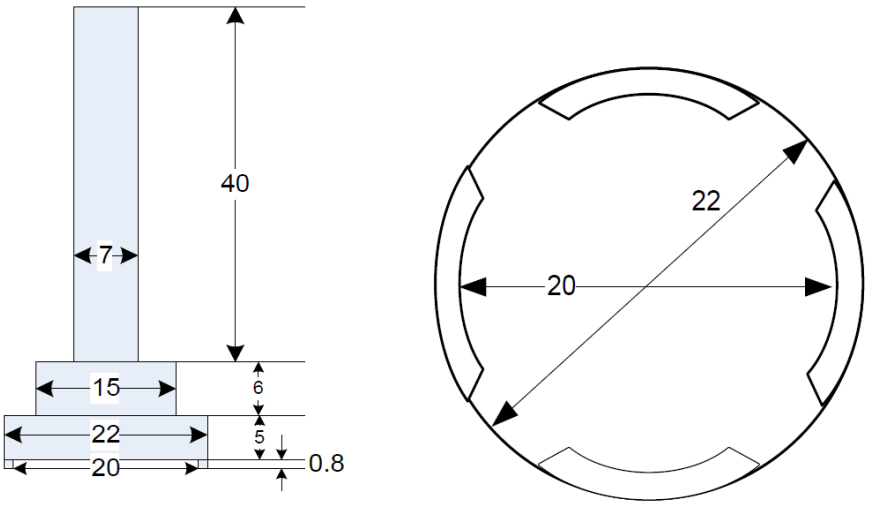


Figure 3:10: Pull-stub front elevation and bottom view (Units in millimetres)

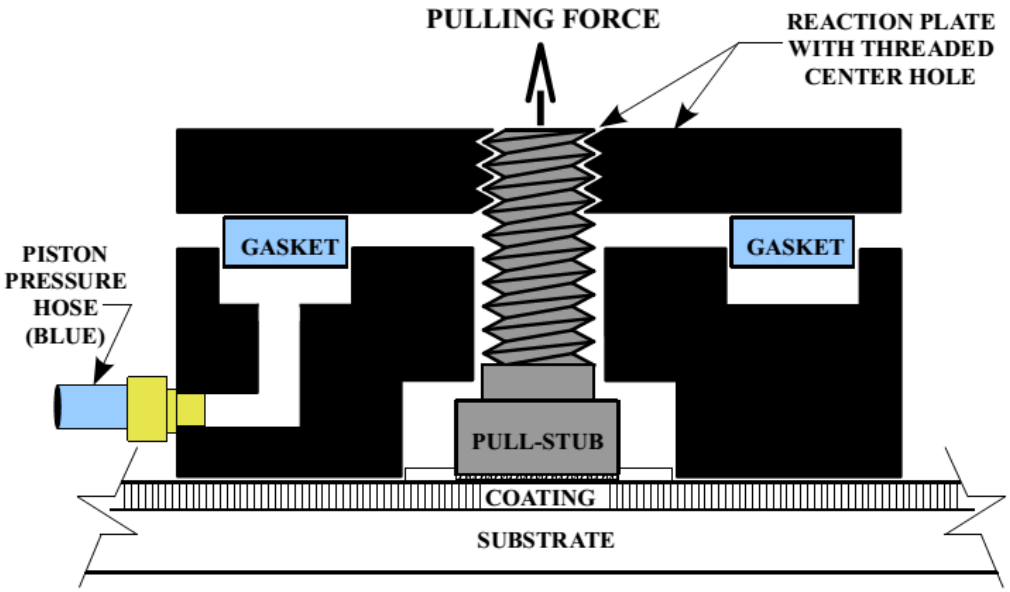


Figure 3:11: Cross-sectional view of PATTI test piston and pull-stub (Quantom 2002)

The aggregate substrates are heated at about 70^{o}C for one hour before testing. In parallel bitumen/mastic samples are also heated at 150-180^{o}C

The liquid bitumen/mastic is then poured on the surface of the substrate and the preheated stub is placed on the top with a gentle pressure.

With the applied vertical pressure, the excessive bitumen/mastic flows out through the vertical channels of the stub as shown in Figure 3:10. This leaves a constant thickness of 0.8mm bitumen film inside the pull off stub.

The sample is allowed to cool for at least four hours and then the excessive bitumen/mastic is trimmed using a heated knife or any other suitable sharp tool.

A piston is placed on the top of the prepared sample and a reaction plate is screwed on the top of the piston as shown in Figure 3:11. Pressure applied by PATTI is transmitted to the piston and an airtight seal is formed between gasket of piston and surface of aggregate. A constant rate of pressure is applied through PATTI using a control valve. Data is recorded by the software in the form of applied tensile pressure against time.

The maximum pressure which separates the bitumen and aggregate surfaces is recorded by the software and Peak tensile strength is calculated using the following equation which is calculated by in-built PATTI quantum gold software.

$$T_{po} = ((P_b \times A_g) - C)/A_{ps} \quad (3.1)$$

Where:

T_{po} = Pull-off tensile strength (kPa)

P_b = Burst pressure (kPa)

A_g = Contact area of gasket with relation plate (m^2)

C = Piston constant (kN)

A_{ps} = Area of pull-stub (m^2)

A sample image of the result recorded by the PATTI software is shown in Figure 3:12. It gives all the required information including Peak tensile strength, Units, size of pull-stub Piston size, image of the aggregate sample after test and much more.

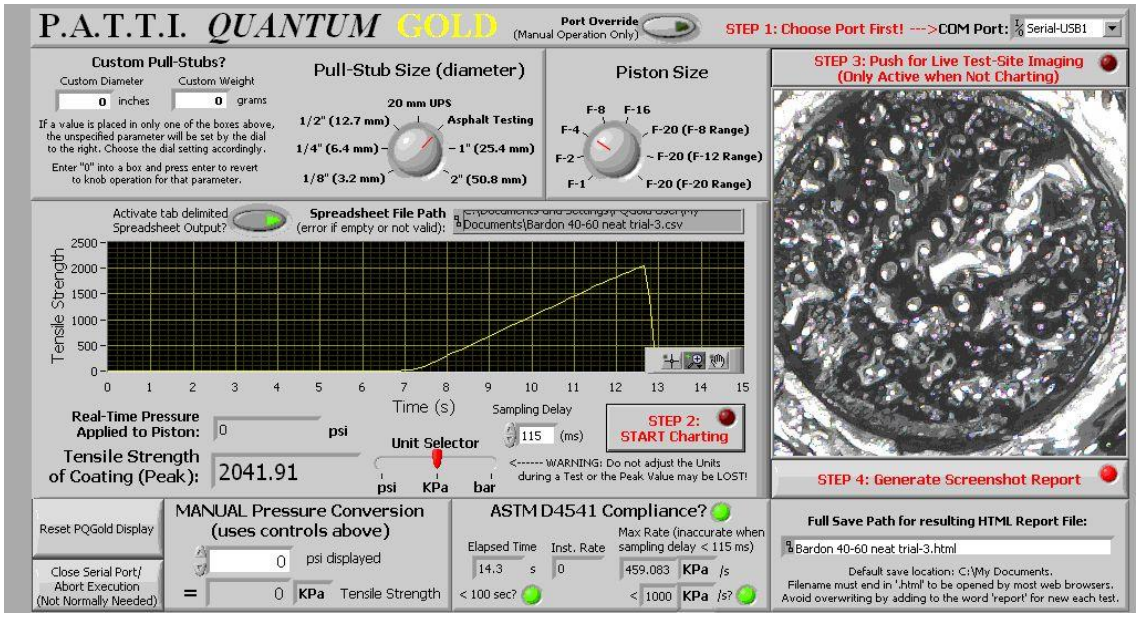


Figure 3:12: Sample output sheet from PATTI software

3.5.2 Peel test

The peel test (ASTM D6862 - 11 2016) is one of the most widely used test method to measure the adhesion of a range of bound joints and laminates using a tension testing machine. The

unbound end of the specimen is clamped in the tension testing machine and a pull-off force is applied at a constant rate. Results are recorded automatically as a function of pull-off load versus distance peeled. It is a quick way to understand the adhesion potential between two materials.

Test assembly consists of a tensile testing machine capable of applying a tensile force at a constant rate of grip separation. This machine should be capable of measuring both force and extension at the same time.

A frictionless slide table is required to provide free flow and smooth motion of the sample during the test. The test assembly is shown in Figure 3:13.

The aggregate substrate is attached to the slide table with the help of clamps on both sides. During the test 90^{o} peel angle is maintained using a low friction, linear bearing system.

Aggregate substrates used for the peel test should be rectangular in shape. The dimensions of the substrate are not defined rigidly. For this research 150 mm long 20 mm wide and 15-20 mm thick, aggregate substrates have been used. The peel arm should have a very good adhesion with the binder so that the fracture should take place within the binder itself or between binder and aggregate rather than taking place at the interface of peel arm and bitumen surface. For the peel arm aluminium foil sheet having a thickness of 0.2 mm has been found satisfactory. The peel arm should have a length of 50 mm more than the length of the aggregate substrate so that the free end can be grabbed by the jaws of the Instron machine easily.



Figure 3:13: 90 Degree Peel Test assembly (Zhang et al. 2016)

A step by step procedure for the sample preparation in the peel test is as follows:

- The aggregate substrate after cutting to the required size is cleaned for any dust.
- The aggregates are heated at 50^{o}C to remove any moisture for 30 minutes. Aluminium peel arm is also heated at 150^{o}C for 10 mins. The bitumen or mastic is heated at 150-180^{o}C.
- On one end of the bonding surface of aggregate, a release film (PTFE) having dimensions 30 mm x 20 mm x 12.7 μm is placed.
- Five equally spaced, 0.25 mm thick wire separators are placed on the top of the aggregate substrate to maintain a uniform thickness.
- Liquid bitumen is applied uniformly on the surface of the substrate and preheated peel arm is placed on the top of bitumen or mastic layer.
- A gentle pressure is applied to the joints to maintain the thickness of 0.25 mm. the pressure should be uniformly distributed over the full surface.
- The sample is then allowed to cool down overnight to 20^{o}C. The extra bitumen and aluminium peel arm are trimmed with a sharp tool.

For each test combination, a minimum of three samples should be tested at 20^{o}C temperature and 50% relative humidity. The sample should be gripped on the top of the moving slide table as shown in Figure 3:13. The free end of the peel arm is gripped by the moving and sensing end of the machine. When machine moves the displacement of the movable end is equivalent to the fracture length for a 90^{o} Peel angle. For a medium penetration grade bitumen a speed of 10mm/min has been found satisfactory but if there is high inconsistency in the results or there is stick-slip behaviour noticed during testing the speed should be adjusted accordingly (Ahmad et al. 2011)



Figure 3:14: Stages during sample preparation of Peel test

The machine records the data in the form of peel force as a function of displacement to initiate and propagate peel fracture. A sample test output has been shown in the Figure 3:15. A minimum peel fracture of 50mm should be established. Peel fracture energy is calculated from peel force which is defined as the average of the peel force achieved over a length of propagation (minimum 50 mm). To get the information about the type of failure, photographs should be taken during and after the test.

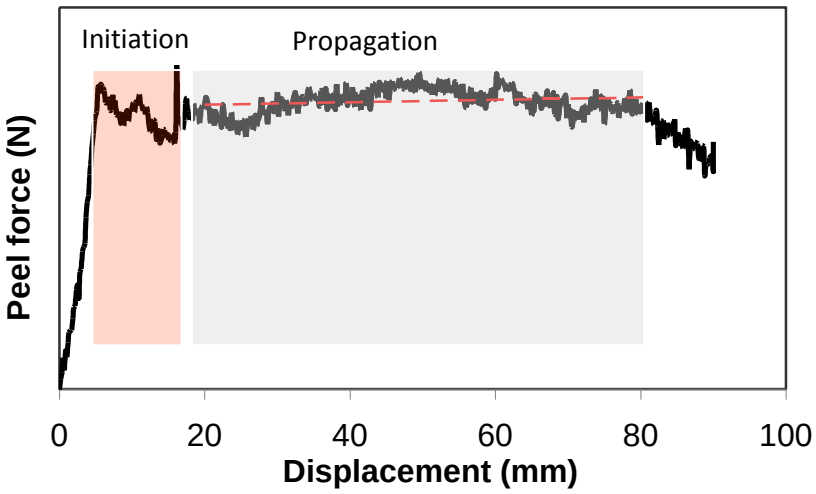


Figure 3:15: Sample Peel test graphical representation

3.6 Summary

The material used in this research includes four aggregate types along with their fillers, one bitumen type and hydrated lime which is used as a filler replacement. Four aggregate types include granite, limestone, basalt and greywacke. One neat bitumen having penetration grade 40/60 has been selected to be used for the project. The testing programme has been divided into three parts namely; component level testing, mixture level testing and adhesion testing. Component level testing involves surface energy testing and testing for the affinity between bitumen and aggregates using rolling bottle test. Surface energy for the aggregates is measured with the help dynamic vapours sorption (DVS) device and for the surface energy of bitumen/mastic, Dynamic contact angle (DCA) measurement are made using Wilhelmy Plate Device. For the assessment of moisture damage at mixture level, SATS test has been used for all four aggregates along with their studied combinations. For Adhesion testing, two tests have been used including PATTI tests and Peel Test. These techniques give an indication of the bond strength between binder and aggregates in dry and wet conditions.

Chapter 4: Component Level Testing

4.1 Introduction

As discussed previously, for the assessment of moisture damage resistance at component level two testing techniques have been used which includes rolling bottles test (RBT) and tests for surface free energy (SFE). The effect of hydrated lime (HL) has been studied by adding it inside the bitumen to make a mastic. With each aggregate type, four combinations of bitumen/mastic have been tested.

To get more reliable and repeatable results in RBT test, image analysis has been used along with eye observation for the calculation of percentage retained bitumen/mastic coating. A comparison has been made between the assessment made by eye and by image analysis for percentage retained bitumen/mastic coating. Surface energy for the aggregates is measured with the help Dynamic Vapours Sorption (DVS) device and for the surface energy of bitumen/mastic, Dynamic Contact Angle (DCA) measurement are made using Wilhelmy Plate Device. For the moisture damage assessment, work of adhesion and work of de-bonding has been calculated from the SFE of aggregates and bitumen/mastic. Furthermore, to represent the moisture susceptibility of a combination in a single value and to incorporate the effect of specific surface area, different energy ratios (ER) have also been calculated.

4.2 Moisture Damage assessment using RBT test

The rolling bottle test (RBT) has been conducted in accordance with BS EN 12697-11 (2012). It is a measure of affinity between aggregate and bitumen and also measures the susceptibility to stripping. The susceptibility to striping gives an indirect indication of the bond strength between the binder and the aggregate. This procedure can also be used to evaluate the effect of moisture for given binder and aggregate combinations as the loose coated aggregates are agitated in water for certain period of time. The results are usually measured by visual inspection in terms of the degree of bitumen coating on loose bitumen coated aggregates after the influence of mechanical stirring in water.

There are many techniques which are used to quantify the affinity between binder and aggregates. Airey and Choi (2002) in a state of the art report on moisture sensitivity test methods on pavement materials summarised 10 different methods on loose coated aggregates

to quantify this affinity in the presence of water. It is difficult to pick one of the best methods out of all. Jorgensen (2002) compared boiling water and rolling bottle tests during a round-robin study and found that the boiling water test can be used differentiate between good and bad combinations of binder and aggregates but on the other hand rolling bottle test can be used to rank these combination, and hence is more precise and reliable. Another comparative study was made by Liu et al. (2013) considering five empirical tests on loose mixtures for performance evaluation including static immersion test, rolling bottle test (RBT), boiling water test (BWT), total water immersion test and the ultrasonic method. Surface free energy (SFE) tests on aggregate and bitumen were also performed to confirm the performance with these empirical methods. RBT and BWT were found to be more sensitive procedures among the five empirical procedures in predicting moisture damage performance. Mixture ranking given by RBT was found to be in agreement with the results of SFE testing. Based on the findings of the previous studies on RBT test it can be said that RBT is one of the most efficient empirical procedures for moisture damage assessment.

Also to support the results of this testing technique some other techniques, such as SFE technique, SATS test, Peel test or some pull off techniques must be employed to get more realistic and precise findings.

4.2.1 Calculation for retained binder coating on aggregates

As per standard procedure, rolling bottle test to measure the affinity between the aggregates and bitumen measures the percentage coating on loose aggregates by visual inspection of two experienced operators and taking the average value of the two observations to get the final coating percentage to the nearest 5%. Sometimes it is not possible to get two operators at the required time or sometimes even if two are available there is a difference in the values suggested by two operators. And even sometimes people consider the results biased as it is based on visual inspection. Many researchers (Groenninger 2008; Grönniger et al. 2010; Källén et al. 2013; Morgenstern et al. 2010) have a view that as RBT results are based on visual inspection so are very subjective and is the main disadvantage of the test method. In some recent studies, arrangements have been made to replace eye observation with the digitally processed images using different software like Image J and Image Pro Plus. But the problem is that one has to make special arrangements for the image capturing including lamps for

lighting, a special platform for the aggregate to put on and capture etc.(Lantieri et al. 2017; Yuan et al. 2015).

An effort has been made to reduce the disadvantage of the method and to make RBT a more reliable and repeatable means of measuring the affinity between aggregate and binder in the presence of water, using image analysis techniques with no special setup, employing Matlab program.

Along with the observation with the naked eye, good quality pictures have also been taken for each combination used in this study. An internally produced code has been developed in Matlab software to analyse the images taken earlier. The full code has been presented in the APPENDIX A for further details. Each image has been divided into three components i.e. bitumen or mastic, aggregates and background.

To get the percentage coating on the aggregates, first of all, the background had to be eliminated from the image. Afterwards, the rest of the area was termed as total area. From this total area, the percentage of the coated aggregates with the bitumen/mastic was calculated and rounded to nearest 5%, which was the required value.

There were three variables in the code. One was the pixel's threshold limit for the aggregates coated with bitumen called bitumen upper limit (bul). The second was the pixel's threshold value for the aggregates without any bitumen coating called aggregate upper limit (aul) and the third was the threshold limit for the background to eliminate this from the full image.

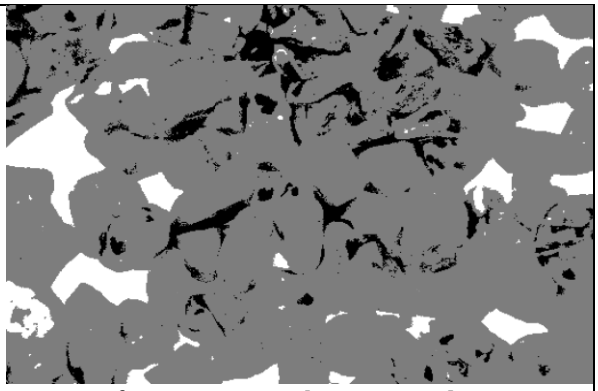
These three variables need changing with the different picture for analysis purposes as the picture quality may be different among all pictures. There are different factors contributing variable quality among the different pictures which include;

- The distance from which each picture was taken.
- The angle from which picture was taken
- Difference in the light in the room at different times
- Image resolution, if taken from multiple devices.

The ideal case would be to capture all the images at the same distance and to keep the capturing device as flat as possible on the top of the sample. Light in the room is also very important, it should not be too bright nor too dim to get best image and one should make an effort to keep light intensity as uniform as possible throughout the image capturing.



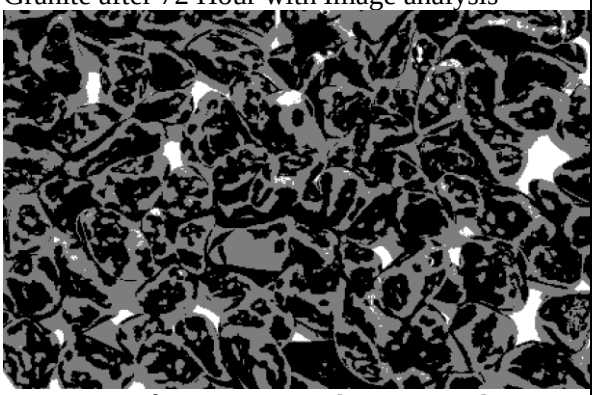
Granite after 72 Hour without Image analysis



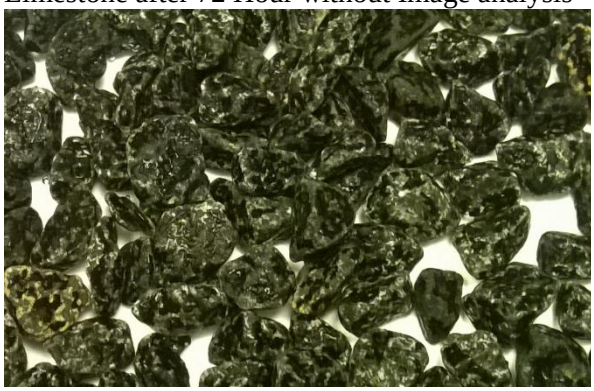
Granite after 72 Hour with Image analysis



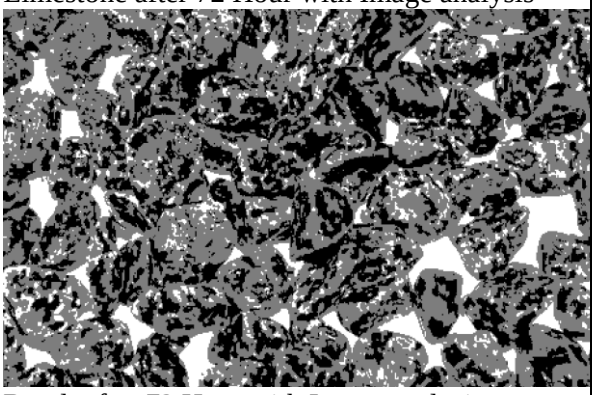
Limestone after 72 Hour without Image analysis



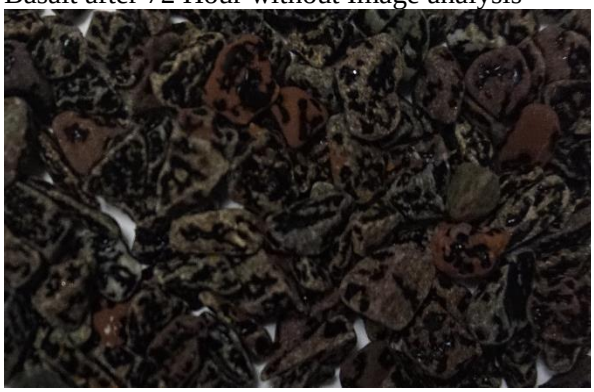
Limestone after 72 Hour with Image analysis



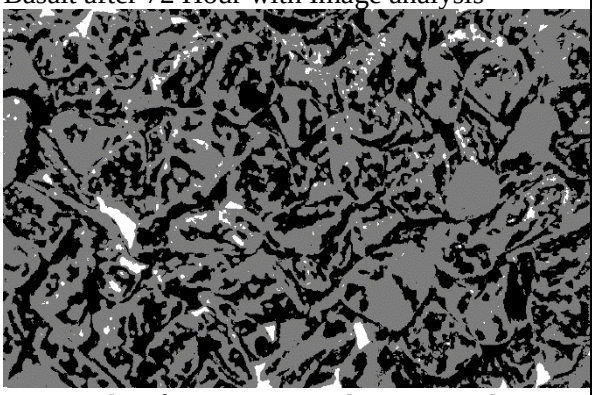
Basalt after 72 Hour without Image analysis



Basalt after 72 Hour with Image analysis



Greywacke after 72 Hour without Image analysis



Greywacke after 72 Hour with Image analysis

Figure 4:1: Summary of Images at 72 hours in RBT before and after analysing with Matlab

It is difficult to keep all the above conditions the same and sometimes it is not possible or practical and that is why a provision has been made in the code to accommodate these variabilities.

Keeping in mind all the above-mentioned precautions quality images have been taken after 6, 24, 48 and 72 hours of agitation in rolling bottles for all the aggregates and bitumen/mastic combinations. After the acquisition of quality images, percentage bitumen/mastic coating have been calculated using the code discussed above.

Images before and after processing with the Matlab program have been shown in Figure 4:1 to illustrate how the processed image will look like and how the background, bitumen coating and aggregate surfaces have been divided. These are the images for neat bitumen in combination with different types of aggregates used in this study, after 72 hours of agitation in RBT. The white portion of the processed picture represents background which has been excluded from the total area. The grey portion shows aggregates without any bitumen coating and the black portion is the bitumen-coated aggregates. A percentage area of the aggregates coated with bitumen has been recorded as a final result.

4.2.2 Material

Four aggregates types have been used along with 40-60 pen neat bitumen and three mastic combinations with each aggregate type. The details about all the combinations used are described in Table 3:3.

4.2.3 Results

The affinity between different aggregates and bitumen/mastic in the presence of moisture has been measured using RBT as discussed earlier. Each of the studied combination has been repeated twice and is found within 5% repeatability range. The allowable limits for the repeatability of the rolling bottles test as reported in the BS EN 12697-11:2012 are 20%, so the results of this research are well in range and can be considered to have a very good repeatability. The results for granite aggregates and different combinations of bitumen/mastic have been presented in Figure 4:2. From Figure 4:2, it can be evaluated that with time, a coating of binder has decreased significantly especially for the 40/60 bitumen and for the mastic containing 0% hydrated lime (50% G). For the mastics having 10 and 20% hydrated lime, it can be seen that

their retained percentage coating is considerably better than neat bitumen and the mastic with 0% HL. Although the mastic with 20% HL has shown slightly better results than the mastic with 10% HL, there is only a small difference between the two types.

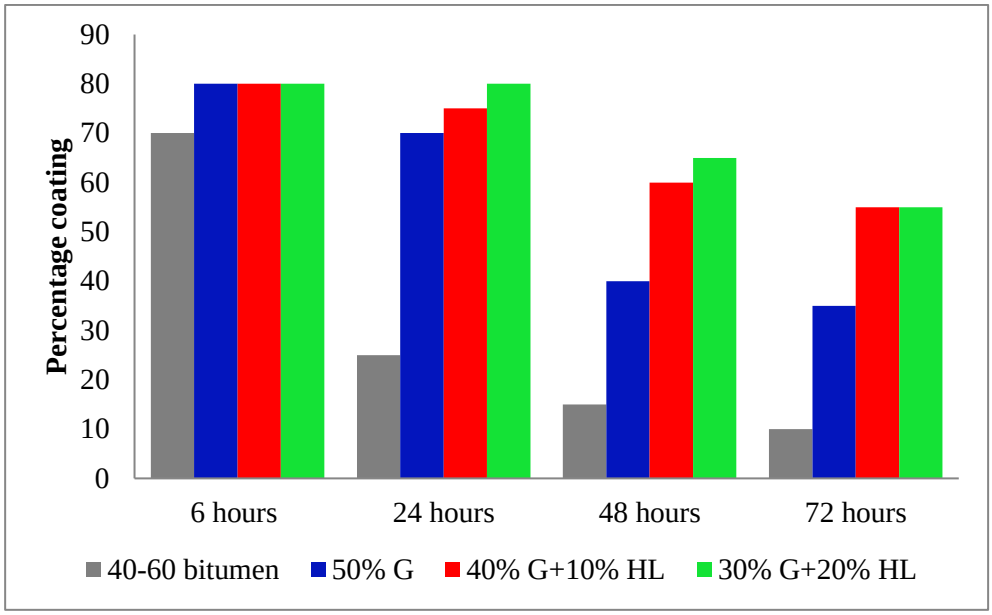


Figure 4:2: RBT test results for all combinations of Granite aggregates using Image analysis

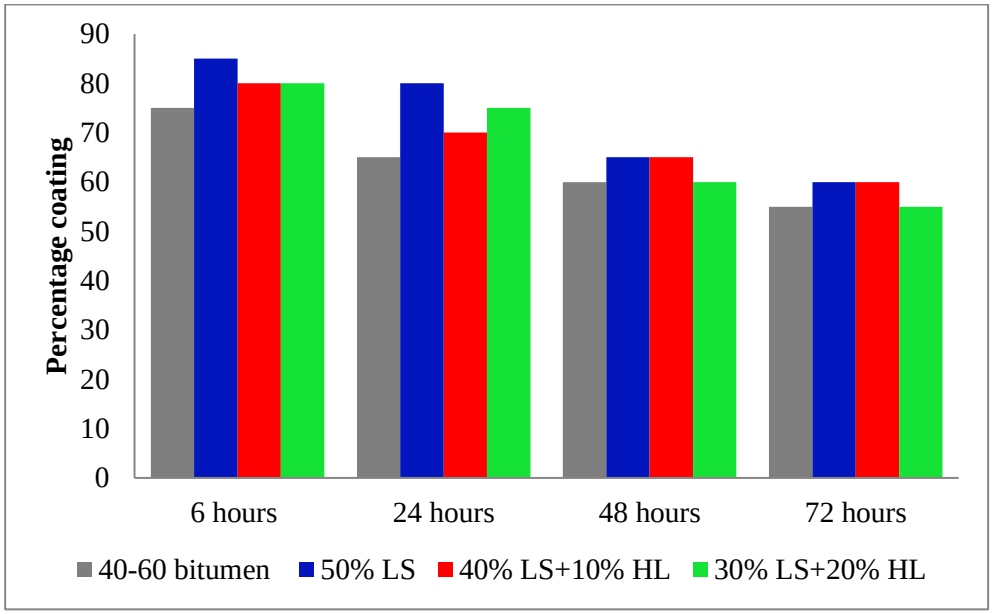


Figure 4:3: RBT test results for all combinations of Limestone aggregates using Image analysis

Similarly, from Figure 4:3 it can be seen that there is no significant difference between all the three mastics and 40/60 bitumen. In contrast to the Figure 4**:2**, the mastic having 0%HL (50%LS) performs slightly better than those containing 20% and 30% HL. There is a marginal difference between neat bitumen and all three mastics used in combination with limestone aggregate. Based on these facts it can be extracted that hydrated lime does not seem to be beneficial in case of limestone aggregate.

