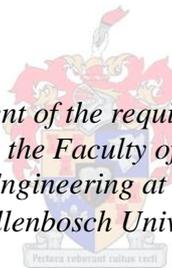


Evaluation of Adhesion Properties in Bitumen-Aggregate Systems for Winter Surfacing Seals Using the Bitumen Bond Strength Test

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ABSTRACT

Flexible pavement designers have a choice of two wearing course: either asphalt concrete or surfacing seals. The latter have been widely used by several countries as their preferred wearing course over other methods, especially countries with a limited number of average inhabitants per square kilometre. Moreover, the surfacing seals were identified as an efficient cost effective road preventive maintenance technique. Surfacing seals in New Zealand, South Africa and Australia cover about 65%, 80% and 90% of their surfaced road networks respectively. The preference of surfacing seals is due to their competitive initial cost and ease of construction.

In South Africa, the life expectancy of surfacing seals varies between 8 and 12 years with an average of 10 years. This has not been the case in a number of surfacing seals constructed in winter, especially when the night recorded temperature is below 10°C. The dominant failure mechanism is ravelling (chip loss) soon after construction due to traffic loading. This chip loss is linked to the poor adhesion bond development rate in the bitumen-aggregate system during winter adverse conditions. In order to address the issue of premature chip loss the need for the development of a robust adhesion test method was identified. For that purpose, recently, researchers in the bitumen industry developed the Bitumen Bond Strength test method. This method was used in this study.

This study intends to contribute to the understanding of binder-aggregate adhesion bond development for winter surfacing seals using the BBS test. Binder type, precoat type and conditioning, aggregate type and curing time are amongst the factors influencing winter seals adhesion bond performance. An experimental matrix involving three types of binder, two types of aggregate, four different precoating fluids, two precoat conditionings and two binder-curing times were then developed and investigated. Winter weather parameters affecting adhesion properties were also taken into consideration during the course of the investigation. Throughout the test, the procedure described in AASHTO TP 91-11 was followed. However, in order to enhance the control of the binder application temperature, a new method for hot applied binder sample preparation was developed as part of this study.

The findings show that there is a significant difference between adhesion properties of the hot applied binders (70/100 and S-E1) and the emulsion (SC-E1). In most of the cases, the hot applied binders performed better than the emulsion. The failure mode observed was found to be linked to the condition of the precoating. The influence of the precoat type and conditioning, and effect of binder curing time were significantly highlighted. The use of a dry precoat benefited the adhesion bond strength up to around 50% relatively to the corresponding non-precoated combination. However, a decrement in the bond strength due to precoating of up to 28.7% was also observed.

A statistical analysis using ANOVA did not illustrate any statistical significant effect of the aggregate type. The interaction effects analysis using ANOVA revealed the aggregate type interacting with

precoat type to be the most influential interaction at level two. The precoat conditioning implication to the adhesion development rate, which influences the time for opening to traffic after construction, was illustrated. Insightful aspects on the compatibility between the binder type and precoat type and conditioning during the aggregate precoating practices and on the time for opening to traffic are highlighted. Finally, the repeatability analysis proved the BBS test to be a repeatable testing method with caution. Recommendations for further studies that could support the conclusions drawn in this study were provided.

OPSOMMING

Buigbare plaveiselontwerpers het 'n keuse van twee deklae: óf Asfalt of oppervlak seëls. Laasgenoemde word algemeen gebruik deur verskeie lande as hul voorkeur deklaag, veral die lande met beperkte aantal gemiddelde inwoners per vierkante kilometer. Verder, is die seëls geïdentifiseer as 'n doeltreffende koste-effektiewe deklaag tegniek. Oppervlakseëls in Nieu-Seeland, Suid-Afrika en Australië dek ongeveer 65%, 80% en 90% van hul padnetwerke onderskeidelik. Die seëls se voorkeur is te danke aan hul mededingende aanvanklike koste en eenvoudige vorm van die konstruksie.

In Suid-Afrika wissel die seël se lewensverwagting tussen 8 en 12 jaar met 'n gemiddeld van 10 jaar. Dit is egter nie die geval van 'n aantal seëls wat in die winter gebou word nie, veral wanneer die aangetekende nagtemperatuur onder 10° C daal nie. Die dominante swigtingsmeganisme is stroping (klipverlies) kort na konstruksie. Hierdie klipverlies is gekoppel aan die power kleef-ontwikkeling van bitumen gedurende die winter. Ten einde die probleem van voortydige klipverlies aan te spreek het die behoefte vir die ontwikkeling van 'n robuuste toetsmetode ontstaan. Om hierdie rede het navorsers onlangs in die bitumenbedryf die “BBS toetsmetode” ontwikkel en is dié toetsmetode in hierdie studie gebruik.

Hierdie studie beoog om by te dra tot die begrip van bindmiddel-klip kleefontwikkeling vir die winter seëls dmv die BBS toets. Die faktore, insluitend maar nie beperk tot bindmiddeltipe, voorafdekking (“PRECOAT”) -tipe en kondisionering, aggregaattipe en kuurtyd beïnvloed winter seëls se kleefeienskappe. 'n Eksperimentele matriks met drie tipes bindmiddels, twee tipes aggregate, vier verskillende voorafdekking-vloeistowwe, twee voorafdekking kondisionering en twee bindmiddel kuurtye is toe ontwikkel en ondersoek. Winter weer parameters wat kleefeienskappe beïnvloed is ook in ag geneem tydens die verloop van die ondersoek. Regdeur die studie is die prosedure AASHTO TP 91-11 gevolg, maar ten einde die beheer van die bindmiddel spuittemperatuur te verbeter, is 'n nuwe metode vir warmspuit-bindmonsters voorbereiding ontwikkel as deel van hierdie studie.

Die bevindinge toon dat daar 'n beduidende verskil tussen die kleefeienskappe van die warm aangewende bindmiddels (70/100 en S-E1) en die emulsie (SC-E1) is. In die meeste van die gevalle het die warmspuit-bindmiddels beter as emulsie gevaar. Daar is gevind dat die swigtingsmeganisme verbind word met die toestand van die voorafdekking. Die invloed van voorafdekkingtipe, kondisionering, en die effek van bindmiddelkuurtyd is duidelik uitgelig. Die gebruik van droë voorafdekking het die kleefkrag tot sowat 50% verhoog relatief tot die ooreenstemmende onbedekte klipkombinasie. Daar is egter ook 'n verlaging van die kleefkrag weens voorafdekking gevind van tot so hoog soos 28,7 persent.

Die statistiese ontleding met behulp van ANOVA het geen statisties beduidende effek van die verskillende aggregaattipe te vore gebring nie. Die interaksie-effek analise, met behulp van ANOVA,

het wel die interaksie met voorafdekkingtipe met aggremaat die mees invloedryke bevestig. Die voorafdekking kondisionering het ver rykende kleefkrag implikasies bloot gelê, wat die tyd vir die opening van die verkeer na konstruksie beïnvloed. Insigwekkende aspekte oor die versoenbaarheid tussen die bindmiddeltipe, voorafdekkingtipe, kondisionering, voorafdekkingpraktyk en tyd tot opening vir verkeer word uitgelig. Ten slotte, die herhaalbaarheidsanalise het die BBS toets as 'n herhaalbare toetsmetode met omsigtigheid bewys. Daar is aanbevelings tot verdere studies, wat uit die gevolgtrekking gekom het, gemaak.

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LIST OF ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

AAPT:	Association of Asphalt Paving Technologists
AASHTO:	American Association of State Highway and Transportation Officials
ANOVA:	Analysis of variance
ASTM:	American Society for Testing and Materials
AwC:	Anionic emulsion with cutters
BBS:	Bitumen Bond Strength
CoV:	Coefficient of variation
CSIR:	Council for Scientific and Industrial Research
CwC:	Cationic emulsion with cutters
DSR:	Dynamic Shear Rheometer
GR&R:	Gage repeatability and reproducibility
hrs:	Hours
kPa:	kilopascal
L.R:	Loading Rate
LCL:	Lower Control Limit
mm:	Millimetre
mm ² :	Square millimetre
MRL:	Material Reference Library
PATTI:	Pneumatic Adhesion Tensile Testing Instrument
PMB:	Polymer Modified Bitumen
PQG:	PATTI Quantum Gold
psi:	Pounds per square inch
SA:	South Africa

SABITA:	Southern African Bitumen Association
SANRAL:	South African National Roads Agency Limited
SBR:	Styrene Butadiene Rubber
SBS:	Styrene Butadiene Styrene
Sec:	Second
SHRP:	Strategic Highway Research Program
SPSS:	Statistical Package for the Social Science
SU:	Stellenbosch University
UCL:	Upper Control Limit
UDSH:	Upside Down Stub Holder
USA:	United States of America
UTM:	Universal Testing Machine
SYMBOLS	
°C:	Degree Celsius
S-E1:	70/100 Penetration grade modified with SBS
SC-E1:	Cationic 65% spray grade emulsion containing 3% SBR latex
SS60:	Stable grade anionic emulsion with 60% residual bitumen
%:	Percent

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Bituminous Surfacing Seals have been widely used by several countries as a pavement surface treatment preferred over other methods. This is due to their competitive initial cost and speed of construction. In Australia, seals have been the mainstay of the road authorities to provide a safe, all-weather, dust-free, rural road network (Holtrop, 2008; Greyling, 2012).

Initially, bituminous surfacing seals were used predominantly as surfacing course (wearing course) in the construction of low volume roads. In the early stages, there was no specific rational design method. F.M. Hanson developed the first scientific approach for the design and construction of surfacing seals in New Zealand around 1935. He quantified the surfacing seal concept, documented the theory of why and how seals worked (Gransberg & James, 2005; Towler & Dawson, 2008; Zoghi, Ebrahimpour & Pothukutchi, 2010). Later, surfacing seals have also been identified as an efficient flexible pavement preventive maintenance technique. Due to that combined application, surfacing seals have increasingly attracted the attention of researchers and Hanson's design concept was improved and adopted by several countries such as South Africa, Australia, the United Kingdom, the United States of America and New Zealand (Zoghi *et al.*, 2010).

In South Africa, about 80% of paved roads have been constructed using bituminous surfacing seals. Due to an aged road network, approximately 10% of the paved road network needs to be resealed each year (SABITA, 2012). However, South Africa as well as other countries that have extreme climatic conditions has experienced several failures related to the construction of bituminous surfacing seals during colder and rainy periods of the year. These failures were observed especially when the minimum night temperature falls below 10° C (this is during the months of May to September in South Africa). This has prompted several road authorities in South Africa to impose sealing embargoes during the colder and rainy period of the year (SABITA, 2012; Van Zyl, O'Connell & Paige-Green, 2012). Similar embargo periods have also been implemented in Australia (May to September) and in New Zealand (1 May to 31st August).

The implementation of embargo periods intends to prevent premature failure of seals. Numerous studies such as the performance grading of bitumen emulsions for sprayed seals by Bahia *et al.* (2008) have identified fattiness (bleeding), fatigue cracking and ravelling (stone loss) as the dominant surfacing seal failure mechanisms. Section 2.5.6 provides a detailed description of these seal failure mechanisms.

As regards winter seals, the predominant failure mechanism is considered to be early stone loss (Van Zyl *et al.*, 2012). Early stone loss is linked to weakness of the binder-aggregate adhesion bond. After construction, the seal needs enough time to develop the appropriate bond strength to withstand traffic loading. In winter, due to low temperature, the development rate of bond strength is low as is discussed in section 2.2.1.4. Once the overnight temperature is low (usually less than 10° C), the binder hardens before it develops the appropriate adhesion bond with the stone. Opening the road section to traffic, very early the following morning causes the weak bond between the binder and aggregates to break easily. Thus, understanding the factors favouring quick adhesion bond development could contribute in the reduction of early stone loss in winter seals.

In South Africa, a number of techniques have been developed to reduce the stone loss associated with winter seals, such as aggregate precoating to improve wettability, selection of aggregate with greater affinity to binders, increasing binder application rate, etc. (Van Zyl *et al.*, 2012). The inability of these techniques to limit the chip loss completely resulted in the implementation of winter seal embargoes. Despite these winter associated failures, as has been aforementioned, about 10% of the road network needs to be rehabilitated each year. In addition to that, the suppliers of construction material are not able to satisfy the demand imposed on them in the remaining period of the year. This resulted in a South African initiative to maximize sealing activity throughout the year, which means that there will be a need to extend the sealing activities into the cold weather period. As part of this initiative, the South African National Roads Agency Limited (SANRAL) launched a project to evaluate and improve the winter seal performance under a project known as “SANRAL Winter Seal Project” (SABITA, 2012).

This research is linked to that project and investigates the bitumen-aggregate adhesion properties for various material combinations. It explores the influence of various bitumen emulsion based precoating fluids on the development of adhesion bond strength. This will ensure the material combinations' suitability during adverse weather conditions.

Adhesion is scientifically defined by ASTM D907 as the state by which two dissimilar surfaces are held together by interface forces which can be either valence forces or interlocking forces or both (Hefer, 2004). The concern on adhesion properties can be traced back to very early scientific eras. Packham (2006) stated that Newton identified two questions to be asked as far as adhesion is concerned. The first one concerns the force that has to be applied to break the adhesion bond. The second is about how different components are held together in the first place. Referring to those two questions, two different terminologies of adhesion are provided based on what you are referring to (Packham, 2006).

The “Practical Adhesion” describes the amount of mechanical energy required to break an adhesive bond. This term can be used to describe the required forces to break an adhesive bond under load in service or the results of adhesion destructive tests in the laboratory.

On the other hand, “Fundamental Adhesion” or True adhesion is used once the reference is taken to the intermolecular forces at the interface of two dissimilar surfaces. In this study, attention is given to practical adhesion through bitumen adhesive bond strength testing.

Although various methods exist for the evaluation of bitumen-aggregate practical adhesion bond strength, most of them do not provide sufficient information to understand the phenomenon (Greyling, 2012). In this study, the Bitumen Bond Strength (BBS) test method, recently developed by the University of Wisconsin-Madison (USA) in partnership with the University of Ancona (Italy) and the University of Stellenbosch (South Africa), to address some of these limitations, will be used for testing.

Definitions of key words

- **Bituminous Surfacing Seals:** consist of application of binder onto the road pavement surface. This is immediately covered with a layer of aggregates and then compacted with a roller to ensure close contact between aggregate and the binder and thus a good adhesion bond between them. The rolling activity initiates the compaction process that is completed later by the traffic and thus a relatively impermeable pavement surface is obtained (SANRAL, 2007).
- **Aggregate Precoating:** consists of application of a bitumen-based fluid to the surfacing aggregate. For environmental reasons, tar-based precoats are no longer used. Precoating is carried out prior to the spreading of surfacing aggregate on the sprayed binder. Precoating reduces the risks of aggregate loss in the surfacing seals by coating the residual dust or moisture on the aggregate surface, and provides excellent adhesion between the aggregate and the binder (Louw, 2004).
- **BBS:** (Bitumen Bond Strength) is a testing method recently developed by the University of Wisconsin-Madison (USA) in partnership with the University of Stellenbosch (South Africa) and the University of Ancona. It is an improvement of the apparatus known as “PATTI” (Pneumatic Adhesion Tensile Testing Instrument). PATTI was initially developed to evaluate the pull-off strength of coatings on solid materials and was later improved and adopted by the bitumen industry. The literature shows that PATTI was used by the bitumen industry for the first time in the late 1990s. At that time, it was used to investigate the adhesive loss of binder-aggregate systems exposed to moisture conditioning (Jenkins, Van de Ven, Greyling, Constable, Abrahams & Stander, 2013).

1.2 PROBLEM STATEMENT

Bituminous surfacing seals are used extensively to provide a waterproof cover for underlying pavement layers. Moreover, they provide a skid resistant, safe, all-weather dust-free pavement surface and protect the underlying pavement layers against traffic wearing forces as well as environmental effects (SANRAL, 2007).

In South Africa, the life expectancy of a pavement surfacing seal varies between 8 and 12 years with an average of 10 years (SABITA, 2012). This has not been the case in a number of bituminous surfacing seals constructed during the cold and rainy seasons of the year, especially when the night recorded temperature is below 10° C. This shortening of pavement service life is considered to be caused mainly by the poor adhesion bonding between the aggregates and the binder when the construction is done during cold and/or rainy periods. Figure 1-1 below shows the temperature distribution throughout the country.

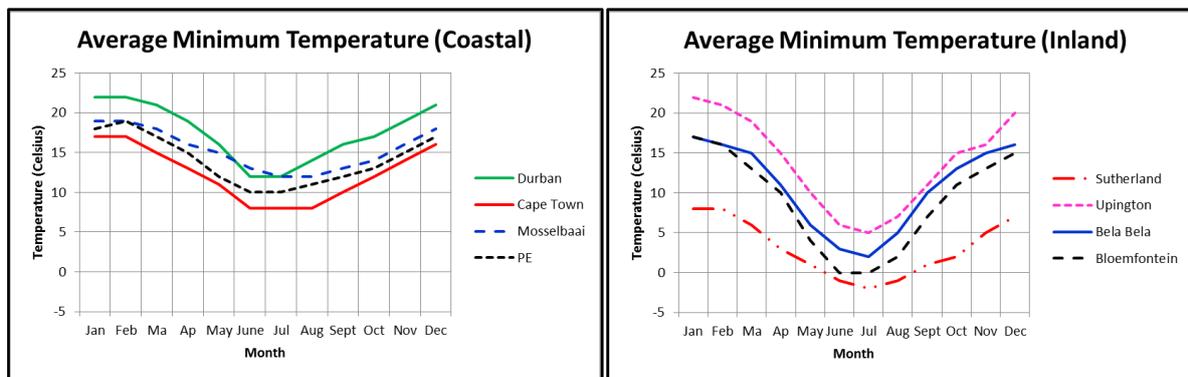


Figure 1-1: Average Minimum Temperatures (Coastal and Inland) (Van Zyl *et al.*, 2012)

In this figure, it appears that chip loss related to winter sealing activities is likely to occur over a large part of the country, especially in Inland areas. Moreover, as mentioned previously, there are a significant number of kilometres to be rehabilitated every year. At the same time, a more constant supply of sealwork needs to be provided for contractors to ensure their profitability. Due to that, the South African trend is towards maximizing sealing activities throughout the year, which will need to extend sealing work to winter periods.

Therefore, there is a need to achieve what is mentioned above and at the same time ensure that the constructed seal meets short- and long-term performance requirements. It is obvious that the scientific understanding of the interaction between various aggregate-binder combinations under winter conditions is necessary.

1.3 RESEARCH OBJECTIVES AND SCOPE

The main objective of this research is to contribute to the understanding of bitumen adhesion properties for winter bituminous surfacing seals. This is achieved through evaluation of the various bitumen-aggregate combination interactions under winter conditions and by exploring the influence of bitumen emulsion based precoats to the bond strength development. The Bitumen Bond Strength (BBS) test is used in this study.

In an effort to achieve the above-mentioned main objective, the following specific objectives were defined:

1. Detailed identification of the factors that significantly influence the bond strength development between various types of aggregate and binders through a literature survey.
2. Evaluation of the compatibility between acidic and basic aggregates with various types of binder commonly used in South Africa at low temperature conditions by using the BBS.
3. Evaluation of the influence of different bitumen emulsion based precoats to the bitumen-aggregate bond strength development at low temperatures by using the BBS.
4. Evaluation of the impact of curing conditions of the precoated aggregates to the bitumen bond strength development at low temperatures by using the BBS.
5. Evaluation of the repeatability of the BBS

The scope of this study is as follows: a critical analysis and review of the past studies related to bitumen adhesion properties in general and to bitumen adhesion, properties for winter surfacing seals in particular were done. An experimental matrix involving the combinations of various types of materials as shown in Chapter 3 was prepared. As shown by the matrix, the study was limited to the use of three types of bitumen, two hot applied bitumen (70/100 penetration grade bitumen and SBS modified bitumen (S-E1)) and one cold applied bitumen (SC-E1). Two types of aggregate commonly used in SA with different surface chemical behaviour were used. These are granite with acidic behaviour and dolerite with basic behaviour. The above-mentioned bitumen was applied to non-precoated aggregates as well as to precoated aggregates. Three types of bitumen emulsion based precoat fluid were used. These are a 30% water diluted non-fluxed SS60, an anionic emulsion with cutters and a cationic emulsion with cutters. All of these precoat fluids as well as the bitumen were supplied by Colas, Cape Town. In addition to this, two types of precoated aggregate curing conditions were used. These are, wet condition, in which the cutters in the precoats were not allowed to evaporate, and dry conditions, by which it is assumed that the cutters in the precoats have evaporated. Low application temperatures of bitumen and testing temperatures were adopted to model the winter seal conditions. Water damage was not part of the investigation in this study.

1.4 THESIS OUTLINE

This research thesis is described into six chapters as follows:

Chapter 1: Introduction

The first chapter provides a brief introduction to the research project, and states the problem that has pushed this research to be carried out. Moreover, this chapter discusses the research objectives as well as the outline of the thesis.

Chapter 2: Literature Review

The second chapter consists of an extensive review of previous studies on bitumen adhesion properties for various aggregate-binder combinations. Studies pertaining to the impact of bitumen based precoating fluid on aggregate-binder interactions were reviewed as well. Special attention is given to the factors affecting the binder-aggregate adhesion bond as well as to the adhesion failure mechanism. This chapter also deals with the review of the surfacing seal design and failure mechanism. Finally, the current available adhesion testing methods are reviewed and the BBS test method is described in particular.

Chapter 3: Research Methodology

This chapter provides a review of the methods, strategies and procedures used by the researcher to achieve the proposed objectives. Furthermore, this chapter describes in detail the experimental design and laboratory testing.

Chapter 4: Results Discussions

This chapter pertains to the discussion of research results. The results from laboratory testing are analysed and scientific interpretations corresponding to the scrutinized results are provided.

Chapter 5: Analysis, Interpretation and Applicability

This chapter examines further and interprets the laboratory test results relative to their applicability in winter seals construction.

Chapter 6: Conclusion and Recommendation

The sixth chapter concludes the thesis and is based on the objectives and the findings of the study. Finally, it provides recommendations for proposed further studies in this field.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

The adhesion of the bitumen-aggregate system is a primary factor that dictates both short and long-term performance of surfacing seals. The winter surfacing seals have long suffered of premature failures. Winter failures such as early aggregate chip loss are linked to poor adhesion between bitumen and aggregate due to unfavourable environmental conditions. Chip loss may take place immediately after construction or during the surfacing seal service life and occurs through primarily adhesive bond failure, although some cohesive failure can occur.

The bitumen-aggregate adhesion bond is a very complex phenomenon to analyse. This is due to a significant number of factors contributing to its development. The contributing factors include but are not limited to aggregate type, binder type, binder curing time and condition. In general, these contributing factors are categorised into material properties, environmental conditions, traffic and construction techniques (Tarrer & Wagh, 1991). In this review, the emphasis is placed on material properties and environmental conditions influencing bitumen-aggregate adhesion bond development.

The Strategic Highway Research Program (SHRP) found that bitumen-aggregate interaction is strongly dependant on material properties. The aggregate physico-chemical properties have shown to have a stronger influence on the bitumen-aggregate interaction than bitumen chemistry (Curtis, Ensley & Epps, 1993). Be that as it may, the material properties influence had been observed but also the specificity of material combinations was one of their findings. As such, it is cogently important to investigate the material compatibility and the right material combination prior to the construction of surfacing seals, especially in adverse conditions. Thus, in order to ensure a good performance of winter surfacing seals, the micro- and macroscopic bitumen-aggregate interactions need to be fully understood.

This review of relevant literature was done in three main sections. Firstly, there was a review of literature pertaining to bitumen-aggregate system adhesion bond development, which was done through a description of primary material factors influencing adhesion bond development. The adhesion bond principles, development and failure mechanisms were then documented. Secondly, a critical review and discussion was carried out on the design, construction and failure mechanisms of surfacing seals. Lastly, there was a systematic description of the current state of the art of methods of measurement of practical adhesion and the method selected to be used in this study.

PART A: BITUMEN-AGGREGATE SYSTEM ADHESION BOND DEVELOPMENT

This section is a review of previous studies pertaining to bitumen-aggregate adhesion bond development. The review was discussed in two main components. The material properties influencing adhesion bond development were reviewed first and literature related to adhesion development principles and failure mechanism followed.

2.2 MATERIAL PROPERTIES INFLUENCING ADHESION BOND DEVELOPMENT

In this section, the focus was placed on the properties of the three types of materials used in this study: the aggregate, seals bitumen binder and precoating fluid.

2.2.1 Aggregate Properties

2.2.1.1 Introduction

The aggregates mineralogical composition and physical-chemical properties such as morphology, surface chemistry, electrical properties, etc. play a vital role in bitumen, aggregate and water interaction (Tarrer & Wagh, 1991). Curtis *et al.* (1993) used the net adsorption test to analyse the bitumen-aggregate interaction. They varied the types of aggregate (known as “SHRP Material Reference Library (MRL) aggregates”) for a particular type of bitumen. The difference in bitumen affinity and water damage tendency was recorded. It was observed that this difference was large compared to the case where the aggregate type was kept constant and the bitumen types varied. Their study concluded that aggregate surface characteristics influence bitumen-aggregate interactions more than bitumen properties.

Thelen (1958) explained the poor adhesion bonding of cold aggregates to bitumen compared to the adhesion of hot aggregate and bitumen by using the interfacial free energy concept. This involves the aggregate surface characteristics. Therefore, it is quite clear that understanding the aggregate surface properties is crucial. This is especially important for winter surfacing seals where the bitumen-aggregate adhesion development is problematic due to adverse weather conditions.

2.2.1.2 Aggregate Mineralogical Composition

The physico-chemical properties of aggregate are similar to those of the parent rock. The properties of the latter as a construction material are controlled primarily by its mineral composition and secondarily by the size, shape and arrangement of, and bond between the minerals (Weinert, 1980). The aggregate consists of a large number of one or more type of minerals that have a definite chemical composition and an ordered internal atomic arrangement. The internal atomic arrangement is facilitated by the fact that each atom is bound to its neighbouring atom by an electrostatic coordination bond, thereby forming a stable three dimensional network known as “lattice” (Mitchell, 1993).

Weinert (1980) reported that even though the number of minerals in aggregate is great, most of them occur as accessories. The other have similar properties in such a way that they can be treated as entities. Those entities, which are almost a dozen, are known as “rock forming minerals”.

The predominant rock forming minerals in rocks that are commonly crushed to get road construction aggregates are “Silicate minerals” which are composed essentially of silicon dioxide (SiO_2), “Calcite minerals (CaCO_3)” and certain sulphur containing minerals.

In the crushing process during aggregate production in the quarry or during construction, the aforementioned electrostatic coordination bonds between atoms are broken thereby creating a new aggregate surface. Prior to the creation of the new surface, the atoms in the bulk system experience uniform forces in all directions. This is due to interactions with neighbouring chemical units as shown in Figure 2-1 (a). In creating the new surface, the atoms exposed to the new surface become deprived of part of their neighbouring atoms as shown in Figure 2-1 (b). This is caused by breaking some of the electrostatic coordination bonds between atoms. The lack of interaction causes an energetic instability to the surface atoms or molecules i.e. the free energy of the system is increased (Thelen, 1958; Hefer, 2004). This molecular description is the basis for the surface energy and chemical concepts that are discussed subsequently as part of the adhesion mechanism as well as for the surface charge development discussed in section 2.2.1.3.

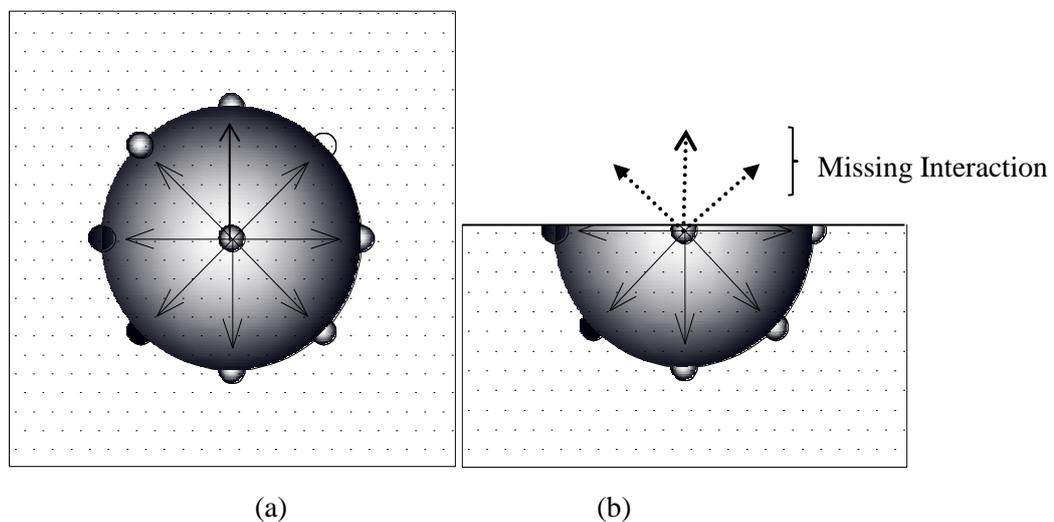


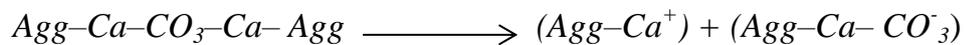
Figure 2-1: Illustration of atoms interactions for (a) the atoms in the aggregate bulk system and (b) the atoms at the aggregate surface adopted from (Venugopalan, 2012).

The energetically unstable surface atoms restore their equilibrium by redirecting some of the forces inward, which consequently reorient the crystal lattices as the chemical units come closer. Another way to restore the equilibrium is to attract contaminants such as dust and organic materials (clay) at the surface. The latter also serve as active sites of the aggregate surface during the interaction with bitumen (Twagira, 2010).

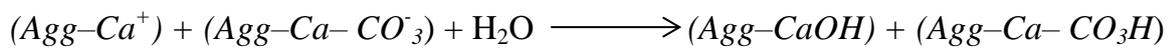
2.2.1.3 Aggregate Chemical Properties

Aggregate surfaces are classified as acidic, basic or neutral based on their ionic strength or acid-base interaction. When the aggregate is crushed in the quarry, some of the coordination bonds between atoms are broken and a multitude of unsatisfied charges occurs on the newly formed surfaces. The surface charge development may serve as the basis for explanation of the basic or acidic behaviour of aggregate surface. The subsequent sections discuss the mechanism of charge development on aggregate surface as has been described by Mertens and Wright (1959).

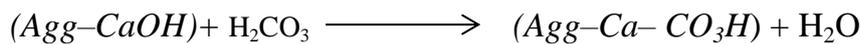
The aggregates composed mainly of calcite minerals (CaCO_3) are known to have basic surface characteristics. The simplified chemical reaction describing the fracturing process of such aggregates with associated formation of countless unsatisfied charges is as follows:



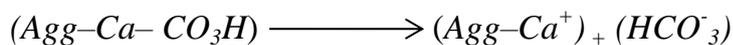
Agg stands for the bulk aggregate structure, with atomic lattice constituting CaCO_3 rhombohedral unit cells, and intermolecular bonds symbolized by “-“. In an effort to satisfy the broken bond, water vapour in the air is adsorbed onto the aggregate surface. The hydration reaction that takes place is described as follows:



The resultant calcium hydroxide adsorbs the carbon dioxide dissolved in the surface water ($\text{CO}_2 + \text{H}_2\text{O} \longrightarrow \text{H}_2\text{CO}_3$). Thus, an additional reaction takes place as follows:

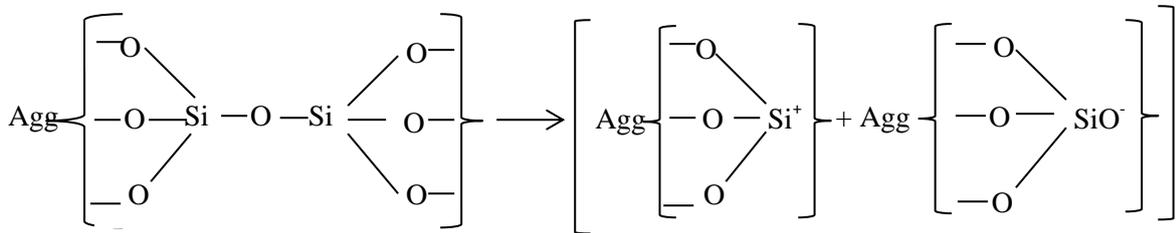


This shows that, irrespective of the type of charge developed directly after cleavage, the final surface chemical composition is represented by the right side of the reaction. Once water is present, this structure dissociates to yield a characteristic electropositive charge on the aggregate surface as shown below:

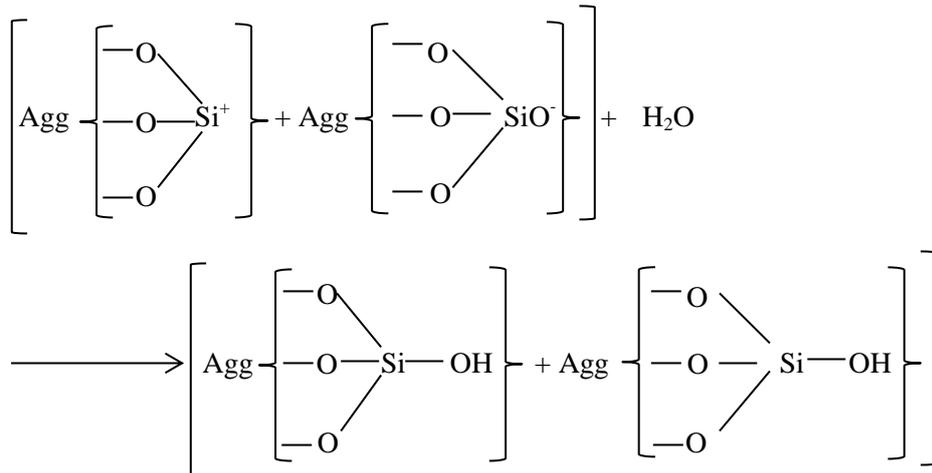


These types of aggregate have shown basic chemical characteristics with a positively charged surface. They are considered to be water hating (Hydrophobic) and are believed to develop a strong fundamental adhesion bond with the bitumen surface which is known to have negatively charged functional groups.

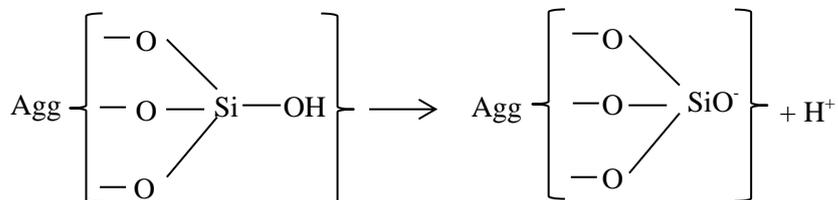
On the other hand, the process of the fracturing and formation of unsatisfied charges of aggregates that are made predominantly of minerals containing silica (SiO_2), such as granite and quartzite, can be presented by a simplified reaction as follows:



With Agg standing for bulk aggregate structure, with the atomic lattice composed of silica tetrahedral unit cells. In response to the developed unsatisfied charges, water is adsorbed onto the surface. The hydration reaction takes place to form a hydroxylated surface (covered with OH, called Silanol groups) as shown below.



Further water adsorption initiates the dissociation of the surface hydroxyl group, by imparting the protons to the contacting water. This results in the development of a negative charge on these hydroxylated surfaces, which is a characteristic of silica surfaces.



The amino, sulphate and carboxyl groups were identified as some of other groups of mineral aggregate surfaces that could dissociate to form charges. Adsorption of H^+ and OH^- ions influences the charge development on these surfaces (Mertens & Wright, 1959).

The level at which the above-mentioned reactions occur depends on the pH of the water which comes in contact with the mineral surfaces. Water with a high pH value (High concentration of OH⁻ ions) favours the dissociation of Silanol groups. Thus, it induces the surfaces to be more negatively charged.

Furthermore, it has also been noted that at low pH values, the surfaces of siliceous aggregates can be positively charged (Gast, 1977; Butt, Graf & Kappl, 2003). Thus, it is clear that the pH value of the contacting water has an influence on the bitumen-aggregate interaction.

Mertens and Wright (1959) stated that natural aggregate surfaces contain elements that cause both electropositive and electronegative features. They provided basalts, diorites and siliceous limestones as examples of aggregates that have an intermediate character. Figure 2-2 classifies aggregates according to surface charges.

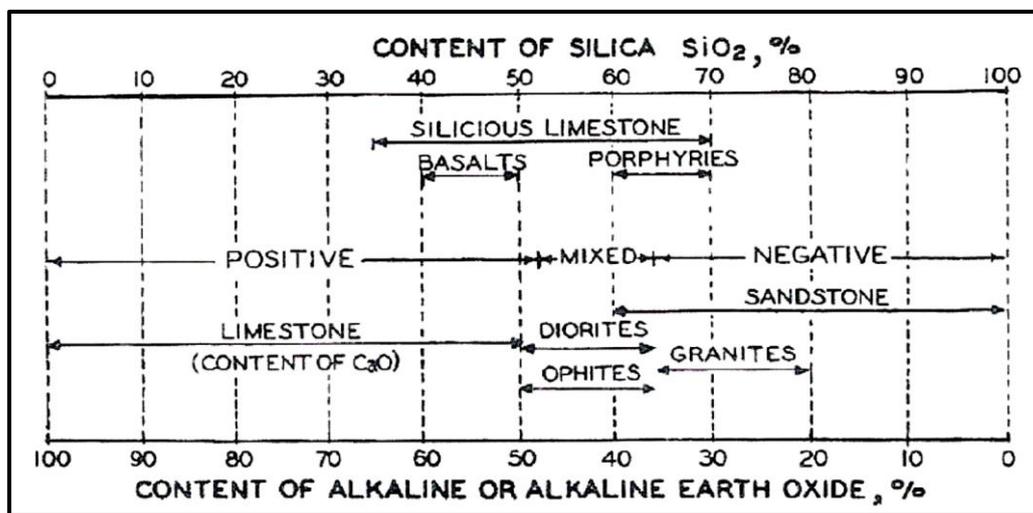


Figure 2-2: Classification of aggregates based on silica and alkaline content (Mertens & Wright, 1959)

Curtis *et al.* (1993) found that the aggregate surface charges determine to some degree the extent of attraction and adsorption of bitumen binder to aggregate surface and thus adhesion between them.

A study by Ross using autoradiographic experiments states that the aggregate surface is heterogeneous and has a variety of sites of different composition and levels of activity. It has also been mentioned that these active sites are frequently charged or contain partial charges that attract and orient the polar components of bitumen binder (Ross, Mirsalis, Loo & Rhee, 1991). The active sites on mineral aggregate surface did not receive as much attention as bitumen by researchers in this field.

Petersen and Plancher (1998) in their literature review presented intuitive reference in this regard. They mentioned that the active sites on aggregate surface range from hydroxyl groups of varying acidities to hydrogen bonding sites of high acidity. They further mentioned that aggregate which

contains some metals such as magnesium, iron and calcium may also form strong electropositive Lewis acid bonding sites. This seems to complement what has been reported by Jamieson *et al.*, namely that chemical sites on aggregate surfaces associated with a high affinity for bitumen include elements such as aluminium, magnesium, iron and calcium (Jamieson, Moulthrop & Jones, 1995).

As previously discussed, the chemical composition of the aggregate is reflected by the charge development during the crushing process and the corresponding active sites strongly influence the adhesion between aggregate and bitumen. The next section discusses other properties that cannot be neglected in the study of adhesion and physical properties of aggregates, such as but not limited to aggregate porosity and roughness.