Now if we look at Figure 4:4 when basalt aggregate is used in combination with neat bitumen and three mastic types, the beneficial effects of HL are obvious, when it seems to improve the affinity between basalt aggregate and mastic. Again the performance of neat bitumen and the mastic with 0% HL (50% Basalt) looks similar to each other but with the addition of 10% and 20% HL, the coating percentage remained at a relatively higher value compared to neat bitumen and the mastic having 0% HL. There is another thing worth noting is that there is really no difference found between the performance of the mastics having 10% HL and 20% HL. In fact, both are giving good results in terms of higher affinity compared to the mastic without HL.

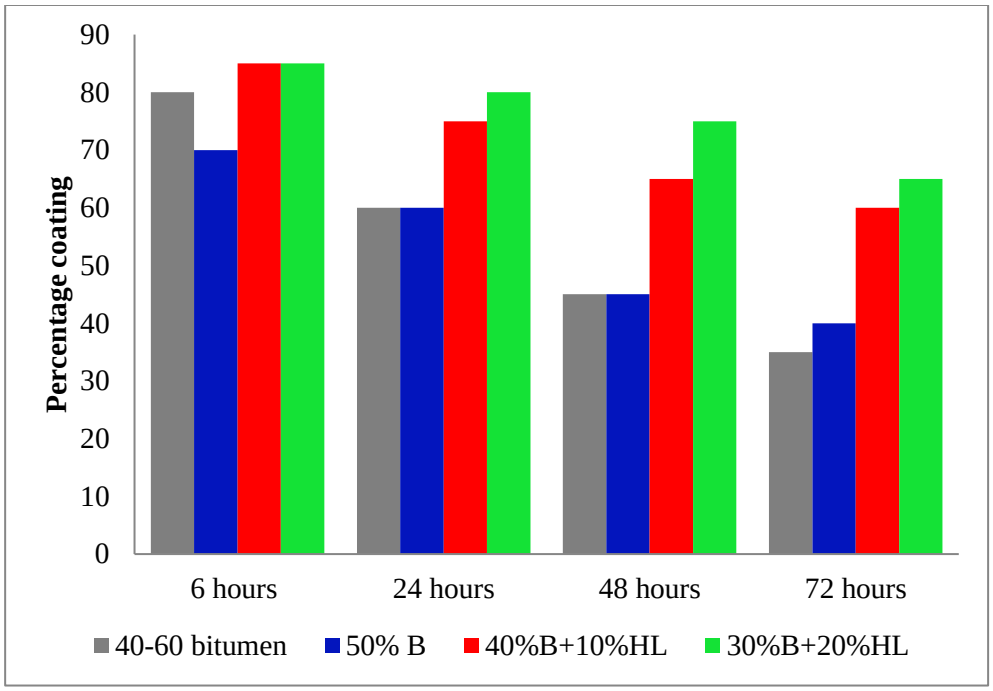


Figure 4:4: RBT test results for all combinations of Basalt aggregates using Image analysis

The results for the greywacke aggregates have been summarised in Figure 4:5 and again the beneficial effects of HL have been noted. Although the results are not as discriminatory as they

were in the case of granite aggregates, there still has been a positive effect of HL addition noted clearly with the addition of 10% and 20% HL in comparison with neat bitumen and mastic with 0% HL.

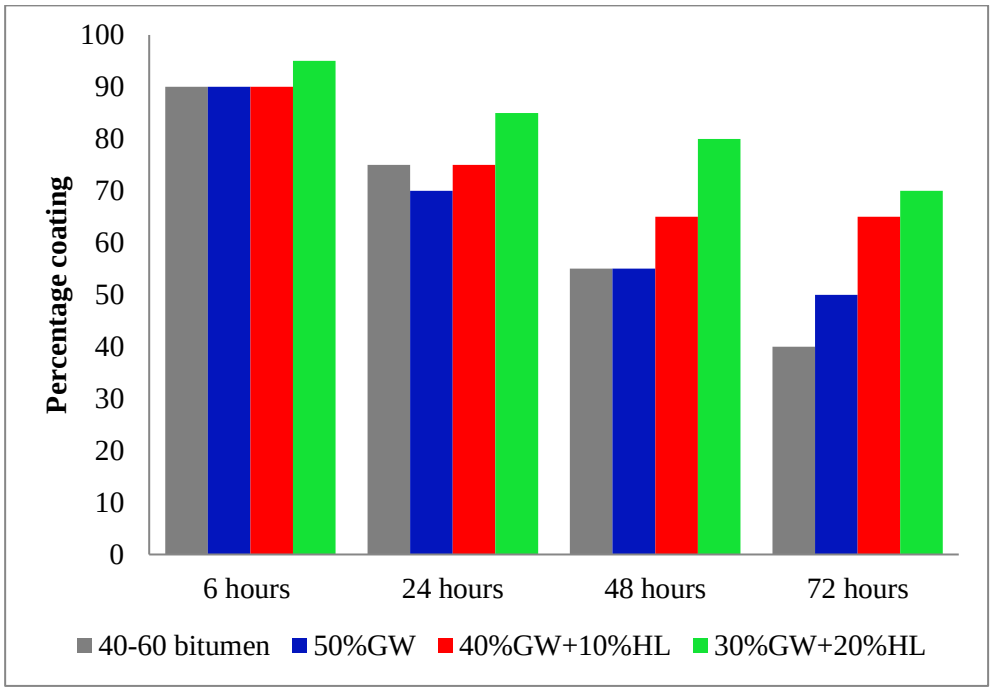


Figure 4:5: RBT test results for all combinations of Greywacke aggregates using Image analysis

By looking at Figure 4:2, Figure 4:3, Figure 4:4 and Figure 4:5 again it is obvious that there has been practically no difference between different combinations within same aggregate type after 6 hours visual or photographic inspection of the samples. After 24 hours this difference is getting obvious in some of the aggregate types for example in the case of granite aggregates the value for percentage coating for neat bitumen, mastic with 0% HL, mastic with 10% HL and mastic with 20% HL is 25, 70, 75 and 80% respectively. In the case of limestone aggregate, the percentage coating values at 24 hours for different combinations are not far from each other. The basalt and greywacke aggregates both show some difference at 24 hours between different combinations within each type but this difference is less obvious compared to granite aggregates.

Now looking at the results after 48 hours of inspection, it can be seen that the granite aggregate is continuously showing more distinctive results between its different combinations. The value for percentage coating for neat bitumen, mastic with 0% HL, mastic with 10% HL and mastic

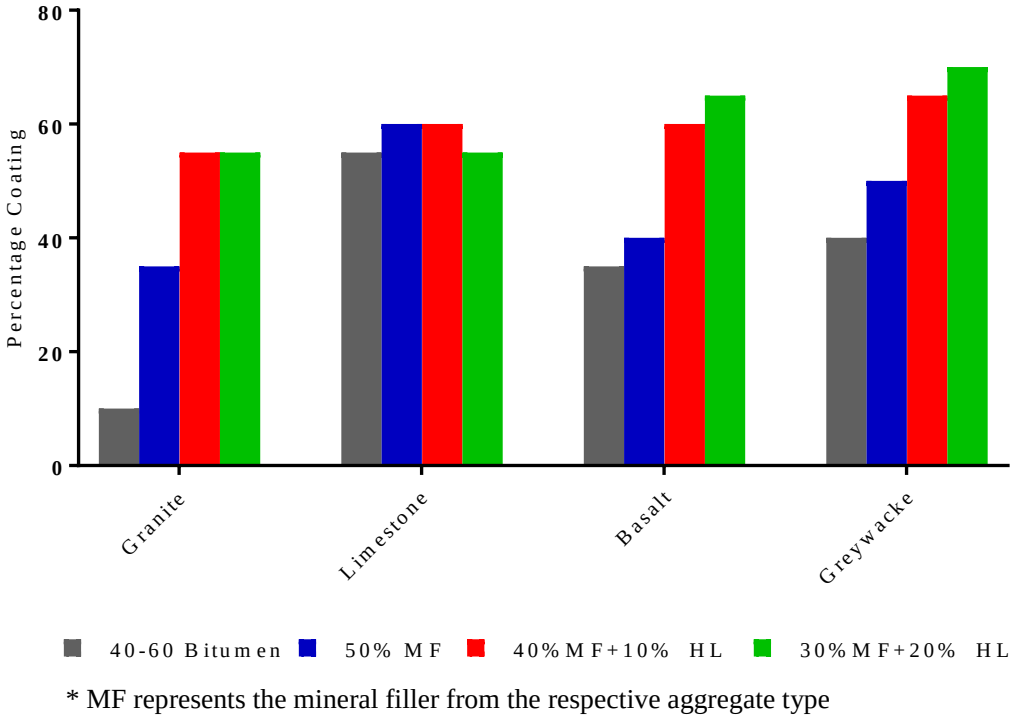
with 20% HL are now 15, 40, 60 and 65% which is again more discriminatory compared to 24 hours' values. Again, limestone aggregates show nearly no difference between its various combinations. Basalt and greywacke results at 48 hours are again similar to each other and both are showing some difference between their various combinations. This difference is a bit more than what was obtained at 24 hours.

The results after 72 hours of RBT agitation give the highest value in terms of the difference between different combinations within same aggregate type. For the granite aggregates, the values for percentage coating for neat bitumen, mastic with 0% HL, mastic with 10% HL and mastic with 20% HL are 10, 35, 55 and 55% respectively. The values for limestone aggregates after 72 hours for its combination with neat bitumen, 50% LS, 40%LS+10%HL and 30% LS+20% HL are 55, 60, 60 and 55 % respectively which is again not very distinctive. Basalt aggregate in combination with neat bitumen ,50% B, 40%B+10%HL and 30% B+20% HL gave percentage coating values of 35, 40, 50 and 65% respectively at 72 hours. Similarly, Greywacke gave values of 40, 50, 65 and 70 for neat bitumen, 50%GW, 40%GW+10%HL and 30%GW+20%HL respectively.

Among all the aggregate types, percentage coating for granite and basalt aggregate are most distinctive. Greywacke also gives a decent distinctive effect of HL addition but limestone on the other hand does not show any effect, in fact all of the combinations are similar to each other.

Now to evaluate the performance of different aggregate types with and without HL, results after 72 hours have been used based on the arguments made above and shown in Figure 4:6. It shows a clear difference between the performances of different aggregate types with their various combinations. Looking at the results of neat bitumen it can be found that it has the worst performance with granite aggregates. On the other hand, neat bitumen performance with the limestone is found as best compared to other three types but basalt and greywacke are giving very similar performance. Similarly, the mastic with 50% mineral filler of the respective aggregate has shown quite variable performance with best performance is for the limestone, then greywacke, then basalt and again the granite performance is worst in comparison to all other aggregate types. Now with the addition of 10% HL with the mineral filler, performance of some aggregate types have jumped to a significantly higher value. For example, the percentage coating for the granite aggregates have jumped 57% compared to the one without HL (50%MF). Similarly, percentage coating increased by 50% in the case of basalt aggregate.

For the limestone aggregates there is a slight decrease noticed with the addition of HL but greywacke followed the same trend of granite and basalt aggregates and showed increment of 30% in coating with the addition of 10% HL. The performance of the mastics having 10% and 20% HL in most of the cases is very similar to each other. This means it may not be worth adding 20% HL in the mastic as it is not improving the performance by the same rate of 10% HL addition and the performance of the two mastics are very similar to each other in most of the cases.



* MF represents the mineral filler from the respective aggregate type

Figure 4:6: Comparison for RBT results after 72 hours between all aggregate combinations

Based on the discussion above it might be a good idea to check the coating left on the loose aggregates for time periods longer than 72 hours as the most discriminating stage has been found at 72 hours in all the aggregate combinations in this particular test protocols.

4.2.4 Comparison between visual observation and process image results

A comparison has been made for granite, limestone, basalt and greywacke aggregates in Table 4:1, Table 4:2, Table 4:3and Table 4:4 respectively between the observations made with the naked eye by the operators and results obtained after image analysis. The values for bitumen upper limit (bul) and aggregates upper limit (aul) have also been presented which are used in the Matlab code for the calculation of percentage coating.

Table 4:1: Comparison of percentage retained coating between visual observation and Matlab analysis for granite aggregate combinations

Time (Hrs)	Sample Name	Percentage Coating by Visual Observation	Percentage Coating by Matlab	bul (value)	aul (value)
6	40-60 Bitumen	80	70	45	120
24	40-60 Bitumen	30	25	35	120
48	40-60 Bitumen	10	15	30	120
72	40-60 Bitumen	5	10	35	130
6	50% G	80	80	60	120
24	50% G	50	70	60	120
48	50% G	40	40	42	120
72	50% G	25	35	45	120
6	40%G+10%HL	90	80	55	120
24	40%G+10%HL	65	75	55	120
48	40%G+10%HL	60	60	50	130
72	40%G+10%HL	50	55	50	120
6	30%G+20%HL	90	80	50	120
24	30%G+20%HL	70	80	50	130
48	30%G+20%HL	60	65	50	130
72	30%G+20%HL	60	55	50	130

Table 4:2: Comparison of percentage retained coating between visual observation and Matlab analysis for limestone aggregate combinations

Time (Hrs)	Sample Name	Percentage Coating by Visual Observation	Percentage Coating by Matlab	bul (value)	aul (value)
6	40-60 Bitumen	85	75	60	140
24	40-60 Bitumen	60	65	60	140
48	40-60 Bitumen	50	60	50	140
72	40-60 Bitumen	45	55	50	140
6	50% LS	90	85	60	140
24	50% LS	80	80	60	140
48	50% LS	70	70	50	130
72	50% LS	60	60	55	130
6	40%LS+10%HL	80	80	60	140
24	40%LS+10%HL	70	70	60	140
48	40%LS+10%HL	60	65	60	140
72	40%LS+10%HL	50	60	55	140
6	30%LS+20%HL	80	80	60	140
24	30%LS+20%HL	65	75	60	140
48	30%LS+20%HL	60	60	60	140
72	30%LS+20%HL	50	55	60	140

Table 4:3: Comparison of percentage retained coating between visual observation and Matlab analysis for basalt aggregate combinations

Time (Hrs)	Sample Name	Percentage Coating by Visual Observation	Percentage Coating by Matlab	bul (value)	aul (value)
6	40-60 Bitumen	80	80	35	80
24	40-60 Bitumen	60	55	35	120
48	40-60 Bitumen	50	45	35	130
72	40-60 Bitumen	40	35	45	130
6	50% B	90	70	50	120
24	50% B	65	60	50	120
48	50% B	50	45	35	120
72	50% B	40	40	70	150
6	40%B+10%HL	90	85	60	120
24	40%B+10%HL	70	75	60	120
48	40%B+10%HL	65	65	45	120
72	40%B+10%HL	60	50	50	120
6	30%B+20%HL	95	85	60	120
24	30%B+20%HL	80	60	45	120
48	30%B+20%HL	70	70	45	120
72	30%B+20%HL	65	65	45	120

Table 4:4: Comparison of percentage retained coating between visual observation and Matlab analysis for greywacke aggregate combinations

Time (Hrs)	Sample Name	Percentage Coating by Visual Observation	Percentage Coating by Matlab	bul (value)	aul (value)
6	40-60 Bitumen	80	80	25	60
24	40-60 Bitumen	60	55	30	85
48	40-60 Bitumen	50	45	25	85
72	40-60 Bitumen	40	35	20	85
6	50% B	90	70	60	100
24	50% B	65	60	60	125
48	50% B	50	45	60	170
72	50% B	40	40	50	150
6	40%B+10%HL	90	85	70	150
24	40%B+10%HL	70	75	60	150
48	40%B+10%HL	65	65	60	150
72	40%B+10%HL	60	50	60	150
6	30%B+20%HL	95	85	25	80
24	30%B+20%HL	80	60	25	80
48	30%B+20%HL	70	70	20	80
72	30%B+20%HL	65	65	25	80

By looking at the above tables it can be seen that the results obtained from the naked eye and the results calculated after image analysis are very similar to each other. Definitely, there will be some difference between the two results as one is just visual (subjective) observation and other is digitally computed using Matlab. The change of error in the results computed using

Matlab would be less compared to the observations made using naked eye by the two operators. So, it is recommended to analyse the results of RBT test using some image analysis techniques rather than just relying on eye observations.

Some of the recent studies on RBT results analysis have reported the difficulties in the image analysis with the aggregates having dark colour like basalt as the pixels of bitumen coated aggregates appears same as pixels of dark coloured aggregate (Lantieri et al. 2017; Yuan et al. 2015) but no such difficulty in analysis was found using the code produces as a part of this research.

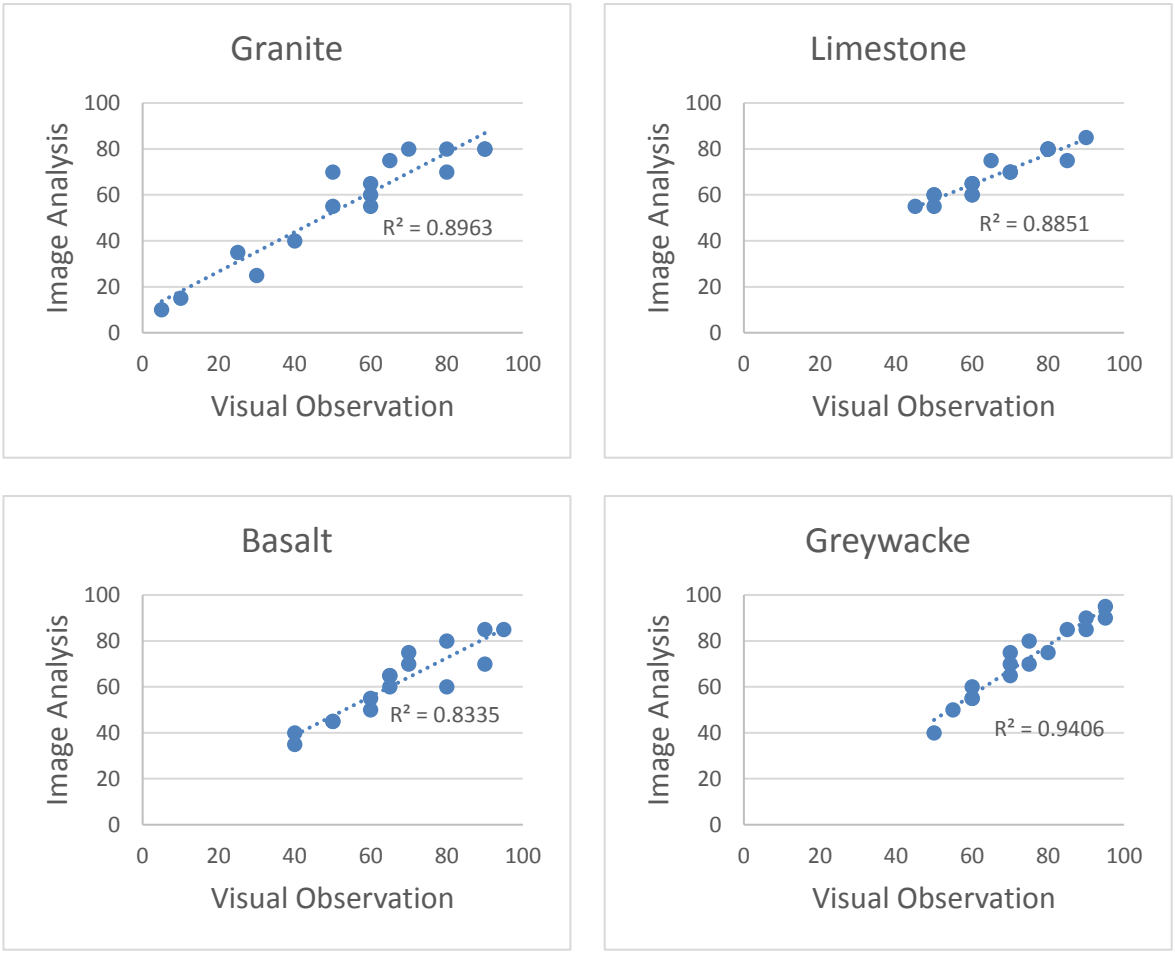


Figure 4:7: Relation between visual observation abd Image analysis results in RBT for all four aggregate types

To further support the statement made above that the results obtained by visual inspection and those computed after image analysis are similar to other, results of each aggregate

combinations obtained by visual inspection have been plotted against results obtained after image analysis and are presented in the Figure 4:7. R square value is calculated for each aggregates type which is a statistical measure of how close the data are to the fitted regression line. In the Figure 4:7 it can be observed that R square value ranges from 0.83 to 0.94 which is not bad and it can be deduced that the two results are not far from each other and can be referred as similar.

A frequency distribution of the difference between the visual and image analysis results for all the four aggregate types have been shown in the Figure 4:8. By looking carefully at the figure it can be observed that the difference between the visual and image analysis results for most of the aggregate types is within + 10%, so again it is concluded that the visual and image analysis results can be referred as similar but the chance of error in the results computed using image analysis is less compared to those obtained by visual examination.

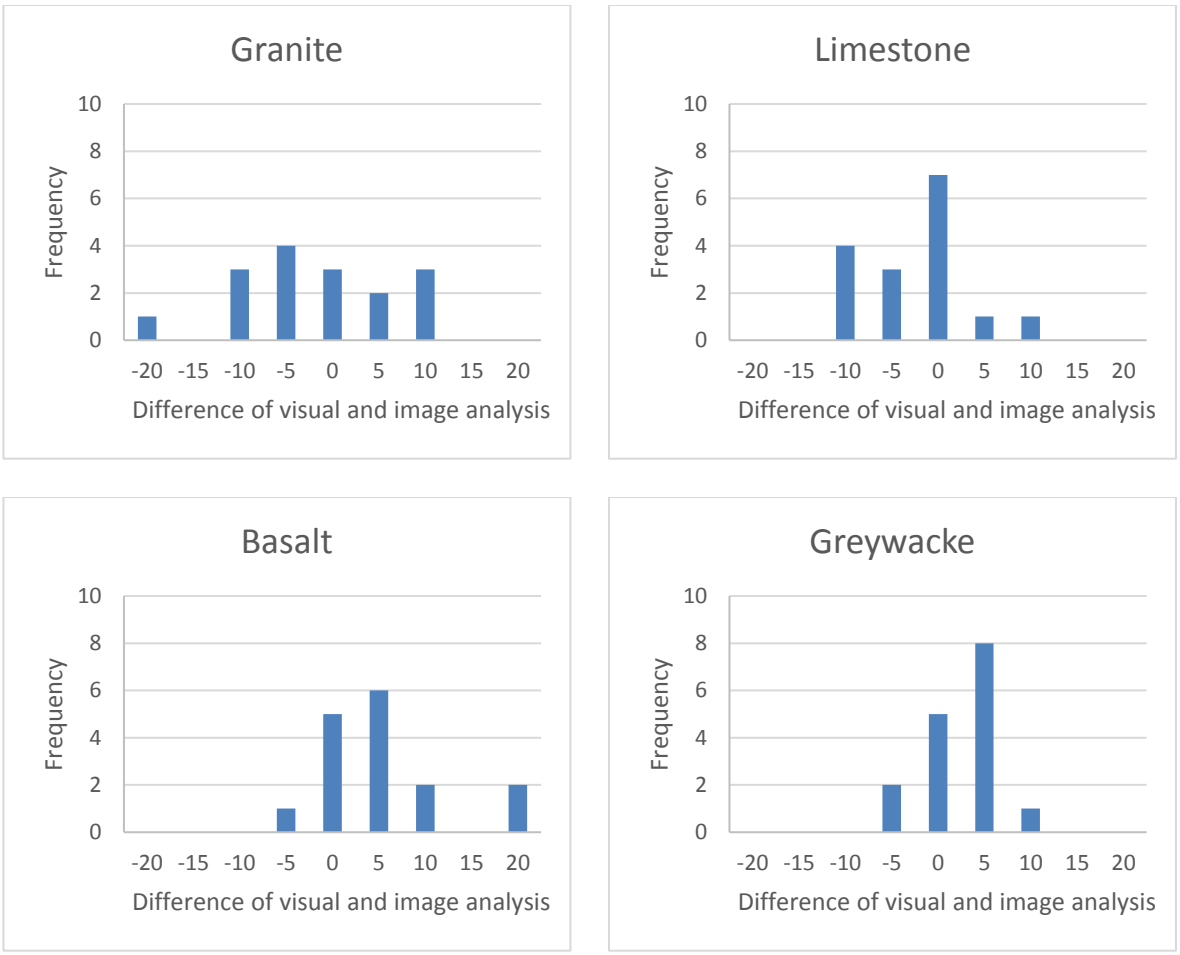


Figure 4:8: Frequency distribution of the difference between the visual and image analysis results for all the four aggregate types

4.3 Surface free energy (SFE) and moisture damage assessment

Surfaces of materials often have unique physical and chemical properties compared to their bulk. Because of this and the special role of surfaces in various fields of science and engineering, the study of surfaces has grown to be a vast subject on its own. As discussed in Chapter 3 the surface energy of the binder and mastic have been calculated using DCA device. For the aggregate, DVS device has been used for the surface free energy calculations. Once the surface energy is known for the two materials, the work of adhesion and work de-bonding is calculated using the equations of thermodynamics. In order to represent the moisture damage by a single value, four energy ratios have been suggested by (Bhasin 2007). The value for all the four types have been calculated to quantity the effect of HL replacement in the different combinations of the studied mastics.

4.3.1 Materials

One neat bitumen and 12 different mastics have been tested with four combinations of aggregates including granite, limestone, basalt and greywacke. The details of mastic notation, composition and the aggregate type with which it has been used is mentioned in Chapter 3.

4.3.2 SFE measurement for bitumen

The surface energy of a liquid or solid cannot be calculated directly. There are various indirect methods available for the measurement of SFE for liquid and solids which can be found in reports (Ahmad and Airey 2010a; 2010b; 2010c; 2010d).

The surface energy of the bitumen and mastics used in this study has been calculated using the Wilhelmy plate method. In the Wilhelmy plate surface energy is measured with the help of contact angle between the bitumen and a liquid of know surface energy based on the kinetic force equilibrium when a thin plate coated with binder is immersed or withdrawn from a liquid solvent at a very slow and constant speed (Adamson and Gast 1997). The contact angle measured when a binder coated thin glass plate is submerged in the solvent of known surface energy is called advancing angle and the angle which this solvent makes with the binder coated glass plate during withdrawal process, called receding angle of contact. Components of surface free energy calculated from the advancing angle are called components of surface energy of

wetting. Similarly, surface energy or its components calculated from the angle of receding are called components of surface energy of de-wetting.

When the binder coated glass plate is suspended in the air, equation 4.1 is true.

$$F = Wt_{plate} + Wt_{bitumen} - V.\rho_{air}.g \tag{4.1}$$

Where;

F	=	Force from the Dynamic Contact Angle analyser (DCA) balance
Wt_{plate}	=	Weight of the glass plate
$Wt_{bitumen}$	=	Weight of the coated bitumen film
V	=	Volume of the bitumen plate
ρ_{air}	=	Density of the air
g	=	Local acceleration due to gravity.

When the binder coated glass plate is submerged in solvent, the balance of DCA machine measures the force in accordance with equation 4.2:

$$F = Wt_{plate} + Wt_{bitumen} + P_t\gamma_L \cos\theta - V_{im}\rho_L g - \left(V - V_{im}\right)\rho_{air} g \tag{4.2}$$

Where;

P_t = Perimeter of the bitumen coated plate

γ_L = Total surface energy of the liquid

θ = Dynamic contact angle between bitumen and the liquid

V_{im} = Volume of the immersed plate.

Change in force can be calculated by subtracting equation 4.1 from equation 4.2.

$$\Delta F = P_t\gamma_L \cos\theta - V_{im}\rho_L g + V_{im}\rho_{air} g \tag{4.3}$$

By rearranging equation 4.3, the contact angle can be calculated from equation 4.4.

$$\cos\theta = \left(\Delta F + V_{im}\left(\rho_L - \rho_{air}\right)g\right)/\left(P_t\gamma_L\right) \tag{4.4}$$

Based on the Young-Dupré equation and the assumption that equilibrium film pressure is neglected for bitumen, equation 4.5 is obtained:

$$\gamma_{Li}(1+\cos\theta_i) = 2\sqrt{\gamma_S^{LW}\gamma_{Li}^{LW}} + 2\sqrt{\gamma_S^{-}\gamma_{Li}^{+}} + 2\sqrt{\gamma_S^{+}\gamma_{Li}^{-}} \quad (4.5)$$

In the equation 4.5 γ_{Li}, γ^{LW}_{Li}, γ^{+}_{Li}, and γ^{-}_{Li} are surface energy components of the probe liquid. The term, θ_i, can be measured using the Wilhelmy plate method. The three unknowns in equation 4.5 i.e. γ^{LW}_{s}, γ^{-}_{s}, and γ^{+}_{s} are the components of surface energy of bitumen or mastic in question and are called; Lifshitz–van der Waals, Lewis base, and Lewis acid components, respectively. To calculate these three unknown components, three liquids with known surface energy components have been used to get a system of three simultaneous equations. For this study glycerol, diiodomethane (Methylene Iodide), and distilled water are selected as solvent liquids. The surface energy components of the three liquids are listed in Table 4:5

Table 4:5: Surface energy components for the Probe liquids used for DCA

Surface Energy	γ_L	γ_L^{LW}	γ_L^{+}	γ_L^{-}
Water	72.8	21.8	25.5	25.5
Glycerol	64	34	3.92	57.4
Diiodomethane	50.8	50.8	0	0

Now if we assume,

$$\begin{aligned} &y_i(x) = 1+\cos\theta_i \\ &a_{1i} = 2\frac{\sqrt{\gamma_{Li}^{LW}}}{\gamma_{Li}}; a_{2i} = 2\frac{\sqrt{\gamma_{Li}^{+}}}{\gamma_{Li}}; a_{3i} = 2\frac{\sqrt{\gamma_{Li}^{-}}}{\gamma_{Li}}, and \\ &x1 = \sqrt{\gamma_S^{LW}}; x2 = \sqrt{\gamma_S^{-}}; x3 = \sqrt{\gamma_S^{+}} \end{aligned} \quad (4.6)$$

the following matrix form of linear simultaneous equations is established:

$$\begin{bmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{bmatrix} \begin{bmatrix} x1 \\ x2 \\ x3 \end{bmatrix} = \begin{bmatrix} y1 \\ y2 \\ y3 \end{bmatrix} \quad (4.7)$$

Equation 4.7 can be solved easily for all three unknown surface energy components of bitumen or mastics. Surface energy components of one neat 40/60 pen binder and 12 mastics have been calculated using Wilhelmy plate method and presented in Table 4:6

Table 4:6: Surface free energy components of all bitumen/ mastic samples

Bitumen/Mastic	Surface energy components (mJ/m^2)			Total Surface Energy γ (mJ/m^2)
	γ^{LW}	γ^+	γ^-	
40-60 Neat	19.040	1.316	0.990	21.323
50%G	23.696	0.350	1.680	25.232
40%G+10%HL	28.072	0.012	2.316	28.411
30%G+20%HL	28.744	0.000	8.865	28.747
50%LS	36.609	0.147	1.490	37.544
40%LS+10%HL	36.665	0.177	2.028	37.865
30%LS+20%HL	37.990	0.095	1.209	38.669
50%B	29.764	0.0278	0.803	30.063
40%B+10%HL	28.955	0.112	0.296	29.320
30%B+20%HL	29.799	0.009	1.424	30.032
50% GW	29.330	0.0004	3.787	29.407
40%GW+10%HL	31.368	0.036	6.934	32.380
30%GW+20%HL	29.400	0.017	7.170	30.112

The base bitumen (i.e. 40/60 Neat) used in this study is of a typical acidic nature which can be compared with the results of some previous studies presented in Table 2:9. Now by looking at the above results carefully, it can be deduced that with the replacement of hydrated lime for granite and greywacke filler there is a significant increase of basic component. On the other hand, the same replacement for limestone and basalt has shown marginal change. Similarly, there has been some small reduction observed in the acidic component in most of the cases. The increase in the basic component of some aggregates is in agreement with the work carries out by Liu et al. (2013).

4.3.3 SFE measurement for aggregates

The surface energy components of material with high energy surfaces such as aggregates can be calculated with vapour sorption techniques such as Universal sorption or dynamic vapour sorption techniques. These techniques use the gas adsorption characteristics of the selected solvents with known surface energy to measure the surface free energy of aggregates indirectly. This method can be used universally for aggregates having different size, shape, mineralogy and surface texture.

Four solvents with known components of surface energy have been used including Octane, Chloroform, ethyl acetate and distilled water. The total surface energy of the probe liquids and their components have been tabulated in Table 4:7

Table 4:7: Surface energy components for the Probe liquids used for DVS

Surface Energy	γ_L	γ_L^{LW}	γ_L^+	γ_L^-
Octane	21.62	0	0	21.62
Ethyl acetate	23.90	0	19.20	23.90
Chloroform	27.15	3.80	0	27.15
Water	72.80	21.80	25.50	25.50

The amount of adsorbed solvent on aggregate surface and simultaneously measured vapour pressure, P, at the surface of aggregate has been measured. By using the BET equations (Li 1997) Specific surface area (SSA) of the aggregate can be calculated.

$$SSA = \left(\frac{n_m N_o}{M} \right) \alpha \tag{4.8}$$

$$\frac{P}{n(P_0 - P)} = \left(\frac{c-1}{n_m c} \right) \frac{P}{P_0} + \frac{1}{n_m c} \tag{4.9}$$

Where;

SSA = Specific surface area of absorbent, m^2

n_m = Monolayer specific amount of vapour adsorbed on the aggregate surface, mg

N_0 = Avogadro's number, 6.022 x 10^{23} mol^{-1}

M = Molecular weight of the vapour, g/mol

α = Project area of a single molecule, m^2

P = Vapour pressure, Pa

P_0 = Saturated vapour pressure of solvent, Pa

n = Specific amount adsorbed on the surface of the absorbent, mg

c = BET constant, unit less.

The spreading pressure at saturation vapour pressure, π_e, for each solvent, can then be calculated using the following Gibbs adsorption equation 4.10

$$\pi_e = \frac{RT}{A} \int_0^{Po} \frac{n}{P} dP \tag{4.10}$$

Where;

π_e = Spreading pressure at saturation vapour pressure of the solvent, $ergs/cm^2$

R = Universal gas constant, 83.14 cm^3 bar/mol.K

T = Absolute temperature, K.

The work of adhesion of a liquid on a solid, W_A, can then expressed in terms of the surface tension (surface energy) of the liquid, γ_l, and the equilibrium spreading pressure of adsorbed vapour on the solid surface, π_e, as shown in Equations 4.11, 4.12 and 4.13.

$$W_A = \pi_e + 2\gamma_l = \Delta G_{sl} \tag{4.11}$$

$$\Delta G_{sl} = \Delta G_{sl}^{LW} + \Delta G_{sl}^{AB} = 2\sqrt{\gamma_s^{LW}\gamma_l^{LW}} + 2\sqrt{\gamma_s^{+}\gamma_l^{-}} + 2\sqrt{\gamma_s^{-}\gamma_l^{+}} \tag{4.12}$$

$$\pi_e + 2\gamma_l = 2\sqrt{\gamma_s^{LW}\gamma_l^{LW}} + 2\sqrt{\gamma_s^{+}\gamma_l^{-}} + 2\sqrt{\gamma_s^{-}\gamma_l^{+}} \tag{4.13}$$

Where the subscripts *s* and *l* refer to solid and liquid, respectively.

Now again in equation 4.13, we have three variables and we can get three simultaneous equations by using three liquids of known components of surface energy which can be solved easily for the aggregate surface energy components.

The surface energy with all the components and specific surface area of four aggregates used in this study are presented in Table 4:8.

Table 4:8: Surface free energy components of all aggregate types

Aggregates	Surface energy components (mJ/m^2)			Total surface energy, γ (mJ/m^2)	Specific surface area, γ (m^2/g)
	γ^{LW}	γ^{+}	γ^{-}		
Granite	68.97	17.41	569.68	199.18	0.3819
Limestone	65.81	0.98	375.72	104.19	0.7173
Basalt	66.66	176.03	214.92	389.01	1.7817
Greywacke	57.00	4.46	1435.62	57.00	1.9450

The surface energy of the four aggregates varies considerably among different types and sources. This difference is not only in terms of the total energy but also among the components. This difference in the energy can be due to the fact that different aggregate varies considerably in the composition depending on their source and type. This difference has a considerable effect on the adhesive properties when bitumen come in contact with aggregates in the form of bituminous mixture. If we look at the components of SFE of granite we find that its basic component is more than its acidic component which should be the other way round but this can

happen as the surface chemistry and bulk chemistry of a material could be different (Kim, 2009). In addition, the results from some other research works show the same type of variation in the acidic and basic components (Ahmad 2011; Liu et al. 2013).

4.3.4 Dry and wet work of adhesion

The main purpose of calculating the surface free energy of bitumen/mastic and aggregates is to determine the interfacial work of adhesion under dry conditions and also wet conditions which is also termed work of de-bonding. Larger values of work of adhesion mean that the mixture is more durable and bond between aggregate and bitumen is stronger. On the other hand, it is desirable to have a mixture having smaller absolute values for work of de-bonding, which means it will have more resistance against moisture damage. A summary of all the combinations of bitumen/mastic and aggregates used in this study have been listed in Table 4:9.

Also for an easy and quick understanding, a graphical representation of these combinations have been drafted in Figure 4:9. The beneficial effects of hydrated lime replacement in mastics with granite aggregates can be seen clearly.

By looking at the results for granite aggregates and comparing the work of adhesion and de-bonding results for mastic having only granite to the mastics with 10 or 20% HL replacement, it can be derived that the work of adhesion has increased by about 9.5% with the replacement of 10% HL and jumped by about 56% with 20% HL replacement. Along the same lines the work of de-bonding dropped down 9% with the replacement of 10% HL and dropped massively again by 51% with 20% HL replacement.

For the limestone aggregates on the other hand the replacement of HL in mastics has not shown any positive results both for the work of adhesion and the work of de-bonding. Although there has been some difference noted between neat bitumen and the other mastics, all the combinations of mastics have almost the same work of adhesion and de-bonding values. Basalt aggregate also didn't show any positive signs of HL replacement to mastics. The results of all combinations of mastics and even neat bitumen are very similar to each other.

Table 4:9: Summary of Work of adhesion and work of de-bonding for bitumen/mastics and aggregates combinations

Description	Work of Adhesion	Work of De-bonding
Granite		
40-60 Neat	130	-148
50%G	148	-132
40%G+10%HL	162	-120
30%G+20%HL	231	-64
Limestone		
40-60 Neat	112	-87
50%LS	146	-62
40%LS+10%HL	154	-57
30%LS+20%HL	143	-64
Basalt		
40-60 Neat	131	-144
50%B	120	-155
40%B+10%HL	113	-159
30%B+20%HL	127	-150
Greywacke		
40-60 Neat	146	-245
50% GW	229	-170
40%GW+10%HL	285	-125
30%GW+20%HL	285	-123

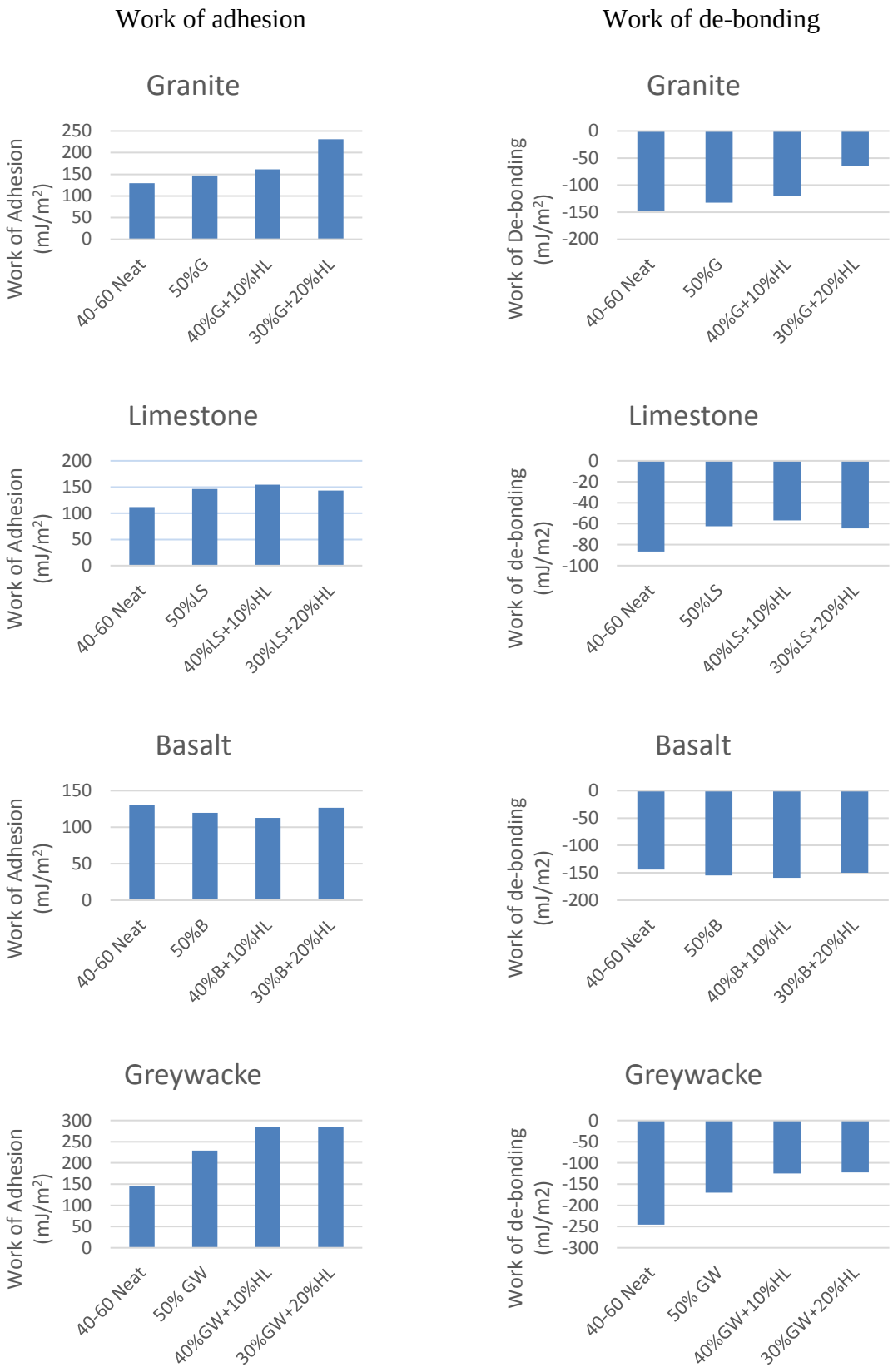


Figure 4:9: Work of adhesion and work of de-bonding for bitumen/mastics and aggregates combinations

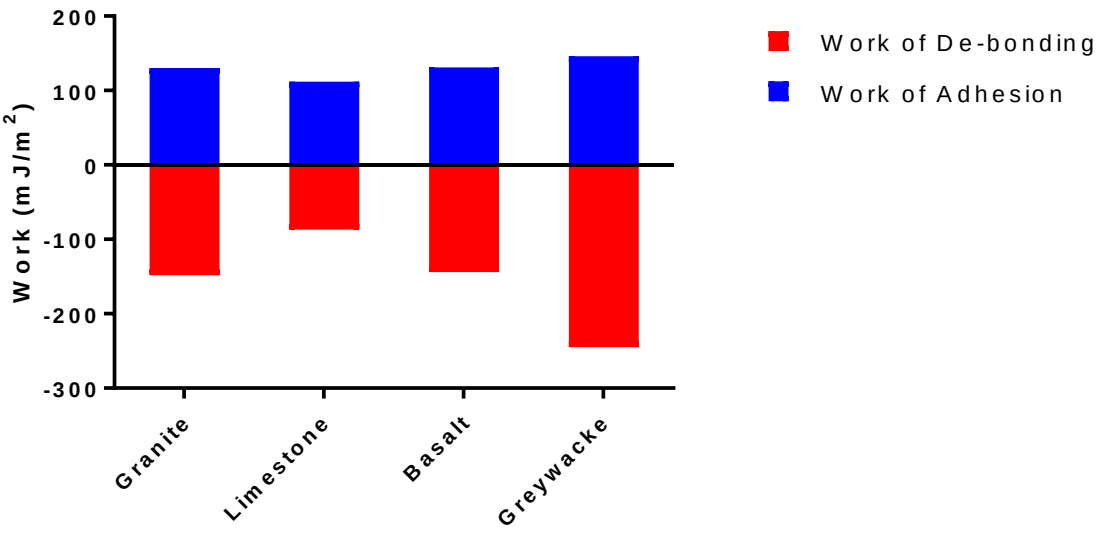


Figure 4:10: Work of adhesion and work of de-bonding results for all aggregates types with neat bitumen

But considering the results of work of adhesion and de-bonding for greywacke aggregate, there are some interesting findings. The greywacke aggregates have shown positive results with the replacement of the greywacke filler with HL. Work of adhesion for the mastics with 10 and 20% HL has increased by 25% compared to the mastic with 0% HL i.e. 50%GW. By analysing the work of de-bonding results a decrease of 26% and 25% has been calculated with the replacement of 10% and 20% HL respectively. This increase or decrease which has been noted with the granite and greywacke aggregates are even more when compared with the 40-60 neat bitumen.

Based on the results of all the aggregates with the neat bitumen each type can be ranked in terms of moisture damage performance separately. Figure 4:10 shows a graphical representation of all the results of all aggregate types with neat bitumen to rank the performance based on the work of adhesion and work of de-bonding.

Results presented in Figure 4:10 are self-explanatory. It can be seen that the values for work of adhesion for all the type of aggregate are very similar to each other. But the results of work of de-bonding in the presence of water clearly shows a performance difference between the four

aggregates types. Based on the above results it can be concluded that limestone aggregates are showing the best performance under moist conditions. Granite and basalt have a very similar performance in the presence of moisture. Greywacke is showing the worst performance based on the values of work of de-bonding compared to other three types used in this study.

Another interesting comparison has been plotted in Figure 4:11 where all the aggregates and mastics have been plotted at one place to see improvement in the performance with the addition of HL among different types of aggregates.

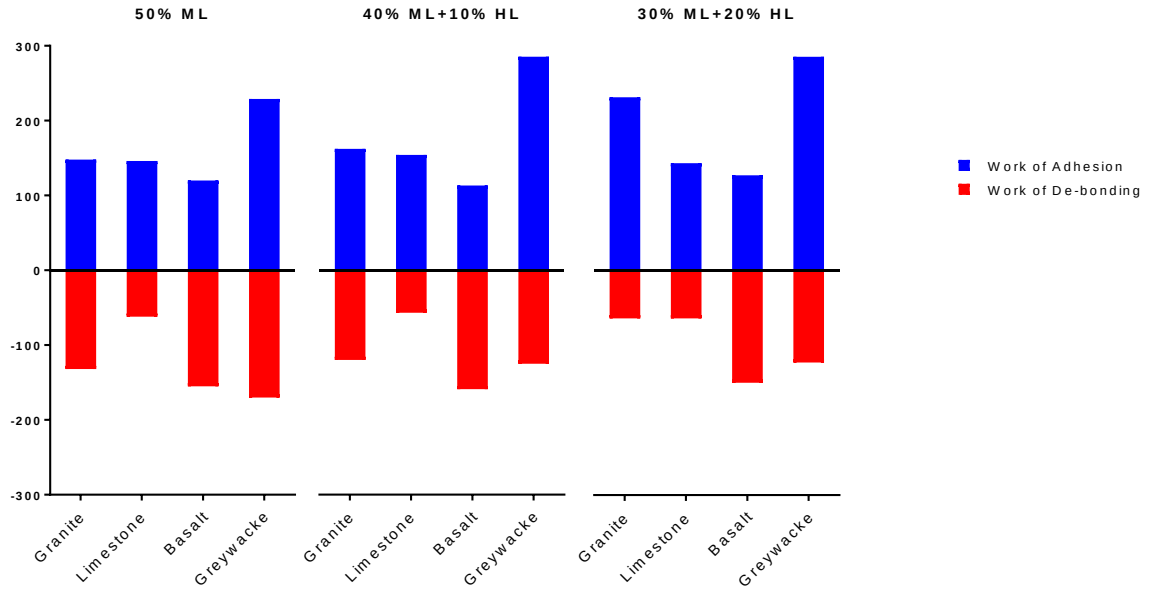


Figure 4:11: HL performance assessment between different aggregates types

From Figure 4:11 if we compare granite and limestone we see that both have similar performance under dry conditions for the combination having 0% HL (i.e. 50% MF) and 10%HL but under wet conditions the performance of limestone aggregates is far better than granite for both the combinations. One thing should be noted that the addition of 10% HL into the mastic when used in combination with respective aggregate type (granite and limestone in this case) is not showing any significant improvement with both aggregate types. Now with the addition of 20% HL to the mastic the performance for granite aggregate has improved significantly and even its performance under dry conditions has exceeded the performance of limestone aggregate. Also in the presence of water the performance of granite aggregate has improved so that it is now nearly the same as the performance of limestone aggregates. As

discussed earlier the limestone and basalt aggregates have shown very consistent results and do not show any noticeable improvement with the addition of HL.

Greywacke aggregate shows the best performance under dry conditions and with the addition of 10% HL the performance under dry conditions has further improved. However, with the addition of 20% HL its performance in dry conditions didn't improve any further. For the wet conditions, greywacke aggregate which showed relatively poor performance compared to the other three aggregates has improved its performance with 10% HL addition to the mastic. The performance of greywacke aggregates with the addition of 10% HL has even exceeded the performance of basalt aggregate. On the other hand, the addition of 20% HL to the mastic has not shown an improved performance compared to one having 10% HL. Based on the surface energy results in terms of work of adhesion and work of de-bonding it is very difficult to make a generalised statement for better HL content. For the granite aggregate the 20% HL addition produced the better performance but in the case of greywacke, 10% HL addition seems to be the better performance compared to 20%. So, it is not a straight forward scenario and HL content can't be generalised for all types of aggregate but will vary between different types of aggregates.

4.3.5 Energy parameters for moisture damage

As discussed earlier for a system of binder and aggregate combination to be more resistant to moisture damage it is desirable to have a higher magnitude of work of adhesion, W_{12} and an absolute lower magnitude for the work of de-bonding W_{123}. Based on the above argument one energy parameter that can be used to assess the moisture damage performance of binder aggregate systems is the ratio of W_{12} and W_{123}.

$$ER_1 = \left| \frac{W_{12}}{W_{132}} \right| \tag{4.13}$$

Where ER_1 is the energy ratio for a particular combination of bitumen and aggregate.

The higher value of ER1 shows a greater resistance against moisture damage. This statement is based on the hypothesis that resistance to moisture sensitivity is in direct relation to work of adhesion, W_{12} and in inverse relation to work of de-bonding, W_{123}. In the calculation of ER_1,

these two parameters have been considered i.e. work of adhesion which is directly proportion and work of de-bonding which is inverse in proportion. Another factor which has an effect on the moisture damage resistance is the wettability. Although the wettability and adhesion are both related to surface energy, both demonstrate different attributes. Adhesion or work of adhesion is the measure of the bond strength at the interface of the two materials. On the other hand, wettability can be defined as the ability of a material to wet or coat the other material. It can be explained by considering two materials for example bitumen and aggregates. The phenomenon of wettability will occur if the cohesive bond energy (work of cohesion) of the bitumen is less than the adhesive bond energy (work of adhesion) between bitumen and aggregates. Greater value of wettability means a better coating of one material on another. So for the problem in question if we get a combination of a binder aggregate system having a better coating (more wettability) it will be more resistant to moisture because better coating means less chance of moisture entering the system. So now another energy parameter can be proposed, which is directly proportional to wettability and inversely proportion to work of de-bonding for a given binder-aggregate system. This energy parameter can be mathematically expressed as:

$$ER_2 = \left| \frac{W_{12} - W_{11}}{W_{132}} \right| \tag{4.14}$$

Where W_{11} is the cohesive bond energy or work of cohesion of the bitumen and other terms are as previously described.

In addition to the ER_1 and ER_2 there are two more parameters which consider the micro-texture of the aggregates. Micro texture of the aggregate can be considered to be directly proportioned to the specific surface area (SSA). Hence the two new energy ratios can be defined as follows;

$$ER_3 = \left| \frac{W_{12}}{W_{132}} \right| * SSA \tag{4.15}$$

$$ER_4 = \left| \frac{W_{12} - W_{11}}{W_{132}} \right| * SSA \tag{4.16}$$

Using all the equations 4.13, 4.14, 4.15 and 4.16, all four types of energy parameters can be calculated for a given combination of bitumen/mastic and aggregate.

The values for all the four energy parameters have been reported in Table 4:10. Little and Bhasin (2006) and Bhasin (2007) in a study on field moisture damage performance versus laboratory surface free energy and intrinsic adhesion defined a set of threshold values for all the energy parameters to separate 'good' and 'poor' performing binder-aggregate combinations against moisture damage performance. These threshold limits are 0.75 for ER_1, 0.50 for ER_2, 0.50 for ER_3 and 0.35 for ER_4. The combinations which do not fulfil the above-mentioned criteria have been highlighted with a blue background.

If we look at all combinations for granite aggregate each combination passes the required criteria of 0.75 and 0.5 for ER_1 and ER_2 but the neat bitumen and mastic with 0% HL failed the criteria of 0.5 for ER_3. Also, three of the combinations i.e. Neat, mastic with 0% HL and mastic with 10% HL, failed the criteria of 0.35 for ER_4. Although some of the material combinations passed while others failed some or the ERs, one thing can be observed that the replacement of granite filler with HL has shown improved results compared to neat bitumen and the mastic having only granite filler. Especially the mastic having 20 percent HL has shown a massive jump of over 200 percent in terms of all four energy parameters.

The energy ratio results for all limestone combinations are very similar to each other. All of the combinations pass the required criteria with a big margin yet the effect of HL addition is not obvious. Addition of 10% HL has shown slightly better results but 20% HL addition has decreased performance relative to both the 0% HL and 10% HL mastics. Overall it can be concluded that there is practically no beneficial effect of HL addition on limestone aggregates based on these surface energy calculations.

Basalt aggregates have also shown hardly any effect of HL addition with the main difference observed between the results of neat bitumen and mastics but there is hardly any difference between the three mastics. Addition of 20% HL has shown a slight improvement but all the results are in a very narrow band.

Table 4:10: Bond energy ratios, ER1, ER2, ER3, ER4 for binder-aggregate combinations

Description	**ER_1**	**ER_2**	**ER_3**	**ER_4**
Granite				
40-60 Neat	0.88	0.59	0.33	0.22
50%G	1.12	0.74	0.43	0.28
40%G+10%HL	1.35	0.88	0.52	0.33
30%G+20%HL	3.61	2.71	1.38	1.03
Limestone				
40-60 Neat	1.29	0.80	0.92	0.57
50%LS	2.34	1.14	1.68	0.82
40%LS+10%HL	2.72	1.38	1.95	0.99
30%LS+20%HL	2.22	1.02	1.59	0.73
Basalt				
40-60 Neat	0.91	0.61	1.62	1.09
50%B	0.77	0.39	1.38	0.69
40%B+10%HL	0.71	0.34	1.26	0.61
30%B+20%HL	0.84	0.44	1.51	0.79
Greywacke				
40-60 Neat	0.60	0.42	1.16	0.82
50% GW	1.35	1.00	2.62	1.95
40%GW+10%HL	2.28	1.76	4.44	3.43
30%GW+20%HL	2.33	1.84	4.53	3.57

The results of greywacke aggregates are interesting as they have shown significant improvement with HL replacement compared to the mastic having only greywacke filler. Although the mastic with 0% HL passes the criteria set by Bhasin (2007); Little and Bhasin (2006), the replacement with 10% HL results in a huge improvement of around 70% for all the four energy ratios. Replacement of 20% HL didn't improve the performance any further as the results of the combinations with 10% HL and 20% HL are very similar in terms of energy ratios.

4.4 Summary

Two test methods have been used for the assessment of moisture damage resistance with the use of HL. Rolling bottles test was successfully performed on all the combinations used in the study and was quite useful in discriminating different combinations. Image analysis technique by using Matlab software was used for the calculation of percentage retained coating after 6, 24, 48 and 72 hours of agitation. The results after image analysis were compared to the results of eye observation and both were found very similar to each other. Hence RBT can now be used confidently for the comparison of different combinations. The beneficial effects of HL addition were clearly quantified with granite, basalt and greywacke aggregates but limestone aggregates didn't respond to the effect of HL addition when tested in the rolling bottles. Based on the results of RBT test, 10% HL (10% in mastic or 1% in mixture) substitution was found as better performing as 20% HL substitution gave very similar results to 10% HL substitution in most of the studied combinations.

As far as results from the surface energy testing are concerned, with the granite aggregate, there was a clear increment in the performance observed with the addition of HL to the mastic. Even with the 10% HL replacement, there were some positive trends but with the use of 20% HL, the was a significant improvement observed in the results. Limestone and Basalt aggregates, on the other hand, didn't show improvement signs with the results obtained from SFE testing. Greywacke aggregates followed the trend of granite aggregates and showed a good improvement when 10% greywacke filler was replaced with HL. With the use of 20% HL there was practically no difference observed in the performance. Calculation of energy ratios (ER_1, ER_2, ER_3, ER_4) demonstrates that most of the combinations are passing the criteria set by Little and Bhasin (2006) and Bhasin (2007) yet a significant increase in the values with the addition of HL was observed especially for granite and greywacke aggregates.

Chapter 5: Mixture Level Testing Using SATS Test

5.1 Introduction:

To study the moisture damage in the asphalt mixes the saturation ageing tensile stiffness (SATS) procedure was developed prior to mechanical testing (Collop et al. 2004). The application of the SATS test was limited to basic aggregates and hard binders initially. Later it was optimised for a universal asphalt mix application (Grenfell et al. 2012).