2.2.1.4 Aggregate Physical Properties

The physical characteristics of aggregate surfaces have shown its capability to influence the adhesion properties of the bitumen-aggregate system. The surface texture which is considered to be the small irregularities of aggregate surface ranges from smooth to rough (Das, 2004). During the rock crushing process, the surface texture of the resulting aggregates is determined by the size of the minerals in a rock, their state of crystallization, their geometrical arrangement as well as by the type of bond between them (Weinert, 1980). Coarse-grained mineral rocks particularly those containing large feldspars, once crushed provide surfaces with numerous flat faces. Therefore, they are more prone to adhesion problems than fine-grained rocks that produce irregular surfaces when crushed.

Pocius (1997) made an insightful contribution by describing the importance of surface roughness to improve adhesion. He mentioned that surface roughness prevents an abrupt plane stress transfer. It facilitates stress transfer across or into the binder, which exhibits viscoelastic and plastic behaviour in most cases. Therefore, due to adhesive characteristics, the surface roughness may favour the dissipation of energy around the crack and in the bulk system (Pocius, 1997). The same author further stated that by increasing the physical contact area, surface roughness can also promote adhesion due to the fact that the bitumen-aggregate interaction is directly proportional to the contact surface area. This seems to be in agreement with what has been reported by (Curtis *et al.*, 1993) that an aggregate with rough surface presents more opportunity for the second bond due to great surface area per unit volume of aggregate.

However, Tarrer and Wagh (1991) stated that the surface texture of an aggregate affects its adhesion properties by influencing the wettability of aggregates, in that it is easier to wet a smooth aggregate surface than it is a rough one. Moreover, in a study on investigation of critical factors influencing bitumen bond strength development, in which various factors such as aggregate type, moisture condition, surface roughness, loading rate, curing temperature and curing humidity were investigated,

there was no statistically significant evidence found to justify the influence of aggregate surface roughness on bond strength development (Miller, 2010).

In addition, in a study on Bitumen Bond Strength by using the Pneumatic Adhesion Tensile Testing Instrument (PATTTI), the combination of different types of binder and aggregates with different surface morphology (smooth and fractured aggregate surface) were analysed.

The results showed that aggregate with a fractured surface affected the bitumen-aggregate bond strength negatively compared to aggregate with a smooth surface (Stander & Jenkins, 2011; Jenkins *et al.*, 2013; Jenkins & Van de Ven, 2013).

Therefore, it is believed that in general, slight surface roughness improves the adhesion properties through mechanical interlocking. However, the level of roughness needed is not well defined in the available literature.

Many researchers have also identified aggregate surface porosity as one of the factors influencing bitumen-aggregate adhesion. Thelen (1958) and AAPT (1958) reported that, once the aggregate is coated by bitumen, the latter penetrates the surface pores and cracks to some extent. Thus, through mechanical interlocking between bitumen and the aggregate surface, the adhesion between them is improved. Aggregates that have a large porous surface such as limestone appear to have a stronger adhesion bond with bitumen than aggregates that possess smaller pores on the exposed surface such as quartzite (Thelen, 1958). This concurs with the postulations of Curtis *et al.* (1993) who found that once bitumen penetrates the pores on the aggregate surface, the intramolecular attractions within the binder provide an interlocking network, and consequently a firm bond.

However, it is also considered that a porous aggregate surface is not always beneficial to the bitumen-aggregate adhesion bond. Jeon and Curtis (1990) stated that aggregate surfaces with a high pore density can act as molecular sieves, separating high and low molecular weight bitumen fractions. Phenomenological observations indicate that when selective absorption occurs, the bitumen remaining on the outside becomes hard and brittle (Curtis *et al.*, 1993).

Furthermore, it was also observed by researchers that contaminants such as dust and water on aggregate surfaces could influence the adhesion properties of aggregate. The presence of dust on the aggregate surfaces prevents effective coating and therefore intimate contact with the adhesive. Figure 2-3 illustrates how the presence of either a water layer or dust on the aggregate surface can influence bitumen-aggregate interaction. Tarrer and Wagh (1991) reported that once aggregate is mixed with bitumen, dust on the aggregate surface has a tendency to trap air and thus the bitumen-aggregate bond is weakened by preventing an intimate contact between them. Moreover, this can favour the stripping

phenomenon by providing a channel between the aggregate and the binder that can easily be accessed by water and displace bitumen.

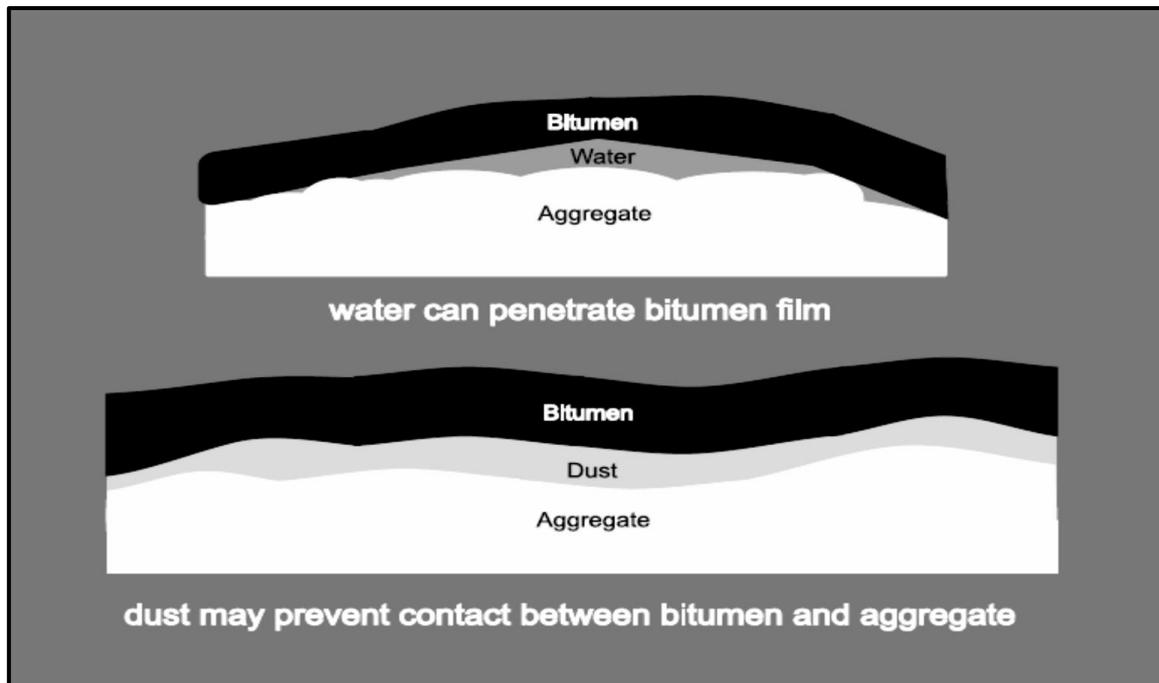


Figure 2-3: Interaction between bitumen and aggregate with the surface coated with dust or water layer (AKZO NOBEL, 2006)

In conclusion, the literature review has shown that the mineralogical composition and surface chemical and physical properties of aggregate do influence the bitumen-aggregate adhesion bond. The influence of winter environmental conditions on aggregate performance is discussed under section 2.3 on adhesion principles and mechanisms. The following section discusses the influence of winter seals bituminous binder characteristics.

2.2.2 Seals Bituminous Binder

2.2.2.1 General Introduction

Bituminous binders used in seal constructions are conventional bitumen, polymer-modified bitumen, cutback bitumen or bitumen emulsion. Various authors define bitumen differently. In this study, Francken's definition of bitumen was adopted. He defined bitumen as "*a viscous liquid or solid, consisting essentially of hydrocarbons and their derivatives, which is soluble in carbon disulphide: it is substantially non-volatile and softens gradually when heated. It is black or brown in colour and possesses waterproofing and adhesive properties. It is obtained by the refinery processes from petroleum and is also found as a natural deposit, or as a component of naturally-occurring asphalt, in which it is associated with mineral matter.*" (Abraham & Jenkins, 2012)

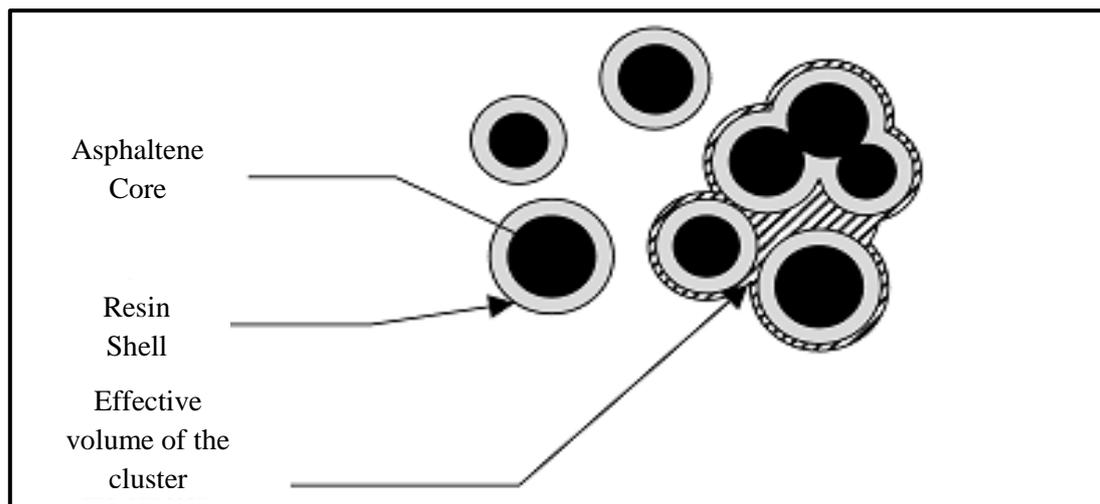


Figure 2-5: A Simplified view of the colloidal model of bitumen: The asphaltenes micelles are pictured to show the shells of the peptizing resins. The oily dispersion medium is the maltene phase (Lesueur, 2009).

During the Strategic Highway Research Program (SHRP) in the 1990's, the colloidal model hypothesis discussed above was discarded by researchers (Lesueur, 2009). They accused this model of inability to predict the bitumen chemistry-physical property-performance relationship.

As part of the SHRP research, a new model was developed which is referred to as the “Dispersed Polar Fluids” (DPF) model or “microstructural model”. In this model, bitumen is considered to be a single-phase homogeneous mixture of many molecules which can be grouped into two main categories, namely polar molecules and non-polar molecules. The non-polar molecules serve as the solvent for the polar molecules, which associate into polar-polar weak networks that give the bitumen its elastic properties. They emphasised on the fact that there are no micelles in bitumen. It has also been mentioned that the polar molecules are uniformly distributed throughout the bitumen and once the bitumen is heated, the weak polar-polar bonds are broken to yield a Newtonian fluid. When disturbed, these interactions break and reform to generate a new bitumen type (Jones & Kennedy, 1991; Lesueur, 2009). Figure 2-6 illustrates the Dispersed Polar Fluid Model.

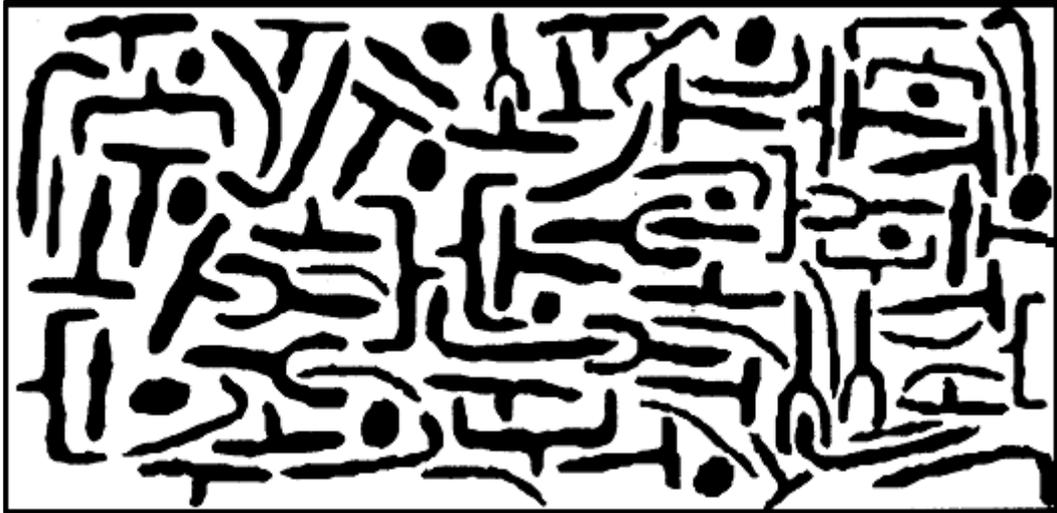


Figure 2-6: SHRP Dispersed Polar Fluid Model for Bitumen (Jones & Kennedy, 1991)

It should be noted that the aforementioned models are both still in use by different engineers and asphalt technologists, to explain the rheological properties of bitumen.

Rheological Properties of Bitumen

Rheology is the study of the flow and deformation of viscoelastic materials that are subjected to either shear or extensional stresses (Schramm & Haake, 1994; Yusoff, Shaw & Airey, 2011).

Generally, the prediction of the engineering performance of any material is based on the understanding of the material's stress-strain behaviour. One of the simplest methods to determine the stress-strain behaviour of any material is the creep test. The mechanical response of bituminous materials in a creep test as shown in Figure 2-7 has revealed that bitumen exhibits aspects of both viscous and elastic behaviour. Thus it is a viscoelastic material and its behaviour characterisation must account for the loading rate and temperature (Anderson *et al.*, 1994).

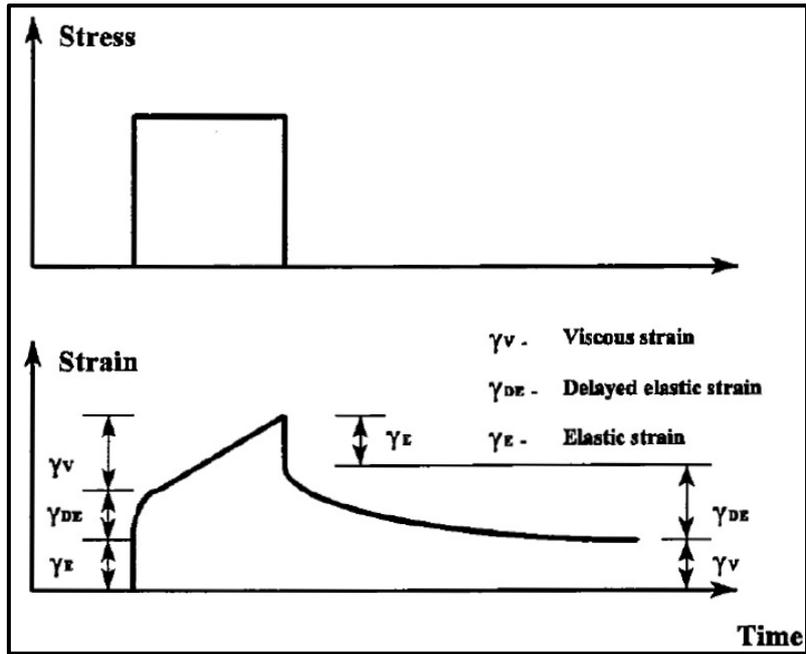


Figure 2-7: Bitumen Viscoelastic response in a creep test (Airey, 1997)

Three commonly used test methods to characterise the viscoelastic behaviour of bitumen exist. These methods are creep, stress relaxation and dynamic mechanical analysis tests. Each of these methods yields a complete characterisation of the viscoelastic behaviour of the selected bitumen, but in different ways (Anderson *et al.*, 1994). Among these methods, nowadays, dynamic mechanical analysis using a dynamic shear rheometer (DSR) is the most frequently used.

The DSR, which appears in various configurations, is a powerful tool to characterise the elastic, viscoelastic and viscous properties of bitumen over a wide range of temperatures and frequencies. A typical stress-strain behaviour characterisation using the DSR for bitumen is shown in Figure 2-8.

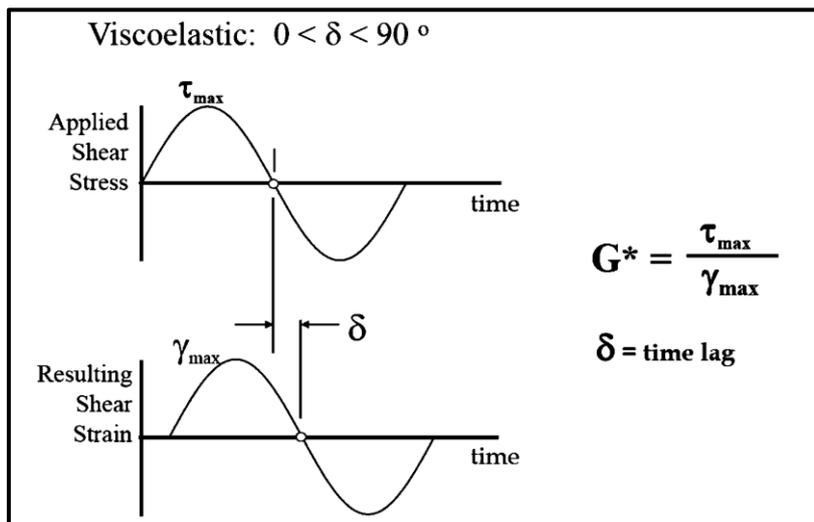


Figure 2-8: Viscoelastic materials response to a dynamic loading (Van de Ven, 2013)

The results of DSR tests for the characterisation of the rheological properties of bitumen are generally presented in the form of complex modulus (G^*) and phase angle (δ) master curves. As shown in Figure 2-8, G^* is the ratio of maximum shear stress to maximum shear strain and δ is the phase angle between stress and strain in harmonic oscillation.

In a dynamic test, if δ for a specific bitumen sample is equal to 90° , this bitumen type may be considered as purely viscous and can be characterised by a coefficient of viscosity. On the other hand if δ is equal to 0° , the bitumen is considered to be purely elastic and its behaviour may be characterised by Young's modulus. If $0^\circ < \delta < 90^\circ$, in this case, the material response combines both viscous and elastic behaviour and the bitumen is called viscoelastic, and the bitumen behaviour in this case is characterised by the values of G^* and δ (Yusoff *et al.*, 2011).

Various research projects in the past have revealed that these rheological parameters (G^* and δ) are strongly dependant on loading rate (time of loading or loading frequency) and temperature for a specific bitumen type. Therefore, in order to provide full characterisation of a specific bitumen, there is a need to carry out rheological tests for all possible temperature and loading rate combinations in the practical range. This was found to be impractical due to a tedious and time-consuming test required.

Fortunately, the time-temperature superposition (TTSP) principle illustrated in Figure 2-9 has provided the possibility of carrying out tests at different temperatures for a specific range of loading frequencies. Using the TTSP principle the results can be converted into a curve known as the "Master Curve" shown in Figure 2-10, which presents the behaviour of bitumen at a specific temperature for an extended range of loading frequencies.

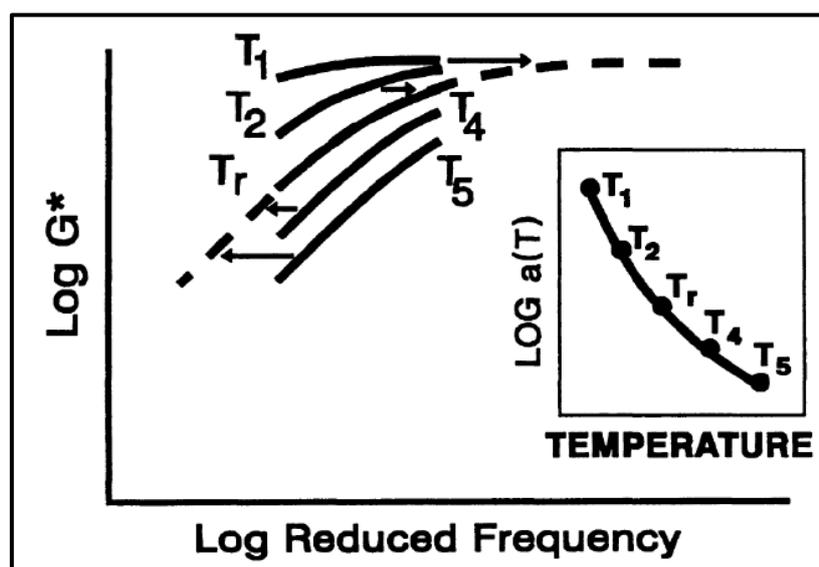


Figure 2-9: Time-temperature superposition principle (Anderson *et al.*, 1994)

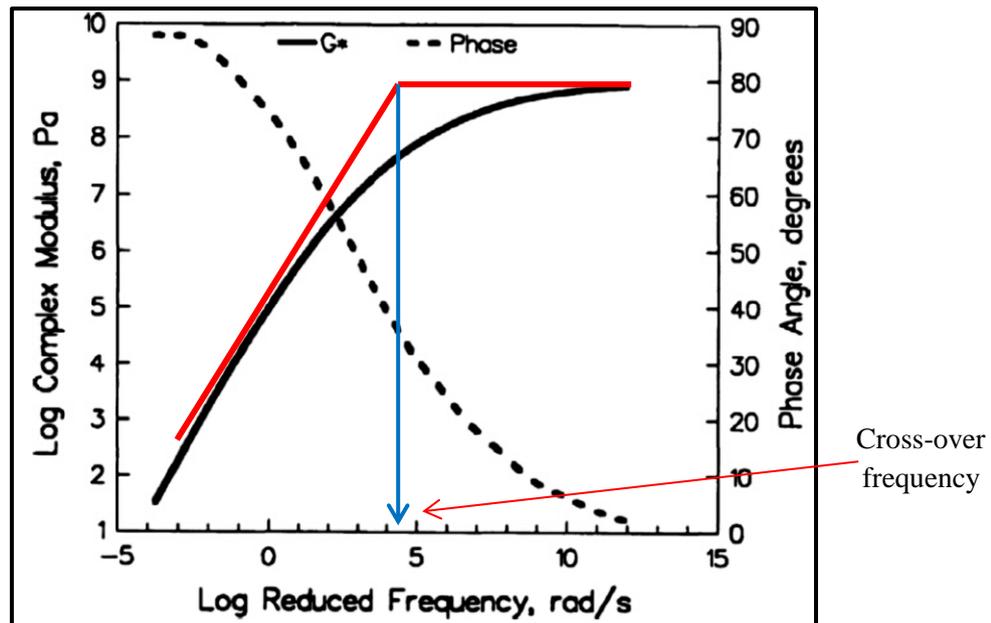


Figure 2-10: Rheological parameters (G^* & δ) master curves (Adapted from Anderson *et al.*, 1994).

The G^* curve in Figure 2-10 illustrates that as the loading rate increases (or the temperature decreases) the complex modulus (representing the stiffness) of bitumen increases up to the level where the complex modulus remains almost constant. This complex modulus is commonly known as “Glassy modulus in shear” that is approximately 1GPa. On the other hand, as the loading rate decreases, the complex modulus decreases too. Up to a level the slope of the curve is almost 1:1, which signifies that viscous flow has been reached. The point of intersection of asymptote passing through the region of viscous flow and an asymptote passing at the glassy point defines the “Cross-over frequency” as shown in Figure 2-10, which is one of the parameters defining the loading rate (time) dependency of the bitumen behaviour (Anderson *et al.*, 1994).

The same authors mentioned that the temperature dependency of bitumen behaviour is defined by the curve of $\log a(T)$ versus temperature shown in Figure 2-9, with $a(T)$ representing the amount of shifting required for rheological parameter from any testing temperature to the reference temperature during the master curve construction process. As rheological properties reflect the flow and deformation behaviour of bitumen, it is obvious that the loading rate and temperature will affect the bond strength development and failure mechanism. The sections below continue with discussions of the implications of bitumen binder properties on bond strength development. However, the influence of loading rate on the bitumen bond strength testing is discussed in section 2.6.7.

Due to its rheological properties discussed above, bitumen at room temperature is either solid or semi-solid. When used for sealing one has to ensure that the bitumen can sufficiently wet the aggregates, it must be made liquid enough to do the job. This can be achieved through three different ways as listed below (Senadheera et al., 2006):

- The viscosity of bitumen can be reduced by heating bitumen to elevated temperatures in a heating kettle or storage tank. This technique is used for conventional pen grade bitumen and modified bitumen and due to that, they are known as “Hot Applied Binders”.
- The viscosity may be reduced also by mixing bitumen with a specific amount of a dissolving petroleum product which act as solvent, the resulting binder is known as “Cutback Bitumen”.
- Lastly, the viscosity reduction can be achieved by combining the base bitumen with water in the presence of some specific chemical compounds known as emulsifiers. The resulting binder is known as “Bitumen Emulsion”.

All of these types of binder can be used as they are, or modified by different modifiers to improve their rheological properties. Worldwide due to environmental concerns, the cutback bitumen is nowadays seldom used. The following section analyses previous studies on the properties of hot applied bitumen and bitumen emulsion, which affect the bitumen aggregate adhesion.

2.2.2.2 Hot Applied Binders

In surfacing seal construction, a binder should be able to develop a strong adhesion bond to the underlying pavement layer and to the aggregates applied over it. The necessary bonding to maintain the aggregate in place should be developed and should be maintained throughout the seal’s service life. The developed bonding must be able to retain aggregates even under adverse conditions (Senadheera, 2006). The literature survey showed that the viscosity of the binder at the time of aggregate spreading (defined by rheological behaviour) and the chemical composition of bitumen were found to be influential binder properties to the adhesion bond development. The section below discusses the previous study related to the influence of these properties.

I. Penetration Grade Bitumen Binder

Rheological Properties

Researchers such as Bagampadde *et al* (2003) and Hicks (1991) examined the influence of rheological properties of bitumen on adhesion bonding development. It was noted that the viscosity of bitumen at the specific time that the bitumen and aggregate come into contact is a critical factor. If at that time the viscosity of bitumen is high, then it may not effectively wet or coat the aggregate surface. The same authors have also mentioned that, in the surfacing seal’s service life, the use of a high viscosity binder can be beneficial against adhesive or cohesive strength loss due to moisture (stripping).

This is due to the fact that high viscosity bitumen usually carries high concentrations of bitumen polar functional groups, which contribute to the bitumen-aggregate interaction and provide a resistance to stripping.

Moreover, as has been mentioned before, once the bitumen comes into contact with porous aggregate, bitumen penetrates into the surface pores and cracks. By mechanical interlocking, this will improve the adhesion between aggregate and bitumen. However, the penetration of bitumen into aggregate pores depends largely on the bitumen's viscosity at the time of contact. If the viscosity of bitumen is high, penetration will be less. In this case, the contribution of mechanical interlocking to the adhesion bond is reduced and poor adhesion can be the result.

Chemical Properties

The chemical composition of bitumen is complex and differs from one bitumen type to another, depending on the crude oil source and the refinery process. Bitumen is a chemically complex mixture predominated by hydrocarbon compounds, with some heterocyclic species and functional groups containing sulphur, nitrogen and oxygen atoms. The elemental analysis of bitumen from various sources has shown a typical elemental composition as carbon 82-85%, hydrogen 8-11%, sulphur 0-6%, oxygen 0-1.5% and nitrogen 0-1% (Read & Whiteoak, 2003). Attempts to correlate bitumen's elemental chemical composition to its rheological properties have never been possible, due to its chemical complexity. However, the bitumen chemical composition can be grouped into two broad chemical compositions namely; asphaltenes and maltenes. The latter ones can be also split into three components, namely; resins, aromatics and saturates. This broad chemical composition of bitumen is used to explain its rheological properties through the colloidal model specified in the previous sections. Figure 2-11 illustrates the broad chemical composition of bitumen.

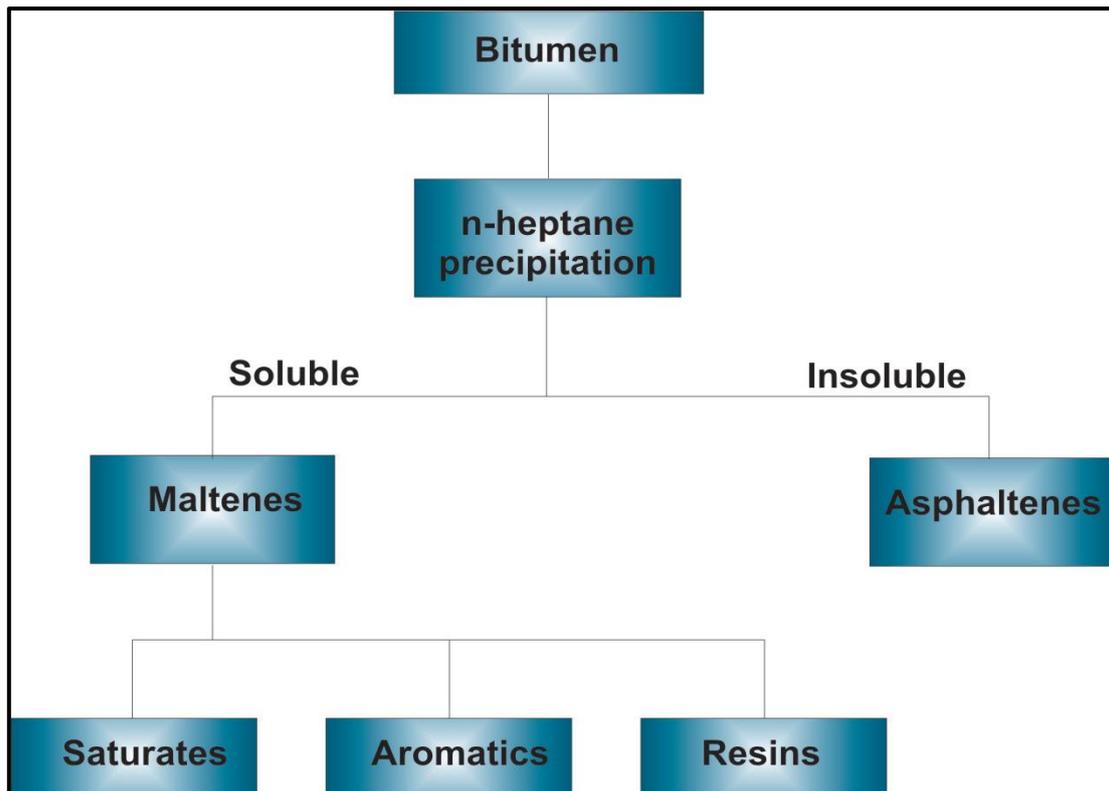


Figure 2-11: Broad Chemical Composition for Bitumen (Sabita, 2007)

Despite the above-mentioned inability to correlate bitumen's elemental chemical composition to its rheological properties, past studies on bitumen-aggregate interactions have shown that the chemical composition plays a significant role in bitumen-aggregate adhesion. The key role players are the elements that contribute to the polarity of some bitumen molecules.

Curtis *et al.* (1993) attested that the chemical functionalities in bitumen molecules are normally present or formed on oxidative ageing as shown in Figure 2-12. They have observed that sulfoxides bitumen functional groups are very sensitive to water displacement. This concurs with what was reported by Petersen (1974) on the carboxylic acid bitumen functional group.

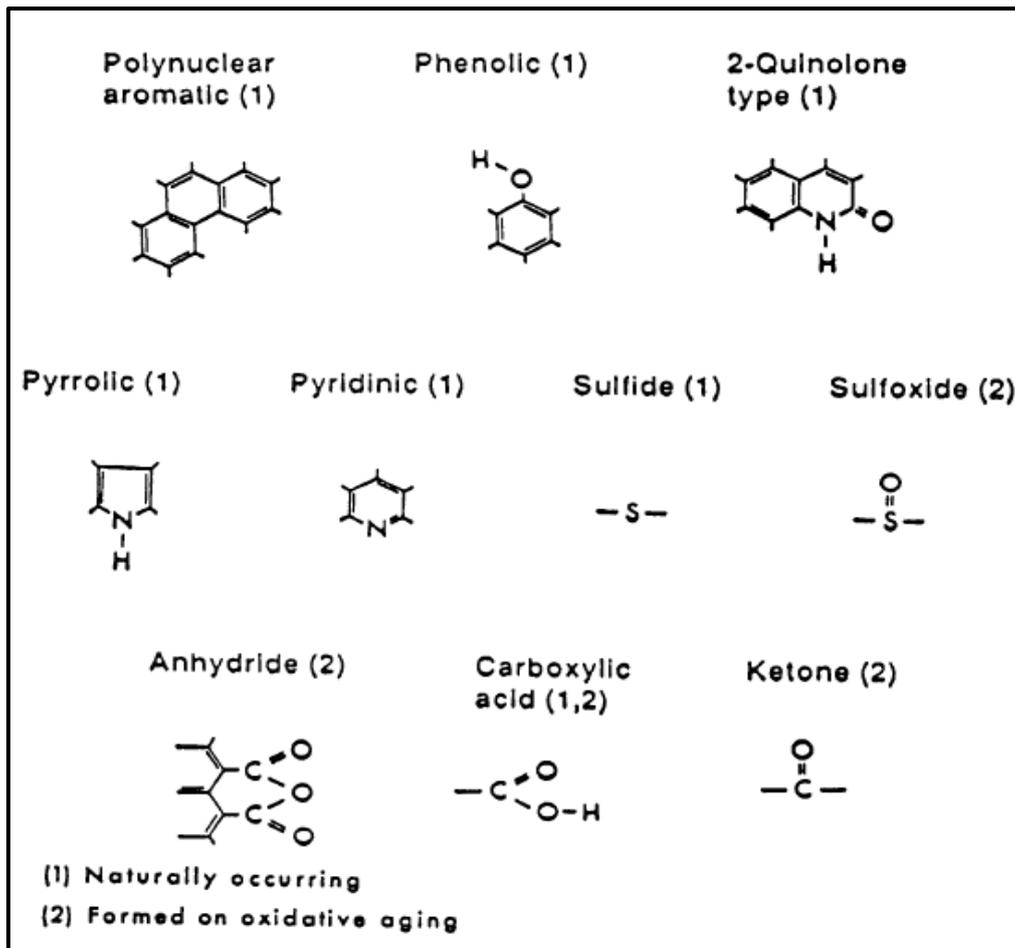


Figure 2-12: Chemical functional groups in bitumen normally occurring or formed on oxidative ageing (Curtis *et al.*, 1993)

Plancher *et al.* (1977) investigated the components of the bitumen functional groups that form the chemical bond with mineral aggregates and studied their sensitivity to water displacement. This was done by extracting bitumen from a field asphalt core by using benzene and benzene/alcohol. After obtaining the benzene and benzene/alcohol extracts, they analysed them by using differential infrared spectroscopy techniques. The study has identified and arranged bitumen functional groups according to their affinity to bitumen as follows (Plancher *et al.*, 1977; Petersen & Plancher, 1998):

Carboxylic acids > Dicarboxylic anhydrides > 2-Quinolone types > Sulfoxides > Nitrogen > Ketones

Surprisingly, another outcome of this study was that most of the highly adsorbed bitumen functional groups are the ones that are readily displaced by water. The water displacement susceptibility was identified and arranged as follows:

Carboxylic acids > Dicarboxylic anhydrides > Sulfoxides > Nitrogen > 2-Quinolone types > Ketones

In 1998, Petersen continued the study by using compound models having functional groups similar to the ones present in bitumen binder and four different types of aggregates. The results confirmed what has been stated in the previous paragraphs. One of the author's conclusions was that the polyfunctionality on the same bitumen molecule, even if the individual functionalities are sensitive to water displacement, may significantly improve resistance of the adsorbed bitumen component to water displacement (Petersen & Plancher, 1998).

II. Modified Bitumen Binder

A conventional bitumen binder is generally altered by using polymer modifiers to improve its performance. Some of the bitumen characteristics that are normally improved by modification include consistency, temperature susceptibility, stiffness and cohesion, flexibility, resilience, adhesion properties and resistance to in-service ageing (Asphalt Academy, 2007).

Yildirim (2007) reported that polymer-modified binders were used successfully at different locations where high stress levels occurred. Among others, he reported on intersections of busy streets, airports, vehicle weigh stations and racetracks. He further stated that the polymers that have mainly been used to modify bitumen are but are not limited to: Styrene-Butadiene-Styrene (SBS); Styrene Butadiene Rubber (SBR), Elvaloy, Rubber, Ethylene Vinyl Acetate (EVA) and Polyethylene (PE).

In South Africa, modified binders are classified in a coded format. The codes are elaborated based on four main aspects as follows (Asphalt Academy, 2007):

- The type of application in which they are intended to be used (Seals (S), Asphalt (A), Crack sealant (C)).
- The type of binder system (if the binder is an emulsion, the letter C will be used. If the binder is a hot applied binder, no letter is used after the letter indicating application).
- The type of modifier (Elastomer (E); Plastomer (P); Rubber (R); Hydrocarbon (H)).
- The level of modification (The higher the number the higher the softening point).

It is well known that the modification of bitumen improves its rheological properties. However, some literature mentioned that modification may negatively affect the adhesion properties, especially in winter periods.

During winter periods, the binder temperature drop rate is very high. This is influenced by different factors such the existing substrate, the ambient air and cover aggregate, which normally have a very low temperature in winter compared to hot bitumen. Furthermore, wind also may contribute. The significant temperature difference between what mentioned above accelerates heat transfer from the binder to the aforementioned cold bodies. This results in a stiff binder in a very short time of binder spray, which may not allow the spread chips to be coated well.

For homogeneous modified binders, the higher the level of modification, the higher the risk of failure due to decreased wetting ability. Moreover, if the stiffness of the binder is high, the contact area will be small, which results in poor bond strength too. (Van Zyl *et al.*, 2012).

Abrahams in his study analysed the adhesion performance of a penetration grade bitumen (70/100) versus a polymer modified bitumen (S-E1) on precoated aggregates. The results of his study showed that the polymer modification does not always improve adhesion. In almost all the cases, the unmodified bitumen (70/100) performed better than the modified bitumen. He mentioned that it is probably due to high viscosity resulting from the binder modification (Abraham & Jenkins, 2012).

Copeland *et al.* (2007) did another study on modified bitumen bond strength. They evaluated the influence of bitumen modification, moisture conditioning and aging on bitumen bond strength. After a series of tests using the Pneumatic Adhesion Tensile Testing instrument (PATTI), they found that modification does not always improve adhesion. Especially after moisture conditioning, the bitumen modification may not increase the resistance of bitumen to moisture damage (stripping). Similarly, long-term aging does not necessarily increase the bitumen bond strength after moisture conditioning (Copeland, Youtcheff & Shenoy, 2007).

However, other researchers, like Khattak, Baladi & Drzal (2007) and Yildirim (2007) found that bitumen modification increases bitumen adhesion properties. Khattak *et al.* (2007) evaluated and compared the bitumen-aggregate adhesion and mechanistic properties of a polymer modified bituminous mixture at low temperatures. In their analysis for adhesion, they used the lap shear test (illustrated in section 2.6.3 of this thesis) and Environmental Scanning Electron Microscope (ESEM). They observed that the lap shear strength was higher for polymer-modified mixtures and increased with increasing polymer content, up to the optimum polymer content and then decreased. Moreover, by using ESEM, he observed that all the failures were adhesive. This implies that the failure in bituminous mixtures at low temperatures may be the result of loss of adhesive strength of the binders (Khattak *et al.*, 2007).

An Engineering brief from 1987 available at the US Federal Aviation Administration Website as cited by Yildirim (2007) discusses the benefits of SBR modified bitumen. It states that SBR improves the properties of asphalt concrete pavements and surfacing seals. Low temperature flexibility is improved, viscosity is increased, elastic recovery is improved and the adhesive and cohesive characteristics of the pavement are improved (Yildirim, 2007).

The literature seems to be contradictory on the contribution of bitumen modification to adhesion properties. This may be caused by the fact that the conditions under which the tests were performed are different. For example once the modified bitumen is applied to aggregate at a low temperature, it

may develop poor adhesion due to its high viscosity at low temperatures. Nevertheless, it does not imply that if you apply it at high temperatures the same observation will be obtained.