Compacted samples are conditioned under high pressure and temperature in the presence of moisture. A pressure vessel is used which can hold five identical samples. The stiffness of the samples is measured before and after the SATS conditioning procedure. The stiffness modulus of the unconditioned samples is calculated using NAT tester. Then the samples are immersed in the water for initial saturation. The pressure vessel is filled with water to between fourth and fifth level. Water saturated surface dried samples are placed in position in the pressure vessel. After that, 0.5 MPa pressure and 85^{o}C temperature are applied for 24 hours' duration. After the conditioning is done the samples are tested for stiffness modulus and a retained stiffness ratio is calculated which is a means of measuring the moisture damage resistance of asphalt mixtures. A detailed sample preparation and test procedure have been discussed in Chapter 3.

This chapter summarises the SATS test results for all the four aggregate types used in this study along with their combination. The Effect of HL has been studied by adding it to the mixture as a filler replacement. A comparative analysis has been made between the combinations having HL to the one without any HL. The moisture damage performance of all the four aggregates has also been compared without the addition of HL to see their performance ranking. In SATS test ITSM is used to find the retained stiffness but some studies claim that ITS being destructive in nature can better discriminate between different combinations. Based on the argument above an attempt has been made to compare ITS and ITSM in SATS test to find out if there is any improvement in the results is observed.

5.2 Material and test conditions

Four aggregates types have been tested as discussed earlier. These include; Granite, Limestone, Basalt and Greywacke. 40/60 pen binder has been used in the preparation of the asphalt mixture. The details for aggregate properties, bitumen properties and the asphalt mixture

variable have been discussed in details in Chapter 3. Modified test conditions have been used to evaluate the effect of moisture damage and estimate any beneficial effect of HL addition. Test conditions are as follows;

Pressure: 0.5 MPa

Temperature: 85^{o}C

Duration: 24 Hours

A total of 12 combinations have been tested with two runs for each combination. Roller compaction has been used to manufacture the slabs having a target air voids between 8 to 10%. From each slab, 5 cores have been trimmed with a 100 mm diameter and 60mm thickness. Only the cores that achieved the target air void of 8-10 % have been used for SATS testing.

5.3 SATS Test Results

SATS test results in terms of retained saturation against retained stiffness have been summarised in Figure 5:1 for the granite aggregates. All three combinations have been put together in one figure to make an easy comparison between them and to see if HL has any beneficial effect against moisture damage resistance. By looking carefully at the results for the granite aggregates it can be seen that although the granite used in this study is not of poor quality in terms of field performance, it has shown some improvements in terms of moisture damage performance with the addition of HL. The difference between the mixture with 0% HL and 1 or 2% HL is not vast but there is definitely some increase in the retained stiffness. This small difference may be due to the fact that granite aggregate used in the study is already of a good quality and although HL improves the overall performance but it is in a narrow band.

Similarly, Figure 5:2 is a graphical representation of retained saturation against retained stiffness for the three combinations of limestone aggregate. Again there is some difference observed with the addition of HL in the mixture. In fact, addition of HL has improved the moisture damage performance of limestone aggregate and the best results have been achieved with the addition of 1% HL. The results of Basalt aggregates are a bit different from that seen for the granite and limestone as shown in Figure 5:3. The addition of HL to basalt aggregates does not seem to make any difference as the results of all the three combinations i.e. 0% HL, 1%HL and 2% HL are very close to each other. Asphalt mixture made with basalt aggregates seems to have good resistance against moisture damage even without the addition of HL so it

can be concluded that HL addition has no beneficial effect if used in combination of basalt aggregates.

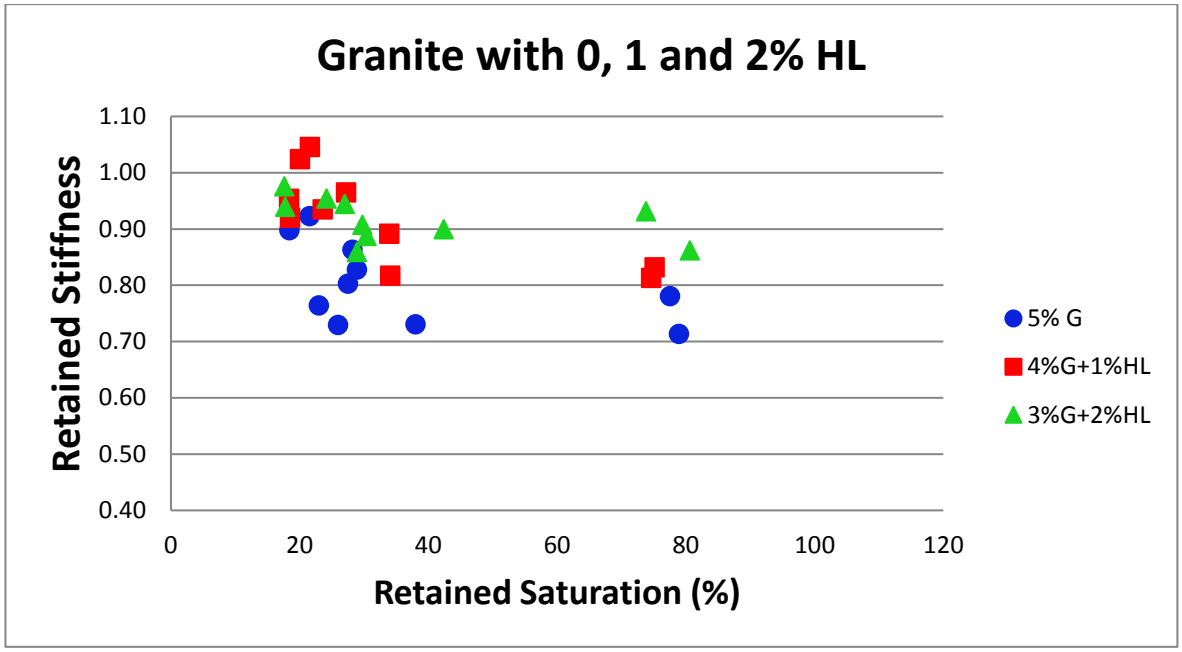


Figure 5:1: SATS test results for all combinations of granite aggregate

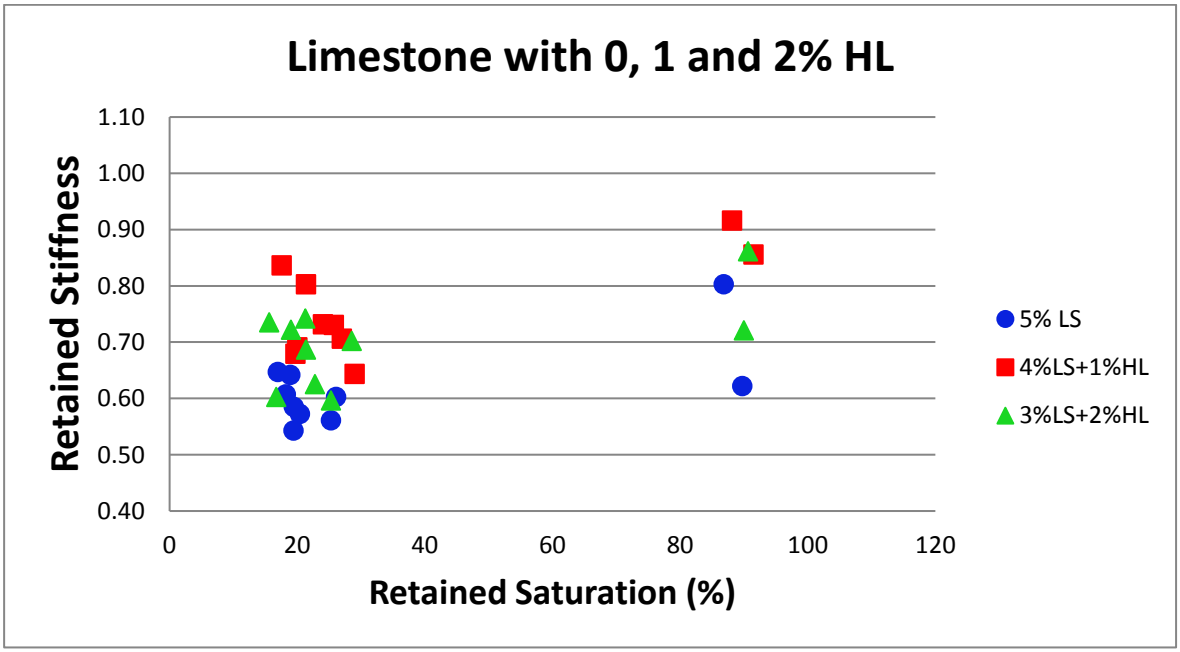


Figure 5:2: SATS test results for all combinations of Limestone aggregates

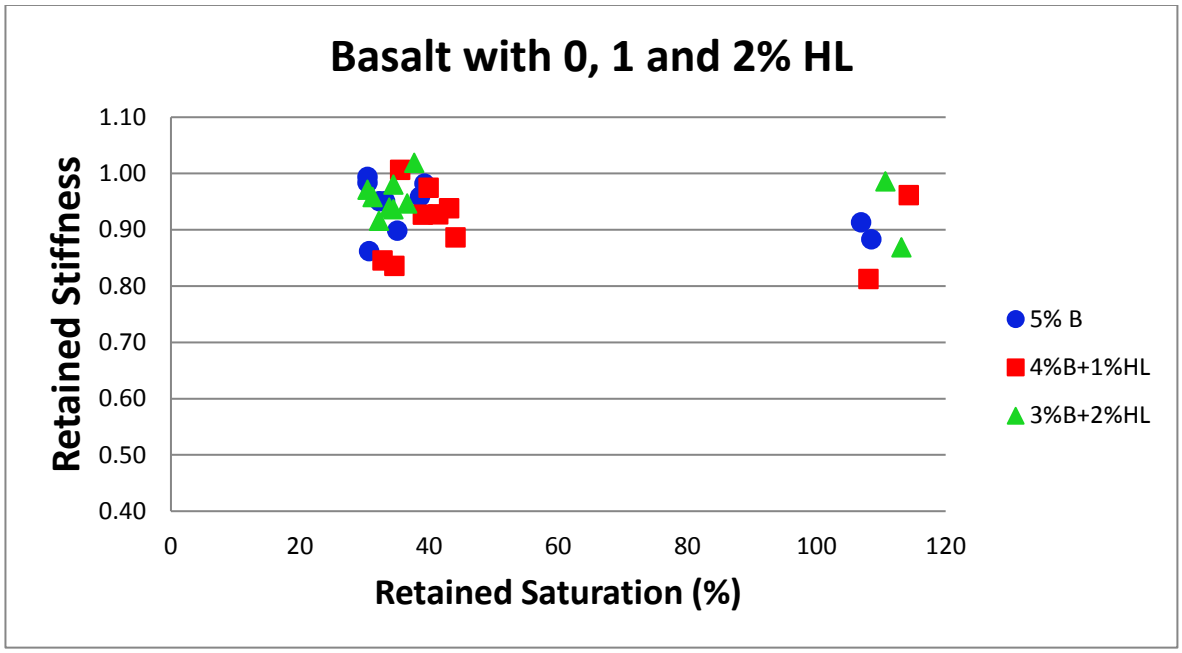


Figure 5:3: SATS test results for all combinations of Basalt aggregates

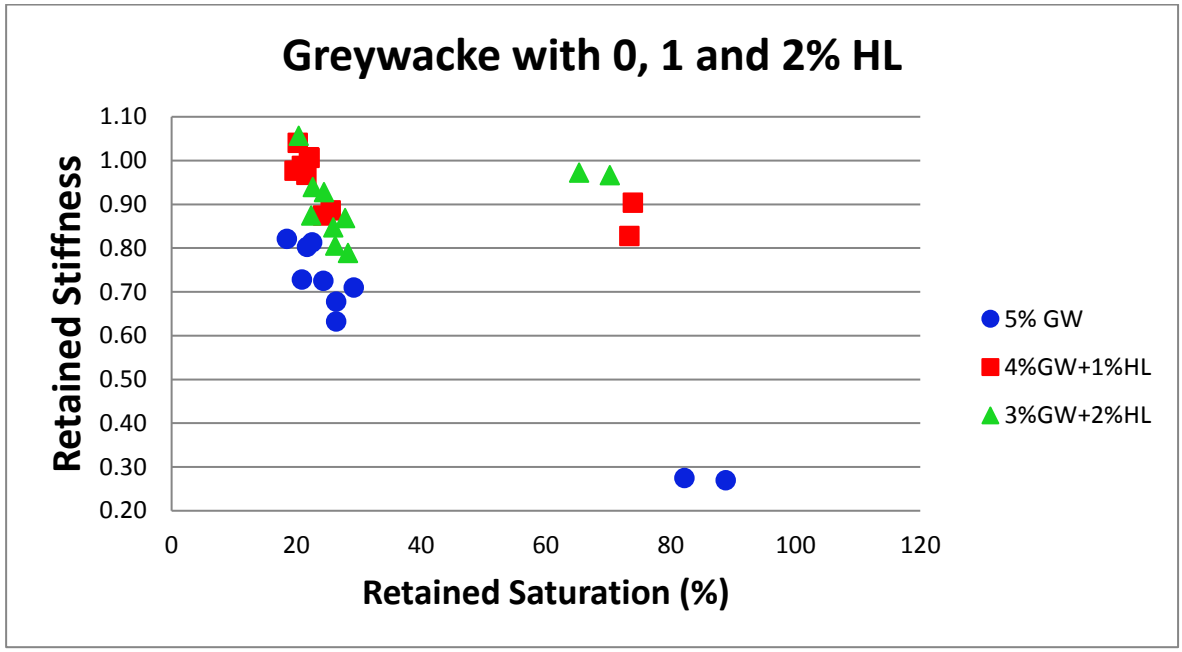


Figure 5:4: SATS test results for all combinations of Greywacke aggregates

The results of greywacke aggregates are presented in Figure 5:4. The effect of HL addition on moisture damage resistance is more apparent for greywacke aggregate. The retained stiffness for the combination having 0% HL is between 0.7 and 0.8 but with the addition of 1% and 2% HL the retained stiffness values are in a higher range of 0.9 to 1.0. Again the mixture with 1% HL gave the highest values but there is a very small difference noted between the moisture damage performance of 1 and 2% HL in terms of retained stiffness against retained saturation.

5.4 Performance of different aggregate types with HL addition

Although there has been a positive effect of HL addition in most of the aggregate types used in this study yet the difference is not very clear in the figures shown above (Figure 5:1 to Figure 5:4). To make an easy illustration of the effect of HL addition on mixtures made with different aggregate types and filler combination an average value has been calculated for the retained stiffness and retained saturation. As mentioned earlier, two runs for each combination have been undertaken which means for each combination 10 cores have been tested. As a general trend the cores at position 1 and at position 5 have the highest retained saturation and hence the lowest retained stiffness compared to positions 2, 3 and 4 (Airey et al. 2005). So for the calculation of average value for each combination a mean of retained stiffness and retained saturation has been taken for the cores at positions 2, 3 and 4 for both test runs. This average value of retained saturation against retained stiffness has been combined in Figure 5:5 for the asphalt mixture consisted of Granite, Limestone, Basalt and Greywacke aggregate respectively.

It can be seen clearly in Figure 5:5 that there has been a definite effect of HL addition in most of the mixtures types. In granite and limestone aggregates the addition of 1% HL has increased the retained stiffness to about 15% and 23% respectively. With Greywacke aggregates a noticeable increment of 33% has been calculated. Also in each case when HL addition seems to be beneficial, 1% HL addition is slightly better than 2% HL addition. Based on these facts it can be concluded that the addition of 1% HL gives better results compared to 2% HL for the studied combination.

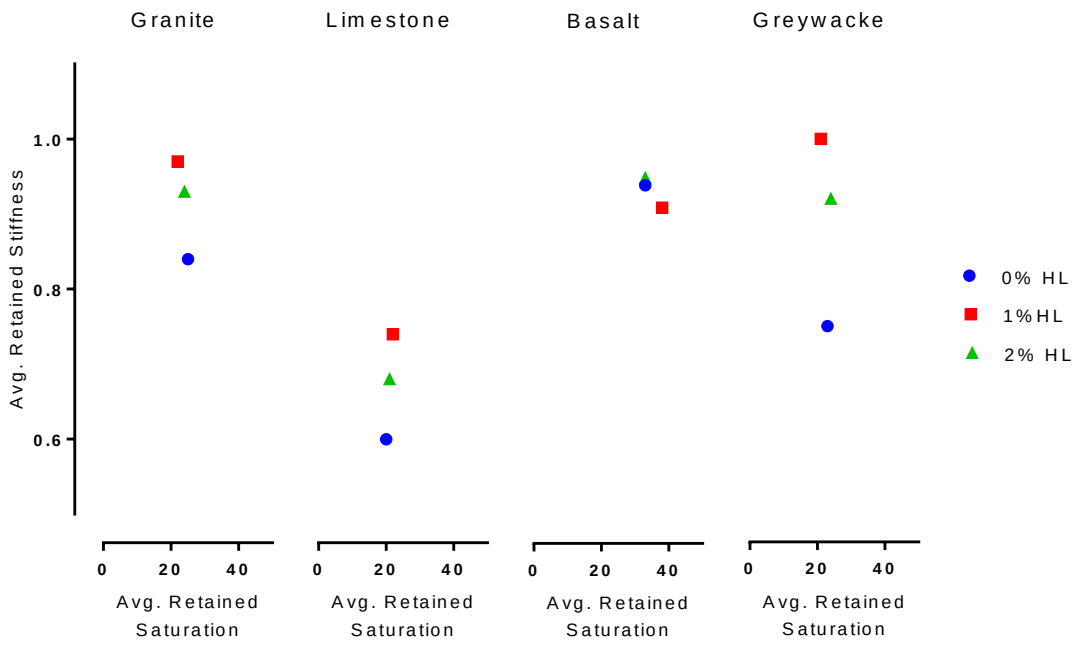


Figure 5:5: Behaviour of different aggregates with HL addition

5.5 Performance comparison among aggregates without HL addition

To look at the individual performance of asphalt mixture without the addition HL a graph has been plotted as shown in Figure 5:6 summarising all four aggregates types. All the four aggregate types although giving good moisture damage performance but basalt aggregates seems to have the best moisture damage performance even without HL addition. Second, best is the granite, 3rd is greywacke and limestone comes last. In most of the studies so far on moisture damage, performance of the mixtures containing limestone aggregates has been found better than those containing granite aggregate (Airey et al. 2005; Airey et al. 2008; Grenfell et al. 2014; Khan et al. 2013; Nejad et al. 2013) but the results from this study are not in agreement with this statement. In another study conducted by (Grenfell et al. 2012), it was found that one granite type was actually performing better than the limestone aggregate. So based on these facts it can be concluded that it is not always true that the moisture damage performance of limestone will be better than the granite aggregates. Some granite aggregates can give better moisture damage performance than limestone aggregates and the performance can vary among different types of granite aggregates depending on their mineralogical composition. Hence it can be stated that moisture damage mechanism is not very straight

forward but is quite complex and different aggregates can behave very differently when used in a mixture.

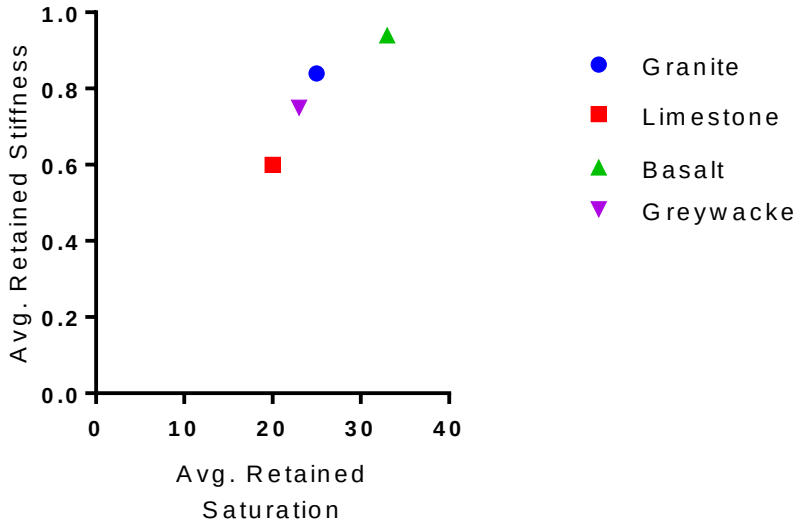


Figure 5:6: Performance comparison among aggregates without HL addition

Another thing which can be extracted out of Figure 5:6 is the increase of retained stiffness with the retained saturation but this interpretation is not true as from many previous studies no evidence of such trend is found (Airey et al. 2008; Grenfell et al. 2012; Khan et al. 2013).

5.6 ITSM vs ITS in SATS test

As per SATS procedure discussed earlier, for each test run five cores are tested for stiffness (ITSM) before and after SATS conditioning. Retained stiffness is calculated by dividing the two stiffness values. In this particular research which is looking at the beneficial effect of HL against moisture damage performance of asphalt mixtures, it has been found that HL improves moisture damage resistance in asphalt mixture for some aggregate types but the difference between the mixture having HL and the mixture without HL was not very distinctive. Various researchers have claimed that ITS results are better and more distinctive than ITSM as it is a more sensitive test in which the sample is loaded to failure (Jenkins 2000; Kim et al. 2007; Oke 2011). To check whether this statement is true for the SATS protocols or not, ITS tests were used instead of ITSM with some combinations.

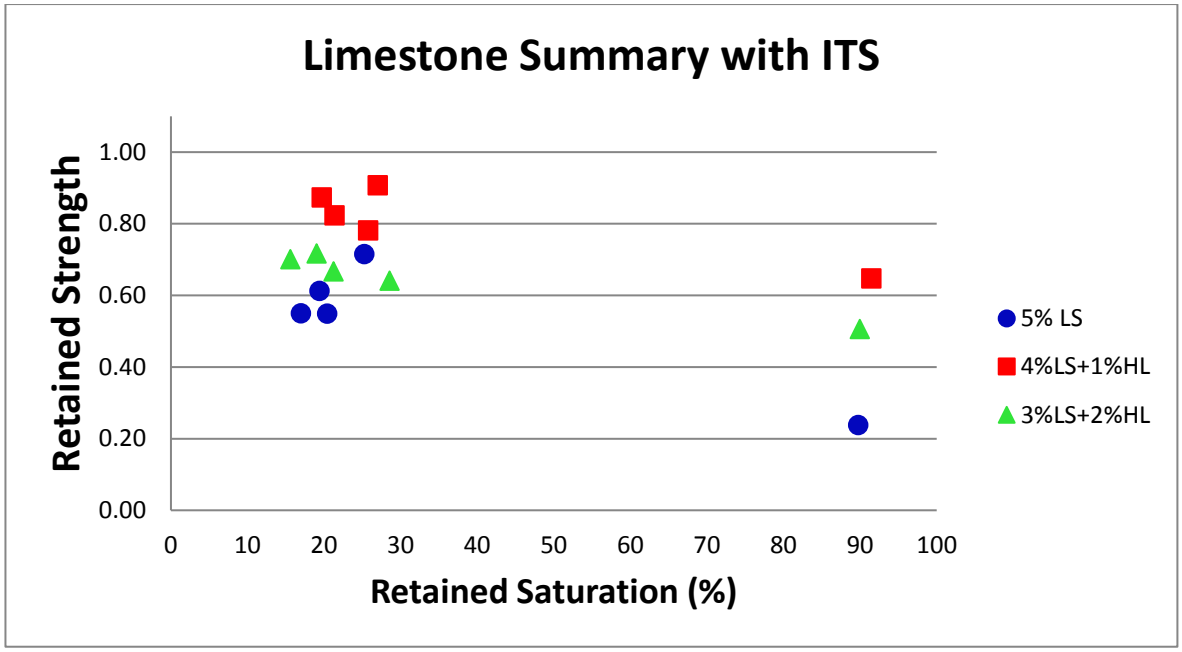


Figure 5:7: SATS test results for limestone aggregates using ITS

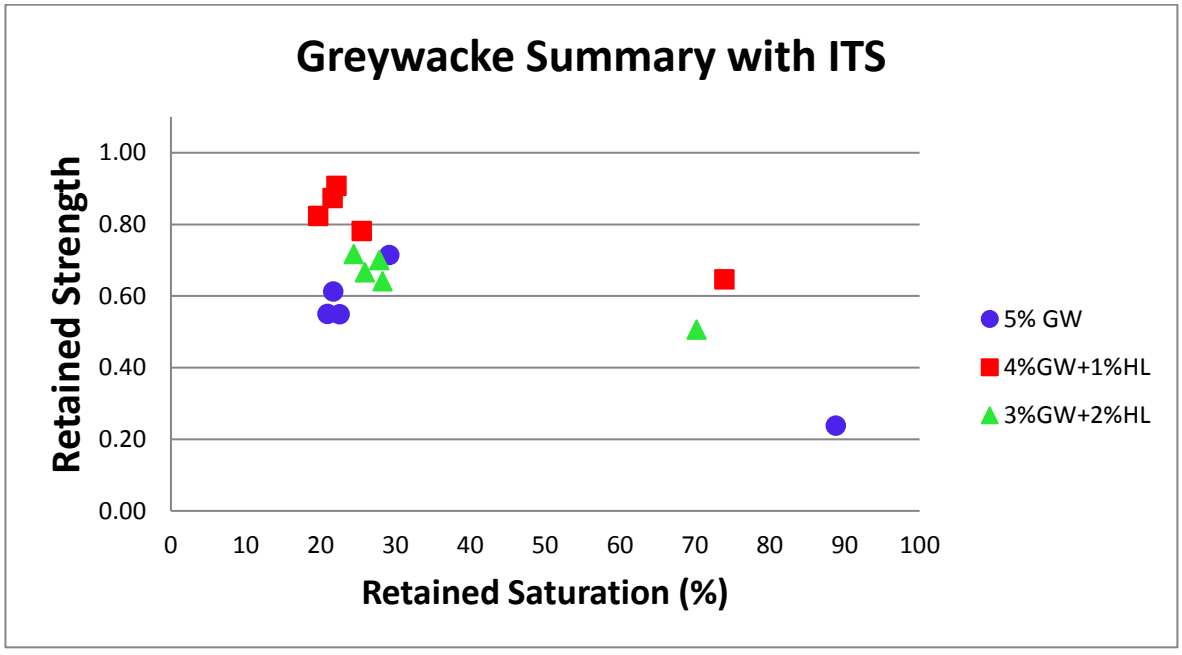


Figure 5:8: SATS test results for greywacke aggregates using ITS

Now the problem was to calculate the retained strength which is the ratio of conditioned to unconditioned cores. ITS being a destructive test it is not possible for the same cores to be used

before and after conditioning as was the case when ITSM was used. To solve this problem, three unconditioned cores were tested with ITS first and a mean value was calculated. After that, the cores conditioned under SATS protocols were tested with ITS and Retained strength was calculated for cores in each of the five positions in SATS machine by diving ITS value for that particular position to the mean ITS value of unconditioned cores.

Limestone and greywacke aggregates have been tested with ITS test to get an idea if testing this way can give more distinctive results. Figure 5:7 and Figure 5:8 shows the results of retained strength against retained saturation for the two aggregate types. If we look at the results for both of the aggregate types and compare it with the original SATS test results presented in Figure 5:2 and Figure 5:4, these look very similar to each other. There has been some difference found for the fifth position but for the all other four positions, there is no significant change noted in the results for both aggregate types. Based on the results shown in Figure 5:7 and Figure 5:8 it can be concluded that ITS being destructive in nature may be more sensitive test than ITSM but after SATS conditioning ITSM is good enough to calculate retained stiffness as retained strength calculated using ITS is very similar to that of the retained stiffness obtained in using ITSM.

5.7 Summary

Three out of four aggregates including granite, limestone and greywacke showed improvement in the retained stiffness but the performance of basalt aggregate remained unaffected with the use of HL. The performance of basalt aggregates was already very good even without HL, this might be one of the reason why basalt didn't show further improvement with the use of HL. The HL content of 1% was observed better than 2% in the three aggregates who showed improved performance with the use of HL.

Moisture damage performance of the mixture without HL addition was also assessed and basalt aggregates was found strongest compared to other three types.

A comparison between ITSM and ITS test after SATS conditioning was also undertaken as some researchers believe that ITS being destructive in nature gives more distinctive results. But the results of ITSM and ITS very found very similar to each other after SATS conditioning in terms of retained strength (using ITS) or retained stiffness (using ITSM).

Chapter 6: Adhesion Testing

6.1 Introduction

As discussed earlier two type of testing techniques, including PATTI test and peel test, have been used for adhesion assessment between binder and aggregates after 0 and 7 days conditioning in water. The results are compared between different combinations to see if there is any beneficial effect of HL found by adding specific percentages of HL to replace the mineral filler of the respective aggregate. The details of each test along with the results and discussion have been presented in the coming sections of this chapter.

6.2 PATTI test

To quantify the practical work of adhesion and any beneficial effect of HL in the mastics, Pneumatic Adhesion Tensile Testing Instrument (PATTI) has been used. It is a pull-off testing technique which is used to evaluate the bitumen-aggregate bond strength. The PATTI is comprised of a portable pneumatic adhesion tester, pressure hose, piston, reaction plate and a metal pull-off stub (Figure 6:1)

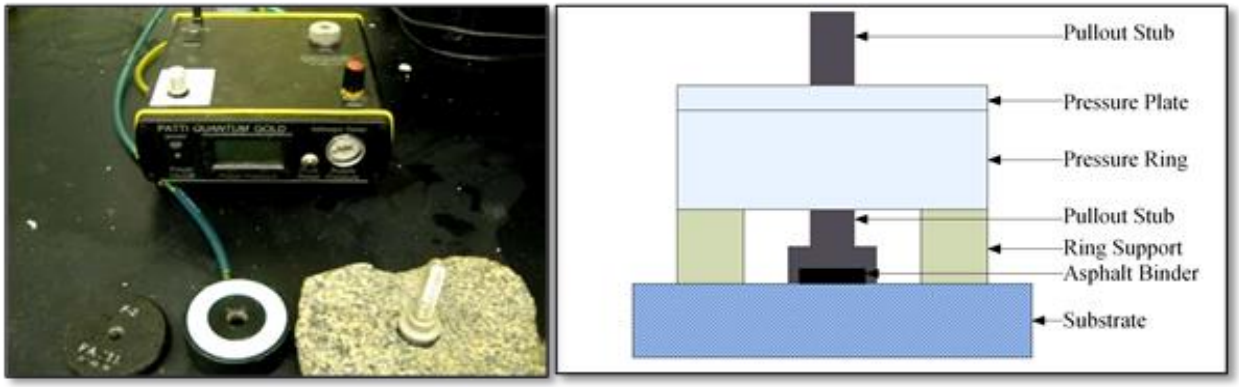


Figure 6:1: Pneumatic Adhesion Tensile Testing Instrument

The portable device displays the results in terms of the pull-off tensile strength, which is a measure of the cohesive or adhesive strength depending upon the type of failure. All the samples were tested after 0 days and 7 days conditioning time under water at 20^{o}C.

Due the unavailability of boulders for the basalt aggregates it was not possible to perform the PATTI test with basalt aggregates so only three aggregates substrates (i.e. granite, limestone and greywacke) have been used with four combinations of binder as discussed in Chapter 3. For each combination 3 repeats have been carried out. Each combination has been tested after 0 and 7 days conditioning under water.

Initially, testing was carried out with a granite substrate with four binder combinations and samples were tested in dry conditions in accordance with the standard PATTI test. Conditioned samples were soaked in water for 7 days at 20°C and then tested within a few hours of taking them out of the water. The results for 0 and 7 days conditioning time are shown in Figure 6:2.

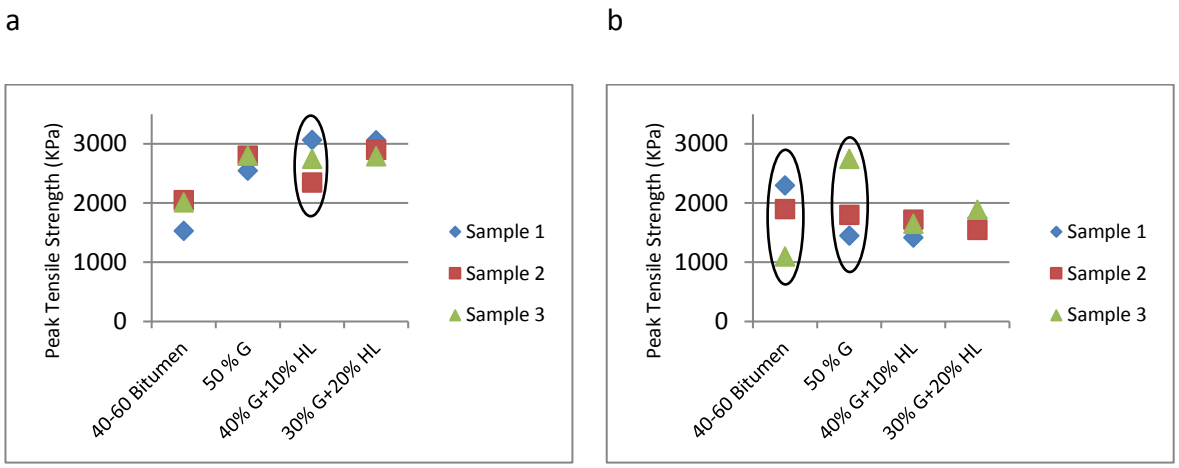


Figure 6:2: Standard PATTI test results with Granite for a) 0 days conditioning b) 7 days conditioning

Results presented in Figure 6:2 shows a high level of inconsistency, especially the results shown in Figure 6:2b for 7 days conditioning which shows a big scatter in the results for some combinations. To achieve more consistency in the results a new technique has been introduced. In this technique, the samples have been tested under water to prevent samples from being dried out. As shown in Figure 6:3 only the substrate and some part of pull off stub is submerged under the water and rest of the procedure remains the same.



Figure 6:3: PATTI test, substrate and pull off stub submerged in water and other test assembly

Testing has been carried out on granite, limestone and greywacke substrates after 0 and 7 days' water conditioning. A comparison of peak tensile strength between 0 and 7 days conditioning for neat bitumen and 3 mastic compositions with granite, limestone and greywacke substrates have been made. Error bars have also been plotted using the ± 1 standard deviation calculation from the three trials in each combination.

Results seem to be quite consistent, but with some combinations, there is still a bit of variability between the three repeats. However, these results are far better than the results using the standard dry conditions during testing. Based on these facts it can be deduced that repeatability of the PATTI test under water is far better than under the standard conditions in which samples are tested out of the water. The previous statement is only true for moisture damage assessment when samples are conditioned for a specific duration in water and are tested for bond strength using PATTI test. Despite the fact that the PATTI has a slightly poor repeatability (Kanitpong and Bahia 2003), it is still a quick and useful tool to give an adhesion indication.

The results for the granite aggregates have been presented in Figure 6:4. In the dry condition, all the three mastics are clearly performing better than the neat bitumen but the difference within different types of mastic used with granite aggregate is not distinct especially between the mastic having 0% HL (50%G) and the mastic with 10% HL (40%G+10%HL). On comparing 50%G mastic with 30%G+20%HL there are some improvement which can be noted in the form of 11% increment in the average peak tensile strength value.

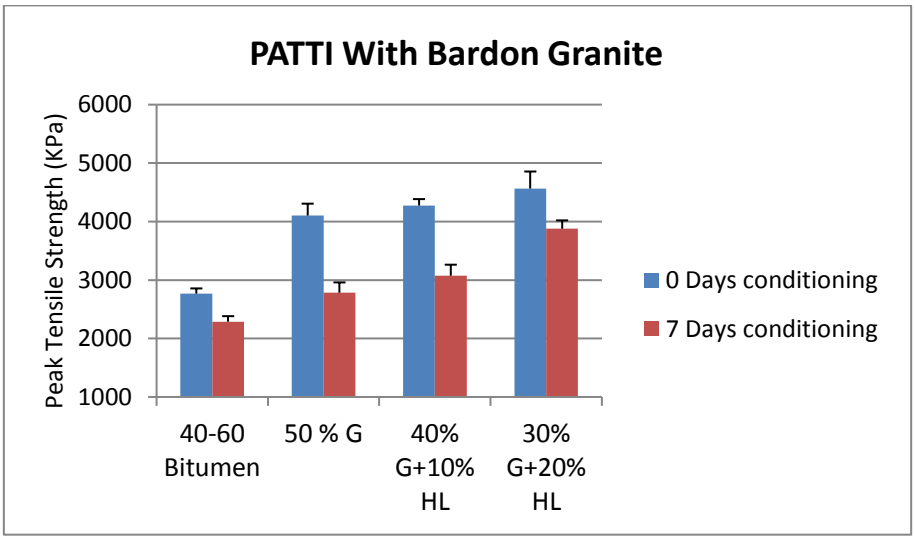


Figure 6:4: PATTI results under water for 0 and 7 Days conditioning with granite substrate

The results after 7 days conditioning in water are clearer and the beneficial effect of HL can be noted easily. Again the mastics are behaving better than neat bitumen. Performance of the two mastics having 0% HL and 10% HL are again similar but an increase of 40% has been calculated in the average peak tensile strength between the mastic having 0%HL and the one having 20% HL. Although there is an increase in the average peak tensile strength in the dry and wet conditions with the addition of 10% HL to the mastic but it is not very distinctive. On the other hand, the addition of 20%HL is showing a clear and distinctive difference in the average peak tensile strength compared to the mastic with 0% HL.

Now the question is why with the addition of granite filler or its combinations with the hydrated lime resulted in an increased peak tensile strength after zero and 7 days' water conditioning? To explain this, we need to refer back to Chapter 4, Table 4.3. If we look carefully at the surface energy components of the neat bitumen we find that this bitumen is acidic in nature as the acidic component of the surface energy is more than its basic component. With the addition of 50% granite filler, there was an increase in the basic component of the surface energy observed while a decrease was observed for the acidic component. Similarly, the addition of 40% Granite filler and 10% HL, to form a mastic, resulted in a further increment of the basic component of SFE and a further reduction in its acidic component. The trend continued with the use of filler having 30% G and 20% HL and the mastic gave an even higher basic component of SFE and similarly, the acidic component was further reduced. This increase in the basic component of SFE and decrease in the acidic component make the bitumen/mastic more favourable to make a strong bond with the acidic aggregates. That is the reason why an improved adhesion has been observed in all three mastics used with the granite aggregates compared to the neat bitumen, especially the mastic having HL.

To check the type of failure, one representative sample from each combination with granite aggregate after 0 and 7 days have been presented in Figure 6:5.

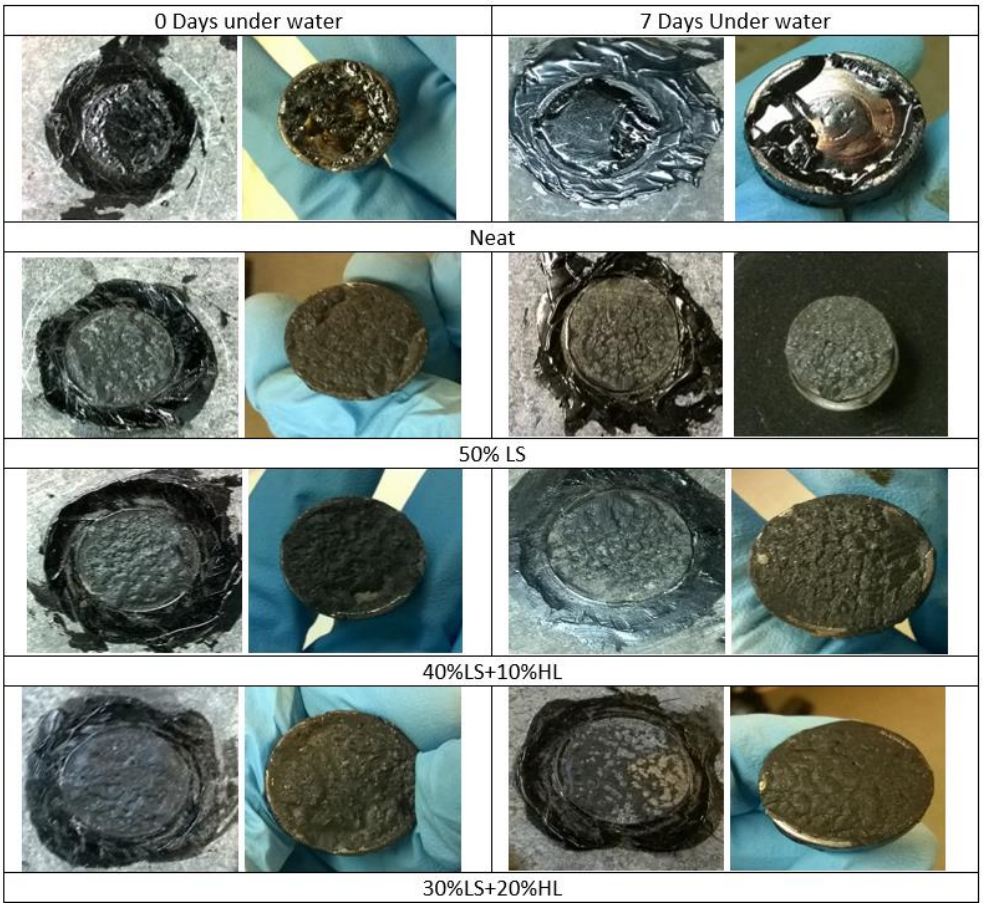


Figure 6:5: Mode of failure for different combinations with Granite aggregates

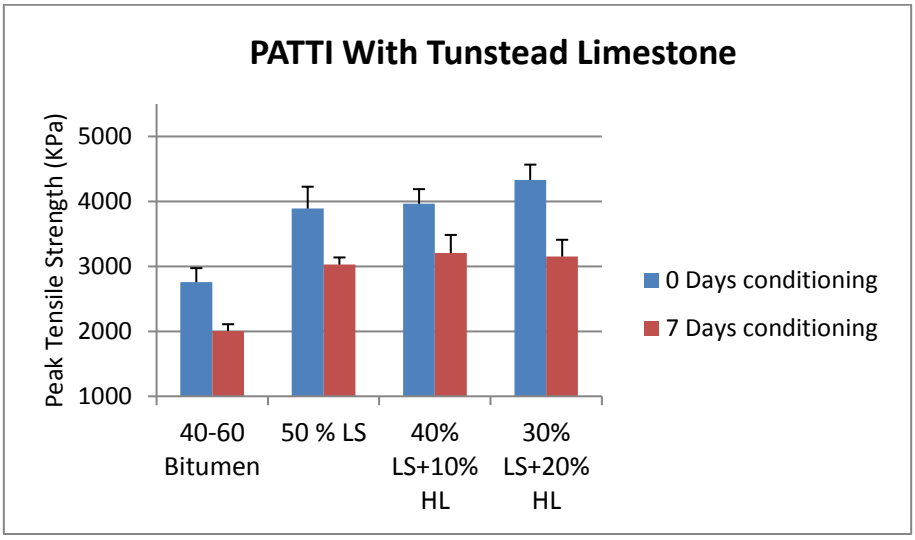


Figure 6:6: PATTI results under water for 0 and 7 Days conditioning with limestone substrate

The failure looks cohesive for the neat bitumen and the three mastics after zero days conditioning but after 7 days' water conditioning it looks a mixed type. For the neat bitumen, it is still cohesive. For the mastics, it is mainly cohesive but in some patches, it looks adhesive as well. This means water conditioning resulted in the loss of adhesive strength between mastic and aggregates. If we look carefully for the type of failure in the three mastic types after 7 days' water conditioning they do not look very different from each other but when it comes to average peak tensile strength, combinations with HL give higher values compared to the one without HL especially the combination having 20%HL. This means that the type of failure is same but the bond between the aggregate and the mastic having HL is stronger in the presence of water.

The results for the limestone aggregate combinations after 0 and 7 days conditioning in water have been presented in Figure 6:6. The results for the samples tested after 0 days conditioning in water look very similar for the three mastics combinations. Although there is a clear increase in the average pull-off strength for the mastic combinations compared to neat bitumen there is hardly any difference noted within different mastics used with the limestone aggregate substrate. The results for the samples tested after 7 days conditioning in water do not tell a different story as again the average peak tensile strength for the three mastics is in a very narrow band. So based on these results it is difficult to say that there is any beneficial effect of HL observed with this particular limestone.

The above results can be explained further by correlating them with results obtained from surface energy (Chapter 4, Table 4.3). There was an increase in the basic component of the mastic having 50% LS filler and a decrease in the acidic component compared to neat bitumen. Also in the mastic having 40% LS filler and 10% HL the basic component was further increased but very slight change was observed in the acidic component. With the use of 30% LS filler and 20% HL in the mastic, an unusual drop in the basic component was observed while the acidic component dropped slightly. So based on surface energy results it can be concluded that the increase in the basic component of the bitumen with the addition of filler will not result in an improved bond strength if used with limestone aggregate as it is already basic in nature. So even if the use of LS or HL filler makes the bitumen more basic in nature yet the adhesion will not improve if used in the combination with limestone aggregate.

The failure mode for all the combinations with limestone aggregates has been presented in Figure 6:7. Under dry conditions again the failure type looks cohesive for neat bitumen as well as for the three type of mastics. Under wet conditioning, failure type is a combination of cohesive and adhesive failure with the cohesive failure dominating in all the combinations. As

was the case with granite, limestone's mode of failure between different mastic combinations looks very similar but the average peak tensile strength is showing a different trend. Granite was showing the beneficial effect of HL addition but here there is practically no effect of HL addition.

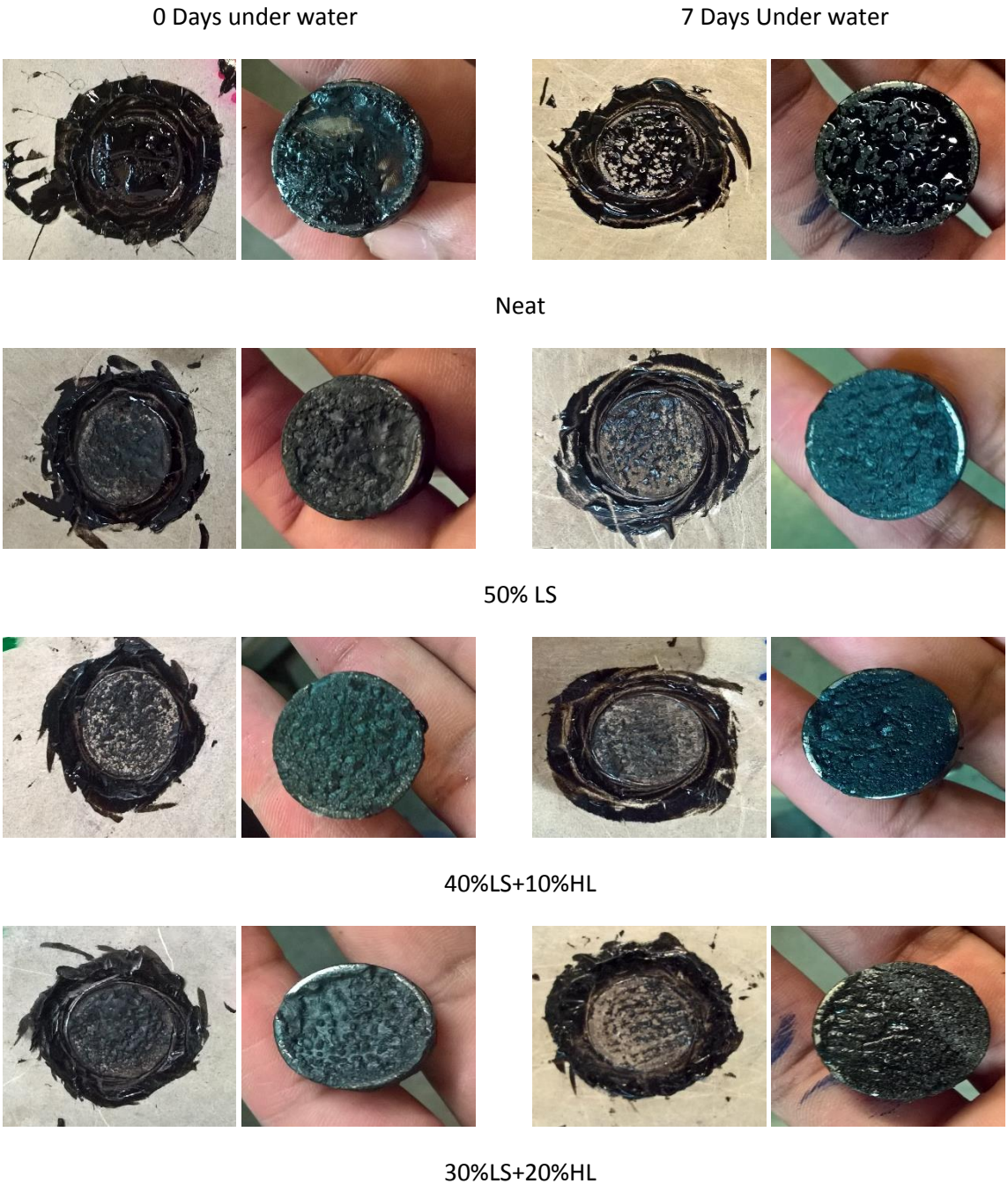


Figure 6:7: Mode of failure for different combinations with limestone aggregates

The case of greywacke aggregates is similar to the granite aggregate where there is a significant improvement of the HL addition compared to the mastic consisting of only greywacke filler

(Figure 6:8). For the 0 days conditioning time, there was a significant increase of 32% with the replacement of 10% greywacke filler with HL. The trend continues with the replacement of 20% greywacke filler with HL and an increase of 38% was achieved compared to the mastic having only greywacke filler (50%GW). With the 7 days' water conditioning there was a significant drop in the average peak tensile strength of 50%GW mastic but the mastics having 10%HL and 20% HL remained steadfast and showed very stable results after 7 days of moisture conditioning. Comparing the results of three mastics after 7 days' water conditioning an increase of 100% and 120% has been found in the mastics having 10%HL and 20%HL respectively. This increment is huge and the effect of HL addition is quite obvious.

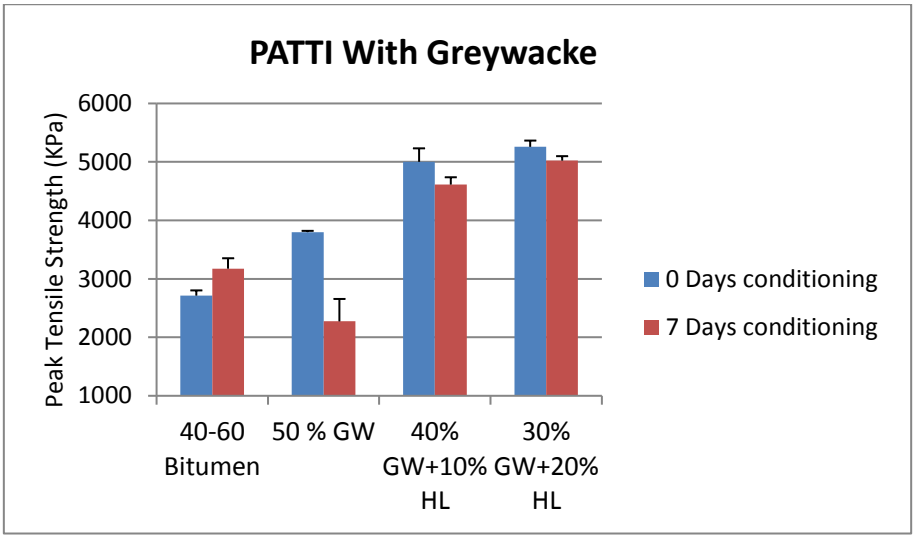


Figure 6:8: PATTI results under water for 0 and 7 Days conditioning with greywacke substrate

To explain the phenomenon associated with results presented in Figure 6:8 once again we have to look back at Chapter 4, table 4.3 where the results of surface energy for neat bitumen and various mastic are presented. An increase in the basic component of the mastic having 50% GW filler and decrease in the acidic component was observed compared to neat bitumen. Also in the mastic having 40% GW filler and 10% HL the basic component was further increased and a slight increase was observed in the acidic component. With the use of 30% LS filler and 20% HL in the mastic, a further increase in the basic component was observed while the acidic component dropped slightly. As a result of this change in the basic component of mastics, the adhesion between the acidic natured greywacke aggregate and the mastic has increased compared to neat bitumen. And with the use of HL, this adhesion has improved further which can be seen clearly in the results of practical adhesion presented in Figure 6:8.

Mode of failure for greywacke aggregates has been summarised in Figure 6:9 for all the four combinations.

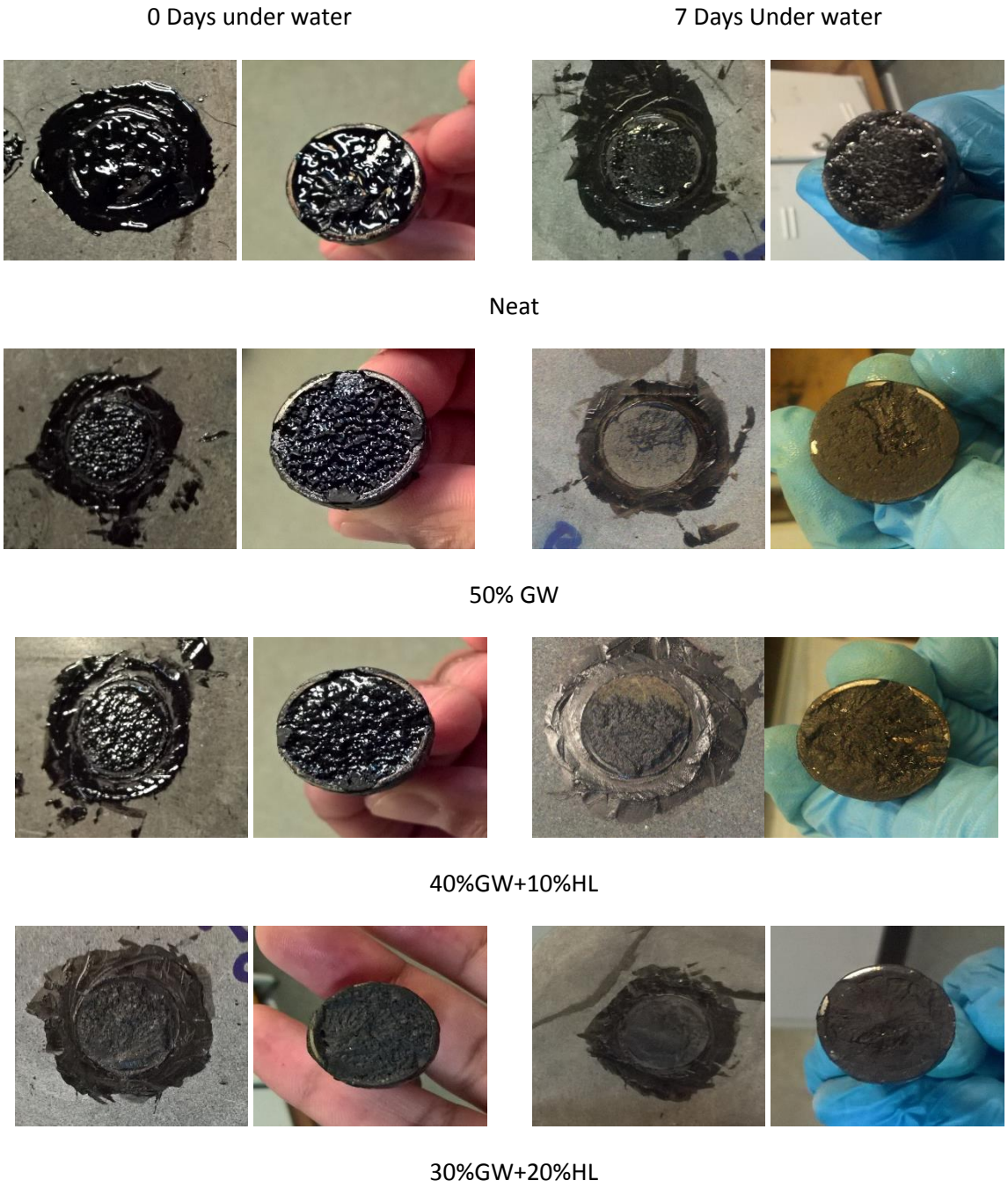


Figure 6:9: Mode of failure for different combinations with greywacke aggregates

The failure type after zero days conditioning is cohesive again in all the combinations of greywacke aggregates with neat bitumen and mastics. However, the mode of failure after 7 days conditioning in water is very interesting. Neat bitumen has failed in cohesion. The mastic

having only greywacke filler gave a mixed failure type with adhesive failure type dominating the cohesive. The mastic having 10%HL also gave a mixed failure mode with mainly cohesive failure and the mastic having 20%HL failed in pure cohesion. If we again compare the three mastics used with the greywacke aggregates, the mastic 50%GW gave the least value for average peak tensile strength and the mode of failure was mainly adhesive. The mode of failure changes to mainly cohesive and a higher value of average peak tensile strength was achieved with the mastic having 10%HL. For the mastic having 20%, HL the mode of failure was purely cohesive and this combination gave the highest average peak tensile strength value. This means the mode of failure is also very important and can be correlated with the peaks tensile strength value in some cases.

Based on the above results it can be stated that HL is not always beneficial for adhesion, and it is the type of aggregate which decides its efficiency. To conclude, with this particular test technique, HL is showing best performance with greywacke, granite comes second while limestone is not showing any beneficial effect in the presence of HL.

6.3 Peel test

One of the most widely used methods to access the bond strength between two materials is the PEEL test. The details of the test method and the sample preparation have been described in Chapter 3.

In the asphalt pavement, moisture damage is a complicated phenomenon which leads to a pavement failure due to cohesion or adhesion loss within the bitumen itself or at the interface of bitumen and aggregate surface. So the study of the fracture properties due to adhesion or cohesion loss is very important. The Peel test has been used successfully to quantify this fracture strength between bitumen and aggregates using a 90^{o} peel test under both dry and wet conditions (Zhang et al. 2015). In the peel test, big boulders are trimmed to a required dimension to get aggregate substrates ready for the test. A new test method for the preparation of aggregate substrates has been developed by (Zhang et al. 2016). In this method crushed aggregates are glued together in a mould and are trimmed to the required size. In this study, this new method of composite substrate peel test has been used for the three types of aggregates (Granite, limestone and basalt). Normal Peel test has been used for one aggregate type (Greywacke) due to unavailability of 28mm nominal sized aggregate. So big boulders were arranged for greywacke and trimmed accordingly.

6.3.1 Material used for Peel test

Four types of aggregates have been used in this test technique including; Granite, Limestone, Basalt and Greywacke. One bitumen type i.e. 40/60 pen neat has been used along with three mastic combinations depending on the type of aggregate used. The details of the source, properties and the combinations of which aggregates and bitumen/mastics have been used are mentioned in Chapter 3.