2.2.2.3 Cold Applied Binders (Bitumen Emulsions)

I. Introduction

Emulsion is a broad term that is used in different fields. It describes a two-phase system, made of water, oil and one or more chemical additives to assist in the formation, stabilization and modification of emulsion properties. The emulsion may be either an oil-in-water (O/W), water-in-oil (inverted) (W/O) or water-in-oil-in-water (W/O/W) emulsion. The O/W type is the emulsion in which water is the continuous phase and the oil droplets are the dispersed phase. The bitumen emulsion used in road construction industry is normally of the O/W type and consists of 40 to 75% bitumen, 0.1 to 2.5% emulsifier and 25 to 60% water (Read & Whiteoak, 2003; James, 2014).

A bitumen emulsion is manufactured by feeding separately but simultaneously, hot bitumen and an emulsifier solution into a colloidal mill. Figure 2-13 illustrates the bitumen emulsion manufacturing process.

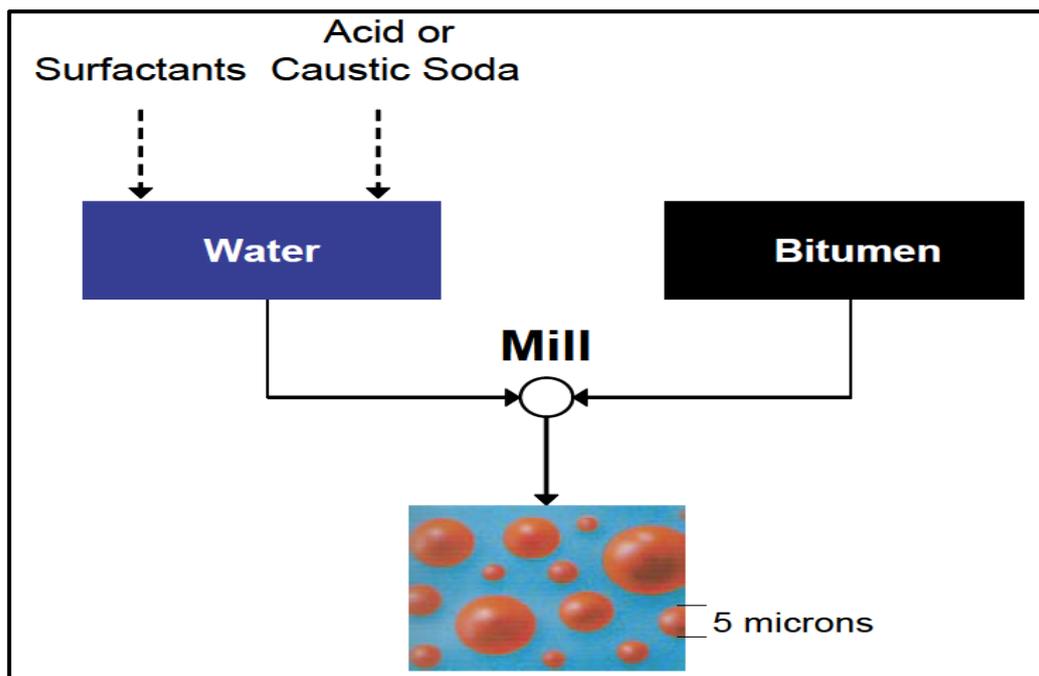


Figure 2-13: Bitumen Emulsion Manufacturing Process (Asphalt Academy, 2009)

The colloidal mill will provide sufficient mechanical energy to break the bitumen into droplets. The emulsifiers that will impart the electrical charges at the surfaces of bitumen droplets will then surround the bitumen droplets. The repulsion of similar charges on the surfaces of the bitumen droplets prevents them to coalesce and they consequently remain dispersed in the water.

II. Classification of bitumen emulsion

Generally, bitumen emulsions are classified according to the type of electrical charges on the bitumen droplets and according to their setting rate. Cationic emulsions have bitumen droplets that carry a positive charge (+). Anionic emulsions have negatively charged droplets (-). Bitumen droplets of non-ionic emulsions do not have any charge and are not generally used in road construction.

Rapid setting (RS) emulsions set quickly when in contact with clean aggregates with a low surface area such as the aggregate used in surfacing seals. Medium setting (MS) emulsions set sufficiently less quickly to the level that they can be mixed with aggregate with a low surface area such as those used in open graded mixes. Slow setting (SS) emulsions will mix with aggregates with a high surface area (James, 2014).

In South Africa, the Technical Recommendation for Highways (TRH7) describes the use of emulsion in the construction and maintenance of roads. In this document, both anionic and cationic emulsions are classified according to the application type into Spray grade, Pre-mix grade, stable-mix grade and special grade. Spray grade is characterised by the rapid breaking of the emulsion on application (RS). It is normally used for seals. Pre-mix grade has sufficient stability to allow mixing with aggregate of 3mm and larger before breaking (MS). Stable-mix grade has sufficient mechanical and chemical stability to allow mixing with stone chippings, natural gravels or soils (SS) (CSRA, 1994).

III. Bitumen Emulsion Properties

The bitumen emulsion properties discussed in this section are those that affect the bond strength of emulsion-aggregate systems in chip seals. The properties examined in the following sections are bitumen emulsion stability, rheological properties (viscosity), breaking and setting and emulsion adhesion properties.

The stability of emulsions during storage, transport and application is important for the performance of the binder. At ambient temperature, the penetration grades normally used in emulsions have a slightly higher density than that of the aqueous phase of the emulsion. Thus, the bitumen particles tend to fall through the aqueous phase, resulting in an upper and lower layer of different bitumen concentration. If this is not well controlled, it may affect the performance of the emulsion (Read & Whiteoak, 2003).

The stability is generally linked to the interfacial properties of the bitumen droplets. Salou *et al* (1998) studied the stability of bitumen emulsions. In this study, the influence of chemical and interfacial properties of bitumen to the stability of the emulsion was analysed. The chemical properties were analysed in terms of asphaltene and resin content. The interfacial properties were studied by measuring the zeta potential of bitumen dispersions in acid water.

The results of the study showed that bitumen with high resins-asphaltenes ratios tend to give a stable dispersions and emulsions. Lower resin-asphaltene ratios tend to give metastable dispersions and unstable emulsions (Salou, Siffert & Jada, 1998).

In practice, the surface dressing emulsions are usually applied by spraying. Thus, their consistencies under spray conditions are of prime importance to ensure appropriate aggregate wetting. The rheological properties of bitumen emulsions were found to be dependent on bitumen content, the particle size and size distribution of the dispersed bitumen droplets, and the condition of the surface of the bitumen particles. The temperature variability has a small influence on bitumen emulsion rheological properties (Lyttleton & Traxler, 1948). This is due to the fact that the temperature variability generally affects bitumen flow properties. However, in emulsion binder the primary component dictating the flow properties of the binder is water instead of bitumen because the bitumen is present in suspended form. Therefore, the influence of temperature variability in normal working conditions is not significant.

Another important property of bitumen emulsions as far as its performance is concerned, is the breaking or setting time. Bitumen emulsions must revert to a continuous bitumen film in order to fulfil their role as an adhesive or binder. In principle, this involves the flocculation and coalescence of the dispersed bitumen droplets and removal of the water. The rate by which this happens depends on the reactivity of the emulsion, the reactivity of the aggregates, mechanical action and environmental conditions such as temperature, humidity, wind speed, etc. (Louw, Spence & Kuun, 2004; James, 2014). Figure 2-14 illustrates the phases of the bitumen emulsion breaking process.

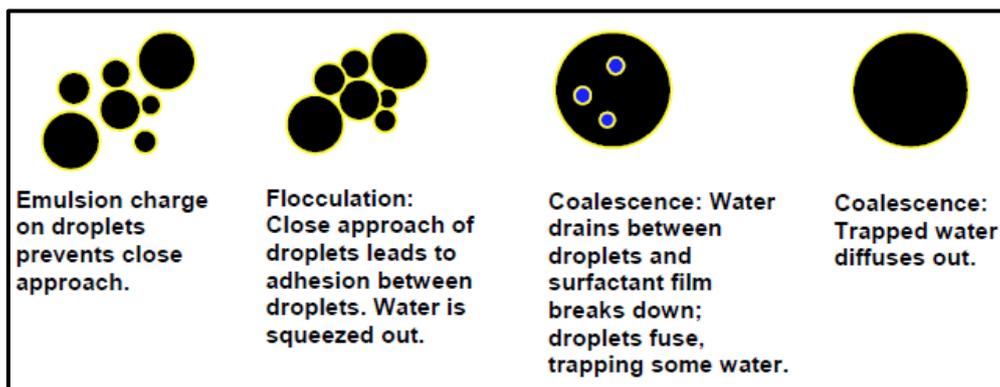


Figure 2-14: Bitumen Emulsion Breaking Process (James, 2006)

In the presence of water, aggregates present a characteristic surface charge, depending on the minerals contained in the aggregate under consideration. Acidic aggregates such as granite and quartzite are known to be negatively charged and basic aggregates such as limestone are positively charged. If the aggregates and bitumen emulsions of opposite charges come into contact, there will be an attraction between oppositely charged particles. This will lead to the destabilization of the surfactant system and

subsequent coagulation of the bitumen particles. This breaking mechanism is absent when aggregate and emulsions of the same charges are used. In this case, coagulation can only take place by evaporation of the water phase.

The last bitumen emulsion property discussed in this section is the adhesion property. As has been reported by Read & Whiteoak (2003), the quality of the bond between the bitumen emulsion and the aggregate depends on the type and amount of emulsifier, the bitumen grade and constitution, the pH of the emulsifier solution, the particle size distribution of the emulsion and the aggregate type. This seems to be in agreement with what has been reported by Bickford (2001), in a simplified visual representation of the reaction of cationic and anionic emulsions on “acid” aggregates. Figure 2-15 illustrates the interaction between bitumen emulsions and acidic aggregates.

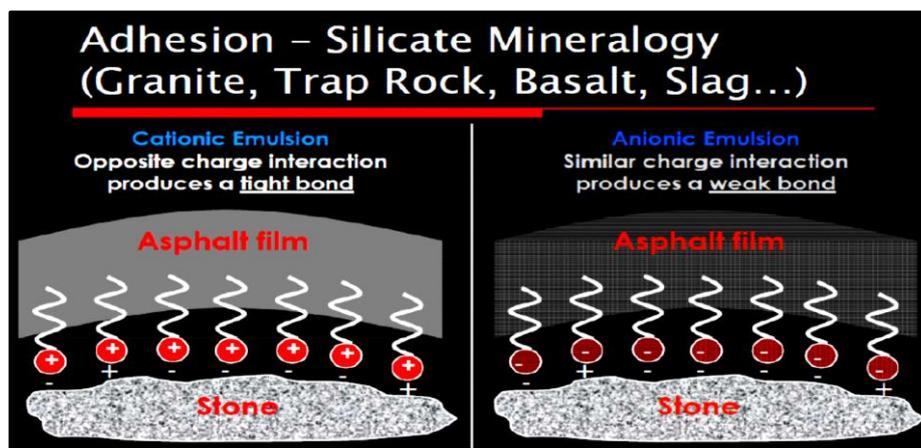


Figure 2-15: Interaction between bitumen emulsions and acidic aggregate (Bickford, 2001).

It is very clear that cationic emulsion will form a very strong tight bond with acidic aggregates due to a strong electrostatic attraction of the oppositely charged particles. The same author has also demonstrated the interaction between basic aggregates like limestone and emulsions. Figure 2-16 illustrates that interaction.

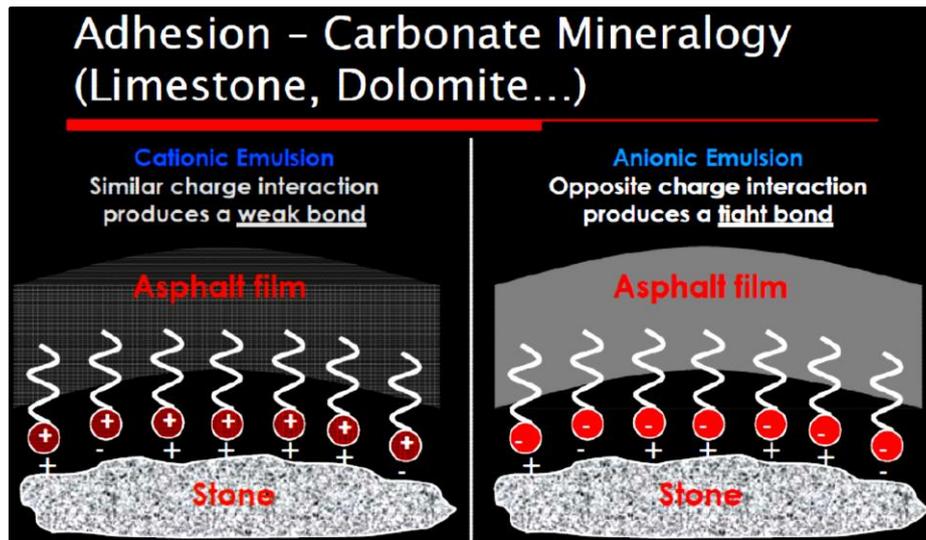


Figure 2-16: Interaction between bitumen emulsions and basic aggregates (Bickford, 2001).

2.2.3 Precoating Fluid

2.2.3.1 Introduction

The presence of dust or water on the aggregate surface may prevent intimate contact between the bituminous binder and applied aggregates. This prevents good adhesion bond development and may result in premature failure of the surfacing seal. The influence of dust on the performance of surfacing seal can be reduced either by washing and drying the cover aggregates by mechanical means or by precoating the cover aggregates prior to spreading. Around 1975, the Pennsylvania Department of Transportation's (PennDOT) specifications allowed the use of cover aggregate with dust up to 2%. Later on, it was realized that even 2% of dust is too excessive for high volume roads and specifications for precoated aggregates were developed to prevent the dust effect (Kandhal & Motter, 1991).

This shows that some specifications allow the presence of dust up to a certain defined percentage, and above that, the aggregate sample fails to meet the requirements. This is in agreement with what is specified in Sabita's Manual 26 (interim guidelines for primes and stone precoating fluids). In this guideline it is specified that if the aggregate sample contains more than 2% of dust, aggregate precoating is essential (Sabita, 2006).

Aggregate precoating is the pre-treatment of aggregates by using fluid materials compatible with bitumen. Generally, it is a petroleum product. Precoating is done to avoid the effect of dust, to improve the initial wetting of aggregate by the bitumen, and to facilitate the use of harder bitumen that will set very quickly and allow early aggregate retention development.

Thus, it improves the adhesion bond between the aggregate and the binder (APRG, 1996). In addition to this, the precoated aggregate results in a surface with a uniform black appearance which forms a sharp contrast with road markings, which makes night driving considerably safer.

Kandhal and Motter (1991) reported that the seal cover aggregate can be precoated with either a bituminous material or a kerosene film prior to spreading. In South Africa, the tar based precoat was replaced by bitumen based precoats due to high health risks associated with tar precoats, such as (Louw, 2004):

- ✓ Irritation to the skin and eyes and sometimes also respiratory irritation may occur
- ✓ The tar precoat has a pungent odour which can raise complaints from residents if the construction is done in residential areas.
- ✓ The risk of skin cancer with a prolonged contact with coal tar pitch.
- ✓ Etc.

The recently released South African Pavement Engineering Manual explains the precoating fluid as follows: “*Precoating fluids consist of low viscosity bitumen based products containing petroleum cutters and a chemical adhesion agent. Their purpose is to precoat surfacing aggregates to improve the adhesion of the aggregate to the bituminous binder*” (SANRAL, 2013). This alludes to the fact that precoating fluids in South Africa are bitumen-based products. The following sections describe the influence of precoats on adhesion of binder-aggregate systems, the application rate and precoating procedures of bitumen based precoats.

2.2.3.2 Influence of Precoating on Bitumen-aggregate adhesion bond; Precoating Application Rate and Precoating Procedures.

Researchers have reported an improvement in the adhesion of aggregate-binder combinations due to aggregate precoating. Parr (1955) as cited by Karasahin *et al* (2011) as well as by Kandhal and Motter (1991) reported that a road of 55km (34miles) long, built with about 1% (precoat equivalent to 1% of the aggregate mass) precoated aggregates in Michigan served for 17 years without any maintenance (Kandhal & Motter, 1991; Karasahin *et al.*, 2011).

Kandhal and Motter (1991) found that the use of precoated aggregate at the rate of at least 90% reduced early chip loss by at least 80% compared to non-precoated aggregates. Karasahin *et al.* (2011) evaluated the performance of pre-coated aggregates with bitumen by using the Vialit Adhesion Test (which is described in section 2.6.2). In this study, four different gradations of aggregates were used (three of basalts and one of limestone). The rate of precoating used was 0%, 0.5%, 1.0%, 1.5% and 2.0% and they reported that precoating reduced the loss of aggregate by 26.9%, 61.2%, 42.0% and 58.8% respectively.

The quality of precoated aggregates depends on the precoating uniformity in the stockpile. The precoat application rate is specified either in terms of % of precoats to be used relative to aggregate mass, or in terms of volume of precoating fluid relative to the volume of aggregates or in terms of % precoated aggregates, obtained at the end of the precoating activity. Generally, the bitumen based precoating products have a higher application rate than tar-based products. In South Africa it is generally recommended to precoat a small sample of the job aggregates with varying quantities of precoats and by visual assessment. The quantity that renders the ideal coating is thus determined (Sabita, 2006). A typical precoating application rate was provided in the guideline as shown in Table 2-1.

Table 2-1: Typical Application rates for bitumen based precoating fluid (Sabita, 2006)

Stone Size (mm)	6.7	9.5	13.2	19
Application rate (Litre/m ³)	13-18	12-17	11-16	10-15

The precoating of the aggregate may either be done in a drum plant that is usually situated at the quarry or on the site using a front-end loader. In the latter case, the aggregates are loaded in the bucket of a front-end loader and the precoating fluid is sprayed evenly over the aggregates by using a watering can. The mixture is then placed on the firm, prepared area and built in stockpiles of 15 to 20m³. The stockpile is then turned over with the front-end loader until the aggregate is uniformly coated. The precoated aggregates are allowed to cure for at least 3 or 4 days (Louw, 2004; Sabita, 2006).

As the normally used precoating fluids contain cutters, early bond development between the precoated aggregates and binder will highly depend on the curing of the precoated aggregates. The curing of the precoated aggregates may depend on curing temperature, humidity and time. In the literature, specifications were found on the curing of precoated aggregates based on curing time only. However, it is believed that the same curing time in summer and winter periods would not have the same effect.

To conclude, this part of the literature survey showed the material properties influencing adhesion between aggregate and the binder. The following section discusses the adhesion principles and mechanisms.

2.3 ADHESION BOND THEORIES AND MECHANISMS

2.3.1 Introduction

The understanding of bitumen-aggregate bond strength involves a consideration of two complex interfacial phenomena namely adhesive and cohesive bond strength. The bituminous binder cohesive bond strength is an inherent material property. Thus it is mainly dependant on the physico-chemical properties of the bituminous binder and due its visco-elastic behaviour it also depends on environmental conditions. However, the adhesive strength concept is far more complicated because it involves two different materials with huge variability in their natures and their interactions are not fully understood.

Once the bitumen-aggregate bond is subjected to a destructive force, whichever is exceeded first will induce the bond failure. Thus the failure may be either adhesive or cohesive according to the nature of rupture. Therefore, both phenomena need to be understood to ensure successful bond strength development. The following sections are devoted to the adhesion phenomenon through the description of forces responsible for adhesion, adhesion development theories and failure mechanisms.

2.3.2 Fundamental Forces of Adhesion

As mentioned in the background of the first chapter of this thesis the emphasis in this study is placed on practical adhesion. This involves the fundamental forces of adhesion as well as the physical and mechanical response of the adherent materials. This section intends to establish a fundamental understanding of interaction forces ultimately responsible for adhesion.

2.3.2.1 Interactions through Electrostatic Forces

Electrostatic forces represent the repulsive or attractive forces between two oppositely charged chemical ions. These forces play a primary role in the formation of ionic bonds. Coulomb's law explains the ionic bond formation. According to this law, an inherent attraction force exists between two oppositely charged ions separated by a distance r as shown in Figure 2-17. This attraction results in a strong bond between ions known as ionic bond. In this process, each ion pair gains electrostatic stabilization energy (Companion, 1979) as cited by (Hefer, Bhasin & Little, 2006).

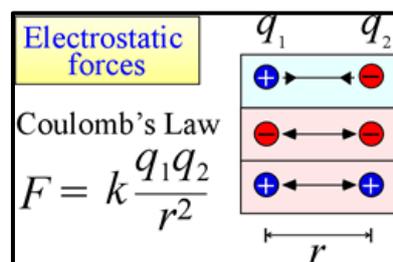


Figure 2-17: Coulomb's Law

2.3.2.2 Interactions through Electron Pair Sharing

The chemical bonding by electron pair sharing takes place by the rearrangement of bonding electrons within the interacting entities to establish balanced electron distributions between interacting entities. This is governed by the electro-negativity of atoms in the structure. Two broad categories of chemical bonding by electron pair sharing exist. In the first category, each atom contributes one of the shared electrons to form a new molecule. The bond resulting from this interaction is known as “Covalent bond”. Covalent bonds have a directional character and interaction depends strongly on the exact position and orientation of the adsorbate with respect to the surface. Covalent and ionic bonds are known to be the strongest and are mostly considered as primary bonds (Good, 1966) as cited by (Hefer *et al.*, 2006).

In the second category, the shared pair is donated by one of the interacting atoms and the other act as an acceptor. In this case, one atom must be in deficit of electrons and this atom is known as the acceptor. The other one must have one unshared pair of electrons and is called the donor. The interaction is known as donor-acceptor interactions and the resulting bond is called “Coordination bond”. Unlike ionic and covalent bonds that can be formed between two atoms, a coordination bond is formed between two molecules or between two ions. Coordination bonds are similar to covalent bonds with partial ionic character.

2.3.2.3 Interactions through Van der Waals Forces

In 1783, J.D. Van der Waals introduced a term that allows explaining of intermolecular forces. He is also the first one to develop an equation of state that describes the force responsible for the deviation of the behaviour of gas from that of an ideal gas. Later on, Keesom (dipole-dipole interactions), Debye (dipole-induced dipole interactions) and London (induced dipole-induced dipole interactions) made great contributions to the understanding of those forces. The Van der Waals force is the sum of these three interactions (Butt *et al.*, 2003) as cited by (Hefer *et al.*, 2006). These forces are small when compared to ionic or covalent bonding forces. However, because these forces are additives, their contribution may be of considerable importance in interactions where large organic molecules are involved.

2.3.3 Theories of Bitumen-Aggregate Adhesion Mechanism

In an attempt to explain the adhesion mechanisms of bitumen-aggregate systems, four main broad theories were developed. These include the mechanical theory developed by Knight (1938), Lee and Nicholas (1954) and Rice (1958); the chemical reaction theory developed by Rice (1958) and Maupin (1982); the molecular orientation theory developed by McBain and Lee (1932), Mack (1957); and the interfacial energy (surface energy or thermodynamic) theory developed by Thelen (1958), Ishai and Craus (1977).

Various researches conclude that due to the complexity of adhesion phenomenon; none of these theories can explain at fully the bitumen-aggregate adhesion mechanism. Rather, it can be explained by the combination of principles developed in these theories (Hicks, 1991; Das, 2004; Hanz, Bahia, Kanitpong & Wen, 2007). The following sections summarise the basic principles that govern bitumen-aggregate adhesion.

2.3.3.1 Mechanical Theory

This theory explains the adhesion mechanism based on several properties of aggregate surfaces such as surface texture, porosity (absorption), surface coatings, surface area and particle size. It explains that once bitumen binder comes into contact with the aggregate surface, the binder is forced into the pores and irregularities of the aggregate surface. As the binder hardens, it provides a strong mechanical interlock with the aggregate surface. Following this logic, it can be argued that rough and irregular surfaced aggregates develop a strong adhesion bond (Hicks, 1991; Hanz *et al.*, 2007). However, it should be noted that this has not always been the case as it was explained in section 2.2.1 of this literature review.

2.3.3.2 Chemical Reaction Theory

According to this theory, once bitumen binder is applied to the aggregate surface, it wets the surface. In the process, the bitumen is adsorbed at the surface and will chemically react with the aggregate. It is suggested that the acidic components of bitumen react with the basic components of the aggregate surface and form insoluble salts. Based on this theory, it is argued that the chemical reaction between most of the bitumen binder and acidic aggregates (e.g. granite, quartz, etc.), is not as strong as the one between the bitumen and the basic aggregates (dolomite, limestone, etc.) (Hicks, 1991; Hanz *et al.*, 2007).

2.3.3.3 Molecular Orientation Theory

This theory states that, once bitumen binder gets into contact with aggregate, the bitumen molecules orient themselves towards the aggregate surface ions to satisfy the energy demands of the aggregate surface (Hicks, 1991). These ions on the aggregate surface may be linked to the aggregate active sites reported by Jamieson *et al.* (1995) and Petersen and Plancher (1998) as has been explained in Section 2.2.1 of this literature survey.

It is common knowledge that water molecules are dipolar and even more polar than bitumen molecules. Thus, water molecules can better satisfy the energy demands of the aggregate surface than bitumen molecules. This can contribute to the explanation of the phenomenon of adhesion loss due to moisture.

2.3.3.4 Thermodynamic Theory

This theory is based on the concept that the bitumen binder adheres to the aggregate surface due to established intermolecular forces at the interface provided that intimate contact is achieved. The magnitude of the established intermolecular forces can generally be related to surface free energies of the materials involved in the adhesive bond. This theory is the most widely used approach in adhesion science as shown by most references on this subject (Kiggundu & Roberts, 1988; Hefer, 2004; Hefer *et al.*, 2006).

The level of contact between liquid and solid materials is generally described in terms of wetting of the surface, spreading of the liquid or the contact angle between them. The shape of a liquid drop on a surface is usually described in terms of the contact angle between them. This provides information on the intermolecular forces of the individual materials involved as well as the interfacial forces between them. The wetting of a surface by a liquid (or spreading of the liquid over the surface) is the process where the binder comes into intimate molecular contact with the substrate surface and establishes fundamental forces of adhesion (Hefer, 2004). Figure 2-18 below illustrates the terms above schematically.

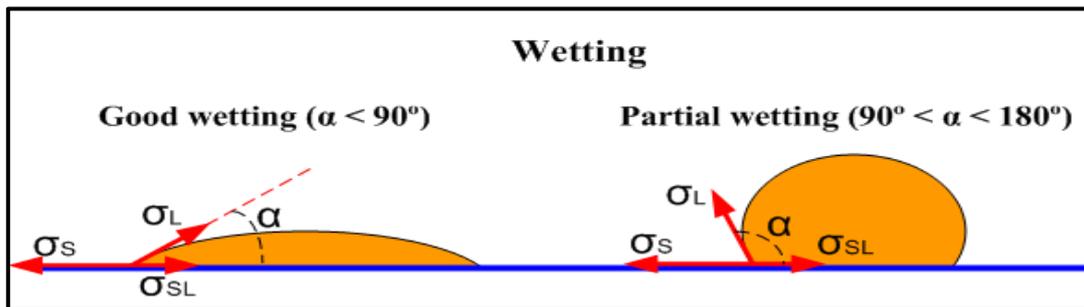


Figure 2-18: Contact Angle, Wetting or Spreading Concept Illustration.

With:

σ_S : Surface Energy of the solid substrate

σ_L : Surface Energy of the liquid (Binder)

σ_{SL} : Interfacial energy between substrate and binder

α : Contact Angle

If the liquid in figure 2-18 is water, the left side shows a water loving (hydrophilic) material and the right side shows an hydrophobic material.

Figure 2-18 also represents the contact angle testing. This concept was used by Young in 1805, to establish an equation of surface tension based on the contact angle formed when a drop of liquid is placed on a perfectly smooth, rigid solid. His equation is based on the triple line in equilibrium (tension in force per unit length). Young's equation is shown below:

$$\sigma_S = \sigma_{SL} + \sigma_L * \cos\alpha$$

Young's equation was combined with an equation developed by Dupré, in 1867 and the resultant equation known as Young-Dupré equation was developed.

$$W_a^{SL} = \sigma_L * (1 + \cos\alpha)$$

With:

W_a^{SL} : Work of Adhesion. The other variables have a similar meaning as previously described.

This equation is the starting point for any method which assesses the adhesion based on contact angle or surface free energy concepts.

Hefer (2004) showed that the thermodynamic theory was used extensively by researchers to relate practical adhesion and fundamental forces of adhesion. However, Merrill *et al* (1991) used peel test experiments for practical adhesion analysis. He compared the practical adhesion with the fundamental work of adhesion obtained based on thermodynamic theory. And the results proved the practical adhesion to be much higher than the fundamental work of adhesion (Merrill *et al.*, 1991). This is due to the fact that because materials are not completely brittle, especially visco-elastic materials such as bitumen, energy dissipation can result from molecular chain stretching, plastic deformation, etc. As has been aforementioned none of these adhesion theories is able to explain fully, the adhesion mechanism and energy used to break a given bond.

2.3.4 Bitumen-Aggregate Adhesion Failure Mechanism

Understanding of both adhesion development and failure mechanisms is important as far as the performance of a surfacing seal is concerned. In the past, researches have identified a number of adhesion failure mechanisms. Most of the researches were conducted on moisture damage of asphalt mixes but some of the theories that have been developed are applicable to adhesion failures in surfacing seals. The adhesion failure mechanisms presented here are those the author finds to be applicable to surfacing seals.

2.3.4.1 Adhesion Failure by Detachment

This type of failure occurs when there is a layer of water or dust between the aggregate surface and the bitumen binder without any clear break in the bitumen film. The presence of water or dust deprives intimate contact between the bitumen and aggregate and thus no adhesion bond will develop between them. The lack of adhesion allows the bitumen binder to be peeled off easily from the aggregate surface under traffic loading. This failure is explained easily by the interfacial energy theory and Van der Waals attractions (Read & Whiteoak, 2003; Hanz *et al.*, 2007). This type of failure can be minimised for winter seals by using cleaned and dried aggregate or precoated aggregates.

2.3.4.2 Adhesion Failure by Displacement

Displacement failure is explained by means of the thermodynamic theory. It occurs when water is introduced at the aggregate-bitumen interface through a break in the bitumen film. The water displaces and replaces the bitumen on the aggregate surface. The system allows the water to replace the bitumen on the aggregate surface in order to establish an equilibrium state of lower surface energy. This can be minimised by using the appropriate binder application rate, to ensure the achievement of the appropriate binder film thickness. For seals this type of failure will be more probable than for asphalt. This is because the aggregates in a seal are not fully coated by the binder. The water can therefore access the interface without the bitumen film breaking and replace it due to the equilibrium law (Das, 2004; Hanz *et al.*, 2007).

2.3.4.3 Blistering and Pitting

An increase in the temperature of a surfacing course results in a reduction of bitumen viscosity. If this is associated with a recent rainfall, the bitumen may creep up the edges of water droplets to form a blister. If the temperature increases again, the blister will expand and burst, leaving a pit which may allow water to access the aggregate surface (Read & Whiteoak, 2003).

2.3.4.4 Adhesion Failure by Chemical Disbonding

This type of failure occurs once water diffuses through the bitumen film. It leads to the formation of double layers of water at the aggregate surface. Depending on the type of aggregate and the pH of water, the presence of water may cause the aggregate surface to be negatively charged against a slightly negatively charged bitumen. This results in two negatively charged surfaces coming into contact, repelling each other. As more water is attracted to the aggregate surface, it may result in disbonding (Read & Whiteoak, 2003).

In the previous sections, an extensively systematic review of the literature pertaining to material properties influencing adhesion was presented. Moreover, the interaction forces responsible for adhesion, adhesion development as well failure mechanisms were reviewed. These literature components provide a general insight in the adhesion performance of surfacing seals. The particularity of the winter seals is generally based on environmental conditions which may work against bond development. Thus it is important to characterise the winter period in the region in order to elucidate if there are any associated parameters which may work against adhesion bond development or which may favour adhesion failure. Therefore, the following sections deal with a brief characterisation of the winter period in South Africa.

2.4 SOUTH AFRICAN CLIMATIC CONDITIONS

South Africa is located in the southern hemisphere, between 22°S and 34°S latitude, thus it is known to be in a subtropical zone. The climatic characteristics are moderated by both the presence of oceans on three sides of the country and the presence of a high-elevated interior plateau.

The climatic conditions vary considerably throughout the country. They range from Mediterranean in the Western Cape to temperate in the interior plateau, and subtropical in the northeast. A small area in the northwest has a desert climate. Temperatures are influenced by variations in elevation, terrain and ocean currents more than latitude. The country has four seasons throughout the year, namely summer, autumn (fall), winter and spring.

Generally, except for the Western Cape that has a wet winter, the country experiences cold and dry winters. The winter in the Highveld of the interior plateau is characterised by dry, sunny days and cold nights; sometimes with heavy frosts. The hot and humid eastern coastal region (Kwazulu-Natal) as well as the Lowveld region has winter weather with dry, sunny and warmish days with no rain. However, the temperature in these regions is higher compared to the interior plateau region.

The average annual air temperatures vary throughout the country. They range from less than 13° C in the interior mountainous areas to 17° C in the broader central and south coastal areas and to 22° C in the eastern, northern and western parts of the country. Maximum air temperatures exceeding 35° C and a temperature exceeding 40° C in the northern and eastern parts of the country is not strange. The minimum air temperatures that are generally recorded in the winter period are found in the Highveld and mountainous regions and may be as low as -8° C. The minimum temperatures mainly occur during June and July (Van Zyl *et al.*, 2012). Figure 1-1 shows that the coastal areas of the country are warmer in winter than the inland areas. This is due to the high altitude of the inland areas compared to the coastal areas. There is however, a striking contrast between temperatures on the eastern and western coastal areas of the country.

This is largely in response to the warm Agulhas Ocean current, which sweeps southward along the Indian Ocean coastline in the east of the country, and the cold Benguela current, which sweeps northward along the Atlantic Ocean coastline in the west of the country.

Pavement temperature is always higher than the air temperature. This is due to the heat absorption capacity of the pavement materials. For thick wearing courses (such as HMA), the maximum pavement temperature is normally recorded at 20 mm below the surface. It was found to be approximately 18° C higher than the air temperature. The minimum pavement temperature is taken at the pavement surface and is approximately 8° C higher than the air temperature (Bahia, 2011) as cited by Kashaya (2013). For thin wearing courses (such as surfacing seals), both the maximum and minimum temperatures are measured at the surface. Both the air and the pavement temperatures influence the adhesion bond development, especially for winter surfacing seals. This is due to the fact that they contribute to the binder temperature, which in turn affects the binder viscosity during aggregate spreading.

Similar to the temperature distribution, the rainfall patterns also vary throughout the country. The average annual rainfall approximates 450 mm and only less than 35% of the country receives more than 500mm per year (South Africa Government Online, 2014).

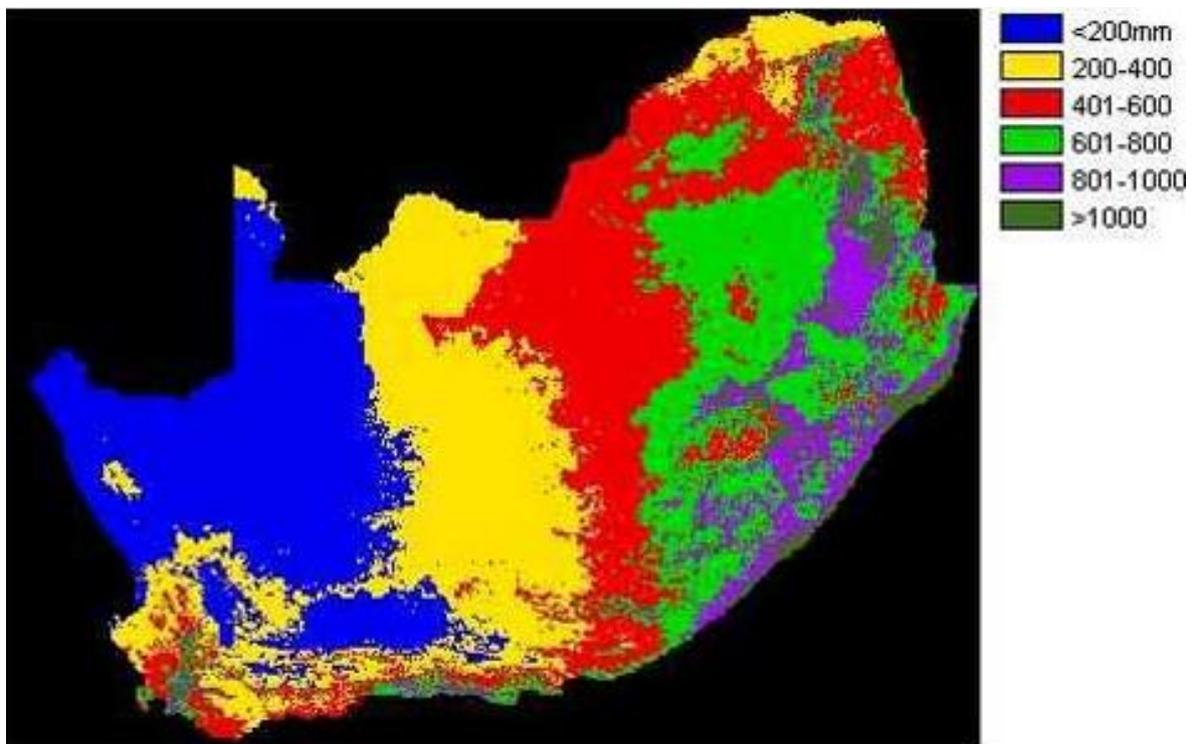


Figure 2-19: Average annual rainfall distribution in South Africa (Van Zyl *et al.*, 2012)

Be that as it may, South Africa may be considered a semi-arid region. However, it should be reminded that based on the surface energy theory, 450 mm of rainfall per annum could be detrimental enough to prevent the development of an appropriate adhesion bond.

SUMMARY ON BITUMEN-AGGREGATE SYSTEMS ADHESION BOND DEVELOPMENT

The knowledge gained from the comprehensive literature review provides an insight on how various material properties influence adhesion bond in seals. As regards to aggregate, the surface chemical and physical characteristics (such roughness, porosity, dust...) were found to influence binder aggregate interactions. However, the aggregate mineralogical composition appeared to be the most influential factor as it is the basis for the aggregate surface characteristics mentioned above as well as for the surface free energy development.

As regards to the binder, the literature found the type and the viscosity of the binder at the time when the aggregate and the binder come into contact to influence significantly the adhesion bond development. In winter the latter may be the most problematic property due to low temperature, thus an investigation on adhesion bond development for different types of the binder under low temperature is needed.

The review of the literature found that four theories are mainly used globally to explain the adhesion bond development mechanisms. These are mechanical, chemical reaction, molecular orientation and thermodynamic (surface energy) theories. The mostly used in adhesion science is thermodynamic theory.

As regards to winter periods in South Africa, it was realised that the country may be considered in two main parts. The western Cape, which is characterised by wet winter (a combination of low temperature and rain) other parts of the country characterised by dry winter (low temperature not combined with rain). It is therefore, important to carry-out an investigation on adhesion bond development at low temperatures under dry and wet conditions.

This literature survey laid the foundation for understanding the theory of adhesion bond development and the contributing material factors. Literature on the South African climatic conditions helped to identify the environmental factors that could have a significant influence on bond development for winter surfacing seals. The following section discusses the design, construction and failure mechanism of surfacing seals in general. An insight in the failure mechanism of winter surfacing seals is also provided.

PART B: ROAD SURFACING SEALS

2.5 SURFACING SEALS

2.5.1 Introduction

When designing a flexible pavement, engineers have a choice of two types of wearing course; either an asphalt concrete wearing course or surfacing seal wearing course. Both these wearing courses can appear in different forms depending on the purpose and choice.