6.3.2 Peel test results using Aluminium foil sheet

In the first place, granite aggregates were tested using composite substrate Peel test under dry and wet conditioning. For the wet conditioning, the samples were soaked in water for 7 days at 20^{o}C before testing. The results for the neat bitumen, Mastic with 0%HL (50%G) and Mastic with 10% HL (40%G+10%HL) have been presented in Figure 6:10.

Based on the results presented in Figure 6:10 it is difficult to discriminate between neat bitumen and the mastic with 0%HL and the mastic with 10% HL. There is no difference between the two mastics and there seems to be zero effect of HL addition. By looking at the results it can be commented that both mastics are in fact behaving worse than the neat bitumen. There is another very interesting thing noted in the result which is the type of failure. If we look at the results of neat bitumen the type of failure is very obvious which is within the bitumen layer and so is cohesive. All three samples failed in cohesion. On the other hand, the failure in both of the mastic types is very unusual and is between the mastic and the peel arm which is not a representation of what we are looking for. One of the prerequisites of the Peel is that the failure should be either within the bitumen/mastic or between the bitumen/mastic and the aggregate substrate. As the type of failure recorded with the mastics differs from the basic principle of the test, the results are not acceptable and of course not comparable. The type of failure suggests that the bond between the mastic itself and the bond between the mastic and aggregate is stronger than the bond between the peel arm and the mastic. Although peel test has been used successfully with a range of neat bitumen using Aluminium foil as the peel arm, the testing of mastic with the peel test seems challenging.

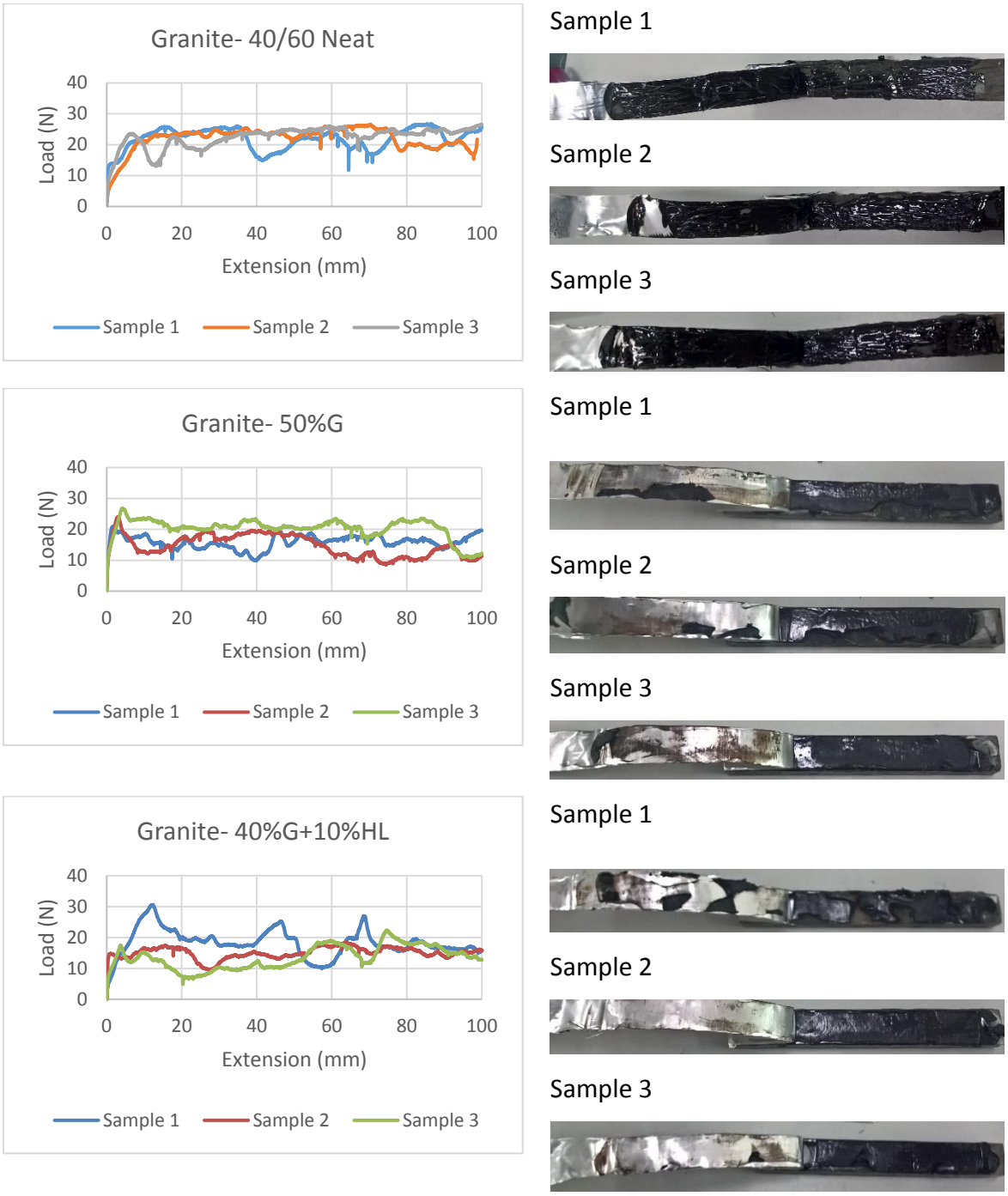


Figure 6:10: Peel test results under dry conditions using Aluminium Peel arm for Granite aggregates combinations

Another set of samples made with 40/60 neat bitumen, mastic with 0%HL (50%G) and Mastic with 10% HL (40%G+10%HL) in combination with granite substrate were conditioned for 7 days under water at 20^{o}C before testing. Peel test results for these samples have been presented in Figure 6:11.

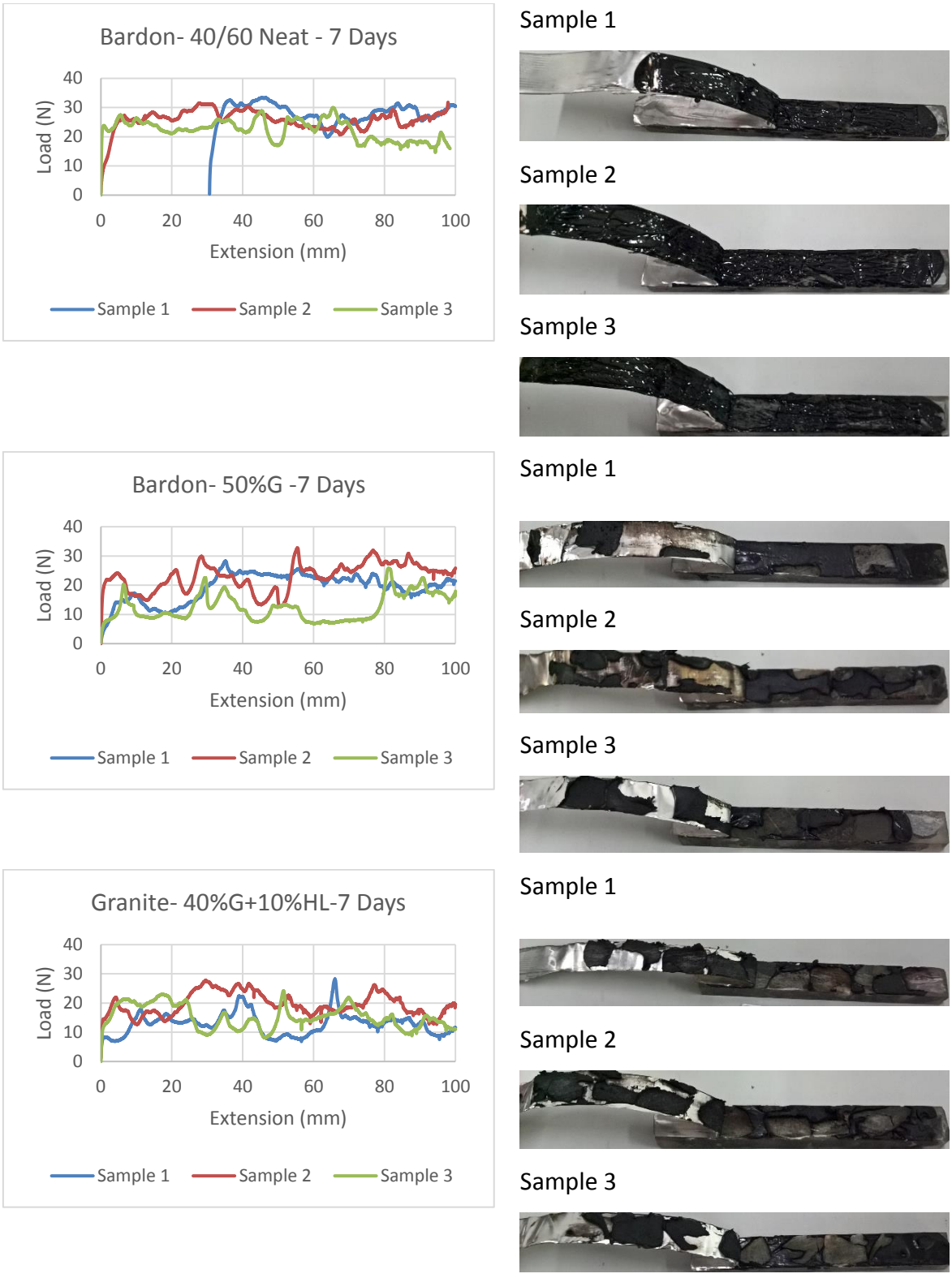


Figure 6:11: Peel test results after 7 days water conditioning using Aluminium Peel arm for Granite aggregates combinations

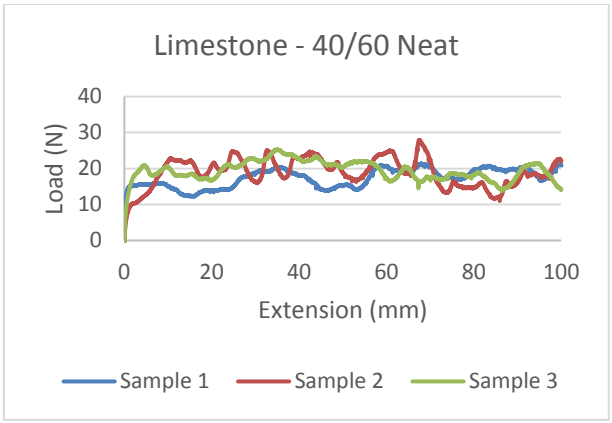
By looking at the results, again all the combinations look quite similar to each other. The combination of the neat bitumen again giving slightly higher results than the two mastics. On

the other hand, the two mastics used are giving very similar results although with a high level of noise being recorded between the results of three runs within the same combination. Type of failure is a slightly different than the one under dry conditions which is interesting. The neat bitumen is failing in cohesion again but the type of failure in the mastic combinations is either adhesive, which is between aggregate and bitumen or it is the failure between the mastic and the aluminium peel arm and there is no cohesive failure recorded in all the combinations shown above. This means that the bond within the mastic itself is stronger than the bond between mastic and aggregate and even between the mastic and aluminium peel arm. Under these conditions, it is not possible to compare the results under dry and after 7 days conditioning under water because if the failure occurs between the bitumen/mastic and the peel arm it is not acceptable, as discussed earlier. So the first challenge was to get a peel arm which can give a stronger bond with bitumen or mastic.

6.3.3 Peel test using sandblasted peel arm

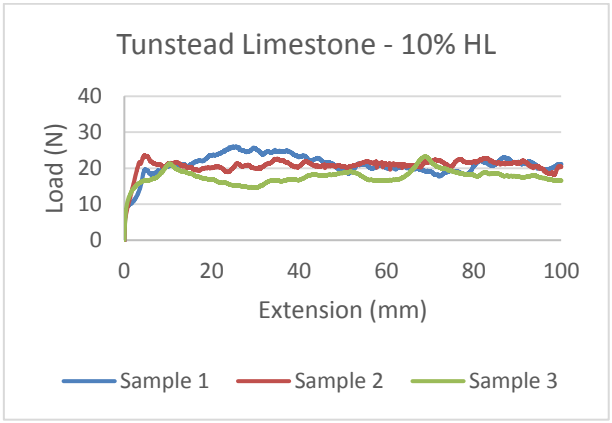
As the results obtained with the use of aluminium peel arm were not acceptable, different options were considered to get better performing peel arm material for this particular case. In the first place, a sandblasted aluminium peel arm was used. The reason behind using sandblasted aluminium peel arm was to get a rougher surface and hence a stronger bond between bitumen/mastic and peel arm.

To check if the sandblasted aluminium peel arm gives any different failure type than the one without sandblasting, limestone aggregates have been tested with 40/60 neat bitumen and the with the mastic having 10% HL (40%LS+10%HL). The test was only conducted under dry conditions and the results have been presented in Figure 6:12. By looking at the results it is difficult to find a difference between the neat bitumen and the mastic. Both give very similar average load values but again the type of failure is different between the neat bitumen and the mastic with 10% HL. Neat bitumen failed in cohesion while the failure in the mastic with 10% HL is again between the peel arm and mastic. Although blackish impression of the mastic on the peel arm can be seen but it is not the mastic and may be from the oils absorbed on the sandblasted surface. The type of failure is again not acceptable and will not tell anything about the bond between the aggregate and the bitumen/binder and hence it is not possible to draw any comparison in dry and wet combinations.



Sample picture





Sample picture



Figure 6:12: Peel test results under dry conditions using sandblasted aluminium peel arm for limestone aggregates combinations

6.3.4 Peel test using Hessian cloth

Based on the results obtained from the aluminium peel arm with and without sandblasting, it can be concluded that the aluminium peel arm is not the right material to be used for peel testing if mastics are to be tested. Alternate materials options were therefore considered. Peel arm material should be rough enough to make a strong bond with binder. Also, it should be soft enough so that it can be easily used to manufacture the sample and can give a 90^{o} Peel off while testing. Wire mesh was one of the choices but it was a bit hard and when some trial samples were tested 90^{o} peel was not achieved due to the hardness of the sample. Also, the samples prepared were not of good quality and the bond which was expected to be stronger was observed to be weaker instead. The Hessian cloth was another choice to be used as a peel arm material. This cloth was soft and rough and was expected to give a strong bond with the bitumen/mastic. Due to its softness, it was also assumed that 90^{o} peel off would not be an issue. A few trials were made under dry conditions to check the suitability of the hessian cloth as a peel arm material. The results were quite consistent and the type of failure was either cohesive

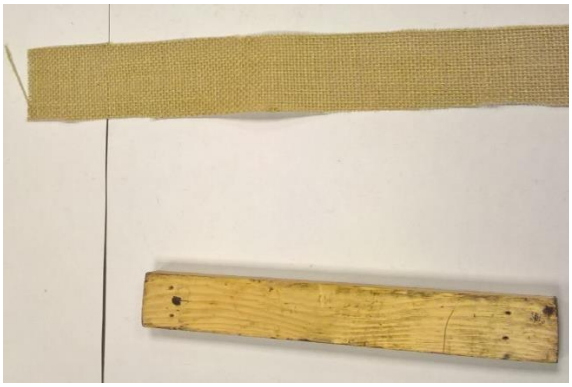
(within the bitumen/mastic itself) or adhesive (between the bitumen/mastic and aggregate). Based on the initial results it was decided to run a full test with all the combinations under both dry and wet conditions to check how the results would be and if any comparison can be drawn. One of the challenges using the hessian cloth was to make consistent samples by maintaining the right thickness. To make the best possible sample using the hessian cloth different steps were undertaken. These steps have been explained in Figure 6:13as follows;



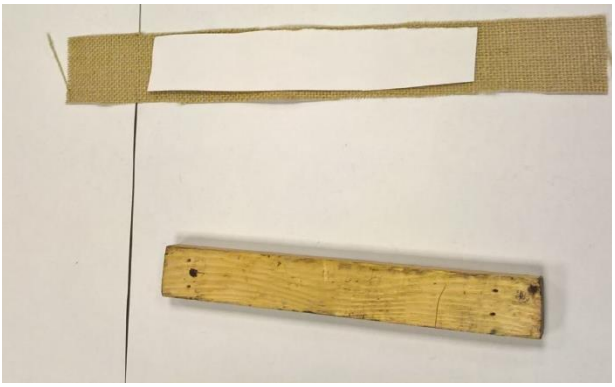
1. Cut the hessian cloth in a relatively bigger size than the size of aggregate substrate



2. Heat the clean and dry substrate in the oven at 70^{o}C for 1 hour.



3. Cut a wooden piece to slightly bigger dimensions than the substrate.



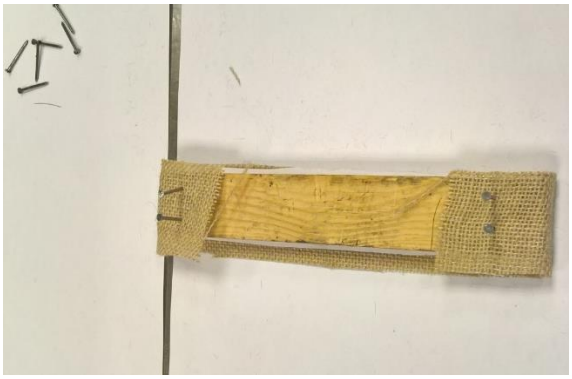
4. Cut a piece of paper to the size of the wooden piece and place it over the hessian cloth.



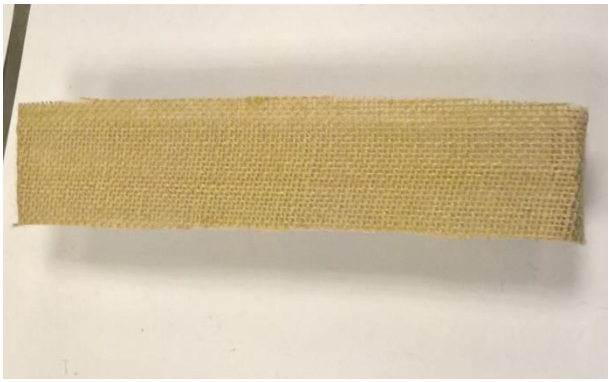
5. Place the wooden piece on the top of the paper



6. Hammer two nails into the wood as shown to clamp the hessian cloth from one side



7. While one side clamped with the nails, stretch the hessian cloth to remove any sag and clamp the other side with the nails again.



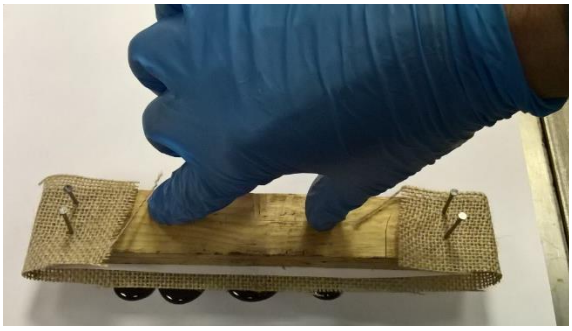
8. Once both sides clamped a sag-free surface will be obtained on the other side as shown.



9. Place five wire spaces to maintain a thickness of 0.25 mm



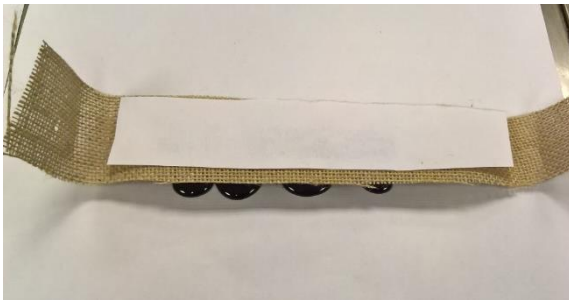
10. Pour the preheated bitumen/mastic on the top of substrate



11. Place the assembly prepared in step 7 on the top of melted bitumen/mastic and apply a gentle pressure.



12. Remove the nails from both of the sides.



13. Remove the wooden piece and the sample will be ready to trim.



14. Trim the extra hessian cloth and bitumen and test the sample as normal.

Figure 6:13: Steps for sample preparation using Hessian cloth in Peel test

In total 96 samples were manufactured for 16 different combinations with 4 aggregate types. Each aggregate type was tested with one neat bitumen (40/60 pen grade) and three mastic types (as detailed in Chapter 3). For each combination six samples were manufactured, three for the dry condition and three for the 7 days' water conditioning.

A summary of results for the granite aggregate combinations has been presented in Figure.6:14. Samples tested in the dry condition and wet condition for each combination have been summarised in one graph. The results look somewhat consistent for the three runs in each combination of dry and moisture conditioned samples. There is a very uniform noise pattern observed in all the mastics, the reason could be the nature of the hessian cloth used as peel arm.

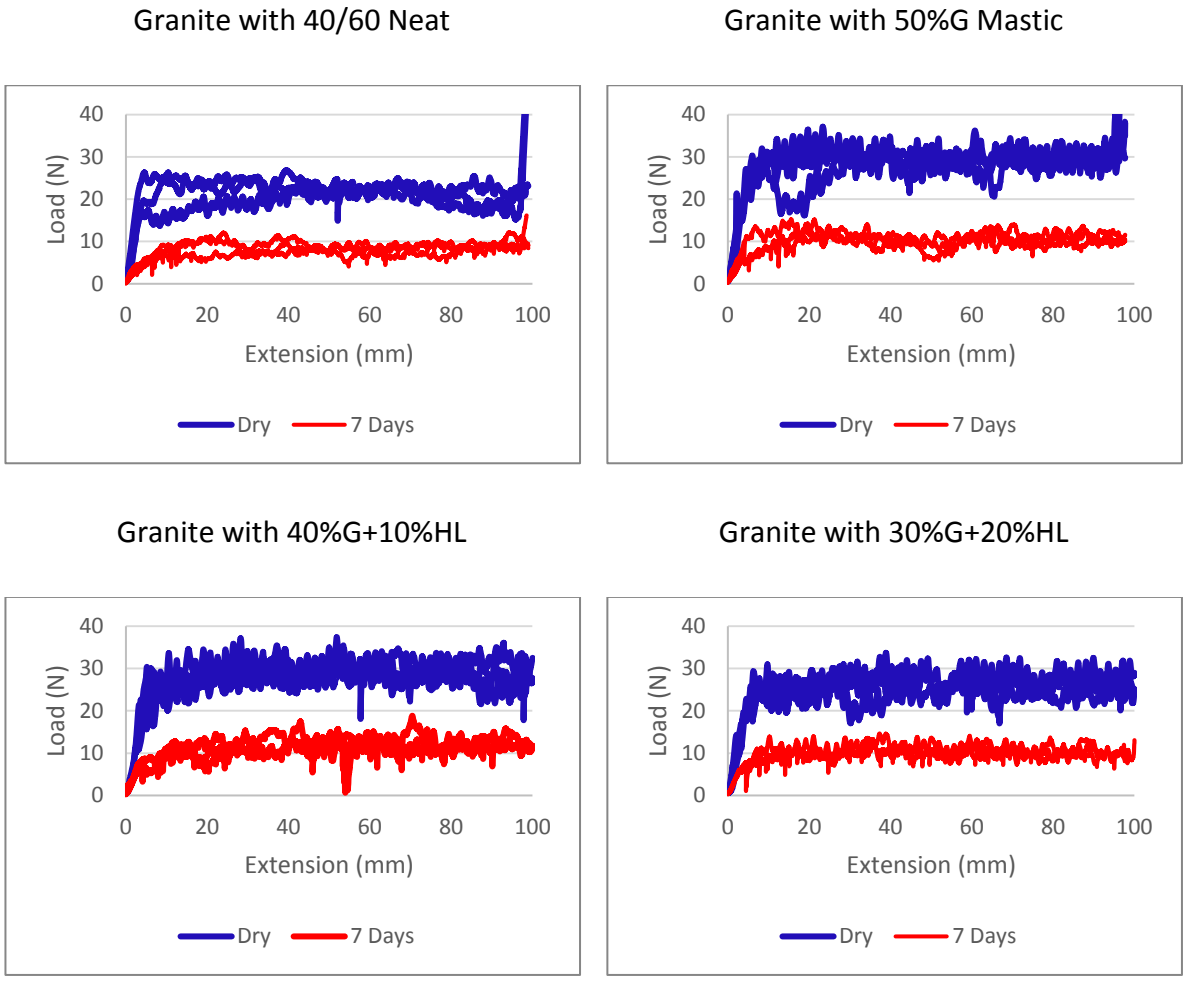


Figure.6:14: Peel test results under dry and wet conditions using hessian cloth peel arm for granite aggregates combinations

By looking at the results under dry conditions there are some differences observed in the neat bitumen and the mastics but it is difficult to find a significant difference within the three mastics. The average value of the load recorded for the neat bitumen is around 20 N (APPENDIX B summarise the procedure for calculation of average load). This value is around 30 N for the mastic with 0% HL (50%G) and for the mastic with 10% HL (40%G+10%HL). The average load value has dropped slightly to 25 N for the mastic with 20% HL (30%G+20%HL). For the water conditioned samples, the average load is about 8N for the neat bitumen and is around 10 N for all three mastics with little or no difference between them. These results for the granite aggregate combinations are still unclear and to do not show any discrimination between the three mastics. Hence it is difficult to see any beneficial effect of HL addition with the results obtained from this test.

The results for the limestone aggregates combinations have been summarised in Figure 6:15. Again the limestone aggregates have been tested with one neat bitumen (40/60 Pen) and three mastics. The further details of the mastics used with the limestone aggregates have been mentioned in Chapter 3.

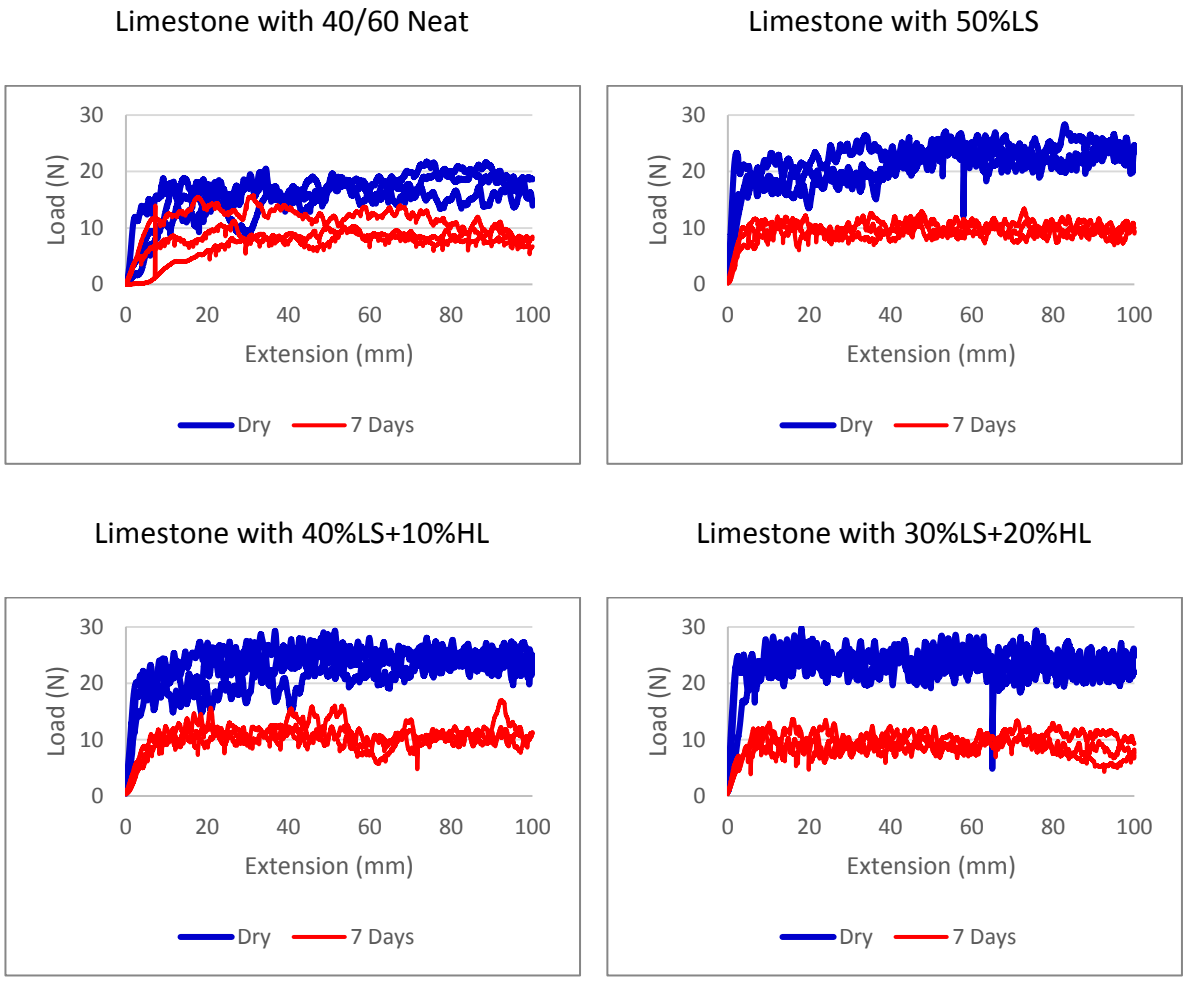


Figure 6:15: Peel test results under dry and wet conditions using hessian cloth peel arm for limestone aggregates combinations

The results of the neat bitumen are a bit more inconsistent between the three runs within the same combination compared to the mastic used with the limestone aggregate. Under the dry condition, the average load value for the neat bitumen is around 18 N. For the 50%LS mastic the average load value is just above 20 N and is around 25N for the mastic with 10%HL and 20%HL. The results under wet conditions for all four combinations including neat bitumen are very similar. The average load value for all the combinations is 10 N which is the same as the

results obtained from the mastic used with the granite aggregates. The conclusion from the results obtained for the combination of limestone aggregates is again similar to that for the granite aggregates. All the results for the dry and wet conditions are very similar to each other and it is not possible to see any beneficial effect of HL addition to the mastics.

Composite substrate Peel test results for the basalt aggregates are represented graphically in Figure 6:16. Like granite and limestone, the basalt aggregates were also tested under dry and wet conditions and tested with one 40/60 neat bitumen and the three combinations of mastic as listed in Chapter 3. The results seem to be repeatable with very similar and consistent noise patterns due to the nature of the peel arm used.

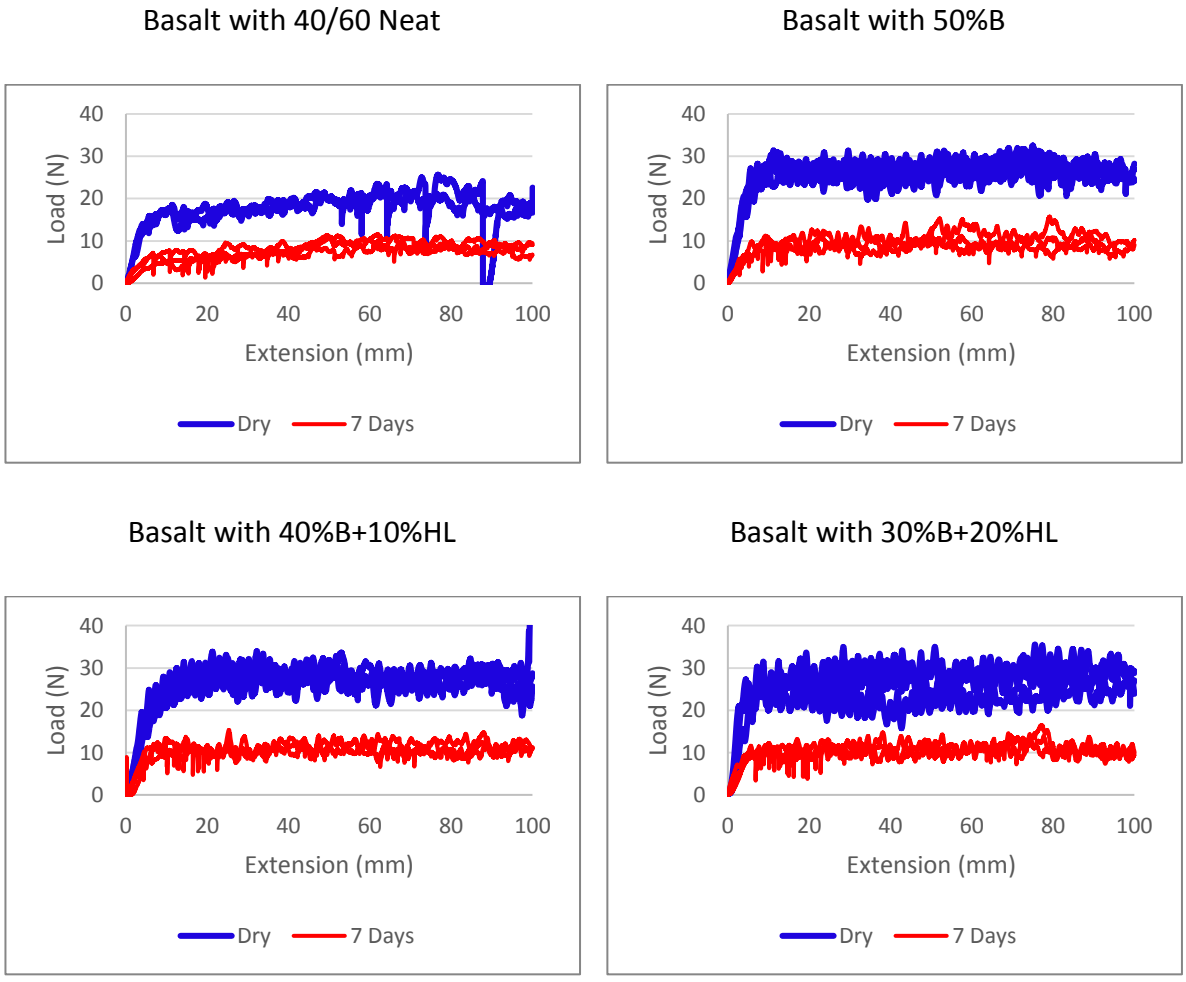


Figure 6:16: Peel test results under dry and wet conditions using hessian cloth peel arm for basalt aggregates combinations

Average load under dry conditions is about 20 N for the neat bitumen. For the mastics 50%B, 40%B+10%HL and 30%B+20%HL the average load value is just under 30 N. Similarly, after

7 days of conditioning under water the results of neat bitumen and other three mastics used with the basalt aggregates gives an average load value of about 10 N. The same results are obtained for water conditioned sample with granite and limestone aggregate. So the conclusion is same again and no effect of HL addition can be identified.

Greywacke aggregates, same as for the other aggregate types, were tested with one neat bitumen which is 40/60 pen and three mastic combinations (Chapter 3). The results are sketched in Figure 6:17 for neat bitumen and three mastics.

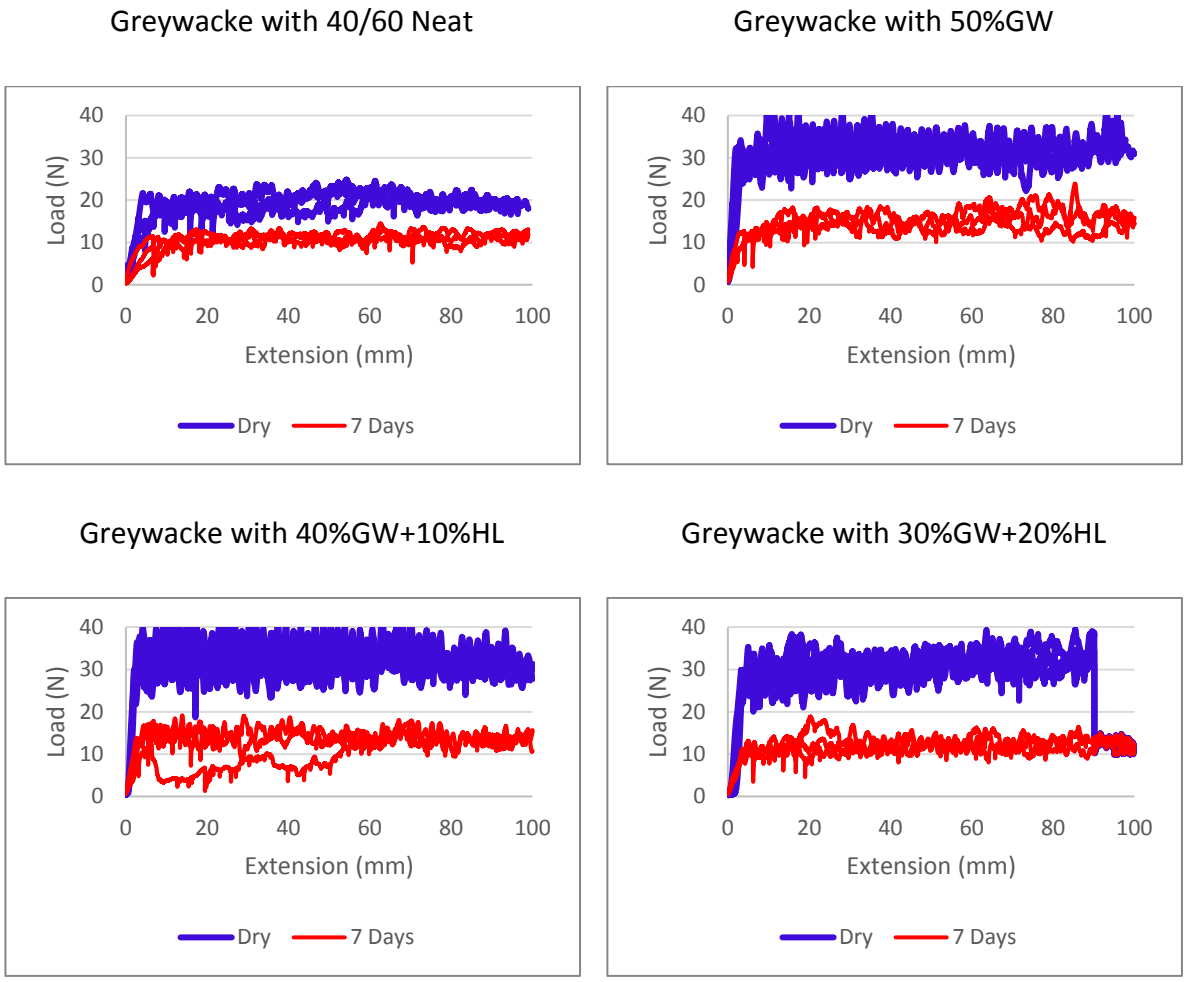


Figure 6:17: Peel test results under dry and wet conditions using hessian cloth peel arm for greywacke aggregates combinations

By looking at the results under dry conditions, neat bitumen gives an average load value of around 20N. Mastics 50%GW, 40%GW+10%HL and 30%GW+20%HL give average load values of about 30, 35 and 30 respectively. For the water conditioned sample, neat bitumen gives an average value of 10 N, mastic with 0% HL (50%GW) and 10% HL

(40%GW+10%HL) gives a value of about 15 N and the mastic with 20% HL gives just about 10N average load value. These results are slightly different than the other three types of aggregates discussed above. In terms of the effect of HL addition it can be seen that there has been a slight improvement with the addition of 10% HL under dry conditions but after the conditioning, the results for the mastic without HL and the mastic with HL are the same.

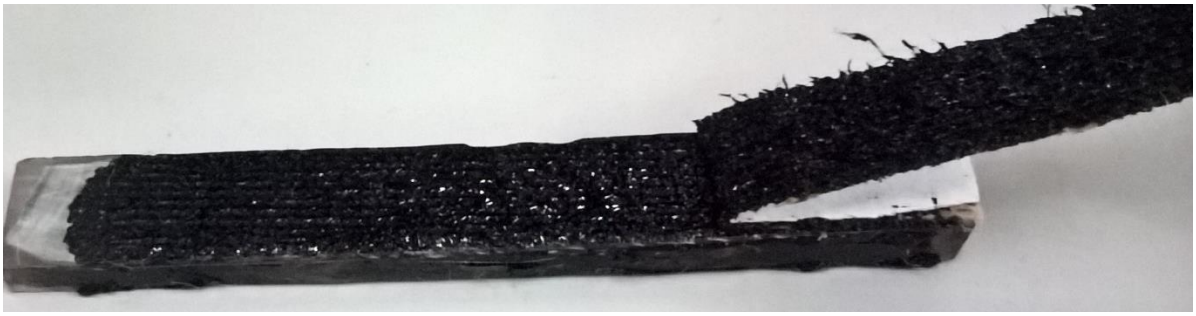
By looking at the results of all aggregate types under dry condition one can observe some difference between average load values among the combinations but under wet condition the average load value for most of the mastic combinations for all four aggregate types are similar, with a value around 10 N. One of the reasons why the average load was the same for most of the combination may be the type of failure and the hydrophilic nature of the cloth used in the replacement of aluminium peel arm.

To get an idea about the type of failure, pictures have been captured after each test. Some of the pictures of granite aggregates have been presented in Figure 6:18 and Figure 6:19 to get an idea how the failure surface looks like with and without water conditioning.

The failure under dry conditions looks very much cohesive in nature for the neat bitumen and the three mastics. But if we look at the type of failure after 7 days' moisture conditioning it looks a bit unclear. For the sample made up using neat bitumen it seems cohesive but for the mastics, it is difficult to judge the failure type as shown in Figure 6:19. In the first place it looks like cohesive failure within the mastic itself but actually, the failure has occurred between the cloth and the mastic. The cloth being hydrophilic in nature absorbed water and its bond with the mastic became weak and hence failure occurred between the cloth and mastic instead of within the mastic itself or between mastic and aggregate substrate.

Based on the results obtained with the use of hessian cloth with different aggregate types, it is difficult to evaluate the suitability of the peel test using any cloth type as a peel arm but there is still a chance that it could be helpful if the samples are to be tested dry (without water conditioning). The aggregates used in this study have shown very similar results under dry conditions for the two techniques used (PATTI test and Peel test) and that is the reason why there is little difference in the results of Peel test when the hessian cloth is used. It will be a good idea to test some of the aggregates which are known to behave very differently under dry conditions based on some other test techniques and then decide whether the Peel test with hessian cloth has a future for stiff or hard binder under dry conditions or not. Obviously as explained earlier, for the sample to be tested after water conditioning hessian cloth will absorb

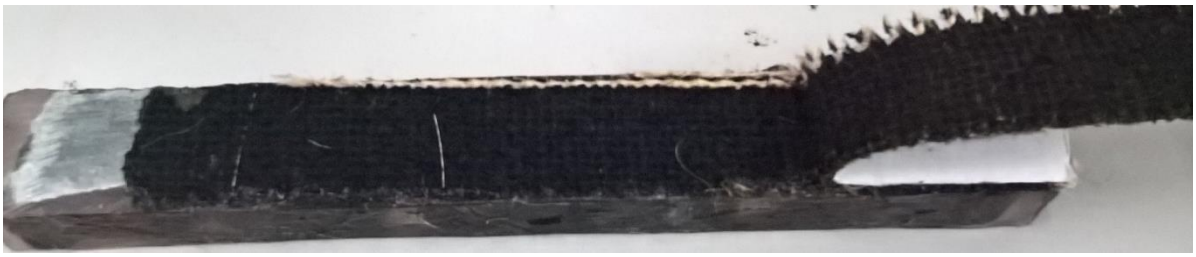
water and the bond between cloth and binder will become weak which will result in a failure between the binder and the cloth which is not acceptable. So under dry conditions, it may be helpful to compare two aggregates having different nature but not under wet conditions.



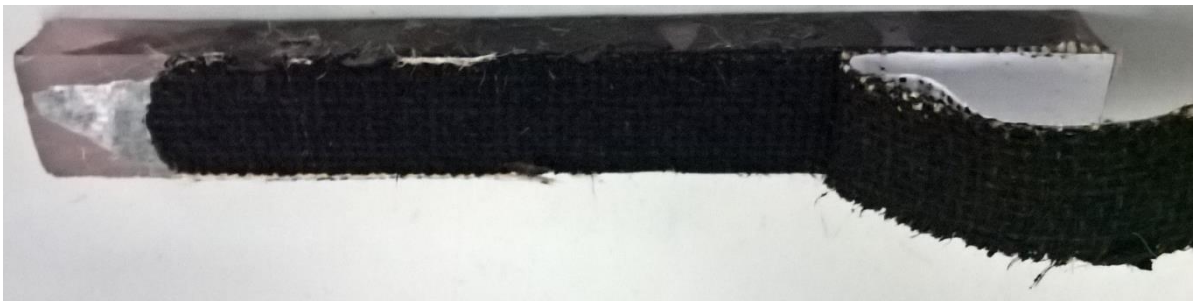
Granite with 40/60 Neat – Dry



Granite with 50%G – Dry



Granite with 40%G+10%HL – Dry



Granite with 30%G+20%HL – Dry

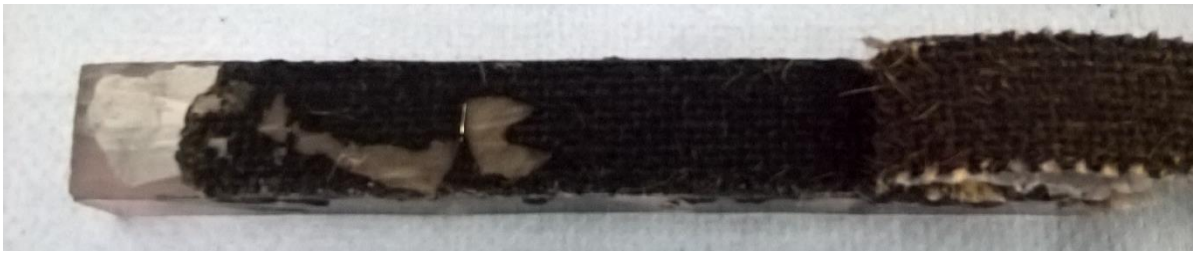
Figure 6:18: Assessment of failure mode under dry conditions for granite aggregate combinations using hessian cloth peel arm



Granite with 40/60 Neat – 7 Days water conditioning



Granite with 50%G – 7 Days water conditioning



Granite with 40%G+10%HL – 7 Days water conditioning



Granite with 30%G+20%HL – 7 Days water conditioning

Figure 6:19: Assessment of failure mode under after 7 days water conditioning for granite aggregate combinations using hessian cloth peel arm

6.4 Summary

To summarise, an effort has been made to quantify the practical work of adhesion between the four aggregates and various combination of bitumen or mastic with the help of PATTI and Peel test. To reduce the poor repeatability of PATTI test, samples were successfully tested inside the water and results were found a lot better in terms of repeatability. Samples were tested after

0 days conditioning and 7 days' condition in water and retained tensile strength was quantified. An improvement in the results for granite aggregate was observed with the addition of HL. Though there was some improvement found with the use of 10% HL in the mastic but with the 20% HL in mastic there was a clear difference observed. Limestone aggregates didn't show improvement with the use of HL but the greywacke aggregates responded strongly in the form improved retained tensile strength with the use of 10% HL. There was no difference observed with 20% HL substitution for greywacke aggregates.

Various trials were made to get the results using Peel test when mastics were tested. Sadly, no attempt was successful. First attempt was made using standard Aluminium peel arm and the failure occurred between peel arm and bitumen mastic which do not tell anything. The second attempt was made by using sandblasted peel aluminium to make the surface rough hence improving the bond between mastic and peel arm but again the failure occurred between peel arm and mastic. Wire mesh was also tried as peel arm but it was not possible to make 90^{o} Peel test due to the hardness of the mesh and also sample preparation was very difficult. Lastly, Hessian was used which was giving better results in terms of consistency under dry conditions but when the samples were tested after water conditioning the results of all the samples were appearing very similar. The reason may be the water absorption of the hessian cloth as the cloth absorbs water when the sample is conditioned before testing and hence the bond between cloth and binder becomes weak which results in a failure between the binder and the cloth and is not acceptable. So under dry conditions, it may be helpful to compare aggregates having different nature but not under wet conditions.

Chapter 7: Comparison between test techniques

7.1 Introduction

A comparison has been made between all the test methods used, for the evaluation of HL effect on moisture damage, for each aggregate type separately. These methods include rolling bottle test (RBT), surface energy technique, Saturation Ageing Tensile Stiffness (SATS) test and pneumatic adhesion tensile testing instrument (PATTI) test. Comparison tables and bar charts for each aggregate type have been produced from the test results obtained. As this study focussed on the effect of HL on moisture damage resistance, this comparison is mainly between the mastics having mineral filler only and the mastic having HL replacing the mineral filler in certain proportions. The details of all the mastic and their combinations have been listed in Chapter 3. Also, as the surface energy results give an indication of fundamental characteristics of the material, so another comparison has been made been between the results of work of de-bonding and the results of other test techniques to find out if all the results are reaching to the same conclusion.

For the RBT test, percentage retained coating has been summarised after 72 hours of visual inspection as the assessment after 72 hours was found most discriminating among different combinations within the same aggregate type. For the surface energy investigation, only the absolute value of work of de-bonding has been compared as the main focus of the study is an evaluation of moisture damage resistance. Average retained stiffness (Ref: Chapter 5) has been used for SATS test to compare combinations with and without HL. Similarly, for the PATTI test retained strength has been used which is the ratio of average peak tensile strength of samples conditioned for 7 days in water to the average peak tensile strength of samples conditioned for 0 days in water. Also for each test type, hydrated lime content has been suggested in accordance with the performance obtained.

7.2 Granite aggregates and HL performance

To evaluate the performance of HL against moisture damage in the presence of granite aggregates a comparison has been made between the studied techniques in the form of Table 7:1and Figure 7:1. Both tabular and graphical representations are presented for quick and easy understanding. For the RBT test, the mastics with HL are performing significantly better than the one without and showing an increase of 57% in the retained coating with the addition of

10% HL. The effect of 20% HL was observed to be exactly the same as 10% HL so in this case the HL content can be suggested as 10% in the bitumen to form a mastic. When this mastic is used in an asphalt mixture having 5% binder content, the HL content in the whole mixture can be referred to as 1% of the mixture. For this reason, the last column (i.e. Suggested HL content) in all four tables in this chapter show 1% or 2% HL content which is referred to as the percentage of the whole mixture having 5% binder content.

Table 7:1: Comparison of different test techniques to assess HL performance on granite aggregates

Test method	Mastic with 50% Mineral filler or Mixture with 5% mineral filler	Mastic with 40% Mineral filler+10% HL Or Mixture with 4% mineral filler+1%HL	Mastic with 30% Mineral filler+20% HL Or Mixture with 3% mineral filler+2%HL	Suggested HL content
RBT	35	55	55	1%
work of de-bonding	132	120	64	2%
SATS	0.84	0.97	0.93	1%
PATTI	0.68	0.72	0.85	2%

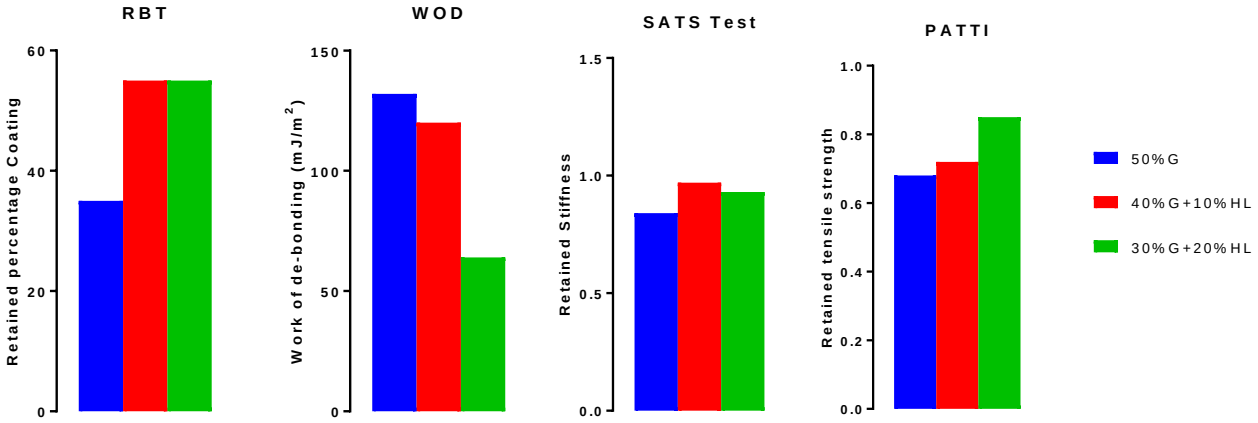


Figure 7:1: Bar chart comparison of the studied test techniques to assess HL performance on granite aggregates

The results obtained from the surface energy testing are listed here in the form of work of de-bonding. With the replacement of 10% HL to the mineral filler, a decrease of 9% in the work of de-bonding was observed but with the replacement of 20% HL, a massive decrease of 51% was calculated, which shows a strong resistance to moisture induced damage. An increase of 15% was observed with the replacement of 1% HL to the mineral filler after SATS test but 2% HL didn't increase the retained stiffness any further and a slight drop in the retained stiffness was observed. Based on the results obtained after SATS test, 1% HL can be suggested as better performing in the mixture for this particular type of granite aggregates. Similarly, for the PATTI test 10% replacement of mineral filler with HL didn't produce distinctive results as only 6% increment in the retained tensile strength was observed but 20% replacement showed a significant increment of 25% in the retained tensile strength compared to the mastic without HL. Two techniques i.e. RBT test and SATS test, are showing 1% HL (10% in mastic or 1% in mixture) as better performing and the other two i.e. SFE technique and PATTI test are suggesting 2% HL (20% in mastic and 2% in mixture) as better performing. So, it is difficult to say which one is true but I will suggest 1% HL content as better based on the mixture level testing using SATS as this technique closely simulates the field conditions.

7.3 Limestone aggregates and HL performance

Evaluation of HL performance with the limestone aggregates is performed in a similar manner to granite aggregates.

Table 7:2 and Figure 7:2 are produced for the comparison among the different techniques employed in this study.

For the limestone aggregates the effect of HL replacement is very different from the granite as hardly any difference was observed among different techniques with the replacement of 1%HL (10% in mastic or 1% in mixture) or 2% HL. RBT, SFE and PATTI tests all show very similar results for the mastic having 10% or 20% HL replacement of limestone filler and with some of the combinations even showing slightly decreased performance with the addition of HL. SATS is the only test which is showing an increase of about 23% in the retained stiffness with the replacement of 1% HL but again with the 2% HL, the performance went down slightly. It is difficult to see any beneficial effect of HL in the case of limestone aggregates as three out of

four techniques are not showing any improvement with the addition of either 1% or 2% HL. So based on the facts discussed above, it can be concluded that HL does not really seem to improve the performance of the limestone aggregates used in this study.

Table 7:2: Comparison of different test techniques to assess HL performance on Limestone aggregates

Test method	Mastic with 50% Mineral filler or Mixture with 5% mineral filler	Mastic with 40% Mineral filler+10% HL Or Mixture with 4% mineral filler+1%HL	Mastic with 30% Mineral filler+20% HL Or Mixture with 3% mineral filler+2%HL	Suggested HL content
RBT	60	60	55	-
work of de-bonding	62	57	64	-
SATS	0.6	0.74	0.68	1%
PATTI	0.78	0.81	0.73	-

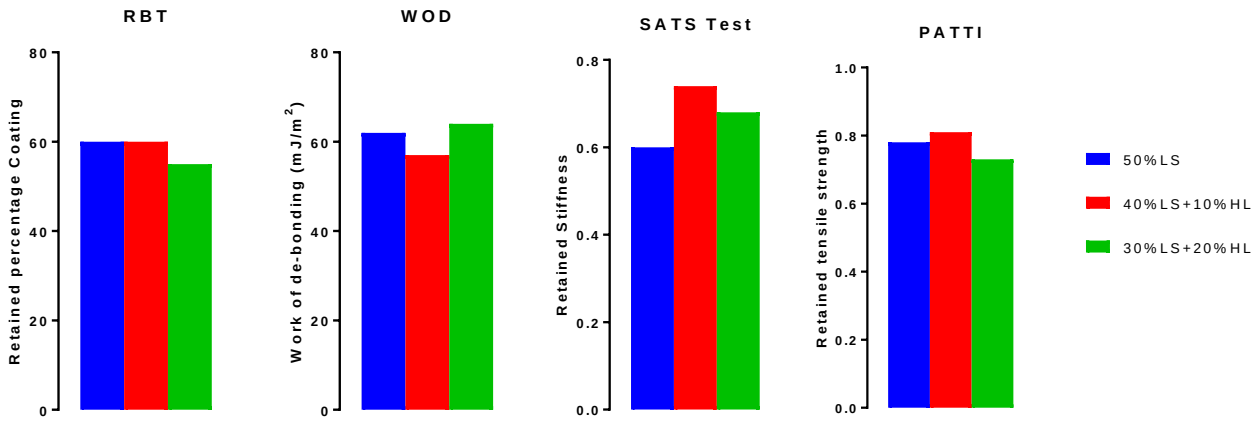


Figure 7:2: Bar chart comparison of the studied test techniques to assess HL performance on limestone aggregates

7.4 Basalt aggregates and HL performance

Assessment of HL performance with the basalt aggregates has been made using three test techniques as discussed in Chapter 3. It was not possible to obtain big boulders and hence aggregate substrates for the manufacturing of samples for PATTI test so the test was not performed. Table 7:3 and Figure 7:3 shows a comparison between the combination with and without HL for the three studied test techniques. The RBT test results show a good increase of 50% with the replacement of 10% HL to the mastic but 20% replacement showed nearly identical results as 10% replacement. The work of de-bonding results derived from SFE technique didn't show any improvement towards HL addition as the results of all the combinations either with or without HL were observed to be in a very narrow band.

Similarly, for the SATS results, there was hardly any difference noted in the performance of the mixtures having 1% or 2% HL compared to the one having 0% HL. If we look carefully the retained stiffness for the combination having 0% HL is already quite high that may be one of the reasons why HL is not showing any further improvement when replacing some of the basalt filler. So the conclusion with the basalt aggregates is somewhat similar to the one drawn with the limestone aggregates and it can be concluded that the beneficial effects of HL addition are difficult to quantify with the studied techniques when basalt aggregate was used.