Asphalt concrete is a mixture of graded aggregate, bitumen binder and filler material that is typically produced in a designated plant using a well-defined mix design procedure. It is generally hot, warm or cold at the time of application on the road. It is applied using an asphalt paver after which it is compacted to a specified density. It is known to be the most durable bituminous surfacing but also the most expensive. It is generally preferred where surfacing seals do not meet the requirements such as on urban streets, heavily trafficked rural roads and at intersections. This type of bituminous surfacing is not discussed further in this report; the emphasis is rather placed on surfacing seals and their behaviour.

Due to international variations, different terminology is used to describe the seal in different regions of the world. It is commonly called “Chip Seal” in the USA and New Zealand, “Sprayed Seal” in Australia, “Surface Dressing” in the United Kingdom and “Surfacing Seals” in South Africa. In this document, the South African terminology is used but in some cases, any one of the above-mentioned terms may be used.

A surfacing seal is defined by the Technical Recommendations for Highways 3 (TRH3) as discussed in section 1.1. In many countries with a considerable size and a limited number of inhabitants per km², this type of road wearing course is predominant. The main reason for its continuous predominance is the corresponding competitive initial cost and ease of construction relative to other wearing courses (Huurman, Scarpas, Kasbergen & Milne, 2003; SANRAL, 2007; Holtrop, 2008).

The surfacing seal in its simplest form is made of one layer of bitumen binder, covered with one layer of aggregate. This makes it very thin to the level that it is not expected to give any significant contribution to the load bearing capacity of the pavement. However, as the surfacing seal is placed over the base, which is one of the pavement’s structural value determinants, the surfacing seal contributes to the pavement’s structural integrity. From the structural point of view, surfacing seals protect the base from moisture and from direct contact with tyres that could undermine the base’s structural capacity. From the functional point of view, the surfacing seals provide a skid resistant and all weather dust-free roads surfacing (Huurman *et al.*, 2003; SANRAL, 2007; Milne & Visser, 2011).

Generally, surfacing seals are used for both new road construction and road preventive maintenance for example patching, crack sealing, edge breaking repair, texture treatment and correction of roughness (Van Zyl *et al.*, 2012). However, even though surfacing seal is considered as a technique of preventing further deterioration while awaiting rehabilitation funds, it should not be used on badly cracked or weathered road surfaces. Such a scenario would be more expensive in the long term as the surfacing seals do not provide additional structural capacity to the road pavement (Gransberg & James, 2005). In practice, the surfacing seals appear in different forms. The section below illustrates various forms of surfacing seals in the field.

2.5.2 Types of Surfacing Seals

In South Africa, the Technical Recommendation for Highways 3 (TRH3) currently describes and dictates the design and construction practices for surfacing seals. This document states that different types of surfacing seals exist worldwide. It points out that the types of surfacing seals commonly used in South Africa are single seals, double seals, Cape seals, slurry seals and sand seals. Figure 2-20 illustrates these types of surfacing seals graphically. TRH 3 discusses other types of surfacing seals such as inverted double seals, chocked seals, split seals, geotextile seals and graded aggregate seals. However, these types of seals are less often used (SANRAL, 2007).

The designers of surfacing seals use different methods as discussed subsequently in the section on leading surfacing seals design methods. However, it appears that all the methods available in the literature relate in one way or another to Hanson's original principles. The subsequent section details the original Hanson's surfacing seal design concept as well as the enhancement made in the TRH3 method.

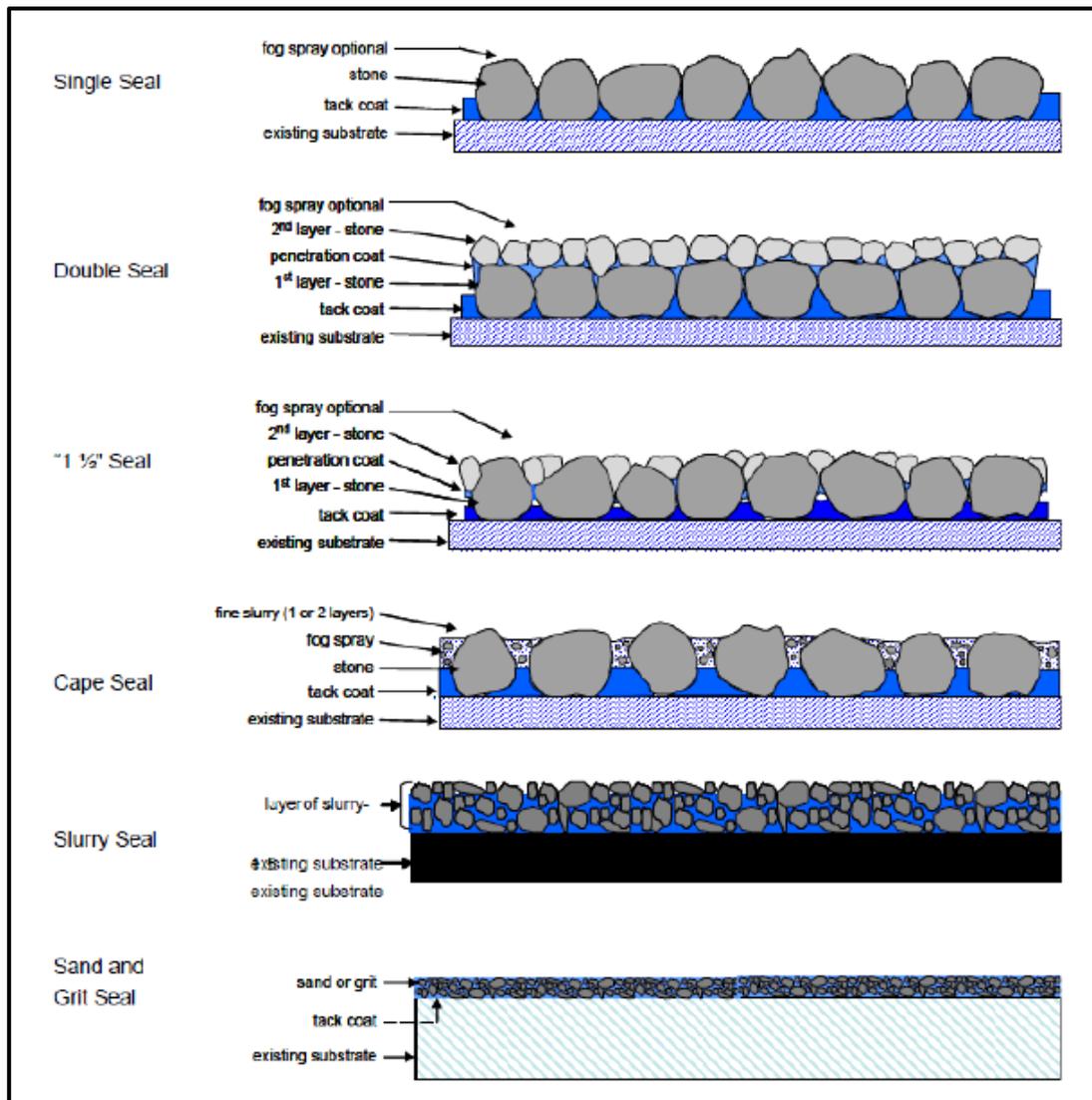


Figure 2-20: Schematic illustration of surfacing seals types (SANRAL, 2007)

2.5.3 Surfacing Seals Design Concept

Available literature shows that despite the application of surfacing seals as discussed in the previous paragraphs, surfacing seal practices have long been based on experience. Nevertheless, nowadays various design methods based on experience and scientific understanding of the performance of surfacing seals are available. The records show that, around the year 1935, the New Zealander F.M. Hanson developed the first scientific approach for the design and construction of surfacing seals. The developed approach was primarily for bitumen binder and particularly for cutback bitumen binder. The basis of his approach was the average least dimension (ALD) of the aggregate spread on the road surface (Gransberg & James, 2005).

The following three principles were the basis for his concept as cited in the paper “history of chip-sealing in New Zealand-Hanson to P/17” (Holtrop, 2008):

Note: The word “voids” describes total voids within aggregate layer, for which part is filled by the binder and another serves to define the texture depth (macro texture).

i. When sufficient chips are placed on a seal binder to ultimately bed into a single layer in shoulder to shoulder contact, the percentage of voids in the initially laid loose state is approximately 50%. This is reduced to about 30% by construction rolling, and to 20% by traffic compaction.

ii. The amount of binder required bears a definite relationship to the volume of voids in the cover stone aggregate; the quantity should be such that between 65 and 70% of the voids in the finally compacted layer of sealing chips are filled with binder.

iii. The average depth of the layer of stone chips forming the cover coat, after construction and traffic compaction is approximately equal to the average least dimension (ALD) of the chips used.”

Figure 2-21 illustrates graphically the above-mentioned principles and the Hanson’s concept in general.

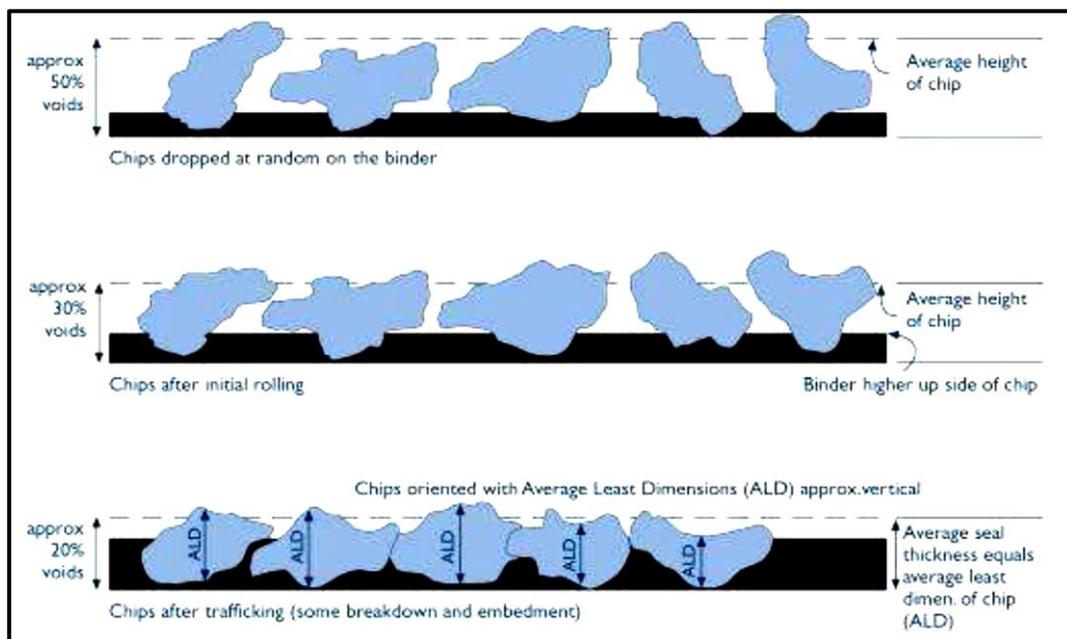


Figure 2-21: Graphical representation of Hanson’s Surfacing Seals Principles (Holtrop, 2008)

This approach remained unchallenged for the next fifty years. Due to the combined application as a wearing course and preventive maintenance technique, surfacing seals increasingly attracted the attention of many researchers. Various countries including South Africa adopted and improved Hanson’s concept.

The current South African surfacing seals design method (TRH3), includes the following enhancements on Hanson's concept (Milne & Jenkins, 2005):

- i. The minimum amount of void to be filled with residual bitumen binder to retain aggregate is 42% for single seals and 55% for double seals (if no embedment is to be accommodated).
- ii. The surfacing seal's void loss under traffic due to wear of the aggregate depends on the aggregate's hardness.
- iii. To ensure adequate skid resistance, the minimum texture depth should be 0.64-0.7mm.
- iv. Embedment under construction represents 50% of the total lifetime embedment.
- v. Adjustment related to traffic and the use of modified binders.

Figure 2-21 illustrates how much the performance of surfacing seals depends on the effectiveness of the binder-aggregate adhesion bond strength. Unlike asphalt concrete in which aggregate particles are fully coated and protected by the binder, the surfacing seals aggregate stability relies on the bonding established by a partial coating of the aggregate surface by the binder. Adequate binder-aggregate adhesion bond development results in a better surfacing seal, thus minimising the risk of ravelling. Moreover, better binder-aggregate interaction could enable designers to optimize the use of materials and reduce the cost and likelihood of bleeding (Senadheera, 2006). Even though material properties and interaction influence the surfacing seals performance through bond development, there are some other important contributing factors as discussed below.

2.5.4 Factors Influencing the Performance of Surfacing Seals

Various factors and their combinations affect the performance of surfacing seals. This section discusses the most influential factors to the short and long-term performance of surfacing seals. The following paragraph presents the dominant factors in groups based on their similarities.

- i. Existing Pavement Condition

The surfacing seal is either placed on compacted gravel or a crushed stone base material as the first seal or over an existing road surface as a reseal. The road base or the existing road surface influences to some extent the performance of surfacing seals. Poor resistance of the existing substrate to aggregate penetration or embedment of surfacing aggregates result in reduction of voids in the seals. This may lead to a fatty or bleeding surface, which in turn will induce skidding problems (SANRAL, 2007).

In a study on bitumen rubber seal behaviour, one of the critical parameters identified was the road surface. The latter provides the support for the seal stone, and in effect the transfer of the applied load through the cover stone to the pavement (Milne & Visser, 2011). Therefore, the surfacing seal's

underlying surface influences the identification of the appropriate seal type, the grade and quantity of the binder to be applied as well as the size of the cover stone and the required pre-treatment.

Furthermore, the flexibility and cracking in the pavement structure also need consideration in the design process. High deflection may result in fatigue failure of the pavement layers and the surfacing. The cracked pavement layers in combination with traffic load repetitions induce reflective cracks in the surfacing seal (SANRAL, 2007).

ii. Traffic and Road Geometry

Some traffic parameters such as traffic volume, axle load and tyre pressure significantly affect the cover stone embedment and consequently the skid resistance performance of surfacing seals. The traffic volume in terms of vehicles per day affects embedment, wearing and polishing of the stone. It reduces the voids in the surfacing seal's aggregate matrix. Typically in the design, the higher the traffic volume, the lower the binder application rate will be. Failure to recognise this will induce skid resistance problems. However, there is also a belief that a surfacing seal requires a minimum number of vehicles per day to keep the binder flexible and elastic. TRH3 recommends approximately 50 vehicles per day for conventional binders. The kneading action of traffic continuously brings fresh bitumen to the surface of the bitumen layer and the exposed and oxidised bitumen inside. This retards the aging process of the bitumen layer and improves flexibility.

Vehicle types and characteristics, speed, traffic distribution and occurrence are other traffic parameters identified to influence the performance of surfacing seals. Slow vehicles (typically at a speed less than 40km/h) cause the surfacing seals to experience longer loading time, and higher horizontal stresses induced as a result of traction, particularly due to the braking or accelerating of heavy vehicles. Together with fuel and oil spillages associated with trucks and slower moving traffic, this may cause flushing and deformation. Traffic occurrence during the surfacing seals service life for example opening the road section to traffic directly after construction, prior to the development of appropriate bitumen-aggregate bond strength, accelerate ravelling (Greyling, 2012; Kashaya, 2013).

The gradient, curves, intersections and road width are some of the road geometry parameters identified which influence the performance of surfacing seals. These geometry parameters cause the surfacing seals to be exposed to high horizontal stresses and longer loading time resulting in ravelling, bleeding and loss of skid resistance (SANRAL, 2007).

iii. Climatic and Environmental Conditions

Several factors related to the climate and environmental conditions can adversely affect the performance of surfacing seals. Frequently reported influential factors include but are not limited to temperature, moisture, ultra-violet radiation and humidity.

Milne and Visser (2011) stated that temperature significantly affects the characteristics of the binder.

TRH3 affirms that hot weather reduces viscosity and cohesion in binders, cold weather makes binder hard and brittle and accelerates ravelling and cracking, while ultra-violet radiation accelerates the aging of binders and thus ravelling. Moreover, Milne and Visser (2011) reported that the presence of moisture influences the performance of surfacing seals through affecting adhesion, weathering of the binder and influencing the seal/base support.

iv. Material Properties

The principal materials used in surfacing seals construction are bituminous binder and aggregates. Their properties are among the leading factors to the performance of surfacing seals. Milne (2004) as cited by Milne and Visser (2011) identified the binder type and application rate as the factors having the greatest influence on seal behaviour. He further attested that the binder type and properties, binder grade, viscosity-temperature relationship and viscosity at application are the most critical elements of the binder in the performance of seals. The most critical elements related to aggregates which influence the seals performance as reported in TRH3 include but are not limited to the size and grading of aggregate, spread rate of aggregate, aggregate cleanliness and aggregate selection.

v. Design, Construction, Supervision and Maintenance

Lack of attention to particular detail during the design, construction and supervision could affect the performance of the resulting surfacing seals. Some surfacing seals are more sensitive to timely maintenance than others. Timely maintenance can significantly extend the service life of surfacing seals and the pavement structure as well.

vi. General Note Related to Winter Surfacing Seals Performance

It has become common knowledge that surfacing seals constructed in winter mostly do not perform well as those constructed during other weather periods. The aforementioned factors also influence seals constructed during the winter period. The poor performance observed for the surfacing seals constructed in winter is linked to climatic and environmental factors that change dramatically during winter. Engineers in the field can hardly control the change of these factors. The climatic and environmental factors interact with other factors such as material properties and pavement conditions. This interaction adversely affects the performance of seals. The dominant failure mode for winter surfacing seals is ravelling (stone loss). This is owing to the development of a weak bond between binder and aggregate due to the poor interaction between them. The poor interaction results from the negative effect caused by the climatic and environmental conditions, which undermines some of the properties of the interacting materials.

In order to ensure a good performance of surfacing seals, consideration of the above-mentioned factors during design is of paramount importance. The following section briefly debates various design methods frequently used in different parts of the world.

2.5.5 Surfacing Seals Design Methods

The design of surfacing seals involves the identification of the appropriate seal type, determination of the type, grade and application rate of the binder and the calculation of the quantity of aggregate to apply over a unit area of the pavement. The review shows that, in the initial stages of surfacing seals construction, practitioners have used a purely empirical approach to their seal designs. Scientific development in the field generated some relevant design methods that vary across the world.

As previously stated, most of these design methods relate to Hanson's concept. The design parameters are determined in relation to a number of factors influencing the performance of seals. The number varies from one design method to another. This section does not set out to describe the listed design methods in full. If more information is needed, the reader can consult the references provided. This section highlights different factors considered in various design methods. Thus, a link between the performance of winter seals and the design process could be understood.

2.5.5.1 South African Method (TRH3)

The TRH 3 design method is based on Hanson's concept of partially filling the voids in the cover aggregate with the binder. The volume of these voids is a function of the average least dimension (ALD) of the cover aggregates. Various improvements were made to Hanson's concept as previously mentioned, to suit South African conditions. The design takes into consideration a number of factors influencing the performance of surfacing seals.

The method uses a number of primary inputs such as traffic volume, preferred new texture depth and road surface hardness to obtain the initial design. Based on the initial results, practical adjustments are made for differences in the climate, gradients, existing texture depth, application temperatures, preferred aggregate matrix and the use of modified binders. This method seems to be a hybrid of the United Kingdom and Australian design methodologies.

Traffic levels and pavement condition are the primary determinants of the appropriate surfacing seal type. Generally, the latter is selected between single seals, double seals, Cape seals and sand seals. This method also measures and evaluates the existing surface hardness and expected aggregate penetration by using a ball penetration test.

Factors such as expected traffic levels, operational road surface, climatic region and aggregate conditions determine the appropriate grade of the binder. The determination of the binder application rate is based on tables and charts that incorporate traffic level, aggregate embedment and the expected new texture depth for a specific aggregate's average least dimension (ALD) value. Figure 2-22 illustrates the factors contributing to the determination of the binder application rate. After determining the binder application rate, some practical adjustments are made to ensure that the application rate is appropriate to the specific conditions.

The aggregate spread rate depends on the purpose of the surfacing seal and the shape and flakiness of the aggregate as shown in Figure 2-23. For the final design aggregate spread rates, TRH3 recommends a trial onsite spreading of the preferred matrix of aggregates by hand.

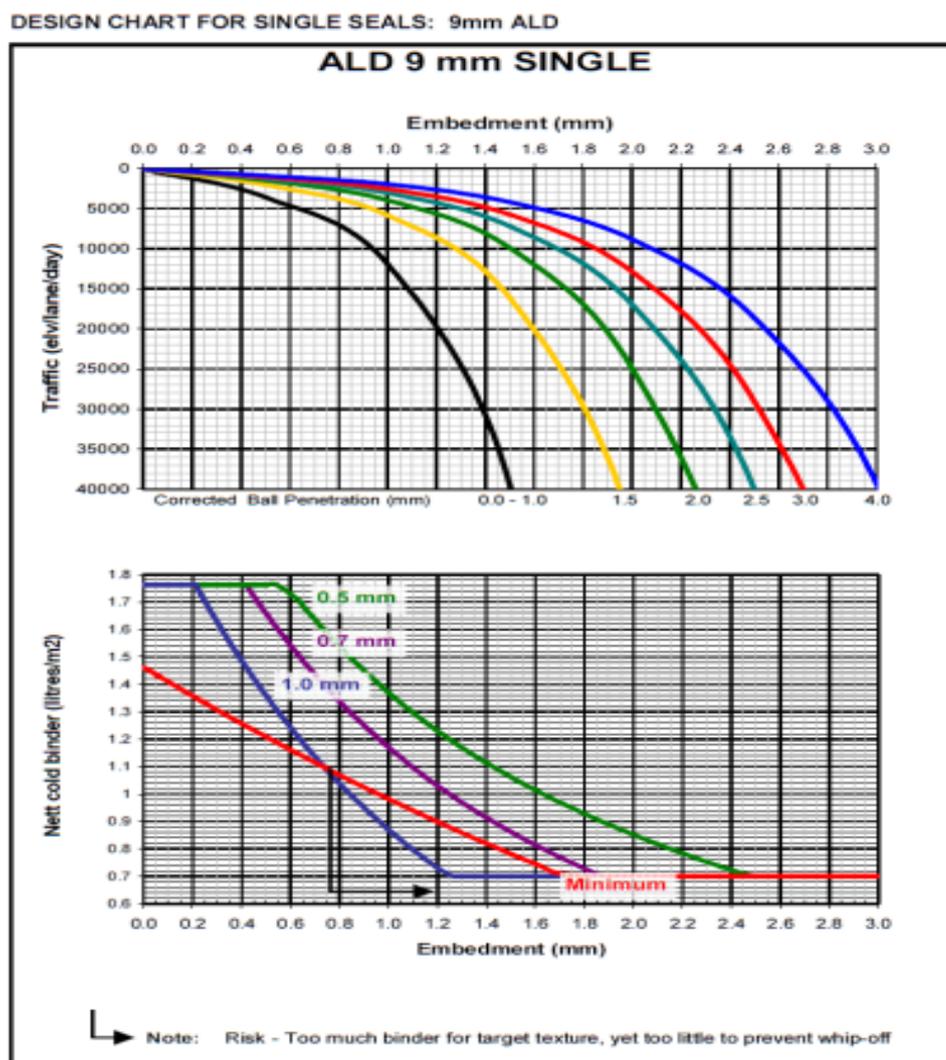


Figure 2-22: Typical design chart for the determination of the binder application rate (SANRAL, 2007)

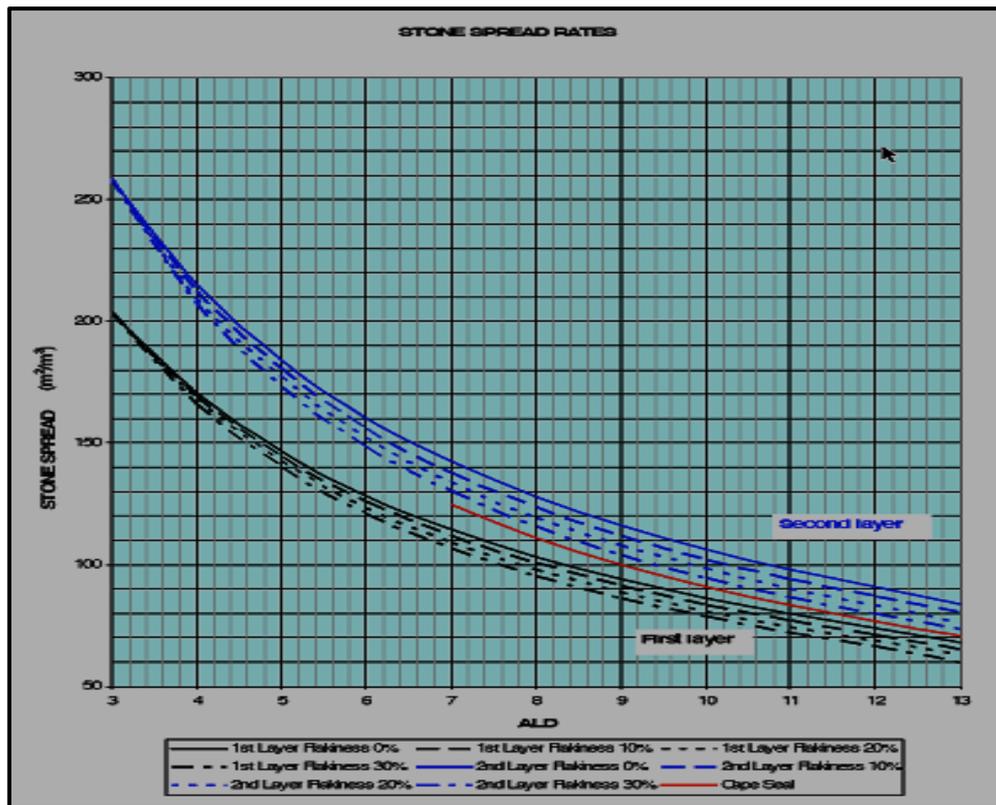


Figure 2-23: Aggregate Spread Rate Design Chart (SANRAL, 2007)

2.5.5.2 McLeod Design Method

Throughout the 1960's in the United States of America (USA), Mr Norman McLeod developed a procedure for surfacing seals design. His design method is partially based on Hanson's design concept and was developed primarily for use with emulsion binders. In the determination of the aggregate spread rate, this method considers the factors such as aggregate gradation, specific gravity, shape and wastage factor. Moreover, McLeod provided a correction factor based on the fraction of voids.

The binder spray rate is dependent upon the traffic volume, existing pavement condition, aggregate gradation, absorption and shape, and the residual bitumen content of the binder. McLeod made it apparent that correction factors for binder lost by absorption of aggregate and the texture of existing surface are recommended. McLeod's concept also gives guidelines for the appropriate type and grade of bitumen for the selected aggregate and the surface temperature at the time of application. This method was adapted by the Asphalt Institute (1979, 1983) and Asphalt Emulsion Manufacturers Association (1981) in the USA.

2.5.5.3 Modified Kearby Design Methods

In 1953 in USA, J.P. Kearby, an engineer with the Texas highway department, developed a method for the design of surfacing seals. This design method was later modified by Benson and Gallaway in 1953 and by Epps and associates in 1974 (Gransberg & James, 2005; Shuler, 2011).

Kearby's philosophy was that "*Computation alone cannot produce satisfactory results and that certain existing field conditions requires visual inspection and the use of judgement in the choice of quantities of asphalt and aggregate*".

In the design process this method requires factors such as dry loose unit weight of aggregate, bulk specific gravity of the aggregate and the results of the board test of the aggregate. The method also specifies some correction factors based on traffic level, substrate surface conditions, season and application temperature (Shuler, 2011). The board test is carried out by placing the aggregates to be used in seal construction on a steel plywood measuring one square yard. Aggregates are placed so that the average least dimension is perpendicular to the board and in a single layer, until no additional aggregate can be added without removing aggregate from the board. Then, the board and aggregate are weighed and the mass of aggregate is determined. The result is presented in pounds of aggregate per square yard of board surface and is used in the calculation of aggregate spread rate.

2.5.5.4 Austroads Sprayed Seal Design Method

This design method is directly related to Hanson's original design concept. Similarly to other methods, the main objective of this design method is that the residual binder will be about 50% to 65% of the height of the cover aggregate 2 years after construction. For determining the aggregate and binder application rates the following parameters are required: aggregate size, shape and orientation, traffic volume, road geometry, pavement absorption, texture depth of the substrate and aggregate embedment into the substrate (Gransberg & James, 2005; Shuler, 2011).

The design involves the determination of the basic voids factor. This is determined by using the aggregate shape adjustment factor and the traffic effects adjustment factor. The voids factor is then multiplied by the ALD of the aggregate to calculate the basic binder application rate. The binder application rate is then adjusted with allowances to cater for the texture and absorption of substrate surface, the aggregate properties and road geometry. The determination of aggregate application rate depends on the traffic level, ALD of the aggregate and chip size (Gransberg & James, 2005; Shuler, 2011).

2.5.5.5 Road Note 39 Design Method (UK)

This method is generally used for surface dressing design in the United Kingdom. It is considered an advanced method due to the use of a computer design program based on decision trees. The design procedure considers the following parameters as critical: traffic level, road hardness, surface conditions and site geometry. Skid resistance requirements and weather conditions are the secondary inputs in the program. The selection of the type of the surface dressing is carried out from single seal, texture slurry plus single seal, racked-in seal, double seal and sandwich seal.

The binder type is selected from either emulsion or cutback bitumen. The selection depends on traffic level and construction season. By defining the construction climatic conditions, an empirically developed factor is assigned and used in the formula of binder application rate calculation. The selection of the aggregate size and type depends on skid resistance requirements, likely weather conditions and road surface hardness. The determination of the binder application rate depends on the size and shape of aggregates, the nature of the existing road surface and the degree of aggregate embedment due to traffic. The aggregate application rate depends on aggregate size, shape and relative density (Gransberg & James, 2005).

This section has demonstrated how much different design methods consider various factors influencing the performance of surfacing seals. It is common knowledge that different binder-aggregate systems may have different bond strength development behaviour. That may affect the surfacing seals performance, especially when chip stone retention behaviour is concerned. Interestingly none of the methods mentioned above has binder-aggregate interaction as a criterion of concern. It is believed that this may increase the risks of poor performance of surfacing seals, especially under adverse construction conditions. In order to have insight in how non-consideration of one or more factors may influence the surfacing seal's performance, the following section presents an overview of dominant surfacing seals failure modes and their causes.

2.5.6 Surfacing Seals Failure Mechanisms

As discussed in section 2.5.1, the main functions of surfacing seals are:

- To serve as a waterproofing layer to protect the underlying layers from moisture ingress
- To provide a skid resistant pavement surface

The surfacing seal is said to fail if it is no longer able to fulfil the functions it was designed and constructed for, unless repaired. Failure mechanisms can be defined as phenomena that cause the surfacing seals to not fulfil its intended purpose. The failure can appear either during construction or during the seal's service life. Failures can either take place early in the seal's life (in the first days or months) or later during the service life (many years after construction). In a study of performance grading of bitumen emulsions for sprayed seals, the following dominant surfacing seals failure mechanisms were identified (Bahia *et al.*, 2008):

2.5.6.1 Fattiness/Bleeding

This is the migration of the liquid bitumen to the road surface, which creates a sticky, shiny and black surface. It is caused by excess in binder relative to the voids in the seal's aggregate matrix. That may result from the embedment of aggregates into the underlying substrate. Moreover, low viscosity of the

binder may accelerate it. Low viscosity of the binder may be due to either high service temperature or by the presence of cutters (Gransberg & James, 2005; Bahia *et al.*, 2008).

2.5.6.2 Fatigue Cracking

This usually appears during the pavement service life due to ageing of the binder. The ageing binder reduces the flexibility and the tensile strength of the seals. Once the stresses induced by traffic exceed the tensile strength of the seal, the latter will start to crack. The cracks facilitate moisture ingress into the pavement and then the structural capacity of the underlying layers is undermined (SANRAL, 2007; Bahia *et al.*, 2008; Milne & Visser, 2011).

2.5.6.3 Ravelling

This refers to the loss of cover stone from the completed surfacing seal. It is the dominant failure in the early life of the seal (a few days after construction) due to loss of adhesion or softness of the binder (Cohesion) (Transit NZ, RCA, Roding NZ, 2005). This failure mechanism is likely to appear in winter, when the adverse weather does not allow quick bond strength development. This happens especially when construction is followed by a cold night believed to be less than 10° C. In this case, the binder hardens before it has time to develop the appropriate adhesive bond strength. Once the road is opened to traffic early the following morning the stone will be easily removed from the seal due to loss of adhesion.

Later during the seal's service life, ravelling can also be observed. In this case it is generally due to ageing of the binder (it became stiff and brittle) (Milne & Visser, 2011). Once a high speed vehicle hits the cover stone, the latter can be easily be removed due to the brittleness of the binder. Another concept that can explain chip loss in this case is moisture damage. The mechanism of moisture damage is clarified by the surface energy concept discussed in section 2.3.3.4. The loss of cover aggregate results in a reduction of the surface texture depth and thus, affect skid resistance. Moreover, ravelling may facilitate the binder to be taken away by vehicle tires and thus, the moisture can penetrate and affect the underlying pavement layers.

The selection of materials that can develop quick and strong adhesion bonds can thus contribute to both short and long term surfacing seal performance. This illustrates the necessity of the assessment and understanding of the adhesion properties in binder-aggregate systems, especially for seals constructed during adverse weather conditions. The assessment of adhesion properties is generally carried out using field and laboratory testing. The subsequent section describes practical adhesion testing methods that are frequently used in the bitumen industry.

PART C: ADHESION TESTING METHODS

2.6 PRACTICAL ADHESION TESTING METHODS

2.6.1 Introduction

Several adhesion-testing methods are available in the literature. They range from those based on sophisticated technology such as the Goniometer (which is one of the methods used to measure adhesion through contact angle measurements. The droplet is placed on the solid surface by using syringe and a high resolution camera captures the image, which can be analysed by using image analysis software) to simple ones such as the pull-off test. As discussed in section 1.1, adhesion is classified as either fundamental adhesion or practical adhesion. Therefore, some of the adhesion testing methods that include but are not limited to contact angle methods, absorption method, contact probe methods, Wilhelm method, and inverse gas chromatography are specific for fundamental adhesion testing. On the other hand, methods such as the peel test, Vialit Adhesion test, lap shear test, blister test, pull-off test, riedel and weber test, etc are practical adhesion testing methods.

This section intends to introduce briefly the practical adhesion methods that are more routinely used for adhesion testing. The following section presents five of the most common testing methods. The Bitumen Bond Strength testing method that was selected for this study is discussed in details here.

2.6.2 Vialit Adhesion Test

Throughout the 1960's, the development of the Vialit test took place in France, to facilitate the evaluation of aggregates and selection of binders in sealing construction. Generally, in this testing method, the binder (often emulsion) is sprayed on a metal tray and aggregates are applied. The tray, binder and aggregates assembly are conditioned depending on the purpose of the investigation, and then placed on the testing frame in an inverted position as shown in Figure 2-24. A steel sphere is dropped multiple times onto the backside of the tray and the resulting aggregate loss recorded (Jordan & Isaac, 2010).



Figure 2-24: Vialit Adhesion Test Apparatus (Louw, Rossmann & Cupido, 2004)

Several studies were carried out using the Vialit test but some of them have shown controversial results. In a study on the evaluation of polymer modified chip seal coats, the Vialit test illustrated the capability to evaluate low/high temperature behaviour of binder-aggregate combinations (Coyne, 1988). However, Louw *et al.* (2004) reported that the test results obtained at 5°C (low temperature) by using the normal Vialit adhesion test were controversial and confusing. This was due to the fact that the adhesion failure generally occurred between the test plate and the binder film. Another study reports that Vialit test results create confusion and controversy, mainly due to the variability and reliability of test results. The main reason for confusion in this study was also the fact that most of the failure modes occurred between the binder film and the test plate (Jordan & Isaac, 2010).

2.6.3 Lap Shear Test

The single lap shear test as shown in Figure 2-25 is carried out by overlapping two flat adherends bonded by the binder and pulling them apart until the bond breaks. This test measures the shear strength of the bond. Due to various variables that need to be controlled, as well as the complexity of the stress distribution along the bond line, the lap test result's consistency is not always ensured.

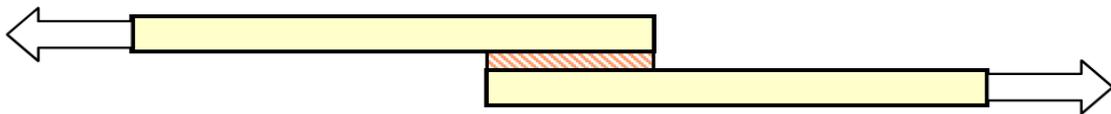


Figure 2-25: Lap Shear Test Configuration (Vickey, 2007)

2.6.4 Peel Test

The configuration of the peel test is as shown in Figure 2-26. The specimen assembly is made of two adherends joined by the adhesive in between. The bottom adherend is solid while the top is flexible. During testing, the flexible adherend is peeled away while the peeling machine records the force applied simultaneously with the corresponding displacement, to characterise the peeling strength of the adhesive. One of the advantages of this method is that the fracture energy can be determined, which facilitates the comparison of its results with other sophisticated methods (Vickey, 2007).

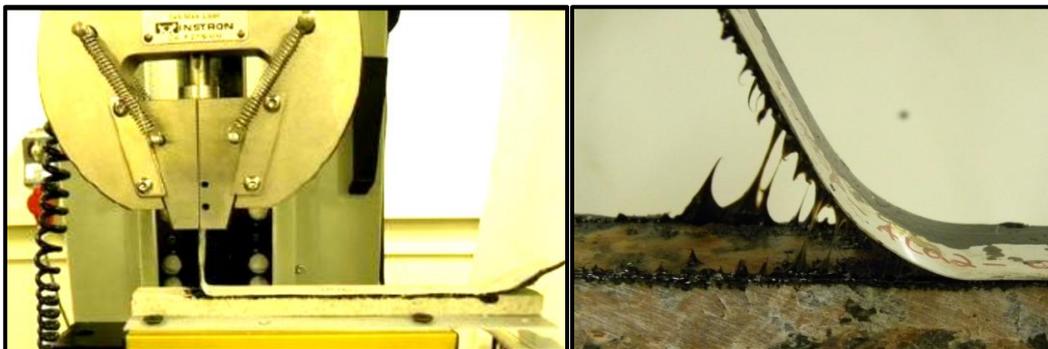


Figure 2-26: Illustration of Peel Test (Airey, 2011)

2.6.5 Riedel and Weber Test Method

This adhesion testing method is described in detail in the South African technical methods for highways one (TMH1). It is a test of the adhesion of bituminous binder to stone aggregate using the chemical immersion technique. The test is carried out by boiling a specific quantity of bitumen-coated aggregate successively in distilled water and in increasing concentrations of Na_2CO_3 , numbered 0 to 9. As water and/or the Na_2CO_3 solution interacts with the bitumen-aggregate system, water replaces the bitumen binder on the aggregate surface. Once adhesion failure takes place to the level that binder is no longer a continuous film but only specks and droplets, the number of the concentration at which this takes place, is reported as the stripping value (Committee of State Roads Authorities, 1986). This test cannot be used in the characterisation of the binder-aggregate adhesion properties in dry conditions.

2.6.6 Pull Test Method

The pull test comprises of two connecting material pieces joined together by the binder or adhesive. When the pieces are forced to move in opposite directions, a normal force to the interface is induced, resulting in predominantly pure tensile stresses along the bond interface. This type of test can appear in various configurations and is known to be simple and inexpensive.

Recently, researchers in the bitumen testing field developed a simple and inexpensive new pull test method. This study adopted and used this newly developed testing method known as “Bitumen Bond Strength (BBS) test” to evaluate the bitumen-aggregate system adhesion properties. The following section describes the working principle of the BBS test in detail.