Table 7:3: Comparison of different test techniques to assess HL performance on basalt aggregates

Test method	Mastic with 50% Mineral filler or Mixture with 5% mineral filler	Mastic with 40% Mineral filler+10% HL Or Mixture with 4% mineral filler+1%HL	Mastic with 30% Mineral filler+20% HL Or Mixture with 3% mineral filler+2%HL	Suggested HL content
RBT	40	60	65	1% or 2%
work of de-bonding	155	159	150	-
SATS	0.94	0.91	0.95	-

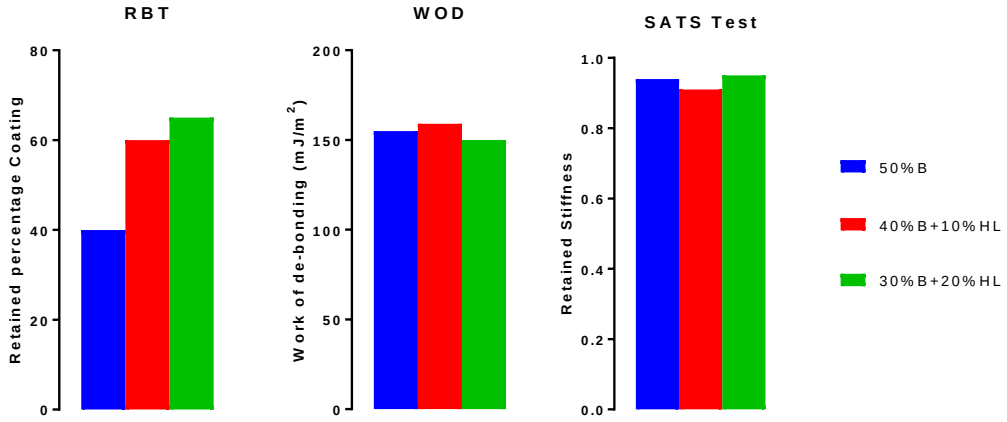


Figure 7:3: Bar chart comparison of the studied test techniques to assess HL performance on Basalt aggregates

7.5 Greywacke aggregates and HL performance

For the assessment of greywacke aggregates together with HL again four test methods were used and for the comparison between all the test method Table 7:4 and

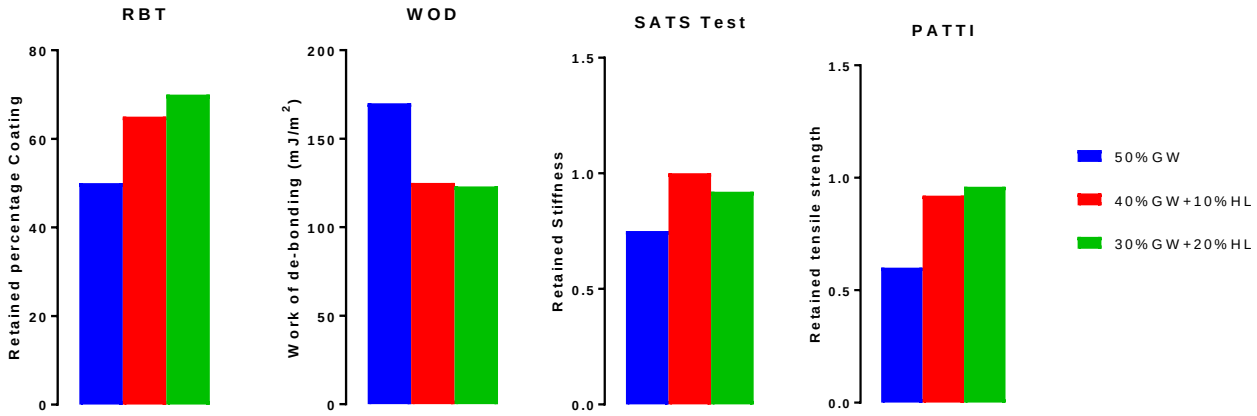


Figure 7:**4** have been compiled. Again if we start with the RBT test results, after 72 hours of agitation in rolling bottles, mastic having 10% HL is giving a good increment of 30% in percentage retained mastic coating compared to the mastic without HL. Performance of the combination having 20% HL replacement was observed to be very similar to the 10% HL replacement. So the HL content was suggested as 10% (10% in mastic or 1% in mixture) using RBT technique. Now for the surface energy results in the form of work of de-bonding a decrease of 26% was observed with the use of 10% HL in the mastic. Also, there was practically

no difference found between 10% HL and 20% HL combinations and hence HL content can be suggested as 10% (10% in mastic or 1% in mixture) using this technique.

For the mixture level testing using SATS test, the combination having 1% HL replacement of the greywacke filler showed a significant increase of 33% but the performance with the 2% HL replacement decreased slightly. Again 1% HL can be referred as suggested HL content for SATS technique when greywacke aggregates are used. Results after PATTI test with greywacke aggregates also shows a beneficial effect of HL replacement. The mastics having 10% HL are showing a huge increment of 53% in the retained tensile strength which improves further with the use of 20% HL but again there is not a significant difference observed between 10% HL and 20% HL. So the suggested HL content, in this case, can again be considered as 10% (10% in mastic or 1% in mixture). So 1% HL can be suggested as a collective HL content for the greywacke aggregates in the light of all the four test techniques used in this study.

Table 7:4: Comparison of different test techniques to assess HL performance on greywacke aggregates

Test method	Mastic with 50% Mineral filler or Mixture with 5% mineral filler	Mastic with 40% Mineral filler+10% HL Or Mixture with 4% mineral filler+1%HL	Mastic with 30% Mineral filler+20% HL Or Mixture with 3% mineral filler+2%HL	Suggested HL content
RBT	50	65	70	1% or 2%
work of de-bonding	170	125	123	1%
SATS	0.75	1.0	0.92	1%
PATTI	0.60	0.92	0.96	1% or 2%

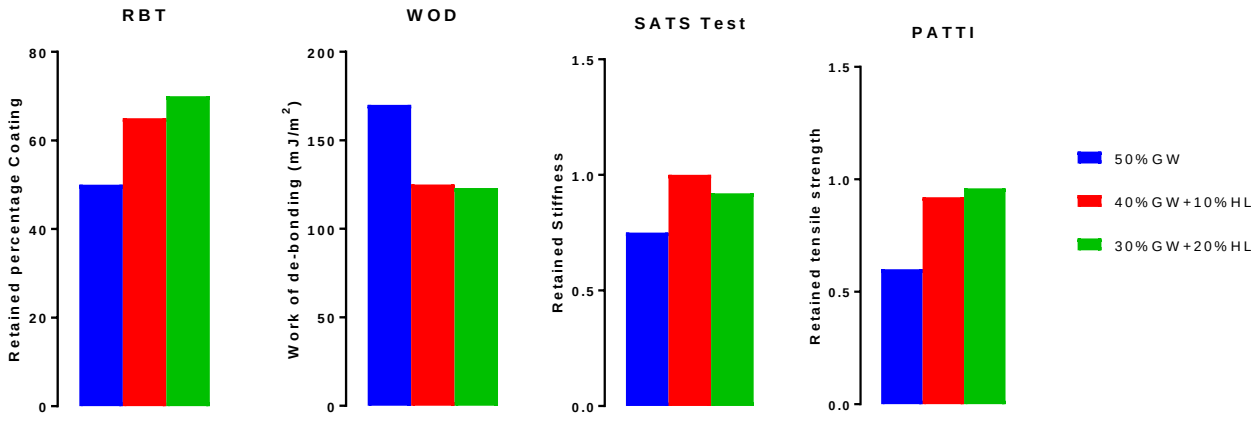


Figure 7:4: Bar chart comparison of the studied test techniques to assess HL performance on greywacke aggregates

Based on the comparison made between the studied test techniques for each aggregate type it can now be concluded that limestone and basalt aggregates used in this study didn't respond well to the replacement of mineral filler with HL but the other two types i.e. granite and greywacke showed a good response and showed a significant improvement in the moisture damage performance with the use of HL. Another good thing observed in the results was the consistency between the results among the different test techniques. The results obtained in each technique are in line with each other and give the same conclusion for most of the combinations studied in this research.

7.6 Comparison between surface energy results and other test techniques

A comparison has been made in Figure 7:5 between the results of surface energy in the form of work of de-bonding and results of rolling bottles test in the form of percentage retained coating for the aggregates and mastics combination. From the Figure 7:5, it can be extracted that the results of both techniques are in line with each other for most of the combinations. A smaller absolute value for work of de-bonding and a higher value of percentage retained coating shows a stronger material against moisture damage so based on this fact if we look at the granite aggregate it clearly shows improvement in the percentage retained coating and a reduction in work of de-bonding for the mastics having 1% or 2% HL.

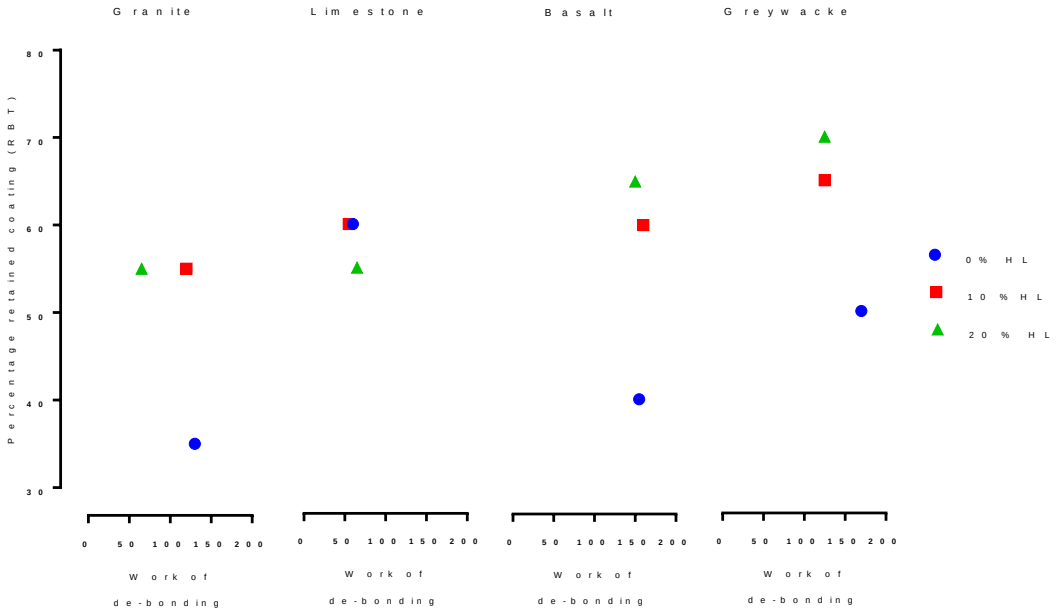


Figure 7:5: Comparison between work of de-bonding and RBT results for four aggregates

Limestone aggregate didn't show any improvement signs with the use of 1% or 2% HL based on the results obtained from both of the test techniques. Basalt aggregates showed improvement in the percentage retained coating, but no reduction was noted in the work of de-bonding for the mastics having HL. Greywacke, on the other hand, showed good improvement in the percentage retained coating and a good reduction in the work of de-bonding for the combinations having HL. In a nutshell, it can be stated that an inverse relationship has been found between work of adhesion and percentage retained coating for granite and greywacke aggregates. Limestone didn't show any improvement with the use of HL, but basalt only showed improvement in the results of RBT test.

Another comparison has been made between work of adhesion and retained stiffness calculated in the SATS in the Figure 7:6 for all the aggregate combinations. Again, an inverse relationship has been found for granite and greywacke aggregate combinations. Both granite and greywacke show improvement with the use of HL in the mastic. Limestone didn't show any decrease in the work of de-bonding with the use of HL but the shown improvement in the retained stiffness. Basalt, on the other hand, does not show any change in either work of de-bonding or retained stiffness with the use of HL.

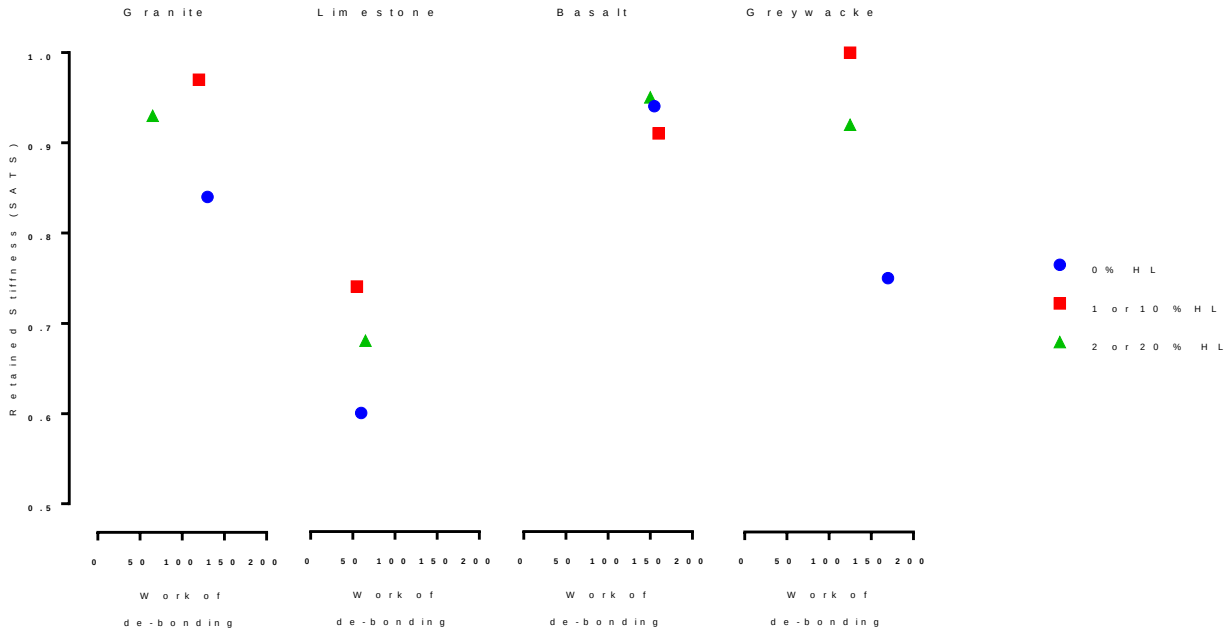


Figure 7:6: Comparison between work of de-bonding and SATS results for four aggregates

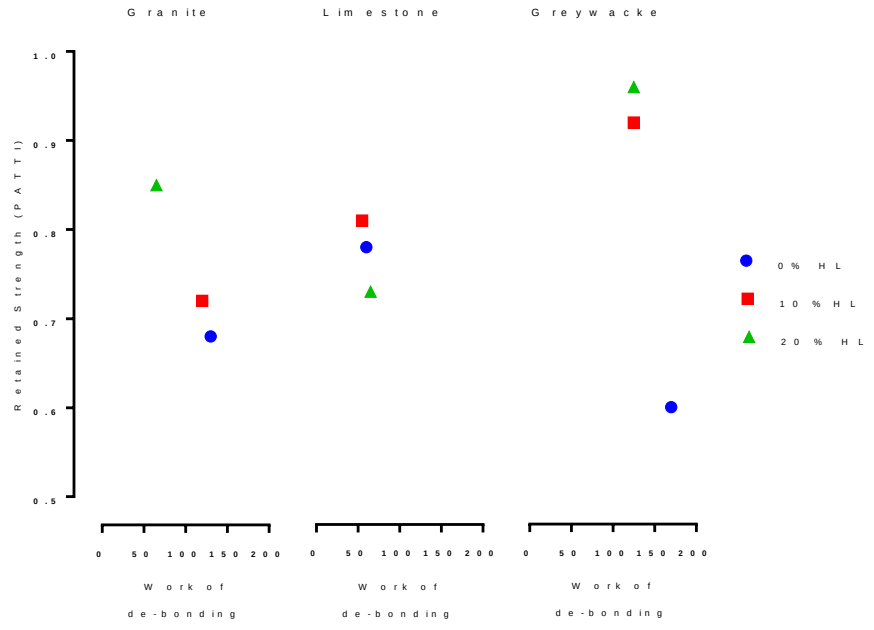


Figure 7:7: Comparison between work of de-bonding and PATTI results for four aggregates

Figure 7:7 shows a comparison between the results of SFE in the form of work of de-bonding and results of PATTI test in the form of retained strength for all the three aggregates and their respective mastic combinations. As discussed earlier that PATTI test was not performed on the combinations of Basalt aggregate that's why only three aggregate types have been presented in the comparison. If we look at the Figure 7:7 carefully, again there exists an inverse relationship between the results of PATTI test and the results of work of de-bonding, for the three aggregate combination. Granite and Greywacke again show a clear decrease in the work of de-bonding with the use of HL and increase in the retained strength computed using PATTI test. Limestone aggregate doesn't show any improvement signs with the use of HL as all the results are in a very narrow band.

Based on the comparison made above it is concluded that the results of the most of techniques are in line with the results of SFE in the form of work of adhesion. Granite and Greywacke shows good improvement with the used of HL based on the results most test techniques.

Chapter 8: Conclusions and Recommendations

8.1 Introduction

The main purpose of the study was to evaluate the beneficial effect of hydrated lime against moisture damage performance of aggregates which are commonly used in the UK. Four aggregates types were studied with the help of some widely used test techniques to evaluate the moisture damage performance. Based on the results obtained and the challenges faced during testing, conclusions and some recommendations for the future work are summarised in this chapter.

8.2 Conclusions

Rolling bottle test was successfully performed on all the combinations used in the study and was quite useful in discriminating different combinations. As discussed earlier in the literature, one of the drawbacks of using the RBT in terms of reliability and accuracy was the visual inspection. To overcome this issue an image analysis technique using Matlab software was used for the calculation of percentage retained coating after 6, 24, 48 and 72 hours of agitation. The results after image analysis were compared to the results of eye observation and both were found very similar to each other. This statement was supported with R square value calculation and frequency distribution. Hence RBT can now be used confidently for the comparison of different combinations.

The beneficial effects of HL addition were clearly quantified with granite, basalt and greywacke aggregates but limestone aggregates didn't respond to the effect of HL addition when tested in the rolling bottles. Based on the results of RBT test, 10% HL (10% in mastic or 1% in mixture) substitution was found as better performing as 20% HL substitution gave very similar results to 10% HL substitution in the most of the studied combinations.

Results from the surface energy testing in the form of work of adhesion, work of de-bonding, and various energy ratios were found to be very interesting. For the granite aggregate, there was a clear increment in the performance observed with the addition of HL to the mastic. Even with the 10% HL replacement, there were some positive trends but the use of 20% HL showed a significant improvement in the results. Limestone and Basalt aggregates, on the other hand, didn't show improvement signs with the results obtained from SFE testing. Greywacke

aggregates followed the same trend of granite aggregates and showed a good improvement when 10% greywacke filler was replaced with HL. With the use of 20% HL, there was practically no difference observed in the performance.

Calculation of energy ratios (ER_1, ER_2, ER_3, ER_4) demonstrates that most of the combinations are passing the criteria set by Little and Bhasin (2006) and Bhasin (2007) yet a significant increase in the values with the addition of HL was observed especially for granite and greywacke aggregates.

Mixture level testing was undertaken using SATS test for all the four aggregates. Three out of four aggregates including granite, limestone and greywacke showed improvement in the retained stiffness but the performance of basalt aggregate remained unaffected with the use of HL. The performance of basalt aggregates was already very good even without HL, this might be one of the reasons why basalt didn't show further improvement with the use of HL. The HL content of 1% was observed to have better performance in the three aggregates which showed improved performance with the use of HL.

Moisture damage performance of the mixture without HL addition was also assessed. Although the performance of all the four aggregate types was found satisfactory but the basalt aggregates was found best compared to other three types.

A comparison between ITSM and ITS test after SATS conditioning was also undertaken as some researchers believe that ITS being destructive in nature gives more distinctive results. But the results of ITSM and ITS very found to be very similar to each other after SATS conditioning in terms of retained strength (using ITS) or retained stiffness (using ITSM).

The results for the limestone aggregates in SATS do not reflect those of the mastic tests. So, it can be concluded that if the mastic tests are designed to predict the performance of mixtures for poorly performing combinations of binder and aggregate, they might not always be suitable for the mixtures that performed well.

To work out the practical work of adhesion PATTI test and Peel tests were used. To reduce the poor repeatability of PATTI test, samples were successfully tested under water (Chapter 6) and results were found to be a lot better in terms of repeatability. Samples were tested after 0 days conditioning and 7 days' condition in water and retained tensile strength was quantified. An improvement in the results for granite aggregate was observed with the addition of HL. Though there was some improvement found with the use of 10% HL in the mastic, the 20% HL in

mastic showed a marked improvement. Limestone aggregates again didn't respond to the effect of HL substitution but the greywacke aggregates responded strongly in the form improved retained tensile strength with the use of 10% HL. There was no difference observed with 20% HL substitution for greywacke aggregates.

Various trials were made to get the results using Peel test when mastics were tested. Sadly, no attempt was successful. First attempt was made using standard Aluminium peel arm and the failure occurred between peel arm and bitumen mastic and the results could therefore not be used. The second attempt was made by using sandblasted peel aluminium to make the surface rough hence improving the bond between mastic and peel arm but again the failure occurred between peel arm and mastic. Wire mesh was also tried as peel arm but it was not possible to make 90^{o} Peel test due to the hardness of the mesh and also sample preparation was very difficult. Lastly, Hessian was used which gave better results in terms of consistency under dry conditions but when the samples were tested after water conditioning the results of all the samples appeared to be similar. One of the possible reasons may be the water absorption of the hessian cloth as the cloth will absorb water when the sample is conditioned before testing and hence the bond between cloth and binder will become weak which will result in a failure between the binder and the cloth which will not be representative of the true moisture damage situation. So under dry conditions, it may be helpful to compare aggregates having different mineralogy but not under wet conditions.

To conclude, the effect of HL either in the mastic or in the mixture has been found to be aggregate type dependent. Granite aggregates showed a good improvement in the performance against moisture damage resistance and all the test methods used in the study supported this statement. Limestone aggregate didn't respond to the addition of HL in the light of most of the techniques considered in this research and the same applied to the basalt aggregates. Greywacke, on the other hand, responded well and showed an improved moisture damage performance with the use of HL and results from each studied test technique were consistent with each other and gave the same conclusion.

8.3 Future Recommendations

On the basis of the work carried out for this research and the conclusions made in the previous section, the following are recommended:

- The aggregates used in this study were either of moderate or good field performance. Although a good improvement in the moisture damage performance of some combinations was clearly observed but it is highly recommended to incorporate aggregates having bad field performance to see how HL improves the performance.

- Four types of aggregates were used in this study along with one type of base bitumen. In order to get a better understanding of how HL behaves with different types of material more aggregate and bitumen types should be studied in order to reach a comprehensive conclusion regarding the effect of HL on commonly used road materials in the UK.

- Only 1% and 2% HL was used for mixture level testing and for all other tests the effect of HL was quantified by adding 10% or 20% HL as a mineral filler replacement in the bitumen to form a mastic. More concentrations of HL can be tried to find out if there is any change in the moisture damage performance with the use of more or less concentration. Also, the increment interval between each concentration can be reduced to get optimum HL content.

- Only one type of HL was used in this study. A comparative study can be made to see if there is any change in the performance with the use of different HL types. For example, Nano-HL is now in the market. It will be a good idea to compare neon HL with the standard HL to check how it affects performance-related properties and if there is any change in the suggested HL content for the two types when tested with the same material.

- So far most of the studies talk about the beneficial effect of HL replacement in terms of the percentage increase or decrease of study parameters and there are very limited studies talking about cost benefits. There is a need for a mathematical model to quantify the cost benefit with the change in each test parameter. If the cost is modelled with the

output of each test result it will be very easy to understand if a specific combination is beneficial or not.

- In the rolling bottle test, image analysis has been used to quantify the percentage retained coating for each observation. It is very important to take high-quality glare free pictures as the results are highly dependent on the picture quality. More advanced image analysis techniques can also be considered for more precise measurements.

- PATTI test was conducted under water to improve the consistency in the results between each trial for the same combination. There are some other things which are very important as well to get consistent results. For example, it is very important to trim the sample correctly and carefully as any extra bitumen or mastic adhering to stub and aggregate substrate can produce big differences in the results. Also, maintenance of the test temperature during the test is equally important as this test is highly sensitive to changes in temperature.

- Peel test for the mastic and stiff bitumen is still challenging. The main issue is the peel arm material. Aluminium, sand blasted aluminium, wire mesh and hessian cloth have been tried but still, the results obtained are not desirable. So there is a need for alternate peel arm material which is good enough to test mastic or stiff binder by fulfilling all the test requirements.

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APPENDIX A

Matlab Code

```
clc % clear screen
im=imread('24.bmp'); % reading the data of the image to be analysied
imtool(im);
gs=double(0.2989*im(:,:,1)+.5870*im(:,:,2)+.1140*im(:,:,3)); % converting the image to grayscale with double system
% imtool(gs);
b=uint8(gs);
% imtool(b);
[m, n]=size(gs); % getting the sizes of the image
for i=1:3:m-2; % apply mean value filter for 2*2 area size
   for j=1:3:n-2;

a=double((gs(i,j)+gs(i+1,j)+gs(i+2,j)+gs(i,j+1)+gs(i+1,j+1)+gs(i+2,j+1)+gs(i,j+2)+gs(i+1,j+2)+gs(i+2,j+2)))/9;
      aa=round(a);
      gs(i,j)=aa;
      gs(i+1,j)=aa;
      gs(i+2,j)=aa;
      gs(i,j+1)=aa;
      gs(i+1,j+1)=aa;
      gs(i+2,j+1)=aa;
      gs(i,j+2)=aa;
      gs(i+1,j+2)=aa;
      gs(i+2,j+2)=aa;
   end
end
gs=uint8(gs); % converting gs from double system to uint8 system
imtool(gs);
c=(gs); % copying the image data to another matrix that is used to show the analysis result
```

```
bul=25; % setting threshold value for the upper limit of the colour of interest
bll=0; % setting threshold value for the lower limit of the colour of interest
b=0; % setting initial value for a counter to be used in the analysis
ag=0;
agul=80;
for i=1:m; % start a loop for analysing data pixel by pixel (rows)
  for j=1:n; % start a loop for analysing data pixel by pixel (columns)
     if gs(i,j)>bul && gs(i,j)<agul;
        ag=ag+1;
     end
     if gs(i,j)<bul;
        b=b+1;
    end
    % in order to show the analysis result, the resulted image is
    % converted to black and white only, then a visual inspection can be
    % conducted to check the matching between the original image and the
    % image after analysis
     if gs(i,j)<bul
        c(i,j)=0;
     end
       if gs(i,j)>bul && gs(i,j)<agul
          c(i,j)=125;
       end
       if gs(i,j)>agul
          c(i,j)=255;
       end
  end
end
% imtool(gs)
imtool(c)
CA240=round((b/(b+ag))*100)
% imhist(gs)
% edges=[-10 -1:0.25:1 255];
% hh= histogram(gs,'Binlimits',[200,250]);
```

APPENDIX B

Calculation of Average Load in Peel test

The average load has been calculated by first taking an average of each repeat over at least 50 mm peel length and then taking the further average of all the three repeats to get the final average load value.

Example calculation

Figure 1C show peel test results for Greywacke aggregate and the mastic having 0% HL (i.e. 50%GW) for three repeats. For the calculation of average load value for this combination, first, we need average value from each repeat over at least 50 mm peel length. For the Sample 1, the average load has been calculated between the extension of 30 mm to 80 mm by taking a mean of all the recorded load values between this extension, which is 28 N in this case. In the same way, average load for the for the 2nd sample is 31 (N) and for the third, it is 30 (N). So now the Average of the three samples will be 29.66 or can be rounded to 30 (N).

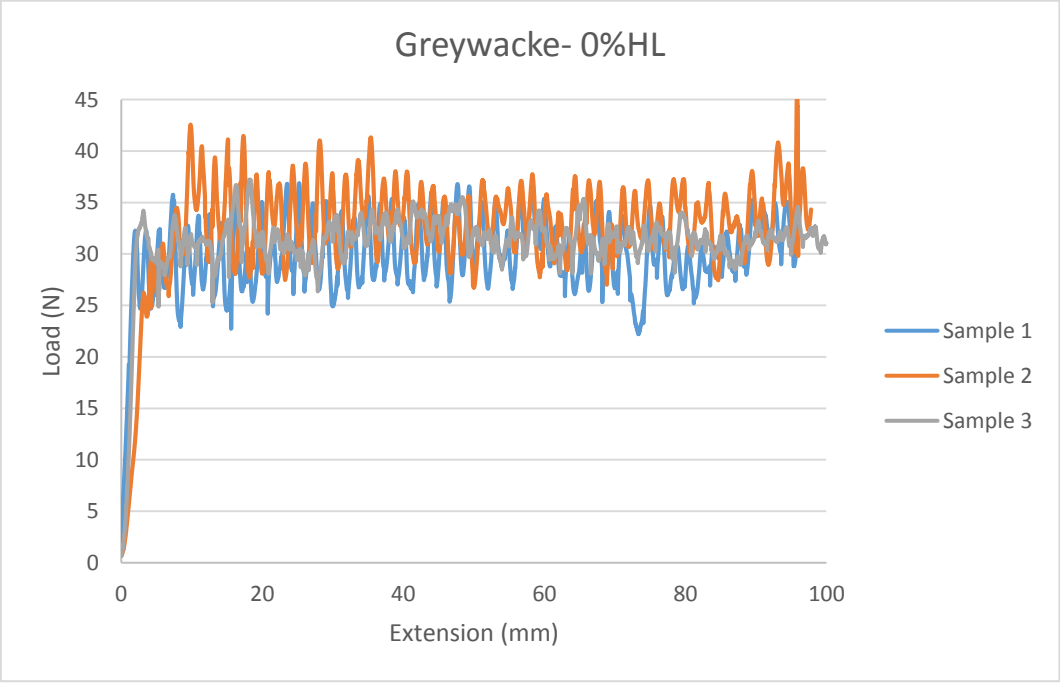


Figure 1B: Peel test results under dry conditions using hessian cloth peel arm for greywacke aggregates and the mastic having 0% HL