2.6.7 Bitumen Bond Strength (BBS) Test

2.6.7.1 Introduction

The development of the BBS test is a recent improvement of an adhesion test known as PATTI (Pneumatic Adhesion Tensile Testing Instrument). The latter was first used in the bitumen industry in the early 1990's to evaluate the adhesion loss of bitumen binder-aggregate systems exposed to various moisture conditions. From the initial tests using PATTI, various shortcomings were observed. The shortcomings included, but were not limited to, the operator dependency of the testing assembly, accurate control of binder film thickness, curing, testing temperature, loading rate determination, testing of a small tensile strength etc (Abraham & Jenkins, 2012 ; Jenkins *et al.*, 2013; Jenkins & Van de Ven, 2013).

The PATTI Quantum Series (Gold Model) is the recent generation of PATTI and is commonly known as the BBS tester (BBS testing machine). The BBS tester was recently developed by the University of Wisconsin Madison, under partnership with the University of Stellenbosch-South Africa to address

some of the shortcomings mentioned above. The most important achievements of this recent development were, but not limited to the following (Greyling, 2012; Jenkins *et al.*, 2013) :

- Modification of the pull stub which made the control of 0.8mm binder film thickness possible,
- Recording the applied pressure over time which made the determination of loading rate possible,
- The use of aggregate substrate which is a real material used in seal construction instead of glass (from glass to aggregates plate substrate)
- Increase of the size of stub, which improved the stability and made testing of low bond strength possible.

2.6.7.2 BBS Components and working principles



Figure 2-27: Illustration of BBS Testing Apparatus Main Components

Figure 2-27 and Figure 2-29 illustrate the BBS test main components and operational principle respectively. The BBS tester applies a pneumatic force to the pull-out stub attached to the aggregate substrate by a film of bitumen binder. The gas from the air compressor flows into the BBS pressurized air chamber and through the hosepipe, then the gas is applied to the reaction gasket. The area of the gasket determines the force transmitted to the reaction plate and thus to the pull-out stub which is screwed to the reaction plate. Pressure is supplied until the force applied to the pull-out stub either exceeds the adhesive force between the bitumen binder and the aggregate substrate or exceeds the cohesive force within the bitumen binder. If either one is exceeded first, it will induce the detachment of the pull-out stub from the substrate. The corresponding pressure known as “burst pressure” is used

to calculate the bitumen bond strength of that particular test, for a specimen prepared under specific conditions. The formula used to calculate the bitumen bond strength is as follows:

$$BBS = \frac{(B_p * A_g) - C}{A_{ps}}$$

With

- BBS: Bitumen Bond Strength (kPa)
- B_p : Burst Pressure (kPa)
- A_g : Contact Area of the gasket to the reaction plate (mm²)
- C : Piston Constant
- A_{ps} : Area of the Pull out Stub (mm²)

Note: Various sizes of piston and reaction plate are available on the market. Each size of the piston is associated to a correction factor known as “piston constant”. In order to get correct BBS values during testing, there is an option in BBS lab view software, where the user have to specify the size of the piston. By doing so, the software identifies the piston constant and A_g to be used in calculation.

The BBS obtained from calculation is used to describe the adhesion property of the binder as well as the compatibility between the aggregate and bitumen binder under consideration. The consistency of BBS test results depends on different factors including consistency in sample preparation and the loading rate control. Sections 3.5 and 3.6 discuss the BBS test sample preparation and BBS loading rate control respectively. However, in this section an insight about the influence of loading rate on BBS test results is provided.

As in section 2.2.2.1, due to the viscoelastic nature of bitumen, the loading rate is one of the primary factors influencing the bitumen binder behaviour. With this in mind, various researchers in the BBS testing field have tried to investigate the influence of loading rate on adhesion bond strength. Miller (2010) found, while he was investigating the significant factors influencing bitumen bond strength development, that the loading rate was one of the factors. He observed that as the loading rate increases the BBS value increases for a specific bitumen type and this agrees with the rheological behaviour of bitumen as debated before. He also demonstrated that a power law can capture the relationship between the loading rate and the BBS development as illustrated in Figure 2-28. Due to the nature of the testing machine, it appears from his results that as the loading rate increases beyond 1000 kPa/s the variability increases. In the range of 690 to 1030 kPa/s a consistent and accurate relationship between BBS and loading rate is obtained. In another study, Greyling (2012) stated that his result seems to agree with Miller’s statement. However, Greyling reported the range of 700 to 800kPa/s to be most appropriate as it improves the sensitivity of the loading rate controller. This is

justified by the increase in variability (coefficient of variation) of the BBS value associated with both higher and lower loading rate relatively to that specified range.

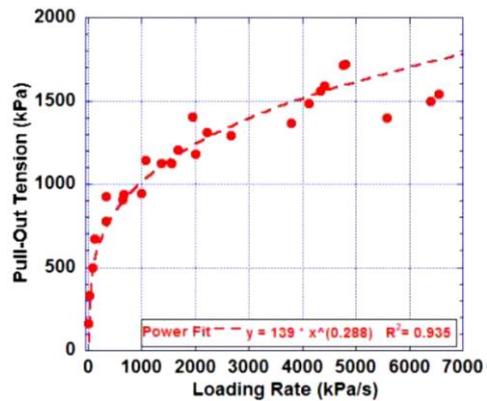


Figure 2-28: Loading Rate-Bitumen Bond Strength Relationship (Miller, 2010)

Due to the viscoelastic nature of bitumen binder, the regression function is expected to provide a plateau value on high values of loading rate. However, it doesn't appear on Figure 2-28, probably due to the type of fitting curve used.

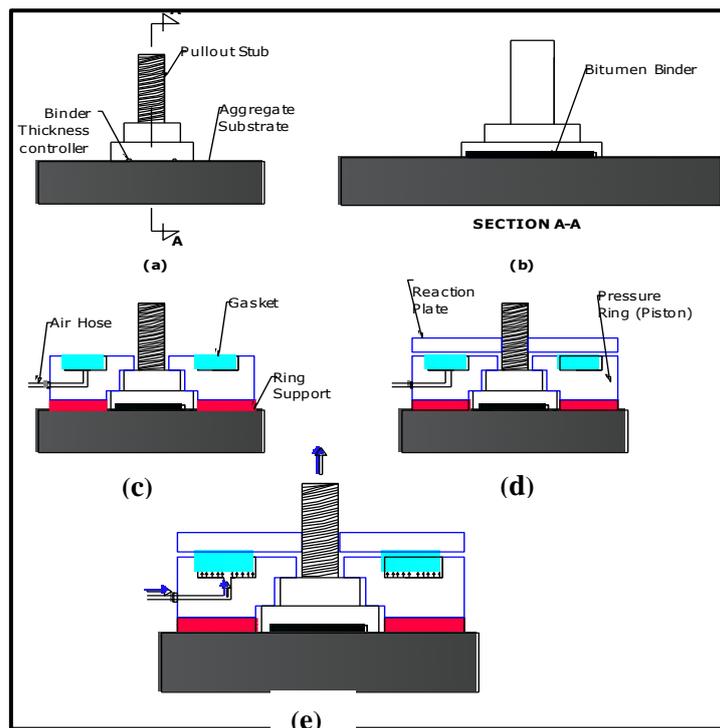


Figure 2-29: BBS test operational principle

Considering the factors influencing the performance of surfacing seals in general and the adhesion bond development in particular discussed in this Chapter and reconciling them with the study objectives elaborated in Chapter 1, a study methodology was developed and is described in the following Chapter.

CHAPTER 3 RESEARCH METHODOLOGY

3.1 INTRODUCTION

Understanding the bitumen-aggregate adhesion bond properties and evaluating the right combination of materials that favours a quick and strong adhesion bond to be developed is very crucial for the good performance of seals in general and of winter seals in particular. Various countries, including South Africa, which have similar climatic condition have experienced several failures associated with surfacing seals construction during the winter period.

Greyling (2012) mentioned that one of the major surface seal failure mechanisms observed is the chip loss (Ravelling). This happens when adhesion bond between binder and aggregate is weak and easily broken by the load generated by traffic. The failure may be either adhesive or cohesive. A number of factors were identified as influencing this failure. Among others, there are binder types, aggregate types, precoating condition, curing time, temperature, etc.

In this study, an experimental matrix was designed attempting to capture winter seals parameters and model them accordingly in order to investigate the influence of different winter conditions on the bitumen adhesion bond strength development. The experimental matrix facilitated evaluation of the appropriateness of different material combinations. It was developed to account for different chemical and physical conditions in binder-aggregate interface and to assess the impacts they could have on bond strength development mechanism.

A wide variety of testing methods, which can be used to evaluate the bitumen-aggregate adhesion properties exist as has been discussed in section 2.6..Although these various bitumen adhesion-testing methods have been mentioned, many of them do not provide sufficient information for the understanding of the phenomenon (Greyling, 2012; Jenkins & Van de Ven, 2013). In this regard, the method recently developed (BBS test) to address the above-mentioned issue was used during the course of investigation.

This chapter describes the bitumen adhesion properties testing methodology by using the Bitumen Bond Strength (BBS) test. The materials selected for this study are described and the experimental matrix is presented. The specimen preparation including aggregate core saw cutting, lapping, cleaning, drying, precoating treatment as well as bitumen preparation are presented and discussed. The working principle of the BBS testing method is clearly explained. The BBS testing procedure, including the loading rate valve set up and the determination of the bond strength and the loading rate from the test data, are presented and discussed. Finally, the conclusions are drawn concerning specimen preparation, loading rate control and pressure supply and their effects on testing results.

3.2 MATERIALS

The subsequent section discusses different types of materials used throughout the investigation. During the investigation, various material combinations were developed to evaluate the effect of different physical and chemical interactions on binder-aggregate adhesion properties.

3.2.1 Bitumen Binder

Three different types of bitumen binder commonly used in surfacing seals construction in South Africa were selected in this study. Two hot applied binders, which are 70/100 Penetration Grade bitumen and a polymer modified bitumen (S-E1) and one cold applied binder, which is a modified emulsion (SC-E1). The latter one was explored more extensively due to its high sensitivity to curing time and temperature and due to its increasing preference as a safer material for field personnel.

- 70/100: It has been selected because it is the most widely used penetration grade bitumen type in South Africa
- S-E1: It is a homogeneous binder, which is produced through modification of the penetration grade bitumen by a thermoplastic elastomer (Styrene Butadiene Styrene). Once the bitumen and the modifier (SBS) are blended, the bitumen elastic behaviour and the stiffness are improved due to the properties of the modifier. Therefore, the modification improves the flexibility of the binder under low temperatures and thus reduces its brittle behaviour. In addition to that, the modified binder is less sensitive to bleeding under high temperature and low loading rate (by increasing the softening point of the binder) (Asphalt Academy, 2007). This type of the binder was selected due to its high application in surfacing seals construction industry.
- SC-E1: This binder is a modified emulsion bitumen, which was produced by emulsifying a 70/100 penetration grade bitumen with 3% kerosene to produce a cationic 65% spray grade bitumen emulsion which was later modified by 3% SBR latex. This means that the residual bitumen contains 3% dry residual SBR polymer. This binder was freshly collected from Colas and was stored with care to prevent flocculation and settlement taking place prior to their use.

Note: In practice, the hot applied binders are heated between 160°C and 190°C and the emulsion binder is heated to about 50°C to 70°C prior to spraying (Van Zyl et al., 2012).

In winter periods due to the large difference in temperature between the road surface and the heated binder, the binder temperature drops very quickly upon its application on the road surface. This has been reported by Senadheera and Yazgan (2008), through Figure 3-1 shown below and it has been mentioned that a spray sealing work on the site of bethal has confirmed it.

They realized that a 70/100 conventional bitumen decreased from 180°C to 45°C within two minutes of being sprayed, at an air temperature of 20°C and base temperature of 30°C (Senadheera & Yazgan,

2008; Van Zyl et al., 2012). This cooling rate seems to be confirmed also by Figure 3-2 provided by Van Zyl (2012) illustrating the temperature reduction of bitumen binder after spraying (Abrahams & Jenkins, 2012).

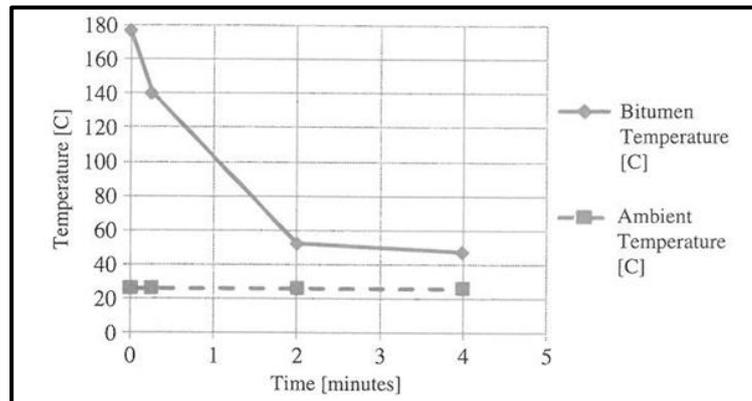


Figure 3-1: Sprayed Binder cooling rate (Senadheera & Yazgan, 2008)

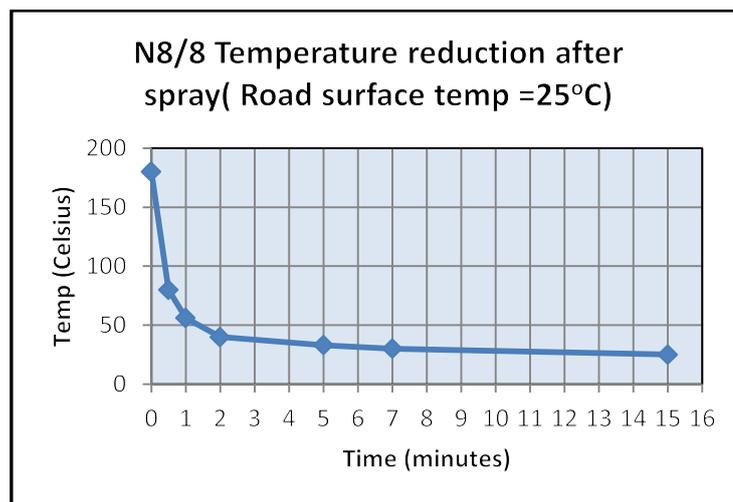


Figure 3-2: Temperature reduction after binder spray on the road surface (Abraham & Jenkins, 2012; Van Zyl et al., 2012)

The aggregate chippings are spread as soon as possible (typically a few minutes) after binder spray, by which time the temperature can be more or less 75°C for hot applied binder (Abraham & Jenkins, 2012) and to approximately 30°C for bitumen emulsion. In winter low road surface temperature and higher wind speed are factors that could further reduce the allowable time to aggregate spreading.

In this research, in order to model the winter seals construction practices and model the probable worst scenarios, the emulsion binder (SC-E1) was applied to the aggregate (precoated or non-precoated) at a controlled temperature of 30°C. The 70/100 penetration grade bitumen and S-E1 were applied to the aggregate at 40°C and 75°C controlled temperatures respectively.

3.2.2 Precoating Fluid

In SA, the bitumen-based precoats that are commonly used are cutback bitumen mixed with some special adhesion agents. These special chemical adhesion agents render the precoats suitable even for problematic silicate aggregates such as quartzite and granite (Louw, 2004). Information from the field practitioner showed that the presence of cutters in the precoating fluid retards the binder setting process. This especially happens when polymer modified bitumen (PMB) is used as a binder during winter seals construction (It appears that this is caused by the cutters in the precoat, which take long time to evaporate during winter condition). In this case, a delay of binder appropriate bond strength development of up to 2 days after construction, is not rare. This is very costly to both the contractor and the road users. It has raised the need to explore the applicability of bitumen emulsion based precoats without cutters and/or with a small quantity of cutters compared to the cutback precoats, with an intention of accelerating the rate of binder adhesion development. Furthermore, the move to the reduction of cutters intends also to limit the cutters environmental impact.

In this research, four different scenarios were analysed. The non-precoated aggregates were examined as well as three different types of Precoats, which are:

- 30% water diluted SS60: This is a water diluted anionic stable grade bitumen emulsion with 60%, unfluxed residual 70/100 pen grade bitumen.
- Anionic emulsion with Cutters (AwC): This was a special emulsion of Colas' cutback precoating fluid (Colcote S). This emulsion precoating fluid consists of about 45% water and 55% by mass of cutback bitumen. On the other hand, the cutback bitumen was made by more or less 50% bitumen and 50% kerosene with some adhesive agents. To cater for the low residual binder content, the calculation in Appendix A was used.
- Cationic emulsion with Cutters (CwC): The cationic emulsion with cutters was made in the same way as the anionic emulsion with cutters described above and the only difference is the type of emulsifiers used.

3.2.3 Aggregates

Two types of aggregate known to have different surface chemistry were used in this study. These are granites that have acidic surface properties and dolerites that are known to have basic surface properties.

3.3 EXPERIMENTAL DESIGN

In order to evaluate the bitumen-aggregate adhesion for different material combinations effectively, an experimental plan based on a fractional factorial experimental design was developed.

The experimental matrix shown in Figure 3-3 was designed to evaluate the influence of the following variables to the development of bitumen-aggregate adhesion bond strength:

- The type of binder (70/100 pen grade bitumen, S-E1 and SC-E1)
- The type of mineral aggregate (one type of granite and one type of dolerite)
- The type of precoating fluid (with no precoat, with 30% diluted SS60, with anionic emulsion with cutters and finally with cationic emulsion with cutters)
- The precoat curing condition (Wet (24hrs @ 20°C and 2hrs @ 15°C) and Dry (48hrs @ 60°C))
- The precoat curing time for a selected number of sets (2hrs and 24hrs)
- The curing time of tack coat especially for the modified emulsion binder (SC-E1) for 2hrs and 24hrs

In this study 3 replicates of each test set was adopted in general, but in order to have an insight on the variability of BBS results as well as on the repeatability of BBS tests, 6 repeats were adopted for 8 testing sets. These sets were selected randomly.

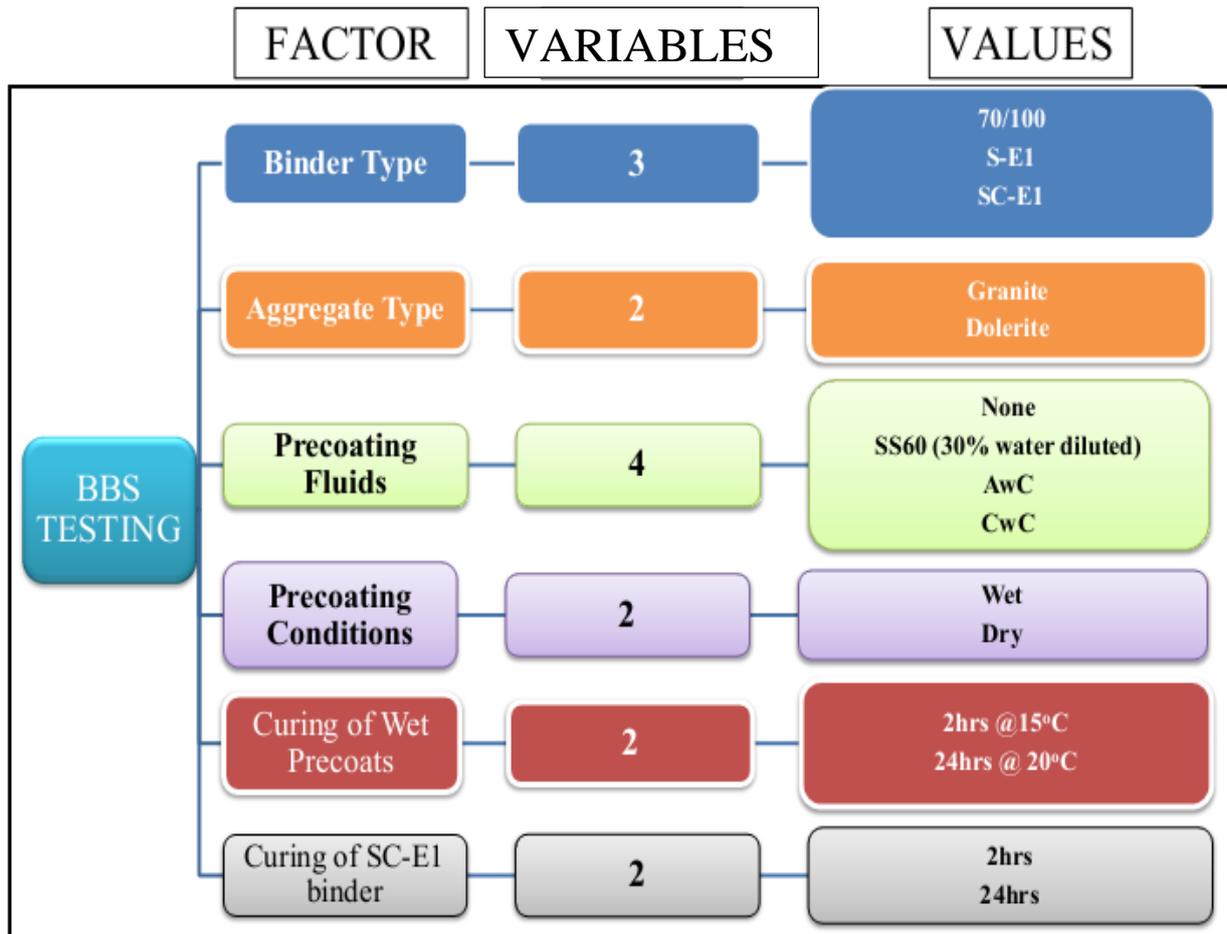


Figure 3-3 : BBS Experimental Design

Note: AwC: Anionic emulsion with cutters

CwC: Cationic emulsion with cutters

3.4 BBS TESTING METHOD

In this study, the Bitumen Bond Strength (BBS) test method was used to evaluate the adhesion properties of the bitumen binder-aggregate system. Figure 3-4 illustrates the set-up of the BBS test.

Generally, adhesion testing results present significant variability, which is caused by the inherent inconsistencies associated with the specimen preparation as well as the testing procedures. The ASTM D4541 standard for pull off testing specifies allowable pull test adhesion testing results discrepancies of up to 41% for intra-laboratory testing and up to 58.7% for inter-laboratory testing (Vickey, 2007).

During and after the BBS test method development, a number of research projects were carried-out to evaluate adhesion properties by using the BBS test. According to the results reported, the variability in BBS results are far less than the limit mentioned above, specified by the standard. This justifies its capability to characterise the adhesion properties of bitumen binder and the bitumen binder-aggregate compatibility.



Figure 3-4: BBS test set up

As previously mentioned this test was developed through partnership of two Universities including Stellenbosch. Stellenbosch University has been involved in this research since 2008. Its involvement has been limited to the assistance to University of Wisconsin Madison in identification and evaluation of suitable equipment, development of a testing protocol and evaluation of the test practicability and repeatability. It also provided inputs gained through experience on chip seals construction (Greyling, 2012).

Therefore this research, apart from being part of the SANRAL winter seals project, is also a continuous involvement of the Stellenbosch University in the development and study of the BBS testing procedure, particularly in the evaluation of the practicality and repeatability of the BBS test.

3.5 SPECIMEN PREPARATION

In this research, the BBS specimens were prepared taking reference to the AASHTO TP 91-11 standard testing method. However, some modifications were made to suite the special conditions that had to be evaluated in the study. The specimen preparation was carried out in three main steps; aggregate substrates preparation, aggregates substrates precoating and binder application.

3.5.1 Aggregates Substrates Preparation

The preparation of aggregates substrates was divided into two main groups:

- **Group 1:**

This consisted of a group of aggregate substrate cores obtained from large rocks using standard lab concrete saw (Greyling, 2012), which needed additional cutting to suit for BBS testing. The aggregates substrates specimens were produced by cutting pieces from the grey granite cores. The Stellenbosch University's (SU) laboratory saw-cutter was used for cutting process. There was two alternatives that could be used, either wet cutting (in which water is sprayed to the aggregate core during cutting process) or dry cutting (In which, no water sprayed in the process). In this study, the wet cutting process was found to be appropriate due to the edge damages that were probable for dry cutting process. The edges damage could affect the smoothness and flatness required for both side of the core to ensure of the levelled standing of specimen during curing and testing phase.

The manual feeding of the substrate to the laboratory saw cutter was found to be risky to the operator. Moreover, the parallel, smooth and spirit-levelled bottom and top faces of the resulting aggregate substrates was not ensured. The improvement was done using a screw driven clamping tool. The clamping was done with care to ensure that the face of the aggregate core was levelled before the cutting process. To achieve that, a spirit level was used during the clamping process for checking prior to cutting and in the case it was necessary, the adjustments were made until the levelled faces were obtained. After levelling the core's faces, the wet cutting was carried-out using a laboratory saw cutter, carefully and slowly at a constant speed to avoid the edge breaking out. The following figure briefly describes the aggregate substrate cutting process.



Figure 3-5 : Laboratory Aggregate Substrate Wet Cutting Process

For quality control, after each cutting exercise the aggregate core was checked for evenness by using a spirit level as shown in Figure 3-6 below. This was done because unlevelled aggregate core faces have an adverse effect on BBS results due to the resulting eccentric loading during testing. In addition to this, in order to be more confident of the quality of the aggregate core from the SU's cutting saw, some representative samples were sent to CSIR-Pretoria for roughness analysis by using laser scanner. Some of the cores analysed for roughness index are shown in Figure 3-6 and the roughness analysis results are shown in table 3-1.

Table 3-1: Results of aggregate surface roughness index analysis by using laser scanner

Particle No.	Surface Texture Index
SU-G1	0.219
SU-G2	0.304
SU-G3	0.359
SU-G4	0.335
SU-G5	0.571
SU-G6	0.403
SU-D1	0.154
SU-D2	0.152

Note: In the scanning process, all the faces of the entire aggregate core were scanned and the roughness presented was not measured on the testing faces only rather on the entire aggregate core.

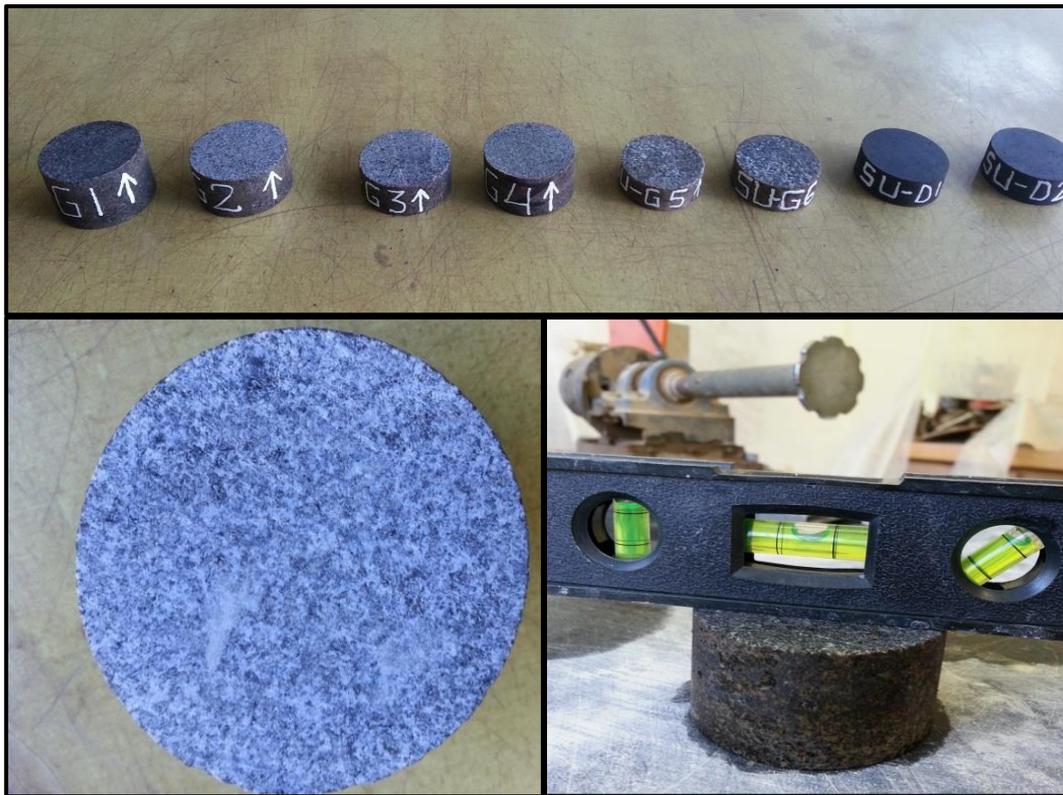


Figure 3-6: Aggregate core surface and Evenness Control after Saw Cutting Activity

Results from various research studies have shown that the variability in roughness of the aggregates substrates influences the variability in BBS results significantly. Thus, the aggregate roughness is considered as one of the factors that affect adhesion bonding between binder and aggregate. Unfortunately in this study the roughness analysis was done for a few representative number of aggregates substrate. This is due to the fact that at the time of testing there was no laser scanner available around Stellenbosch and the time which was taken for a sample to be sent to CSIR-Pretoria could not allow for all the aggregates substrates to be sent and returned in the appropriate time compared to the study time frame.

In this study, after the cutting process the aggregates substrate were lapped by using 1000 grit silicon and it was assumed that the lapping provided the same roughness for all the specimens. Nevertheless, it is recommended that for future studies, an arrangement would be made in such a way that all the aggregates substrates to be used for BBS testing should first be analysed for roughness and this will facilitate the quantification of influence of substrate roughness to the adhesion bond development.

To ensure that all the dust resulting from either cutting or lapping process as well as any adhesive substances which could be present on substrate's surfaces are removed, the aggregates substrates were cleaned by keeping them in water at 60°C for 60 minutes in an ultrasonic cleaner shown in Figure 3-7. The aggregates specimens were then placed in a forced-draft oven at 60°C for 60 minutes to dry.

Once the aggregates specimens were dried, they were allowed to acclimatize to room temperature that was between 18°C and 27°C and thereafter they were subjected to the appropriate temperature depending on which type of the binder they would be used with.



Figure 3-7 : Ultrasonic Cleaner

- **Group 2:** This consisted of a group of aggregates used for BBS testing which were granite and dolerite, as was discussed elsewhere in this methodology. These aggregates were already prepared from the quarry's stone during the previous studies and their preparation procedures were similar to the one described in Group1. Their appropriateness to BBS testing were first verified by checking whether both faces are levelled. The aggregates core found levelled were selected for further preparation. The aggregates were cleaned using mineral turpentine to remove bitumen from the previous tests. Dried in a forced-draft oven for 15 minutes and lapped by 1000 grit silicon, and then placed in an ultrasonic cleaner at 60°C for 60 minutes. This was to ensure that the aggregate surface is free of all the dust, any adhesive material from previous tests or any turpentine used during aggregates cleaning. Upon completion of cleaning, the substrates were placed in a forced-draft oven for 60 minutes at 60°C to dry. After they were allowed to acclimatize to room temperature that was between 18°C and 27°C. Thereafter, they were subjected to the appropriate temperature depending on which type of the binder they would be used with. As aforementioned some representative sample were sent to CSIR for roughness analysis.

3.5.2 Aggregates Substrates precoating

Four different conditions of aggregates substrate were analysed in this study, these are non-precoated aggregates and precoated aggregates considering three different types of precoats. The precoats used are bitumen emulsion based precoats as explained in section 3.2.2. The precoats SS60 sourced from Colas was an anionic stable grade bitumen emulsion with 60% unfluxed, 70/100 pen grade residual bitumen. Prior to precoating, it was diluted with 30% water by mass of the emulsion, which resulted

into a precoat with about 46.2% unfluxed, 70/100 pen grade residual bitumen. This precoat was less viscous compared to the other two.

The anionic emulsion with cutters and cationic emulsion with cutters precoats were produced by Colas' laboratory by emulsifying the normal bitumen based precoating fluid. These emulsions consisted of 45% water and 55% cutback bitumen by mass.

The cutback bitumen consisted of 50% of 70/100 pen grade bitumen and 50% kerosene with some adhesive agents. The cutback emulsification reduced their residual bitumen content to about 27.5%. Thus it was necessary to use a high application rate in order to get the residual binder content that approximates the one of the diluted SS60 or of normal bitumen based precoats. This was facilitated by the high viscosity of these precoats that allowed the use of thick layer of precoat.

Upon completion of preparation of the precoating fluid, the aggregate cores were labelled and checked for temperature by using a laser thermometer (The required temperature was different depending on the type of binder to be applied on the aggregate. The 70/100 was applied to the aggregate surface of $20^{\circ}\text{C}/\pm 1^{\circ}\text{C}$, S-E1 was applied to the aggregate surface of $25^{\circ}\text{C}/\pm 1^{\circ}\text{C}$ and SC-E1 was applied to the aggregate surface of $15^{\circ}\text{C}/\pm 1^{\circ}\text{C}$). These aggregate surface temperatures were selected based on the lowest allowed aggregate temperature for each binder type on site in South Africa (Braidennhan, 2013). The aggregate core surface was precoated by dipping it into the precoating fluid and allows the excess precoat to run off for some time until the precoats are uniformly distributed over the aggregate core surface. The precoats application rate was determined referring to section 2.2.3.2 and adopted to this study by calculating the corresponding thickness to be applied on each aggregate core as shown in appendix A.

The precoated aggregates were placed in the curing environment depending on the precoat curing conditions targeted. There were two different precoat curing conditions. The dry condition (where it is assumed that the cutters were allowed to evaporate) which was achieved by keeping the precoated aggregates in a forced draft oven for 48hrs at $60^{\circ}\text{C}/\pm 0.5^{\circ}\text{C}$. The wet condition (where it is assumed that cutters were not allowed to evaporate) which was achieved by keeping the precoated aggregates in UTM controlled climatic chamber at $20^{\circ}\text{C}/\pm 0.1^{\circ}\text{C}$ or $15^{\circ}\text{C}/\pm 0.1^{\circ}\text{C}$ as shown in Figure 3-8 and Figure 3-9, respectively. The field precoat curing temperature is not constant as the process is carried-out at ambient temperature. However, the selected temperatures simulate the winter condition by not allowing the cutters to evaporate ($24@20^{\circ}\text{C}$) and the summer by allowing cutters to evaporate ($48@60^{\circ}\text{C}$).

Both apparatus were first pre-set on the appropriate temperature to ensure that the specimens were placed at the required temperature. The specimens were kept with care to ensure that they stand on a flat and levelled surface to keep the uniform distribution of the precoat throughout the curing period.

Each aggregate was weighed before and after precoat application as well as after the curing time to get an insight on the effect of curing. The related results are discussed in Chapter 4. After curing, the precoated aggregates were allowed to acclimatize to the required temperature. The required temperature was related to the type of the binder to be applied to the particular aggregate as explained in section 3.2.1.

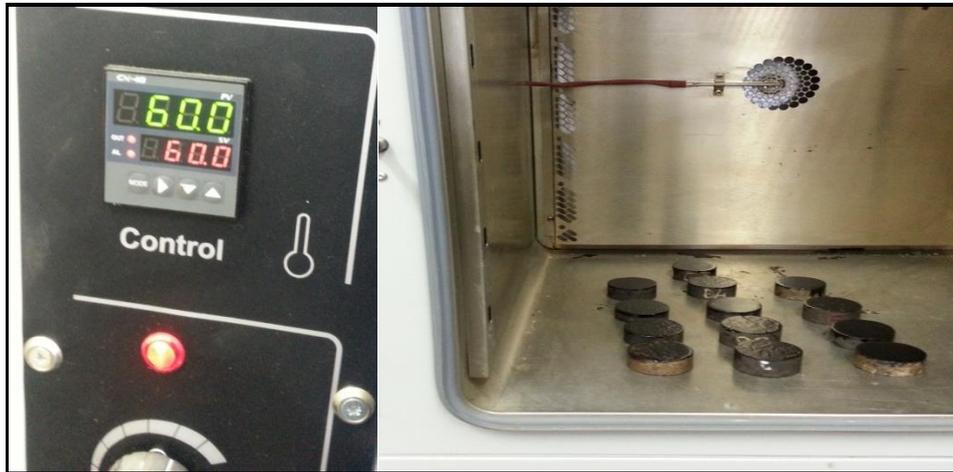


Figure 3-8: Precoat Dry Condition Curing in Oven



Figure 3-9: Precoat Wet Condition Curing in UTM Chamber

3.5.3 Binder Application

Three different types of seal binder were used in this research. Two of them (70/100 and S-E1) were hot applied binder and another one (SC-E1) was a cold applied binder.

- **Hot Applied Binder**

As explained in Section 3.1, the testing was done by trying to capture the winter seals parameters and model them accordingly. One of the primary factors in winter seal construction that influences negatively adhesion bond development is the high viscosity of the binder at the time of aggregates spreading. This problem is generally caused by the high binder temperature drop rate associated with the winter period as explained in section 3.2.1. This leaves a very narrow window to the practitioners for the aggregate spreading, which sometimes is not possible to be respected practically.

This has raised the need for exploration of the bitumen bond development in those worst cases, which required binder temperature change to be accurately controlled under laboratory conditions. To achieve this, the modification of the standard BBS testing sample preparation for hot applied binders as described by AASHTO TP 91-11 was required. This is because it does not facilitate the binder application temperature to be controlled, which was very important aspect in modelling winter seals construction.

For the accurate control of binder application temperature, there was a need for a system, in which the stub is placed in such a way that the binder on the top of it can be monitored by an appropriate temperature control instrument. This was achieved by developing a stub holder named “**Upside Down Stub Holder (UDSH)**”, which can keep the stub in an upside down position. This had made possible the application of the binder on the stub at the binder application temperature used on the field. It facilitates monitoring for binder temperature change by using a laser thermometer to any temperature you may need it to be applied to the aggregate substrate, which was not possible by using the current method.

In the current method, the temperature of the binder at the time of application to the aggregate substrate (which reflects the temperature of the binder at the time the chip stones are spread for sealing field conditions) could not be predicted because the binder application process is time based controlled and not temperature based controlled.

It is specified that the binder is poured into the mould cavities and cooled for 15minutes prior to trimming of the binder and 20minutes after trimming and sometimes refrigerated for 15minutes. This means that the temperature of the binder at the time of application is dependent on the binder quantity placed in the mould, the laboratory ambient temperature, as well as the weather conditions at the time of testing.

The developed UDSH is made in a U-shaped metallic tube. The tube was adjusted to ensure that the stub could be positioned in a straight and levelled position. The UDSH was made into metal for the bitumen application temperature to be controlled at any temperature that the operator may wish. This is because it was realized that in most of the cases bitumen application temperature is either high or low compared to the laboratory ambient temperature. In order to control the binder application temperature accurately, the UDSH needs to be subjected to a temperature that is around $\pm 3^{\circ}\text{C}$ of the targeted temperature. The developed stub holder is as shown in Figure 3-10. The accurate binder application temperature control was made possible and easy by the UDSH. Thus, the latter has facilitated the accurate modelling of winter seals conditions.



Figure 3-10: The Profile view a pull-out stub, developed Upside down Stubs holder and stub inside the holder

After set up of the apparatus, the binder application process was carried out as follows:

- i. Thirty minutes prior to the end of pre-coated aggregates curing period, the binder containers were placed in a pre-heated forced draft oven at 160°C for 70/100 and 170°C for S-E1. Simultaneously, the cleaned pullout stubs from the ultrasonic cleaner were placed in a second forced draft oven kept at 60°C .
- ii. Once the aggregates are cured and acclimatised to the aggregate spreading temperature and the binder was softened to the required temperature, the UDSH was heated. The latter was heated at around $40^{\circ}\text{C}/\pm 3^{\circ}\text{C}$ in the case the binder to be used was 70/100 and at around $75^{\circ}\text{C}/\pm 3^{\circ}\text{C}$ in the case the binder to be used was S-E1.
- iii. The UDSH was then removed from the oven and the pullout stub placed in upside down position and the binder immediately applied to the pullout stub and by using the laser thermometer, the binder temperature reduction was monitored as shown in Figure 3-11.



Figure 3-11: Binder Application on the top of the Stub and Temperature Reduction Monitoring

- iv. Once the binder temperature reached 40°C for 70/100 or 75°C for S-E1, the pullout stub was removed from the stub holder. This is done with care to ensure that the pullout stub remains in a vertical straight position. The pullout stub with the binder is then applied to the aggregate substrate and press the pullout stub firmly until all the four rims of the stub, which control the binder thickness to 0.8 mm, touch the aggregate surface. This is in order to ensure that the excess binder runs out completely.
- v. The excess binder adhering to the pullout stub sides was removed by using hot knives to ensure that its influence on the BBS results is avoided.
- vi. The specimen was then cured for 24hrs at 25°C in a temperature-controlled chamber as shown in Figure 3-12.



Figure 3-12: Curing of Tack Coat for precast and non-precast aggregates

- **Cold Applied Binder**

Bitumen emulsion mould development:

In the previous sections, the technique used for hot applied binder application procedure was described. In an attempt to use the same procedure on the binder to be applied cold, it was realised that the emulsion can be applied on the stub in the UDSH by using an eyedropper. During the temperature reduction, monitoring a hard skin is formed on the top of the emulsion layer. The formed hard skin encapsulating the emulsion layer, allows the pullout stub to be removed from the UDSH and apply it to the aggregate substrate core. Unfortunately, once the hard skin makes contact with aggregate surface, the hard skin breaks immediately. The emulsion which is still in liquid form, flow through the pullout stub binder thickness control openings and no binder can remain between the stub and aggregate. Therefore, the above-described technique was found to be inappropriate for bitumen emulsion testing due to its low viscosity at the time of application. The method described in AASHTO TP 91-11 and method presented by Greyling (2012) for emulsified binders were consulted and adopted to this study.

Due to the fact that the silicon moulds for emulsion as described in AASHTO TP 91-11 were not available in the laboratory, prior to binder application process, the moulds were first developed with some modification as described below:

- i. The casting moulds were first developed, with 20mm and 35mm internal and external diameters respectively. The moulds have the depth of 1mm.
- ii. The bitumen emulsion silicon moulds were then developed by using the silicon and its catalyst products. After 16 hours curing at the ambient temperature and 4 hours in the oven at 65°C, the silicon moulds were demolded from the casting moulds. Figure 3-13 shows some steps of silicon moulds manufacturing process.

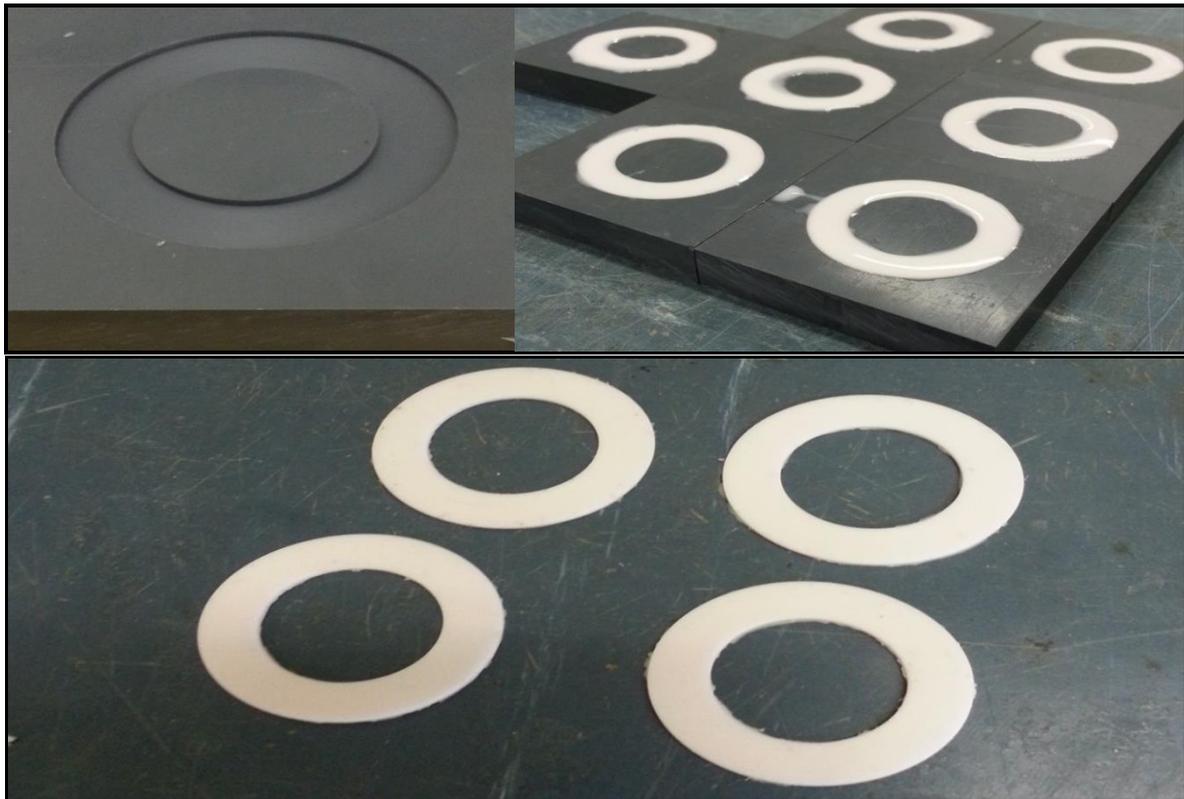


Figure 3-13: The developed casting mould, Silicon curing in the casting mould and Silicon moulds ready to be used after demoulding

Upon completion of silicon moulds manufacturing, the application of SC-E1 on the aggregates surface was carried-out as described below (Greyling, 2012):

- i. Thirty minutes prior to the end of the precoated aggregates curing period, the emulsion was heated in a forced draft oven at $60\pm 2^{\circ}\text{C}$ for not more than 60 minutes to avoid premature breaking of the emulsion. The container was covered to avoid the formation of a hard layer/skin on the top of the emulsion.
- ii. Once the precoated aggregates were cured and acclimatized to the manufacturing temperature, the silicon mould was placed on the top of the aggregates and weighed.
- iii. The emulsion that was heated in a small tin can was removed from the oven and its temperature reduction monitored by using a laser thermometer. Once the temperature reached 30°C , $0.4\pm 0.05\text{g}$ of the heated emulsion was poured immediately into the silicon mould placed on the aggregate core surface, by using a medicine dropper and the weight of the assembly was recorded. The aggregate, binder and silicon mould assembly was as shown in Figure 3-14
- iv. The aggregate, binder and silicon mould assembly was cured in an environmentally controlled chamber at 25°C for 24 hours or 2 hours depending on the curing time under consideration.

- v. While the aggregate, binder and silicon mould assembly was curing, the pullout stub was heated in a forced draft oven at $60\pm 2^{\circ}\text{C}$.
- vi. Once the assembly was cured, its weight was recorded and the silicon mould was removed around the cured binder as shown in Figure 3-15 . The heated pullout stub was applied on the residual bitumen and presses it firmly to ensure that the excess binder is allowed to run out. It was done in such a way that all the four rims of the pullout stub which control the binder film thickness to 0.8mm, touched the aggregate's surface.
- vii. The excess binder adhering to the pullout stub sides was removed by using hot knives to ensure that its influence to the BBS results is avoided.
- viii. Finally, the aggregate, binder and pullout stub assembly was kept in the environmental chamber at 25°C for 1 hour to allow the assembly to acclimatize to the testing condition, waiting for testing.



Figure 3-14: Aggregate Substrate, Silicon Mould and Binder Assembly



Figure 3-15: Bitumen emulsion residue after curing time and removal of the silicon mould

3.6 BBS TESTING PROCEDURE

The testing procedure followed in this research for the analysis of bitumen adhesion properties for winter seal has been based on “*The standard method of test for determining the asphalt binder bond strength by means of the Asphalt Bond Strength (ABS) Test*” under the designation of AASHTO TP 91-11.(AASHTO, 2011)

Prior to this research, several studies have been carried-out using the BBS test. These studies report on the different factors such as test temperature and loading rate that contribute to the consistency of the BBS test results. The factors are related either to specimen preparation or to testing procedures (Abraham & Jenkins, 2012; Jenkins et al., 2013).

Among the factors related to testing procedure, the frequently reported are:

- ✓ Consistency in test temperature and humidity
- ✓ Consistency in loading rate

In order to evaluate and propose some mitigation measures to the issues mentioned in the previous paragraph, prior to commencement of a full-scale test, a trial phase consisting of about 50 tests was implemented. Some strategies to reduce the BBS test results variability were then identified and implemented in the full-scale testing procedure.

▪ **Test Temperature and Humidity control**

Abrahams & Jenkins (2012), during their research have found that the test temperature has a dramatic effect on the bitumen bond strength results obtained. During the trial-testing phase of this research, it was realised that the test temperature fluctuation was inevitable as long as the testing was being carried out in a normal laboratory ambient conditions. This is because the room ambient temperature was varying between 18°C and 27°C and the humidity was not even controllable in spite of how much it affects the results as has been reported by Miller (2010).

To avoid the test results inconsistency due to testing temperature variability and given that the targeted testing temperature in this study is 25°C, all the BBS tests were carried out in an environmentally controlled room. In this room the temperature and humidity was controlled as shown by the temperature indicator in Figure 3-16.



Figure 3-16: Temperature Indicator inside of an environmentally controlled room

- **Loading Rate Control**

Abrahams and Jenkins (2012); Greyling, (2012), have repeatedly reported that the loading rate has a significant effect on the BBS testing results. It was also mentioned that the BBS tester's loading rate valve knob is very sensitive and difficult to control, which results in very high variability of BBS results for different test sets. This was strongly observed during the trial-testing phase.

In order to control accurately the force applied on the pull out stub during BBS testing, one needs to be able to control the air pressure delivered by the testing machine per unit time (the loading rate). To achieve this, the rate valve knob (Loading Rate Regulator) is used. Figure 3-17 illustrates the position of the loading rate regulator on the BBS machine. This valve knob is a needle valve that is used to regulate the size of the orifice and the gas flow rate from the BBS machine internally pressurized air chamber. However, in an effort to reduce the adhesion tests results' variability, this needle valve has posed several problems (Vickey, 2007).

The first problem is that a small change in this needle valve position induces a significant change in loading rate. This shows the lack of sensitivity in order to achieve a reproducible loading rate.

Secondly, the air pressure is supplied either from a high-pressure CO₂ cartridge or from an air compressor. If the air compressor is used, care should be taken to not supply more than 1035kPa (as indicated on the supply pressure gage as 150 psi); otherwise, the machine would be damaged. Therefore, once it reaches 1035kPa, you have to stop the compressor. The finite amount of gas stored in an internally built pressurized air chamber or in the CO₂ cartridge diminishes with time, which causes the loading rate change for a constant orifice size, determined by the position of the rate valve knob (Vickey, 2007).

In an effort to reduce the variability in the loading rate, during the trial-testing phase the rate valve knob was re-fixed. This has slightly improved its sensitivity but the problem was still not addressed to the level needed.

An amazing improvement was made once it was realized that the sensitivity of the rate valve knob strongly depended on the amount of air pressure supplied to the BBS tester from the compressor. Generally, the BBS tester operates under pressure that is between 690 kPa (100psi) and 1035kPa (150psi). Nevertheless, in a series of testing it was observed that the loading rate control caused problems when the pressure supplied to the BBS tester was approaching either side of its capacity. The pressure quantity approaching the allowable lower limit or the allowable higher limit caused troubles for loading rate control.

The probable reason for this is that, if the pressure supplied to the testing machine is approaching the highest specified limit, the rate valve knob starts to be somehow overloaded. The rate valve knob is destabilized in such a way that the inability to control the constant flow of gas from the air pressurized chamber increases. Similarly, if the pressure supplied is approaching the lower allowable limit, it seems that there is not enough air inside the chamber to ensure a constant flow of gas. It was realized that once the compressed air pressure supplied to the tester is between 760 kPa (110psi) and 860kPa (125psi) for a specific rate valve knob set up, the loading rate control could be optimized because the valve sensitivity improves. It can be recommended that prior to any BBS testing phase, for a given loading rate valve knob set up, the air pressure range which can be used to optimize the loading rate control should be first checked.

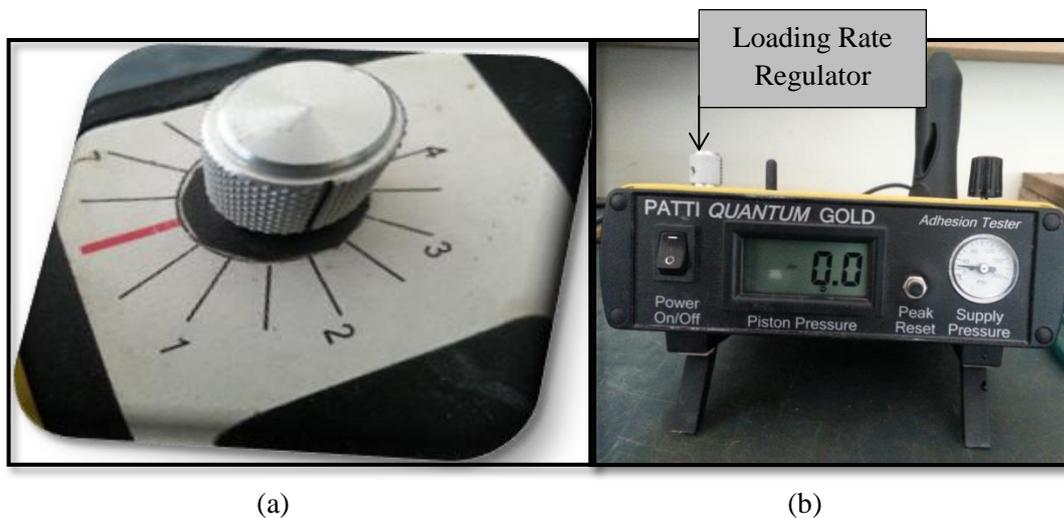


Figure 3-17: Illustration of (a) the top view of the Loading Rate Regulator, (b) the location of Loading Rate regulator on BBS Machine

The targeted loading rate in this study was 750kPa/s. By working under the above mentioned pressure range (110psi-125psi), the loading rate was kept around the targeted one but in general it was more or less 750 ± 35 kPa/s.

After the trial-testing phase, the full-scale testing program was then implemented by following the steps described below:

- i. Twenty minutes prior to the end of curing period of the specimens, the BBS testing machine components are checked and set up. The pressure is supplied to the testing machine and care is taken to ensure that the supplied air is in the range of 760 to 860 kPa (110 to 125psi as indicated on the supply gage). The loading rate is then calibrated by setting the appropriate size of the orifice using the rate valve knob. There are two techniques that can be used for loading rate calibration, either by using a dummy sample or by using a clamping system as shown in Figure 3-18. This is not a once-off process, rather the regulator is set and the test is run with dummy sample or clamped system. The loading rate is then calculated and it is checked whether it is the targeted one. If not the process is repeated by resetting the regulator until the targeted loading rate is attained. Once a dummy samples are used, the loading rate is set at the targeted loading rate. However, once the clamping technique is used, the loading rate is set at about 30-50kPa/s ahead to the targeted loading rate and the value was dependent on the pressure supplied to the testing machine.



Figure 3-18: Calibration by using clamping system technique

- ii. Once calibration was completed, each aggregate substrate temperature was recorded and for the reason that the curing and testing temperature was the same, there was no high variability observed. The observed temperature was around $25 \pm 0.5^\circ\text{C}$.
- iii. Then, by using PQG software, a number of parameters were entered to instruct the machine on how to control data acquisition from testing. The parameters entered are the size of the pull-out stub, size of the reaction plate, specimen identifications, unit of BBS results, etc.
- iv. Upon completion of all set up, the ring support was then placed around the pull-out stub to avoid the risk of eccentric loading. The pressure piston was then placed around the stub and resting on the ring support and care was taken to not induce pre-mature strains or stresses.

The reaction plate was then screwed around the pull-out stub with care not to disturb the pull out stub. Thereafter, it was unscrewed and turned for one quarter to ensure that there is a small gap between the reaction plate and the pressure piston. The complete set up is shown in Figure 3-19



Figure 3-19: Specimen set up prior to testing

- v. Once set up is completed, the test is run by switching on the testing machine and pushing the run knob until the stub is detached from the aggregate substrate.



Figure 3-20: Specimen after BBS test

- vi. Finally, upon completion of the test, a screenshot is displayed and a text tab delimited file containing the test results is saved. The later can facilitate the test results to be reviewed and imported into a spreadsheet program for further analysis.

3.7 CONCLUSIONS

The consistency of the data resulting from a testing method, determines the statistical confidence associated with the testing results. Adhesion properties testing are mostly associated with inherent variability due to a significant number of contributing variables that are difficult to be controlled. In this study, with an effort to evaluate the adhesion properties of bitumen for winter seals and with an intention of reducing the variability in the test results in order to increase the statistical confidence associated with them, a number of conclusions were drawn regarding the specimen preparation and testing procedures. They are as follows:

- Preparation of the aggregate substrate's faces prior to the application of the precoat is one of the key factors that determine the consistency in adhesion test results. It was realized that non-spirit levelled aggregate faces, regardless of their smoothness, provide inconsistency in the adhesion test results. It is recommended to check for the flatness of each of the aggregate substrates to be used in testing, by using a spirit level.
- The loading rate has a significant influence on the BBS results to be obtained on a specific test. Therefore, effort should be made to keep consistency in loading rate for a given set of tests. For this to be achieved, care should be taken during pressure supply to the BBS machine because the sensitivity of the loading rate knob valve seems to be related to the supplied pressure.
- The curing and testing temperatures of the binder-aggregate assembly should be controlled with care. Failure to do that affects significantly the consistency of the BBS results. This is due to the viscoelastic nature of the bitumen binder used as adhesive.

CHAPTER 4

BBS TEST RESULTS DISCUSSIONS

4.1 INTRODUCTION

This chapter presents and discusses the outcome of the laboratory testing phase described in Chapter 3. Section 3.3 examines the variables influencing the development of adhesion bond strength that are under scrutiny for the interest of this study. These variables are linked to the objectives of the study discussed in section 1.3. In order to relate this chapter and the aforementioned sections, the laboratory testing results are organised and presented to illustrate the influence of each particular variable.

To achieve this, test results tabulations and graphical presentations are used in each specific section. It should be noted that this type of analysis is not able to depict the influence of interactions between variables. With this in mind, a statistical analysis method was adopted so as to have an insight on how the interaction between variables influences adhesion properties.

The interaction analysis contributes to the understanding of the compatibility of material combinations under winter conditions. The univariate analysis of variance (ANOVA) was found to be appropriate for this analysis. This is concerned with the analysis of variance of a set of data with multiple independent variables that influence a single dependent variable (response).

Moreover, the statistical analysis technique was used to examine the repeatability of Bitumen Bond Strength (BBS) test. For any measurement system, the sources of variabilities are part-to-part variation, repeatability (variability due to testing machine) or reproducibility (variability due to operators). For the BBS test in this study, only the repeatability was investigated. Figure 4-1 illustrates an overview of the outline of this Chapter.

This chapter is organised in two main components. The first part evaluates the BBS testing apparatus through evaluation of the BBS loading rate control and BBS testing apparatus repeatability analysis. The second consists of results of material testing for variables influencing the adhesion bitumen bond strength development. In the second part, prior to variables influence analysis, the observations made after the precoat curing were presented.

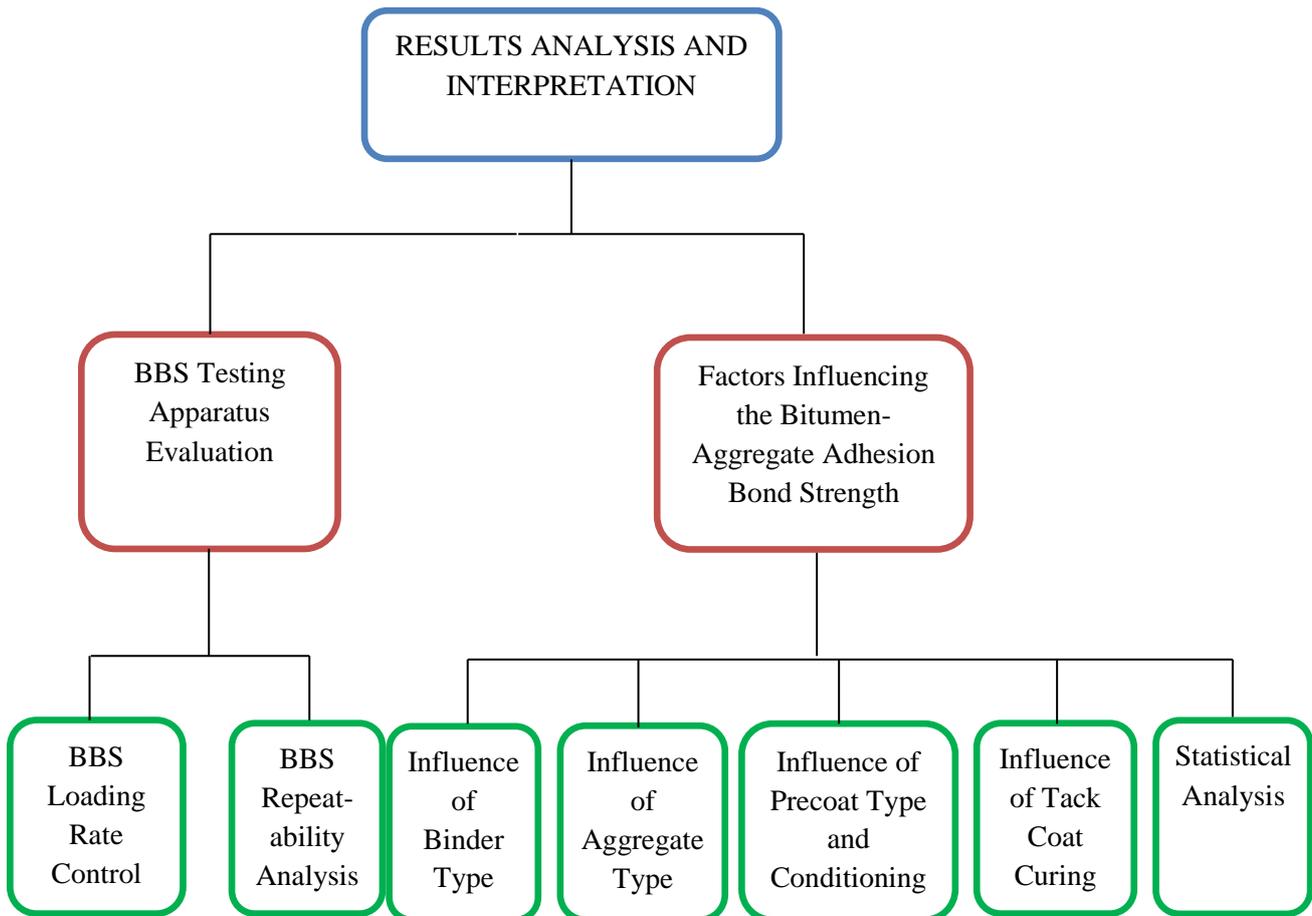


Figure 4-1: Outline of BBS test results Discussion, Analysis and Interpretation

The results are organised in tables 4-2, 4-3 and 4-4 to facilitate a preliminary visualisation of the influence of different variables used in this study. To achieve this, two techniques were used. The first technique was to organise the data, in such a way that the specimens made of similar material combinations except one variable under scrutiny are arranged together. This was to facilitate a direct preliminary comparison between variables. In this regard, the results were organised in three main sections based on the bitumen binder type that was used. Within one binder type, results are organised based on aggregate type, precoat type, precoat conditioning and curing time of the tack coat.

The second technique was the use of colour code as shown in Table 4-1. This technique is based on a philosophy of considering the combination for each binder type and non-precoated aggregate as a standard case. Thus, the non precoated aggregate combination was considered as reference for each particular group of results. In a specific group of results, a particular combination performance was ranked and an appropriate colour code was assigned on it. This was based on the increase or decrease undergone relatively to the corresponding non-precoated combination. To achieve this, a parameter named “Change in BBS (Δ BBS)” was used and its range and the corresponding colour code are as shown in Table 4-1.

Due to a significant amount of data generated during the laboratory testing phase as shown in appendix B, it was found impractical to present all of them here. Therefore, it is highlighted that in Table 4-2, Table 4-3 and Table 4-4 only averages of BBS and Loading rate (L.R) were presented. In order to provide an insight about the results variability to the reader, the number of replicates and the coefficients of variations (CoV) associated with BBS and Loading Rate results were presented. Furthermore, the failure modes were also provided based on the number of replicate in each specific combination.

Table 4-1: Change in BBS (%) for material combination relatively to the reference case-Colour Code.

Change in Bitumen Bond Strength	Performance	Colour Code
$\Delta\text{BBS} > 15\%$	Strongly Improved	
$5\% < \Delta\text{BBS} < 15\%$	Improved	
$-5\% < \Delta\text{BBS} < 5\%$	No Change	
$-5\% > \Delta\text{BBS} > -15\%$	Decreased	
$\Delta\text{BBS} < -15\%$	Strongly Decreased	

In Table 4-2, Table 4-3 and Table 4-4, the combinations were presented in a coded format. Each code was formulated to show the aggregate type, precoat type, precoat conditioning, curing time of the applied binder and finally the test temperature, which was the same for all. For more clarification, an example of interpretation for G-SS60-Wet-24hrs@25C is shown below.

G: Granite (Aggregate Type)

SS60: 30% diluted SS60 (Precoat Type)

Wet: Precoat Conditioning: It should be noted that, for some specific combinations 15°C was specified in brackets. This was intended to highlight the combinations in which precoat wet conditioning was done by curing the precoated aggregate at 15°C for 2 hours, instead of using the normal 20°C for 24hour as specified in section 3.5.2.

24hrs: Curing time in hours for the assembly after binder application

25C: Specify test temperature as 25°C

In the failure mode column, the codes used are:

A: Adhesive failure

C: Cohesive failure

C-A: 50% Cohesive, 50% Adhesive.

In the failure mode column, the number in front of each failure mode code specifies the number in the total number of replicates that experienced such specific type of failure.

Table 4-2: Summary of BBS Test Results on S-E1

SUMMARY OF BBS TEST RESULTS FOR S-E1										
Combinations			S-E1							
			Number of Replicates	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	Time to Break (Sec)	Performance	Failure Mode
G-Non	Precoated-24hrs	@ 25C	3	1183.4	9.1%	761.9	4.5%	1.8		2 A, 1 C-A
G-SS60	Wet (15C)-24hrs	@ 25C	3	1374.0	4.8%	753.8	0.7%	2.0		2 A, 1 C-A
G-SS60	Wet-24hrs	@ 25C	3	1409.3	6.8%	761.7	1.9%	2.2		2 C-A, 1 A
G-AwC	Wet-24hrs	@ 25C	6	972.5	9.6%	776.9	1.8%	1.3		6-A
G-CwC	Wet-24hrs	@ 25C	3	1063.3	10.0%	748.0	4.3%	1.5		2-A, 1C-A
G-SS60	Dry-24hrs	@ 25C	3	1411.1	2.0%	777.8	0.9%	2.2		3 C
G-AwC	Dry-24hrs	@ 25C	3	1748.9	1.5%	753.5	1.2%	2.6		2 C, 1 C-A
G-CwC	Dry-24hrs	@ 25C	3	1406.6	8.7%	762.0	2.1%	1.8		1 C, 1 A, 1 C-A
D-Non	Precoated-24hrs	@ 25C	3	1206.0	11.1%	729.8	2.2%	1.9		3 C
D-SS60	Wet (15C)-24hrs	@ 25C	3	1309.9	10.2%	764.4	1.3%	1.9		3-A
D-SS60	Wet-24hrs	@ 25C	3	1363.2	2.6%	771.9	1.7%	2.0		2 A, 1 C-A
D-AwC	Wet-24hrs	@ 25C	3	1010.9	2.3%	759.4	2.2%	1.6		2 A, 1 C-A
D-CwC	Wet-24hrs	@ 25C	6	1079.3	7.2%	752.3	2.5%	1.9		5-A, 1 C-A
D-SS60	Dry-24hrs	@ 25C	3	1496.0	12.2%	773.9	3.7%	2.2		1 C, 2 C-A
D-AwC	Dry-24hrs	@ 25C	3	1736.3	3.6%	755.4	2.8%	2.6		2 C, 1 A
D-CwC	Dry-24hrs	@ 25C	3	1713.7	9.2%	750.3	0.8%	2.6		3 C

Table 4-3: Summary of BBS Test Results on 70/100

SUMMARY OF BBS TEST RESULTS FOR 70/100										
Combinations			70/100							
			Number of Replicates	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	Time to Break (Sec)	Performance	Failure Mode
G-Non	Precoated-24hrs	@ 25C	3	1687.5	9.7%	776.4	1.3%	2.4		2 A, 1 C-A
G-SS60	Wet (15C)-24hrs	@ 25C	3	1802.2	4.7%	750.5	0.4%	2.6		3 C-A
G-SS60	Wet-24hrs	@ 25C	3	1848.3	7.8%	783.3	3.8%	2.8		3 C-A
G-AwC	Wet-24hrs	@ 25C	3	1652.3	10.4%	759.2	0.3%	2.4		3-A
G-CwC	Wet-24hrs	@ 25C	3	1608.0	8.4%	752.4	1.6%	2.4		3-A
G-SS60	Dry-24hrs	@ 25C	3	2071.4	0.9%	756.2	5.0%	3.1		2 C, 1 C-A
G-AwC	Dry-24hrs	@ 25C	6	2074.6	3.1%	748.6	2.1%	3.0		4 C, 2 A
G-CwC	Dry-24hrs	@ 25C	3	1880.8	7.8%	749.0	1.6%	2.6		2 C-A, 1 A
D-Non	Precoated-24hrs	@ 25C	3	1886.2	8.7%	757.9	2.1%	2.7		3 C
D-SS60	Wet (15C)-24hrs	@ 25C	3	2078.7	17.2%	749.0	1.5%	3.2		1 A, 1 C, 1 C-A
D-SS60	Wet-24hrs	@ 25C	3	1940.4	11.4%	744.1	2.7%	2.9		2 A, 1 C-A
D-AwC	Wet-24hrs	@ 25C	3	1331.6	8.3%	777.2	5.5%	1.9		3-A
D-CwC	Wet-24hrs	@ 25C	3	1900.7	3.0%	770.8	0.6%	2.7		2 A, 1 C-A
D-SS60	Dry-24hrs	@ 25C	3	2217.8	3.5%	746.5	1.7%	3.4		2C, 1 C-A
D-AwC	Dry-24hrs	@ 25C	3	1980.2	2.4%	763.6	4.0%	2.8		2 C, 1 A
D-CwC	Dry-24hrs	@ 25C	6	2183.0	6.5%	752.3	3.0%	3.1		3 A, 2 C-A, 1 C

Table 4-4: Summary of BBS Test Results on SC-E1

SUMMARY OF BBS TEST RESULTS FOR SC-E1										
Combinations			SC-E1							
			Number of Replicates	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	Time to Break (Sec)	Performance	Failure Mode
G-Non Precoated-2hrs@25C			3	233.1	6.7%	783.0	2.1%	0.5		3-A
G-SS60-Wet (15C)-2hrs@ 25C			3	477.0	10.8%	767.6	1.3%	0.8		3-A
G-SS60-Wet-2hrs@ 25C			3	368.7	3.3%	744.6	1.0%	0.7		2 A, 1 C-A
G-AwC-Wet-2hrs@ 25C			6	303.1	14.4%	751.4	1.6%	0.6		4 C
G-CwC-Wet-2hrs@ 25C			3	328.0	10.7%	748.2	1.1%	0.6		3 C
G-SS60-Dry-2hrs@25C			3	202.4	10.7%	751.5	2.6%	0.4		2 A, 1 C-A
G-AwC-Dry-2hrs@25C			3	428.2	10.7%	755.0	1.7%	0.8		2 A, 1 C
G-CwC-Dry-2hrs@25C			3	316.2	12.7%	771.9	3.0%	0.6		3-A
G-Non Precoated-24hrs@ 25C			3	1225.9	8.2%	749.6	1.9%	1.9		3-A
G-SS60-Wet (15C)-24hrs@ 25C			3	808.5	14.0%	759.2	0.8%	1.2		3-A
G-SS60-Wet-24hrs@ 25C			3	854.6	13.8%	755.1	1.6%	1.2		3-A
G-AwC-Wet-24hrs@ 25C			6	876.8	5.7%	755.6	1.3%	1.4		6-A
G-CwC-Wet-24hrs@25C			3	823.9	13.2%	766.4	0.5%	1.3		3-A
G-SS60-Dry-24hrs@ 25C			3	967.5	7.6%	747.9	1.4%	1.5		3-A
G-AwC-Dry-24hrs@25C			3	1063.3	17.8%	759.3	1.6%	1.7		2 A, 1 C-A
G-CwC-Dry-24hrs@ 25C			3	1435.5	4.0%	763.3	0.3%	2.1		1 C, 2 C-A
D-Non Precoated-2hrs@25C			3	219.1	9.3%	763.0	4.6%	0.6		3-A
D-SS60-Wet (15C)-2hrs@ 25C			3	499.6	9.0%	758.0	0.6%	0.8		3-A
D-SS60-Wet-2hrs@25C			3	499.6	9.0%	758.0	0.6%	0.8		3 C
D-AwC-Wet-2hrs@ 25C			3	374.9	14.6%	766.0	1.4%	0.8		2 C
D-CwC-Wet-2hrs@ 25C			6	435.9	8.5%	772.1	1.0%	0.8		6 C
D-SS60-Dry-2hrs@ 25C			3	465.3	6.7%	752.9	0.8%	0.8		3 C
D-AwC-Dry-2hrs@25C			3	633.3	9.1%	749.2	1.4%	1.0		3-A
D-CwC-Dry-2hrs@25C			3	588.1	5.1%	749.5	1.4%	1.0		3-A
D-Non Precoated-24hrs@ 25C			3	1289.4	9.1%	751.0	0.4%	2.0		3-A
D-SS60-Wet (15C)-24hrs@ 25C			3	1172.6	1.8%	753.2	0.5%	1.7		3-A
D-SS60-Wet-24hrs@25C			3	1212.3	12.8%	751.6	1.2%	1.8		3-A
D-AwC-Wet-24hrs@ 25C			3	1034.4	5.4%	741.9	0.6%	1.6		3-A
D-CwC-Wet-24hrs@25C			6	947.2	11.4%	750.0	0.7%	1.5		6-A
D-SS60-Dry-24hrs@ 25C			3	918.8	8.9%	766.6	1.8%	1.5		3-A
D-AwC-Dry-24hrs@25C			3	1155.4	7.6%	768.4	1.4%	1.6		2 C, 1 C-A
D-CwC-Dry-24hrs@25C			3	1477.9	3.9%	745.5	0.5%	2.2		3 C

Note: Generally, it is believed that the time to break was supposed to be equivalent to the BBS value over the loading rate for a specific test. However, observations of the results obtained as shown in tables above do not prove that. For example in table 4-4 second row, $477.0/767.6 = 0.6$ sec whereas the value recorded from the machine is 0.8 sec. It means that throughout the test, the pressure supplied to the pullout stub fluctuates. The level of fluctuation appears to be related to the level of pressure supplied to the BBS machine as has been explained based on Figure 4-2 in the subsequent sections. This inequality shows the inability of the loading rate valve knob to control successfully the uniform flow of pressure throughout the test. It is noted that according to AASHTO-TP 91-11, the BBS loading rate is calculated by determining the slope of the line connecting the 20% and 80% values of the maximum BBS value of a specific test.

4.2 BBS TESTING APPARATUS EVALUATION

4.2.1 BBS Testing Apparatus Loading Rate Control

As previously stated, the present study makes use of bituminous materials as adhesives. The states of the adhesives and the room temperature, at the time of testing allowed the adhesives to behave as visco-elastic material. Thus, it is reasonable to care about the loading rate in the characterisation of the adhesive behaviour under such conditions.

Due to the nature of the testing system, maintaining the constant loading rate throughout the testing phase is not always possible. The troubles associated with the loading rate control and their causes were discussed in section 3.6. This section examines the results obtained during an investigation on how the pressure supplied to the BBS testing machine can influence its loading rate during testing. To control the loading rate, the rate valve knob (Loading Rate Regulator) is used.

During the investigation, a pressure of 813 kPa (118 psi) was supplied to the BBS machine and the machine was calibrated to achieve the targeted loading rate of 750kPa/s. This was achieved by making one full turn of the regulator and continues for the second turn up to the mark 2 as shown in Figure 3-17. Then, the test was run and the loading rate was determined as described in AASHTO TP 91-11.

The loading rate regulator was kept in the same position and the pressure was changed to 703 kPa (102 psi) and 958 kPa (139 psi). The tests were run on these two different pressure levels and the corresponding loading rates were determined. Figure 4-2 and Figure 4-3 illustrate graphically, the BBS test results and the loading rate determination, for three different pressure levels while the loading rate regulator was kept in the same position. The vertical straight lines on Figure below represent the failure of the bond and thus, the end of the test.

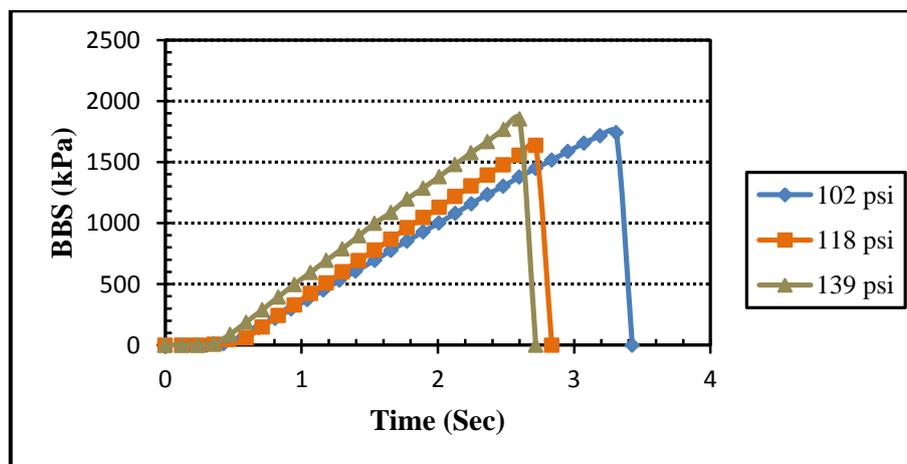


Figure 4-2: BBS results for 3 different levels of pressure while the position of the loading rate regulator is constant and on the same type of material combination.

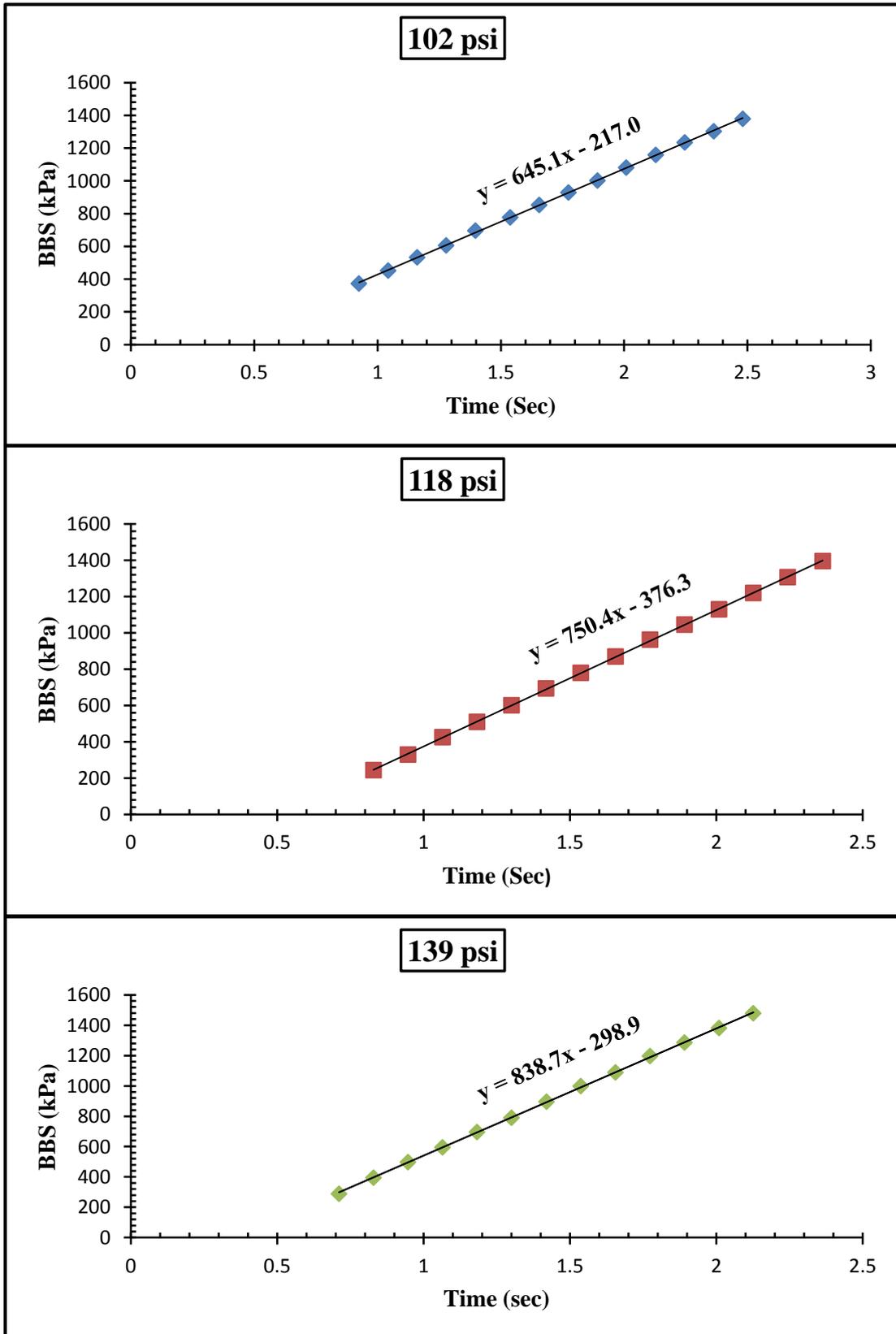


Figure 4-3: Loading Rate for 3 different levels of pressure while the position of the loading rate regulator is constant and on the same type of material combination.

Figure 4-2 and Figure 4-3 illustrate that, the loading rate in a given specific test depends strongly on the pressure level used in the test. Figure 4-2 shows that for a given loading rate regulator set up position, the increase in pressure supplied to the BBS causes an increase in loading rate. This is illustrated by the slope of the loading variation with time. Figure 4-3 reinforced this statement by illustrating the real values of the loading rate as 645.1kPa/s for 102psi, 750.4kPa/s for 118psi and 838.7kPa/s for 139psi. Based on these values, the correlation between the loading rate of BBS test and the pressure supplied from the compressor was estimated as shown in Figure 4-4. It should be noted here that, the BBS loading rate values mentioned above were determined by using the loading rate determination method described in AASHTO TP 91-11.

In this method, the loading rate is considered as the slope of the line between 20% and 80% of the maximum BBS recorded. At very high or low pressure, a small adjustment of the rate regulator caused an abrupt change in loading rate which made it difficult to control under these conditions. This has favoured the use of the middle pressure (around 118psi) because then the rate regulator sensitivity was good enough and more precise loading rate could be obtained.

In this study based on the targeted loading rate, the pressure was supposed to be kept as 118 psi and the loading rate regulator kept in the position shown in Figure 3-17. This was however not possible given that the pressure could not be kept constant throughout the test. Thus, in an effort to control the loading rate under the predefined position of the loading rate regulator, the practical pressure range that helped to reduce the variability in the loading rate was identified. The identified pressure range was 110 to 125 psi and was used throughout the test.

The identification of the appropriate pressure range helped to control the loading rate in a very close range around the targeted value as shown in Table 4-2, Table 4-3 and Table 4-4. The graphical representation of the loading rate used in this study is presented in the following figures. Due to a significant number of test performed, the loading rate cannot be presented in one figure. The graphical representation was divided into four categories that are based on the precoat type used during testing as illustrated in figures 4-6, 4-8, 4-9 and figure 4-10. In each category, the combinations are symbolised by numbers to facilitate graphical representation. Prior to the presentation of each figure, the table that describes the symbols and their corresponding combinations is presented. In order to provide an easy way to locate and quantify the variation in BBS loading rate used in this study, the typical histograms of the loading rate values, the mean values and the standard deviation values are presented based on the data used in figure 4-6, as it appears in figure 4-7.

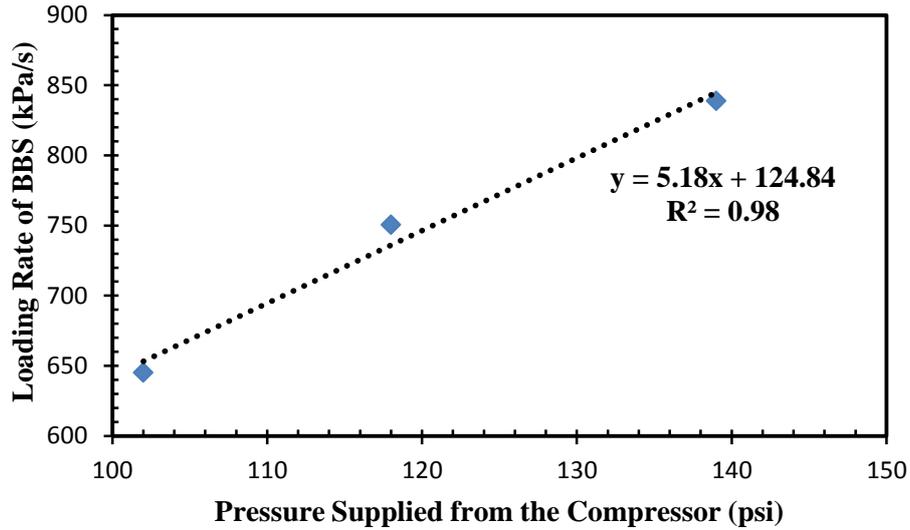


Figure 4-4: Correlation between the Pressure Supply from the Compressor and the BBS loading Rate

It is a common knowledge that the loading rate influences the behaviour of visco-elastic material. In this regard, based on the testing set where six replications were tested on the same combination, a trial was made to correlate the BBS obtained and the loading rate used. The results did not show good correlation between the BBS and the corresponding loading rate as shown in Figure 4-5. This suggests that, in addition to the loading rate, there are other factors influencing the BBS results. It is believed that, the specimen preparation factors and material heterogeneity contribute a lot. Consequently, this could not allow establishment of a loading rate correction factor to bring the tests loading rate values to a targeted loading rate value to make comparison of BBS value more accurate.

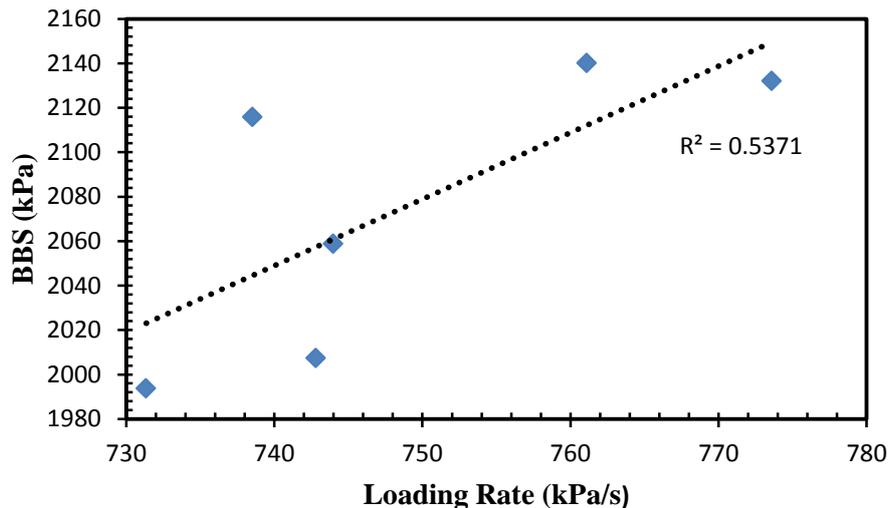


Figure 4-5: Correlation between the BBS and the BBS loading Rate for G-AwC-Dry-70/100-24hrs Combination

Table 4-5: Combinations and their corresponding symbols for non precoated Category

Symbol	Combination	Targeted Loading Rate (kPa/s)	Average Test Loading Rate (kPa/s)
1	G-non precoated-70/100-24hrs	750	759.1
2	G-non precoated-S-E1-24hrs		
3	G-non precoated-SC-E1-2hrs		
4	G-non precoated-SC-E1-24hrs		
5	D-non precoated-70/100-24hrs		
6	D-non precoated-S-E1-24hrs		
7	D-non precoated-SC-E1-2hrs		
8	D-non precoated-SC-E1-24hrs		

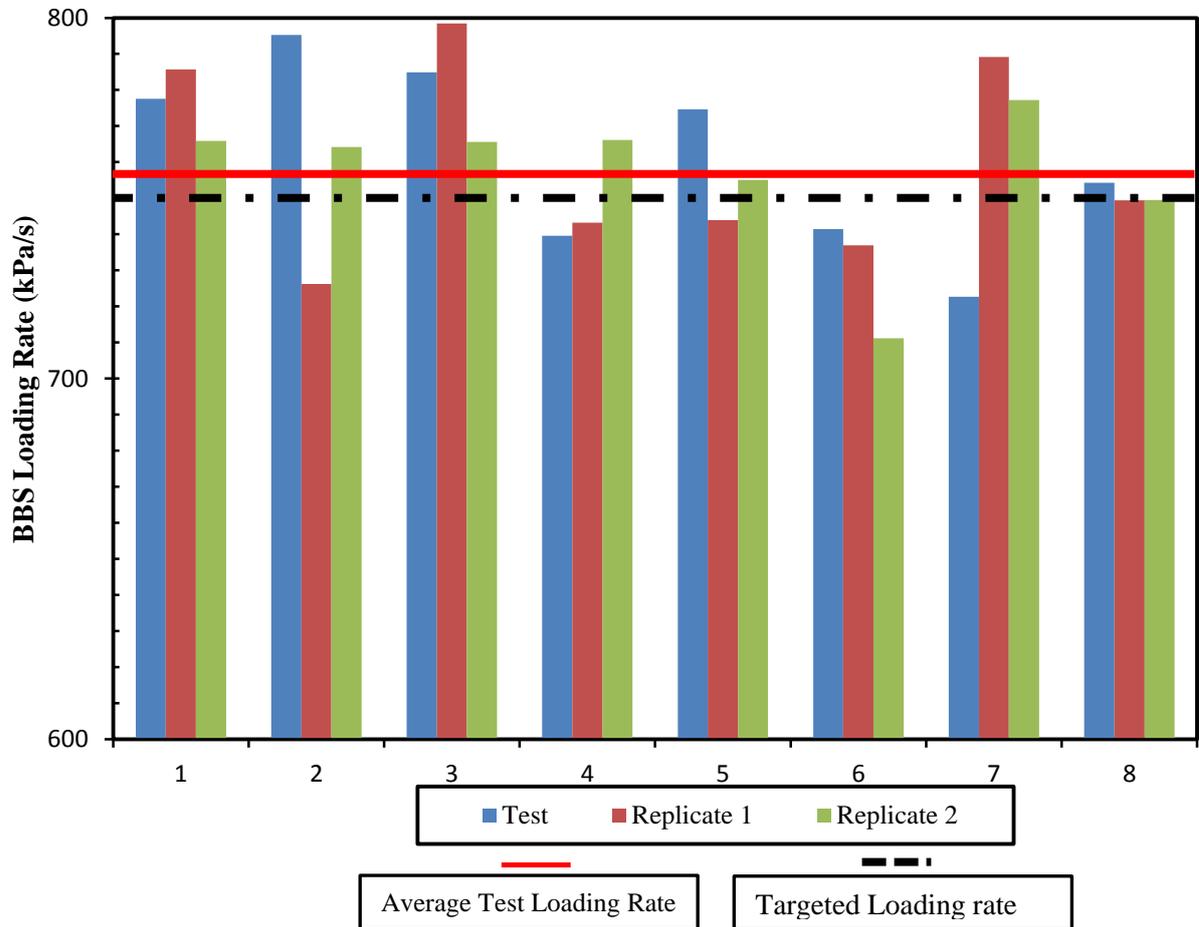


Figure 4-6: Representation of BBS Loading Rate in non-precoated combinations testing.

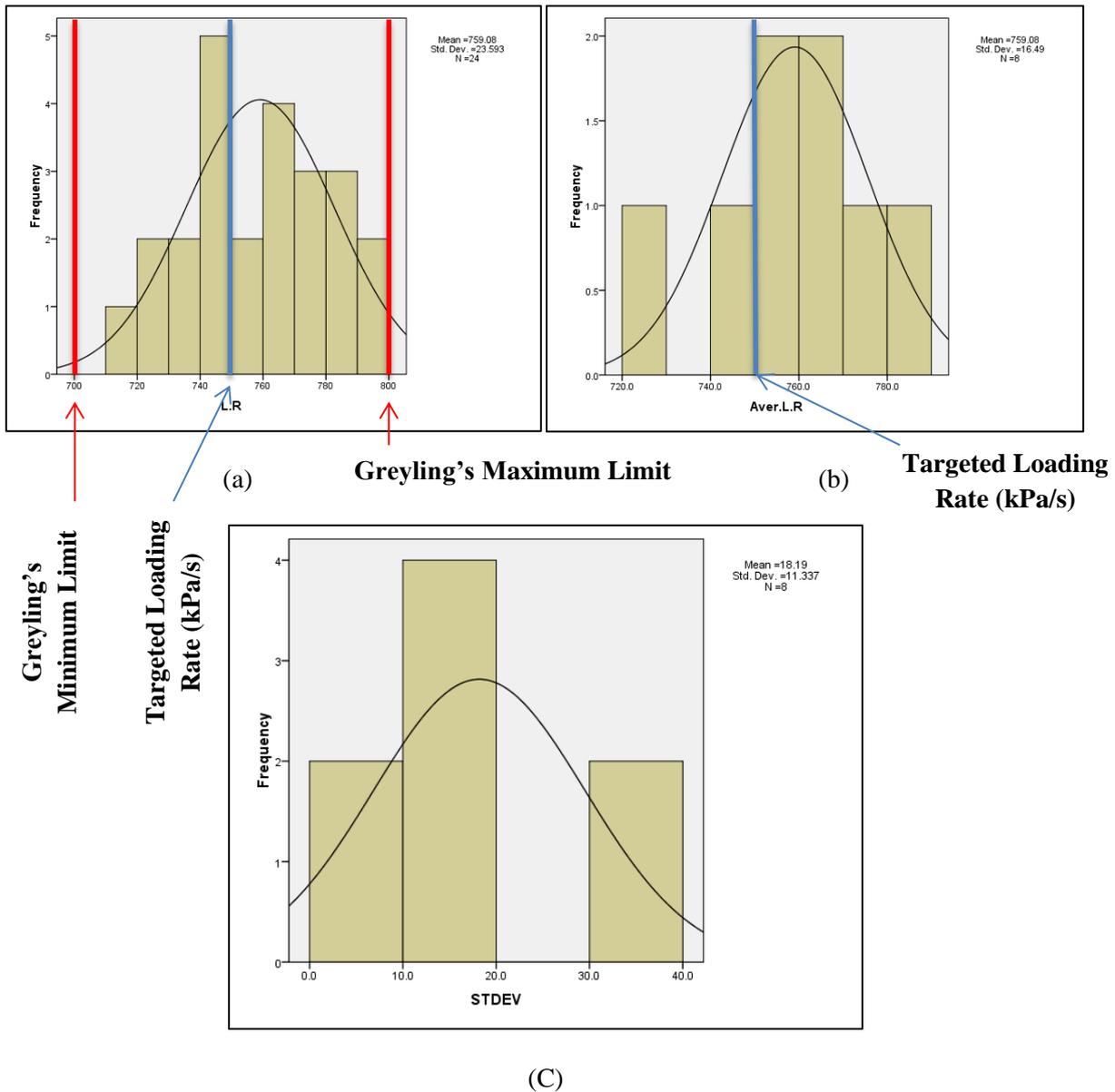


Figure 4-7: Typical Histograms of (a) Loading Rates, (b) Average Loading Rate, and (c) Standard deviation

Note: L.R: Loading Rate (kPa/s), Aver.L.R: Mean Value of loading Rate (kPa/s) and STDEV: Standard Deviation.

Observation of figure 4-7, illustrates that the entire set of data of loading rate for this testing batch falls within the limit specified by Greyling (2012). It appears that, the data have more or less a non-skewed distribution around the target value and the peak value is very close to the target value of 750kPa/s. Thus, on the point of view of specification, the control of the loading rate was quite good. However, in order to achieve these results it required to calibrate the machine every time before testing. This step is very tedious, time consuming, and may undermine the attractiveness of this testing method to practioners. Therefore, the improvement on loading rate control is required and proposed as mentioned in recommendations for further studies.

Table 4-6: Combinations and their corresponding symbols for SS60 (30% water diluted) precoated Category

Symbol	Combination	Symbol	Combination	Targeted Loading Rate (kPa/s)	Average Test Loading Rate (kPa/s)
1	G-SS60-70/100-Wet (15C)-24hr	13	D-SS60-70/100-Wet (15C)-24hr	750	758.1
2	G-SS60-70/100-Wet-24hrs	14	D-SS60-70/100-Wet-24hrs		
3	G-SS60-70/100-Dry-24hrs	15	D-SS60-70/100-Dry-24hrs		
4	G-SS60-S-E1-Wet (15C)-24hrs	16	D-SS60-S-E1-Wet (15C)-24hrs		
5	G-SS60-S-E1-Wet-24hrs	17	D-SS60-S-E1-Wet-24hrs		
6	G-SS60-S-E1-Dry-24hrs	18	D-SS60-S-E1-Dry-24hrs		
7	G-SS60-SC-E1-Wet (15C)-2hrs	19	D-SS60-SC-E1-Wet (15C)-2hrs		
8	G-SS60-SC-E1-Wet (15C)-24hr	20	D-SS60-SC-E1-Wet (15C)-24hr		
9	G-SS60-SC-E1-Wet-2hrs	21	D-SS60-SC-E1-Wet-2hrs		
10	G-SS60-SC-E1-Wet-24hrs	22	D-SS60-SC-E1-Wet-24hrs		
11	G-SS60-SC-E1-Dry-2hrs	23	D-SS60-SC-E1-Dry-2hrs		
12	G-SS60-SC-E1-Dry-24hrs	24	D-SS60-SC-E1-Dry-24hrs		

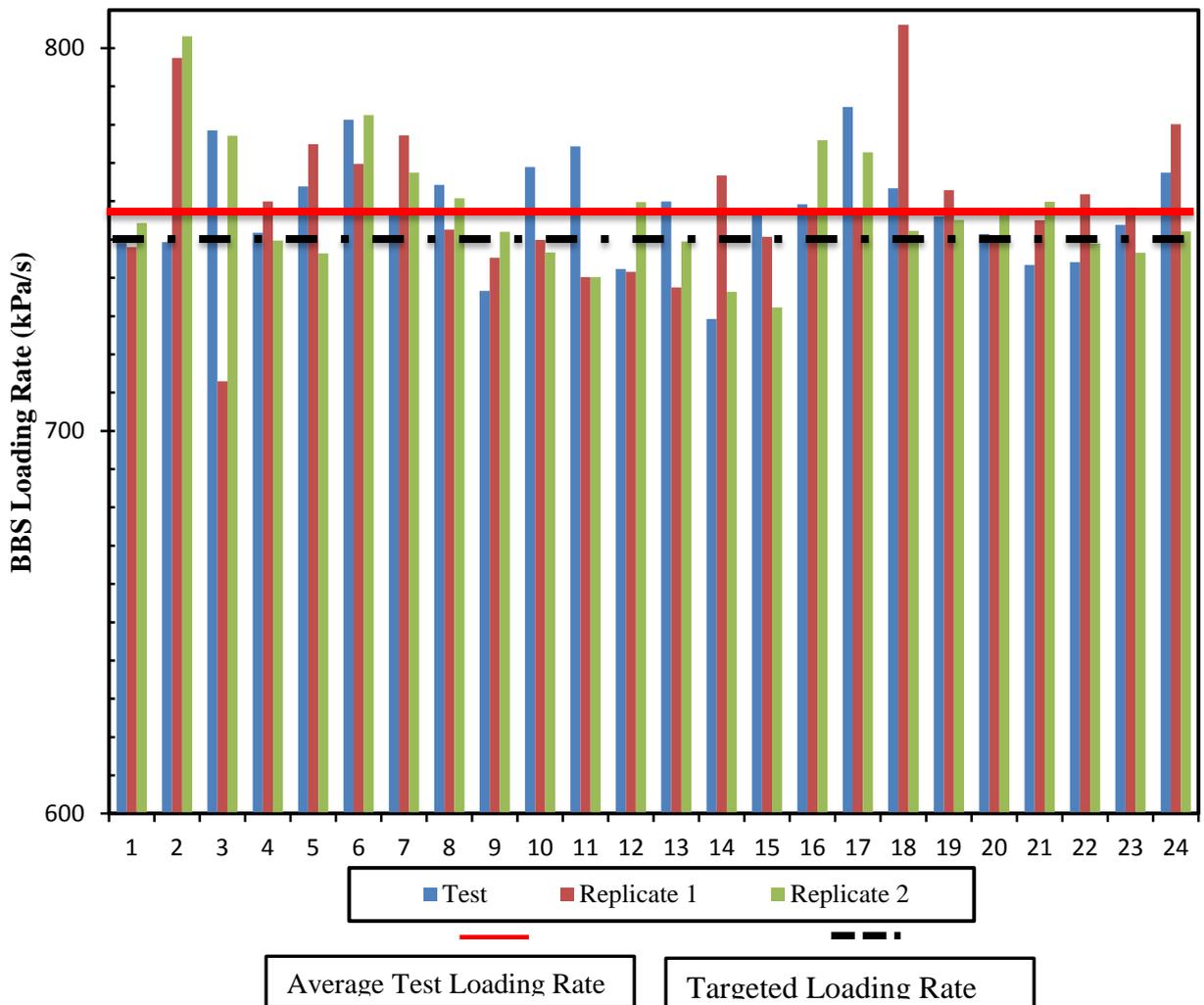


Figure 4-8: Representation of BBS Loading Rate in SS60 (30% water diluted) precoated combinations testing.

Table 4-7: Combinations and their corresponding symbols for AwC precoated Category

Symbol	Combinations	Symbol	Combinations	Targeted Loading Rate (kPa/s)	Average Test Loading Rate (kPa/s)
1	G-AwC-Wet-70/100-24hrs	9	D-AwC-Wet-70/100-24hrs	750	758.8
2	G-AwC-Dry-70/100-24hrs	10	D-AwC-Dry-70/100-24hrs		
3	G-AwC-Wet-S-E1-24hrs	11	D-AwC-Wet-S-E1-24hrs		
4	G-AwC-Dry-S-E1-24hrs	12	D-AwC-Dry-S-E1-24hrs		
5	G-AwC-Wet-SC-E1-2hrs	13	D-AwC-Wet-SC-E1-2hrs		
6	G-AwC-Wet-SC-E1-24hrs	14	D-AwC-Wet-SC-E1-24hrs		
7	G-AwC-Dry-SC-E1-2hrs	15	D-AwC-Dry-SC-E1-2hrs		
8	G-AwC-Dry-SC-E1-24hrs	16	D-AwC-Dry-SC-E1-24hrs		

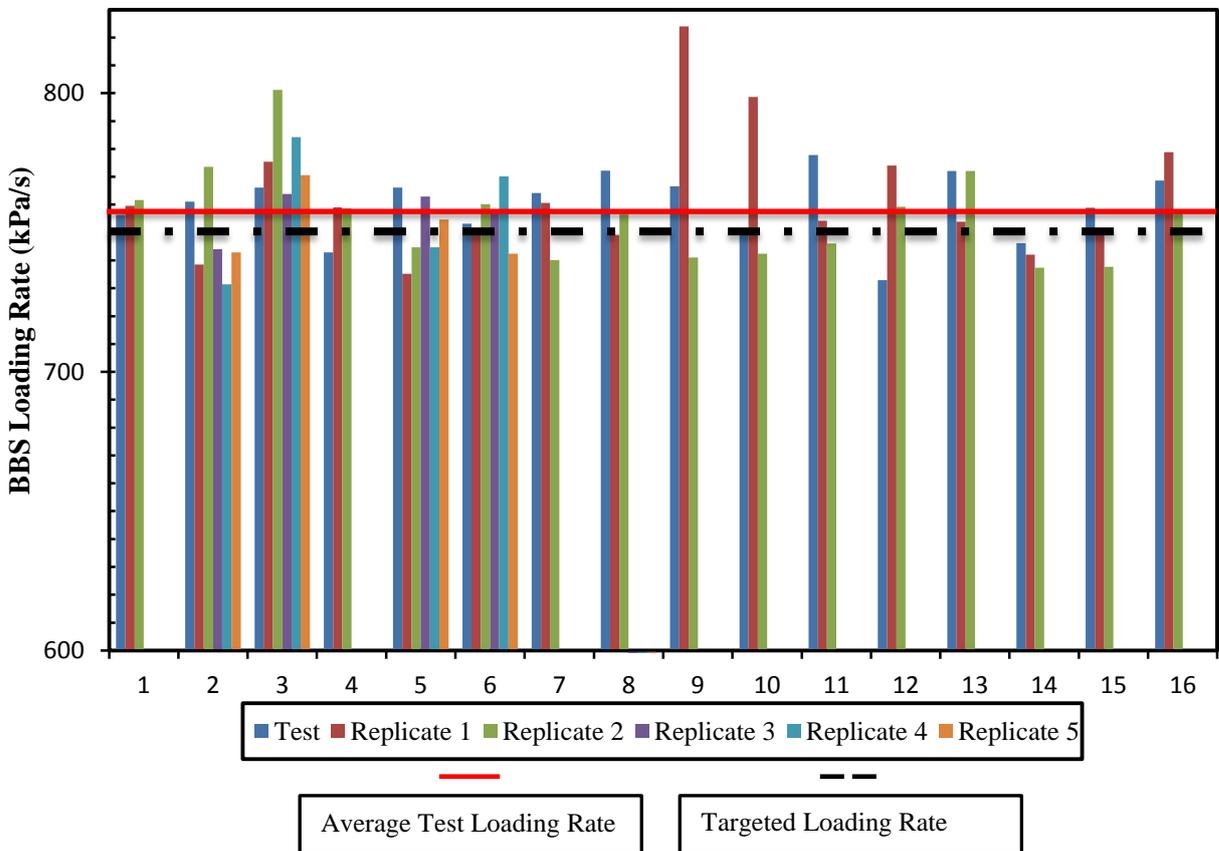


Figure 4-9: Representation of BBS Loading Rate in AwC precoated combinations testing.

Table 4-8: Combinations and their corresponding symbols for CwC precoated Category

Symbol	Combinations	Symbol	Combinations	Targeted Loading Rate (kPa/s)	Average Test Loading Rate (kPa/s)
1	G-CwC-Wet-70/100-24hrs	9	D-CwC-Wet-70/100-24hrs	750	756.5
2	G-CwC-Dry-70/100-24hrs	10	D-CwC-Dry-70/100-24hrs		
3	G-CwC-Wet-S-E1-24hrs	11	D-CwC-Wet-S-E1-24hrs		
4	G-CwC-Dry-S-E1-24hrs	12	D-CwC-Dry-S-E1-24hrs		
5	G-CwC-Wet-SC-E1-2hrs	13	D-CwC-Wet-SC-E1-2hrs		
6	G-CwC-Wet-SC-E1-24hrs	14	D-CwC-Wet-SC-E1-24hrs		
7	G-CwC-Dry-SC-E1-2hrs	15	D-CwC-Dry-SC-E1-2hrs		
8	G-CwC-Dry-SC-E1-24hrs	16	D-CwC-Dry-SC-E1-24hrs		

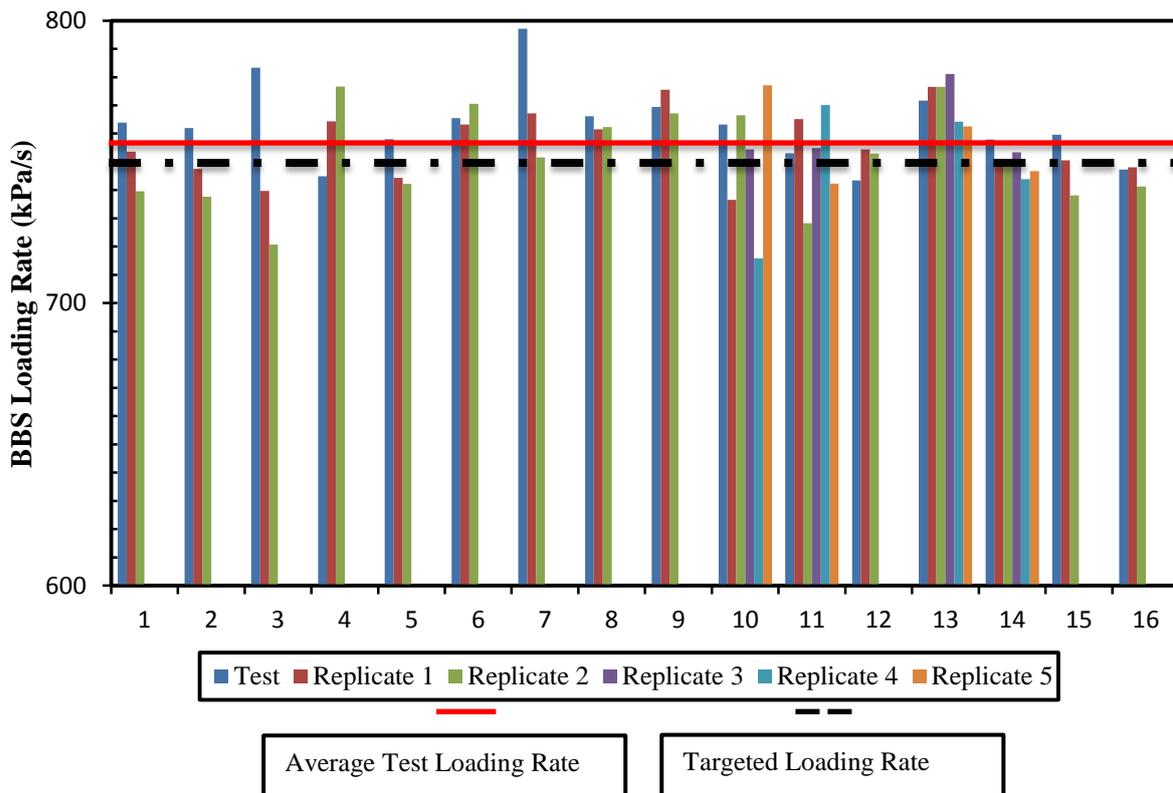


Figure 4-10: Representation of BBS Loading Rate in CwC precoated combinations testing.

In Figures 4-6, 4-8, 4-9 and 4-10, it is clear that, for the above four categories the average loading rate was very close to the targeted loading rate. The analysis of the results presented above shows that more or less 80% of the tests performed, were carried out with a loading rate that is in the range of 750 ± 20 kPa/s. Moreover, it was observed that 5% of the tests were carried out at a loading rate that is out of the range of 750 ± 35 kPa/s but only 0.1% is out of ± 50 kPa/s of the targeted loading rate. Even though a significant effort was made to reduce the variability in loading rate, it is clear that testing on a predetermined loading rate is practically impossible by using the BBS test apparatus in its current status. Therefore, there is a need to investigate how much the variation of a unit loading rate influence the change in BBS value by using a test like DSR tensile testing for which the loading rate can be controlled accurately as mentioned in recommendations.

4.2.2 BBS Testing Apparatus Repeatability Analysis

In any analysis, the tests results are always associated with some variations. For the analyst to ascertain and trust the suitability of the obtained test results, he/she must be able to quantify and understand the results variabilities and their sources. This helps to know whether the measurement system needs an improvement or not. The sources of variations in a given tests set results are different, but they can be categorized as shown in Figure 4-11.

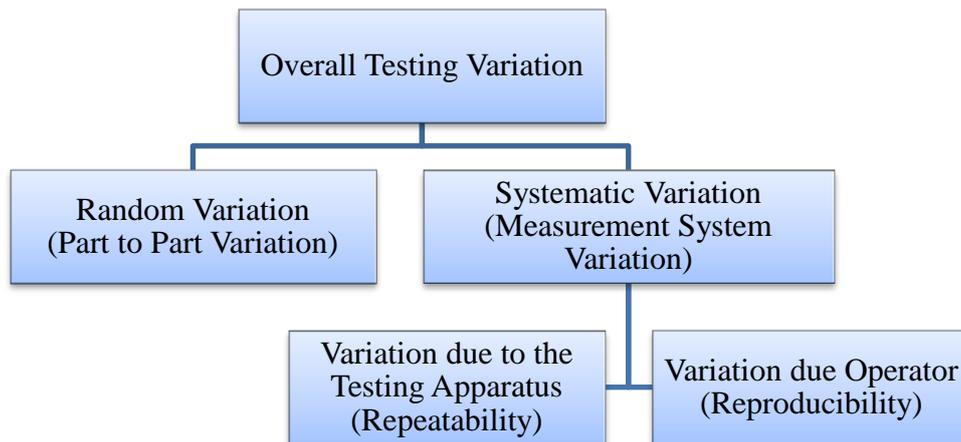


Figure 4-11: Illustration of Sources of Variation in any Tests set Results (Variation Components)

In this study, the emphasis was placed on the analysis of variations due to the testing apparatus. It is generally known as “Repeatability”. As has been mentioned in section 3.4, the BBS test was recently developed through a partnership of three universities including Stellenbosch University.

Part of this research serves as a continuous involvement of Stellenbosch University into BBS test development, by investigating the practicability and repeatability of the BBS test. The repeatability of a measurement system is also known as the inherent precision of the testing apparatus.

It is defined as the variation observed when the same item is measured multiple times by the same person (Operator). This is nonetheless only possible for a non-destructive test, because for destructive test like BBS test, it is practically impossible to measure one item multiple times. In the case of non-destructive test, most of the time the repeatability is analysed together with other variations through Gage repeatability and reproducibility (GR&R) study. In GR&R studies, the analysis methods frequently used are range and average method, and analysis of variance (ANOVA) method. The latter is known to be the most accurate method and was adopted for this study.

Douglas and Keith, (2002) in article titled “Measurement System Analysis and Destructive Testing”; have shown that if some assumptions are made, the same analysis method can also be used for destructive test. Here, this method was adopted after developing the following assumptions:

- i. The BBS specimen prepared under the same conditions by using the same materials are alike enough, in such a way that, they can be considered as one item. The variations observed in the measurement of BBS of such specimen are representative of the testing apparatus error rather than item-to-item variability.
- ii. Each group of similar specimen is assumed to be homogeneous. This is because the within-part variation will be used to estimate the repeatability of the measurement system.

To achieve this, a detailed material experiment was developed in this study. Eight sets of material combinations were selected randomly and are named “Parts”. In each part, six similar specimens were prepared and are considered to be like one item and are called “Trials”. Only one operator carried-out all the tests in every part. Thus, in this study, a total of 48 observations were used for repeatability analysis.

In the analysis of the repeatability, the Minitab software was used through the function of Gage R&R study. It should be noted that, by using the ANOVA technique, the variability observed in a test are analysed through their respective contributing components. The variation components are shown in Figure 4-11. In this study, the intention was to analyse the BBS test repeatability. Given that, only one operator carried-out all the tests, in the analysis the operator’s factor was omitted as it is related to reproducibility. The following figures are the results of BBS repeatability analysis. The Minitab report is first presented as Gage R&R study-ANOVA Method. Thereafter, the graphical representation follows.

Gage R&R Study - ANOVA Method

* NOTE * There are no operator values, or they are all the same. The operator factor will be omitted from the analysis.

Gage R&R for E2E Time (Measurement Data)

Gage name: BBS Test

Date of study: 13/08/2014

Reported by: ETwagirimana

Tolerance:

Misc:

One-Way ANOVA Table

Source	DF	SS	MS	F	P
Case Inspect	7	19727850	2818264	399.115	0.000
Repeatability	40	282452	7061		
Total	47	20010301			

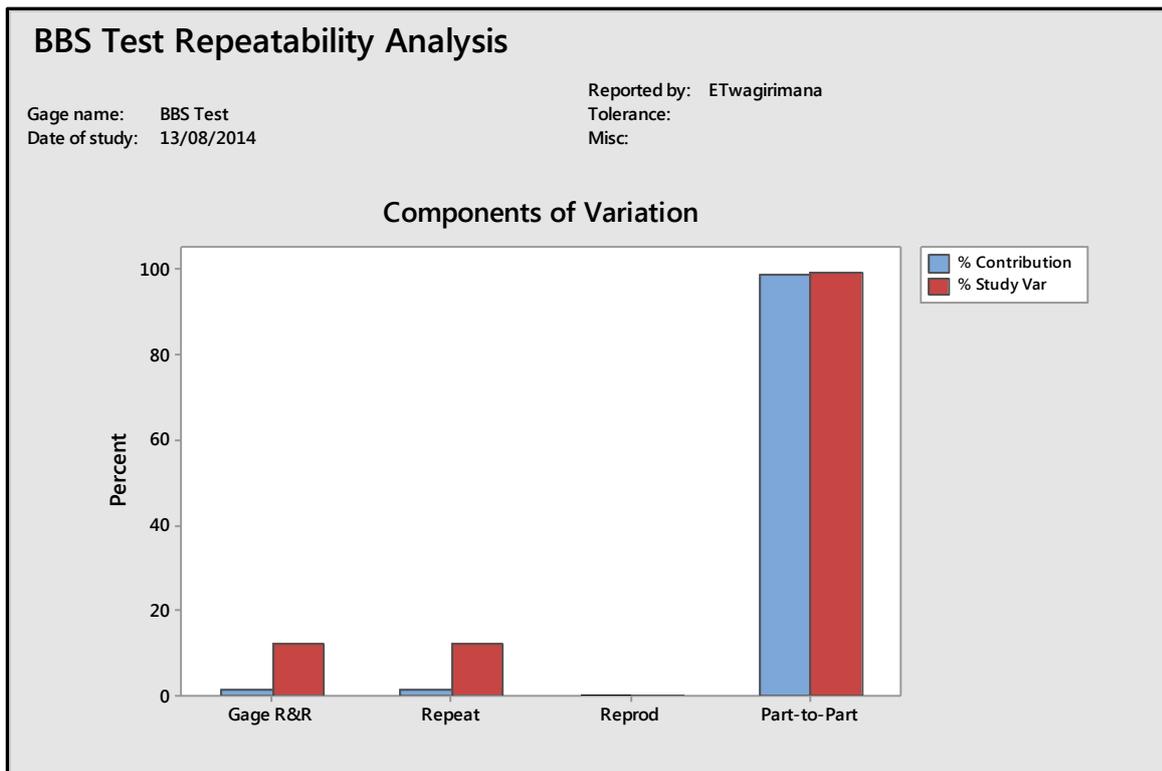
α to remove interaction term = 0.05

Gage R&R

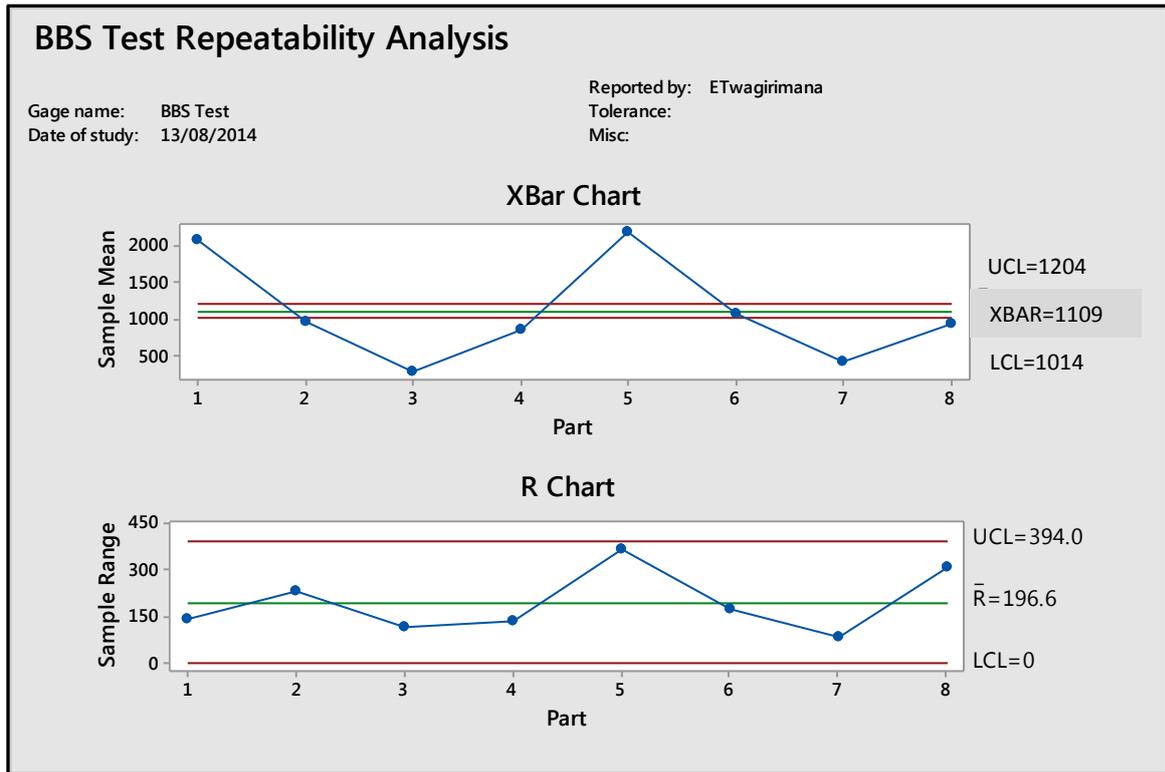
%Contribution		
Source	VarComp	(of Var Comp)
Total Gage R&R	7061	1.48
Repeatability	7061	1.48
Part-To-Part	468534	98.52
Total Variation	475595	100.00

Study Var %Study Var			
Source	StdDev (SD)	(6 × SD)	(%SV)
Total Gage R&R	84.031	504.19	12.18
Repeatability	84.031	504.19	12.18
Part-To-Part	684.495	4106.97	99.25
Total Variation	689.634	4137.80	100.00

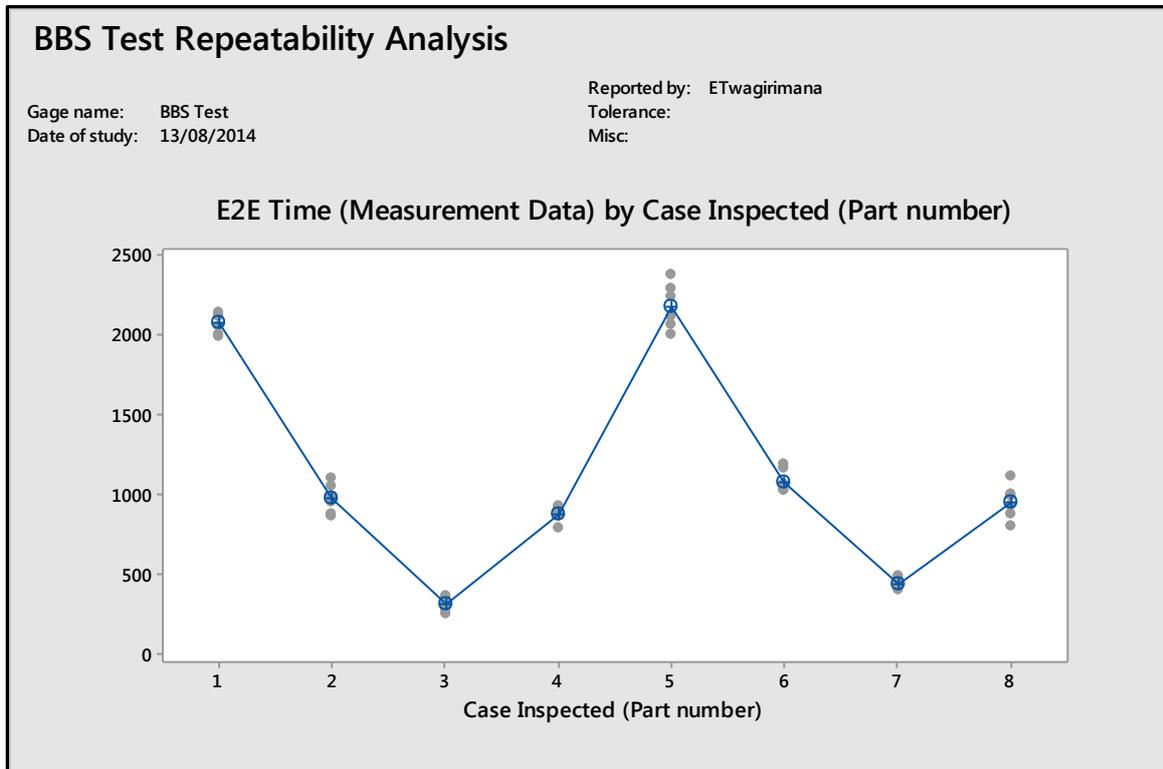
Number of Distinct Categories = 11



(a)



(b)



(c)

Figure 4-12: BBS Test Repeatability Analysis Results

The interpretation of BBS test repeatability analysis is based on the 3 golden rules of Gage R&R. These rules are as stated below (Douglas & Keith, 2002; Raushan, 2011):

- i. Gage R&R as a percentage of contribution toward total variation should be smaller than part-to-part variation.
- ii. Gage R&R as a percentage of tolerance toward total variation:
 - If less than 10% of total variation, then measurement system is accepted.
 - If between 10-30% of total variation, then measurement system may be accepted with caution.
 - If more than 30% of total variation, then measurement system is rejected.
- iii. Number of distinct categories should be equal to or great than 4.

Prior to relating the results in the report to the 3 golden rules of Gage R&R, the graphs obtained in the analysis are first interpreted. The Figure 4-12 (a) represents the components (sources) of variations that were observed in the tests results. Therefore, this figure illustrates the percentage from total for different components. The figure shows also that, the Gage R&R variation is small compared to the part-to-part variation. Thus, the first golden rule of Gage R&R is fulfilled. This means that most of the variation observed in the tests results are from part-to-part or from difference in material combinations, rather than from the measurement system.

Another observation that can be made from the Figure 4-12 (a) is that, the contribution of reproducibility is equal to zero. This is due to the fact that, all the tests were carried-out by one operator. Thus, the reproducibility could not be determined and the Gage R&R in this study corresponds to the BBS testing apparatus repeatability. The contribution of the measurement system (testing apparatus) is very clear but it is very small compared to the contribution of par-to-part variations.

The red columns represent the % study variation of each component relatively to the total variation. The observation shows that the % study variation of Gage R&R is 12.18%, which falls in the second option of the second Gage R&R golden rule. This means that, the BBS test is acceptable with caution. It is thought that, this is caused by the variation of loading rate and some aspect of specimen preparation. It should be pointed out that; the 10-30% range was fixed based on non-destructive test. Thus, it may be somehow tolerable to have some small deviations for destructive test.

Figure 4-12 (b) illustrates two charts. R-Chart also known as “Range Control Chart” is used to analyse the repeatability of the measurement system. XBar chart is used to analyse the part-to-part variations. In the R Chart, each point represents the range of variation for a specific part (group of six similar specimens). As no single point is falling outside the range control limits, it proves that an operator can run tests repeatedly with BBS testing apparatus without problem.

In the XBar chart, each point represents the average of the six observations in each part. It is clear that only one point falls within the control limit. Others are outside the control limits, which prove that there is a significant variation between parts. Therefore, the BBS testing apparatus is able to detect the variations in bond strength due to various material combinations. Figure 4-12 (C) represents the measurements by case inspected. From this figure, the part that has the most or the least observations can be identified. The more tightly clustered the points, the least variation in the measurement.

Based on the tests run by using BBS testing apparatus the following statement can be made in regards to the BBS test repeatability:

- i. The first golden rule of Gage R&R is fulfilled as shown by the graphs discussed above and the values shown in the Gage R&R study: ANOVA Method report surrounded by a blue circle.
- ii. The second golden rule of Gage R&R is fulfilled with caution as discussed previously and shown by the value surrounded by a red circle in the Gage R&R study: ANOVA Method report.
- iii. The third golden rule of Gage R&R is fulfilled as shown by the value surrounded by the green circle in the Gage R&R study: ANOVA Method report which is greater than 4.

Therefore, it can be concluded that BBS test is a repeatable test as shown on figure 4-12 (b). However, some caution should be made to avoid some variability which can result from loading rate variability and sample preparation inconsistency.

4.3 FACTORS INFLUENCING THE BITUMEN-AGGREGATE ADHESION BOND STRENGTH

In this section, the influences of the factors that are under scrutiny on the bitumen-aggregate bond strength are discussed, analysed and interpreted individually. The graphical representation is used to depict the influence of each independent variable on the dependent variable (or response), which is BBS in this study.

To achieve this, similar material combinations are grouped together. The influence of the variable under consideration is determined by keeping other variables constant for that particular case. Thereafter, the change in BBS due to the variation in that particular variable under consideration is examined and interpreted. In order to analyse the compatibility between various material combinations and the interactions between different variables, the statistical analysis of variance (ANOVA) was used. The following sections discuss the influence of each individual variable. Prior to discussing the influence of the variables, the observations made during precoating phase will first be presented and discussed.

4.3.1 Aggregate Precoating

The precoat fluid types used in this study and how they were applied were described in sections 3.2.2 and 3.5.2 respectively. It should be reminded that the precoat application rate adopted was mimicking the field precoating as explained in literature study in section 2.2.1.3. The intention here is to present the observations made during and after aggregate precoating as well as after precoat curing.

In this study for the precoating uniformity quality control, the weight of aggregate was recorded before and after precoating for all 216 specimens. The weight after precoat curing was also recorded to investigate the effect of precoat curing condition. The records for precoat application rate control are shown in Appendix A. The records show no statistical significant difference between the precoat applied. Thus, the precoat application rate is considered to be uniform throughout the test.

During the precoat application phase it was realised that, the viscosity of precoats were different. The 30% water diluted SS60 is less viscous than other two precoating fluids. This renders it able to flow easily and facilitates its uniform distribution to the aggregate surface. The high viscosity of anionic emulsion with cutters (AwC) and cationic emulsion with cutters (CwC) precoats caused them to require more time for uniform spreading on the aggregate surface. To link this with the precoating process on the field as described in section 2.2.1.3, care should be taken for AwC and CwC. This concerns the precoat spray while the aggregates are in the bucket of the front end of the loader as well as during the stockpile turning over process. Otherwise, there might be a risk of non-uniformity of aggregate precoating, especially at cool weather.

As has been aforementioned, immediately after precoating, the precoated aggregates were cured in a specific condition. This was dependent on the condition to be investigated, either wet or dry. In this section the author has preferred to use the figures to illustrate what was observed immediately after precoating and after precoat curing.

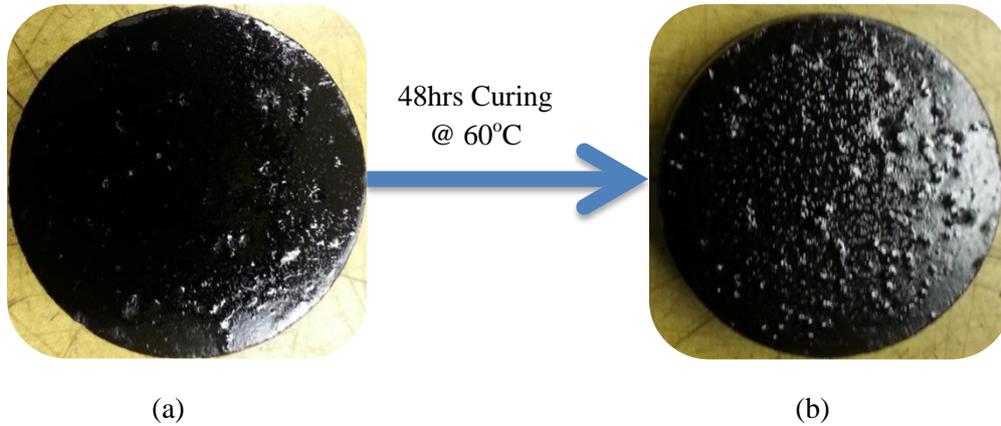


Figure 4-13: Visualisation of (a) Dolerite freshly precoated with SS60 and (b) its appearance after 48hrs @ 60°C.

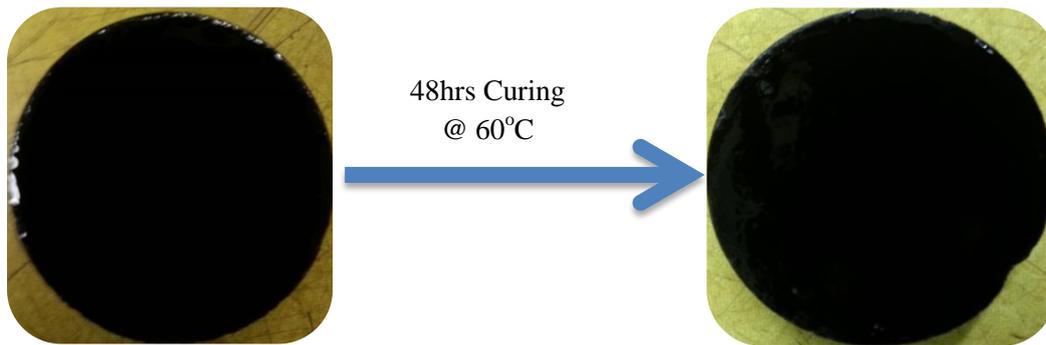


Figure 4-14: Visualisation of an aggregate surface precoated with AwC before and after 48hrs curing @ 60°C

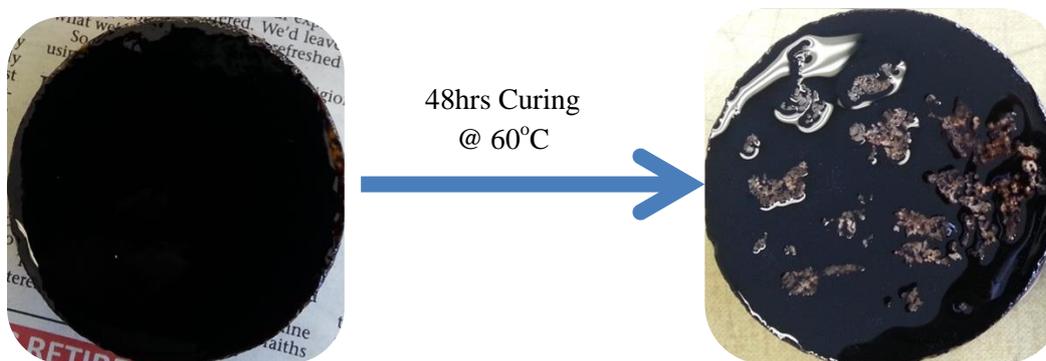


Figure 4-15: Visualisation of an aggregate surface precoated with CwC before and after 48hrs curing @ 60°C

The observations of the precoated aggregates before and after precoat curing have revealed different behaviour of precoat types relatively to curing conditions. In the following section, the observations are discussed based on the precoat type.

- i. SS60 (30% water diluted): This precoat type due to its low viscosity, after its application on the aggregate surface, it looks like water paint. After a short period, of time of curing, it dries and if you touch it, no colour mark left on your finger. There was no visible difference observed between the wet and dry cured SS60 precoated aggregates. Its advantage is that, it can be used within a very short period of time after precoating, which can be less than 3 or 4 days described in section 2.2.1.3. The time may depend on the environmental conditions the combination is exposed to, during curing time. There is no risk of aggregate to stick together because the residual binder is not tackier. Figure 4-13 illustrates the SS60 precoated aggregate before and after dry curing.
- ii. Anionic emulsion with cutters (AwC): This precoat fluid was more viscous compared to SS60 (30% water diluted). The appearance of the precoated aggregate after wet and dry conditioning was the same. However, the wet cured aggregates were tackier than the dry cured aggregates. This is due to the fact that, the cutters were not allowed to evaporate under wet condition curing (24hrs @ 20°C) and the residual bitumen remained softened. In winter period, the ambient temperature reach hardly 20°C, which shows that, the situation may be even worse. The dry cured aggregates have shown slight tackiness behaviour. This is because the cutters were allowed to evaporate under dry conditions (48hrs @ 60°C) but probably not all has evaporated.
- iii. Cationic emulsion with cutters (CwC): Similarly, to AwC, this precoat fluid was more viscous than SS60 (30% water diluted). The CwC and AwC have shown almost similar appearance and tackiness under wet curing conditions. However, under dry curing conditions, the behaviour was completely different. Figure 4-13, Figure 4-14 and Figure 4-15 prove that CwC precoat under dry curing condition has a particular interaction with aggregate compared to the aforementioned precoats. Figure 4-15 shows how the CwC precoats applied on aggregate surface and cured under dry conditions, “open-up” during curing and agglomerate themselves in some specific areas, leaving others unattended.

In an effort to interpret this behaviour, there is need to step back in the Chapters of literature and methodology. In methodology, section 3.2.2 and 3.5.2, the composition of CwC was described in details. One thing to be noted here is the fact that CwC is a cationic emulsion with some cutter contents. Therefore, the bitumen droplets in this emulsion are positively charged.

The section 2.2.1.3, has described the concept of charge development on the aggregate surface and the presence of active sites on the aggregate surface. It has been mentioned that the aggregates containing silica (SiO₂) have a negatively charged surface. Based on these theories, the above-mentioned behaviour may be interpreted as follows:

There is inherent repulsive force between positively charged droplets in a cationic emulsion. There are also active sites of significant difference in levels of activity on the aggregate surface. The localised negatively charged active sites with strong activity level attract the positively charged bitumen droplets as described by molecular orientation theory in section 2.3.3.3. At this curing temperature the emulsion residue is very soft and can easily open-up due to oriented forces. Thus, the bitumen droplets will concentrate themselves on the strong aggregate active sites and the non-competitive aggregate active sites remain unattended. The high viscosity of the residual of CwC under wet curing condition prevented the open-up of the precoat and the above-mentioned behaviour was not observed for wet curing condition. It should be pointed-out that, the active sites oriented forces of attraction are absent in the case of AwC and SS60 as they are negatively charged. Thus, the open-up behaviour was not observed for the AwC and SS60 precoats. The opening-up behaviour was observed on both granite and dolerite aggregates. The influence of open-up behaviour on the adhesive bond strength between bitumen binder and aggregate is discussed in the subsequent section.

4.3.2 Effect of Binder Types

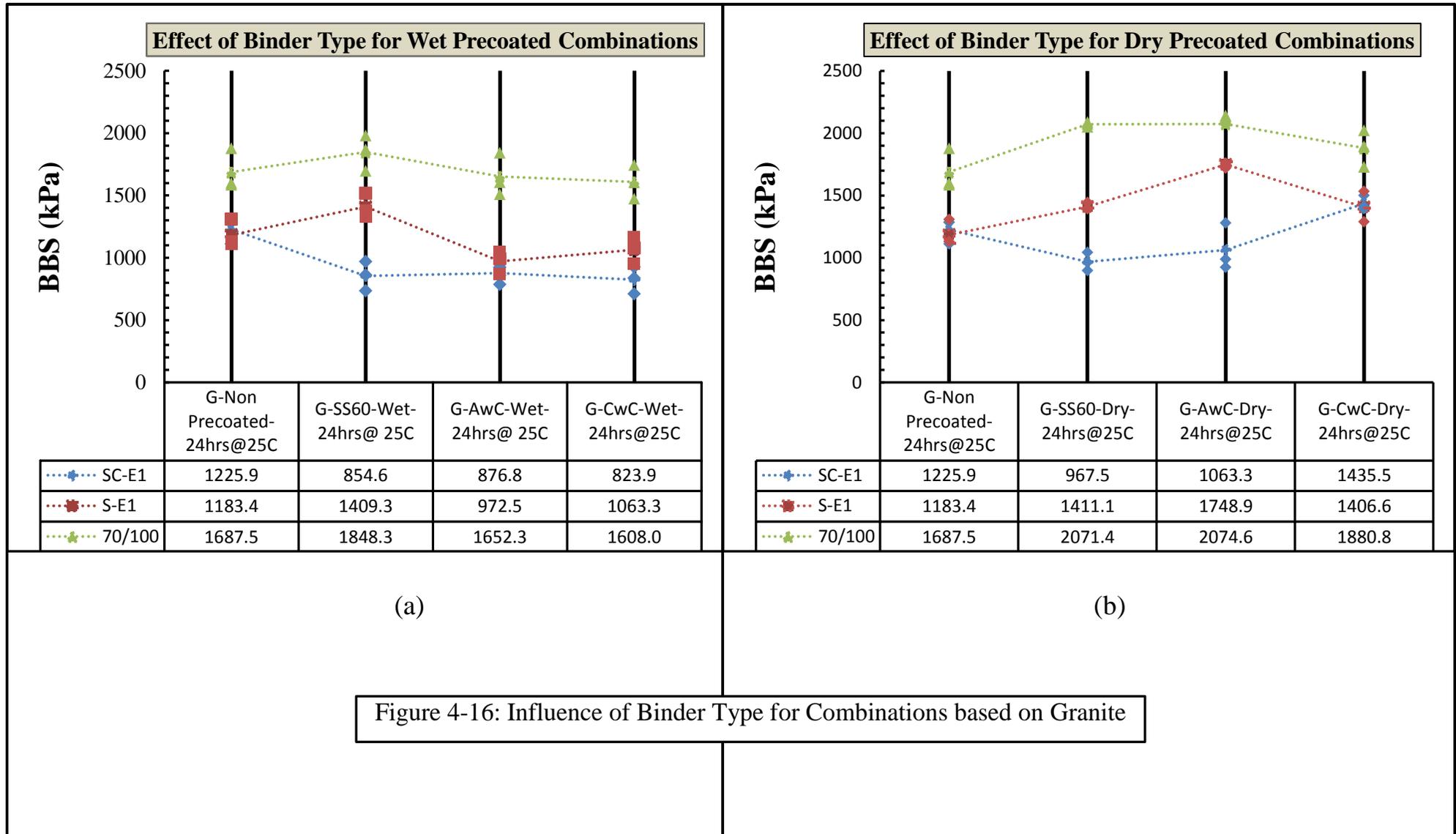
As has been aforementioned in section 3.3, two hot applied binders (70/100 and S-E1) and one cold applied binder (SC-E1) were used in this study. Due to the number of variables and material combinations used in this study, there is a need to categorize combinations, in order to be able to depict the influence of binder type. In this regard, the combinations were grouped into two main categories based on aggregate types, which are granite and dolerite.

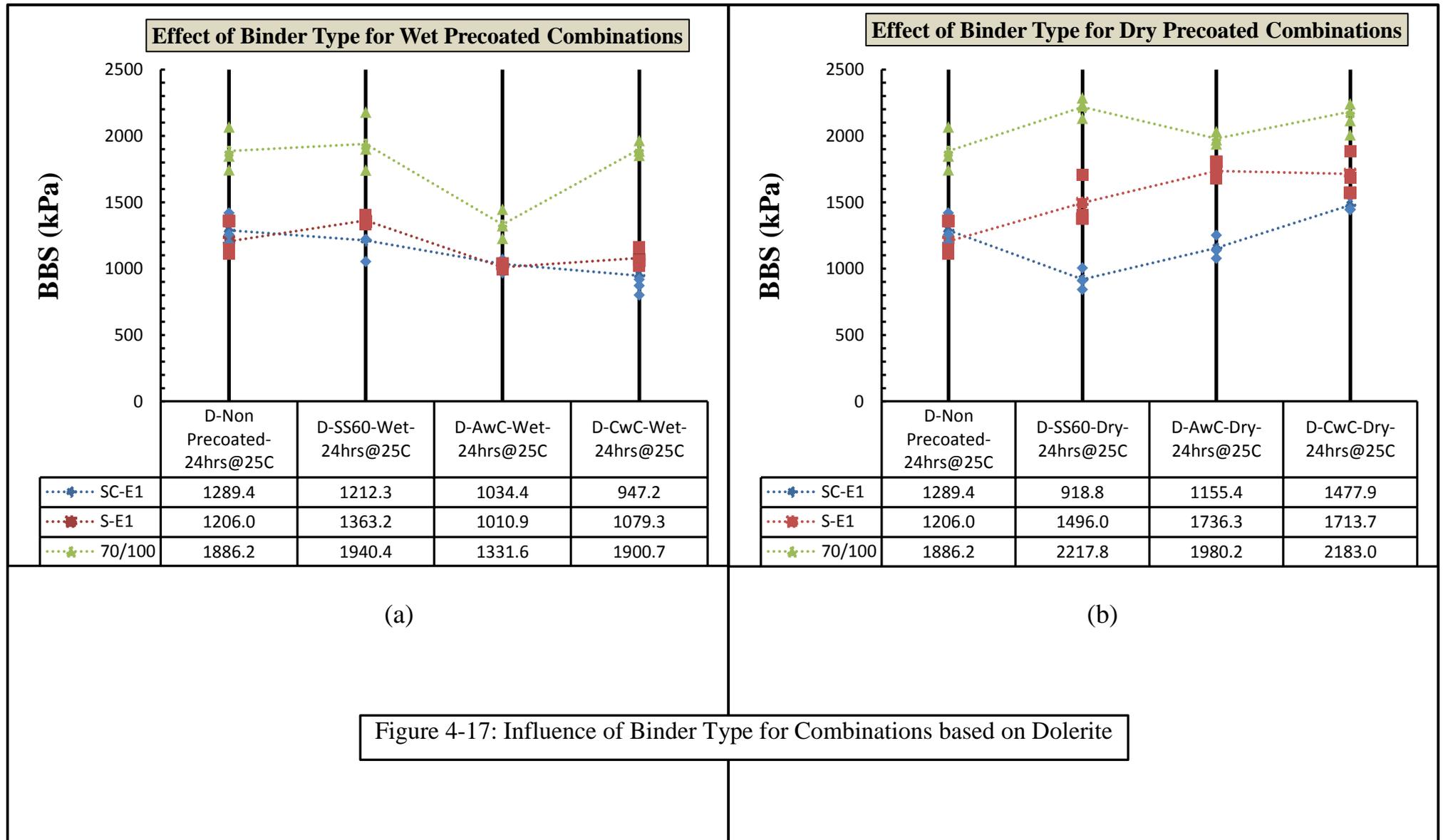
Moreover, under each main category, the combinations were grouped into two main sub-categories based on the precoat conditioning type; these are either wet or dry condition. Therefore, the effect of binder type was presented here into four different sub-categories. Within each sub-category the material combinations are grouped into four different classes which are based on precoat fluid type. Thus, varying the binder type on each material combination class helps to depict very easily the influence of binder type on bitumen-aggregate bond strength under the testing conditions considered in this study.

Prior to the presentation of binder type influence, the data used to provide the graphical representation are shown in Table 4-9. The table 4-9 is a rearrangement of tables 4-2, 4-3 and 4-4.

Table 4-9: Summary of data used for analysis of influence of the bitumen binder type on bitumen-aggregate bond strength

INFLUENCE OF BINDER TYPE ANALYSIS														
GRANITE														
Combinations			SC-E1				S-E1				70/100			
			BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV
G-Non Precoated-24hrs@25C			1225.9	8.2%	749.6	1.9%	1183.4	9.1%	761.9	4.5%	1687.5	9.7%	776.4	1.3%
G-SS60-Wet (15C)-24hrs@ 25C			808.5	14.0%	759.2	0.8%	1374.0	4.8%	753.8	0.7%	1802.2	4.7%	750.5	0.4%
G-SS60-Wet-24hrs@ 25C			854.6	13.8%	755.1	1.6%	1409.3	6.8%	761.7	1.9%	1848.3	7.8%	783.3	3.8%
G-SS60-Dry-24hrs@25C			967.5	7.6%	747.9	1.4%	1411.1	2.0%	777.8	0.9%	2071.4	0.9%	756.2	5.0%
G-AwC-Wet-24hrs@ 25C			876.8	5.7%	755.6	1.3%	972.5	9.6%	776.9	1.8%	1652.3	10.4%	759.2	0.3%
G-AwC-Dry-24hrs@25C			1063.3	17.8%	759.3	1.6%	1748.9	1.5%	753.5	1.2%	2074.6	3.1%	748.6	2.1%
G-CwC-Wet-24hrs@25C			823.9	13.2%	766.4	0.5%	1063.3	10.0%	748.0	4.3%	1608.0	8.4%	752.4	1.6%
G-CwC-Dry-24hrs@25C			1435.5	4.0%	763.3	0.3%	1406.6	8.7%	762.0	2.1%	1880.8	7.8%	749.0	1.6%
DOLERITE														
D-Non Precoated-24hrs@25C			1289.4	9.1%	751.0	0.4%	1206.0	11.1%	729.8	2.2%	1906.1	15.0%	757.9	2.1%
D-SS60-Wet (15C)-24hrs@ 25C			1172.6	1.8%	753.2	0.5%	1309.9	10.2%	764.4	1.3%	2078.7	17.2%	749.0	1.5%
D-SS60-Wet-24hrs@25C			1212.3	12.8%	751.6	1.2%	1363.2	2.6%	771.9	1.7%	1940.4	11.4%	744.1	2.7%
D-SS60-Dry-24hrs@25C			918.8	8.9%	766.6	1.8%	1496.0	12.2%	773.9	3.7%	2217.8	3.5%	746.5	1.7%
D-AwC-Wet-24hrs@25C			1034.4	5.4%	741.9	0.6%	1010.9	2.3%	759.4	2.2%	1331.6	8.3%	766.3	3.3%
D-AwC-Dry-24hrs@25C			1155.4	7.6%	768.4	1.4%	1736.3	3.6%	755.4	2.8%	1980.2	2.4%	763.6	4.0%
D-CwC-Wet-24hrs@25C			947.2	11.4%	750.0	0.7%	1079.3	7.2%	752.3	2.5%	1900.7	3.0%	770.8	0.6%
D-CwC-Dry-24hrs@25C			1477.9	3.9%	745.5	0.5%	1713.7	9.2%	750.3	0.8%	2183.0	6.5%	752.3	3.0%





The analysis of Figure 4-16 and Figure 4-17 illustrate that generally, the hot applied binders performed better than the cold applied binder. Under wet conditioning of precoat, which is more or less simulating the precoat curing in winter, no clear difference was observed between S-E1 and SC-E1 performance as illustrated in Figure 4-16 (a) and Figure 4-17 (a). The exception was observed for the combinations in which SS60 was used as a precoat. It seems that there is a generalized incompatibility between SS60 as a precoat and SC-E1 as a binder relative to the corresponding non precoat combination.

The interpretation that is given to the difference in performance for 70/100 and S-E1 is the fact that modification increases the softening point of S-E1, which decreases the wettability and thus results into poor bond development. However, there is a need to investigate the interaction between the cutters and the polymer. This is due to the fact that the performance of S-E1 on SS60-Wet compared to its performance on other precoat fluid in wet conditions could be questioned.

The results of SC-E1 could be interpreted by the fact that, the presence of water did not allow a quick bond development. Thus, the investigation on extended curing time is required to be able to draw a solid conclusion. The hot applied binder performed better than cold applied binder but in all the cases 70/100 is the highest performer.

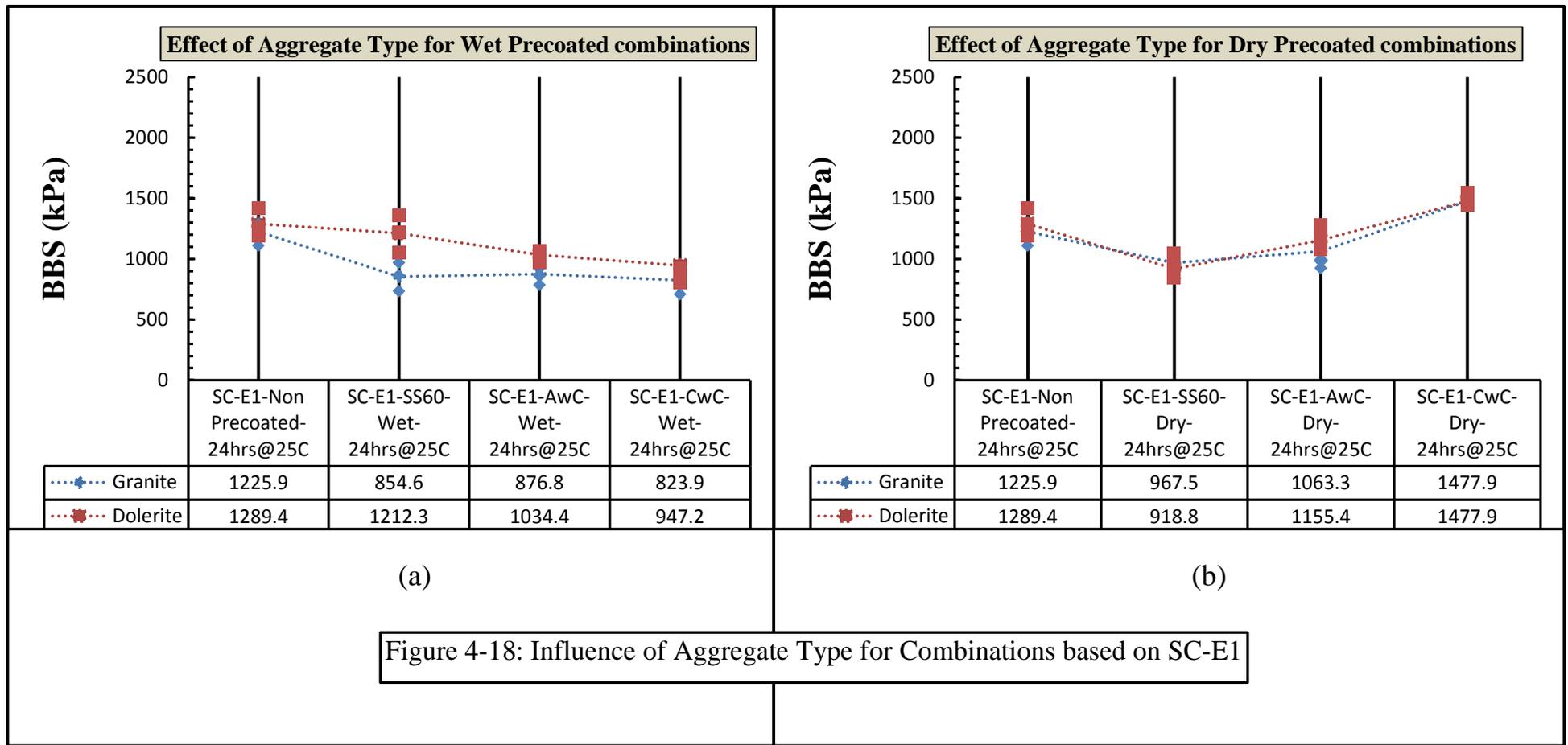
4.3.3 Effect of Aggregate Types

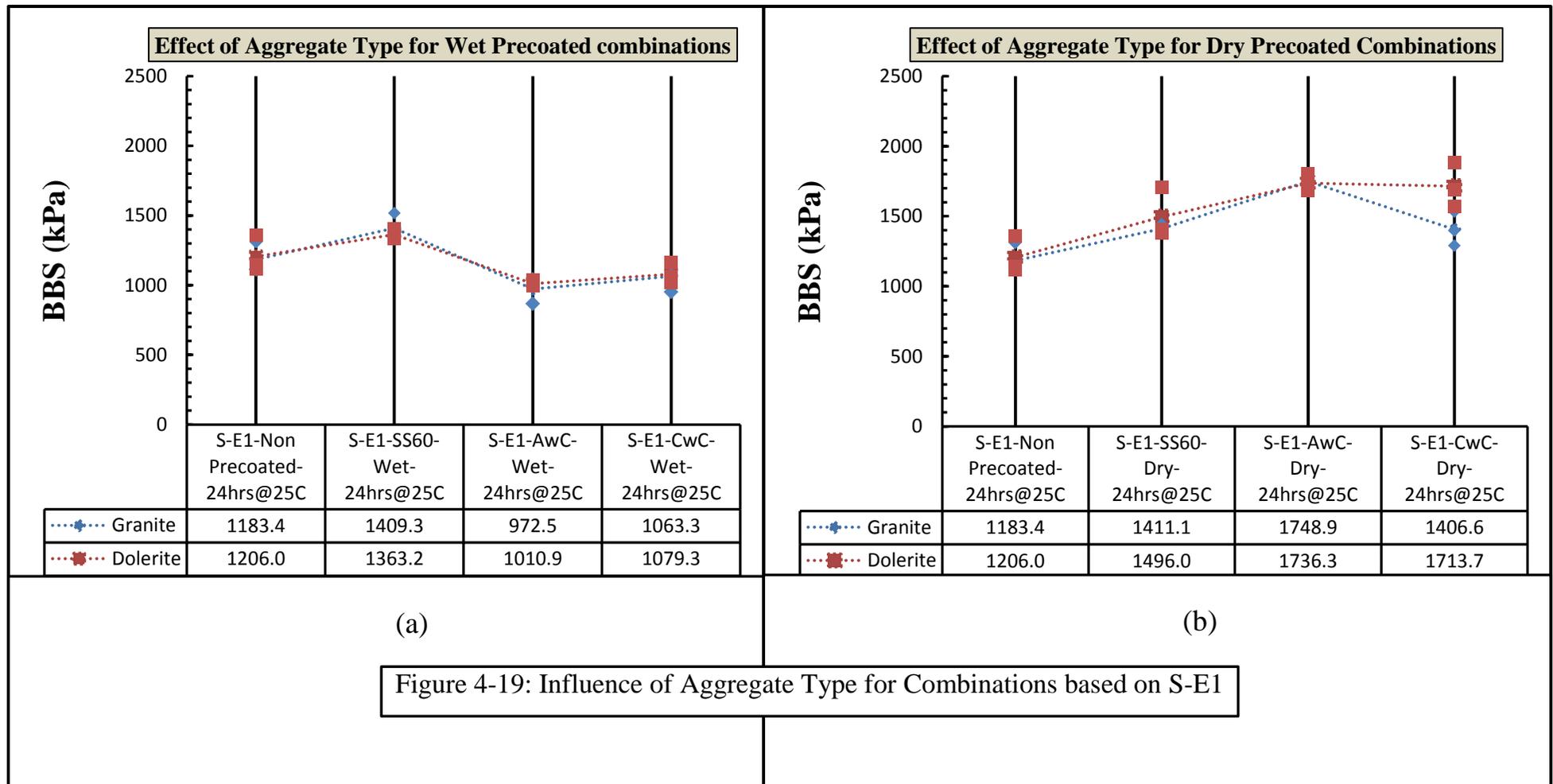
In this study, two types of aggregates were used. There is granite that is known to have an acidic behaviour and dolerite known to have a basic character. Similarly, to what was explained in the analysis of effect of binder type, here also the material combinations were grouped into categories.

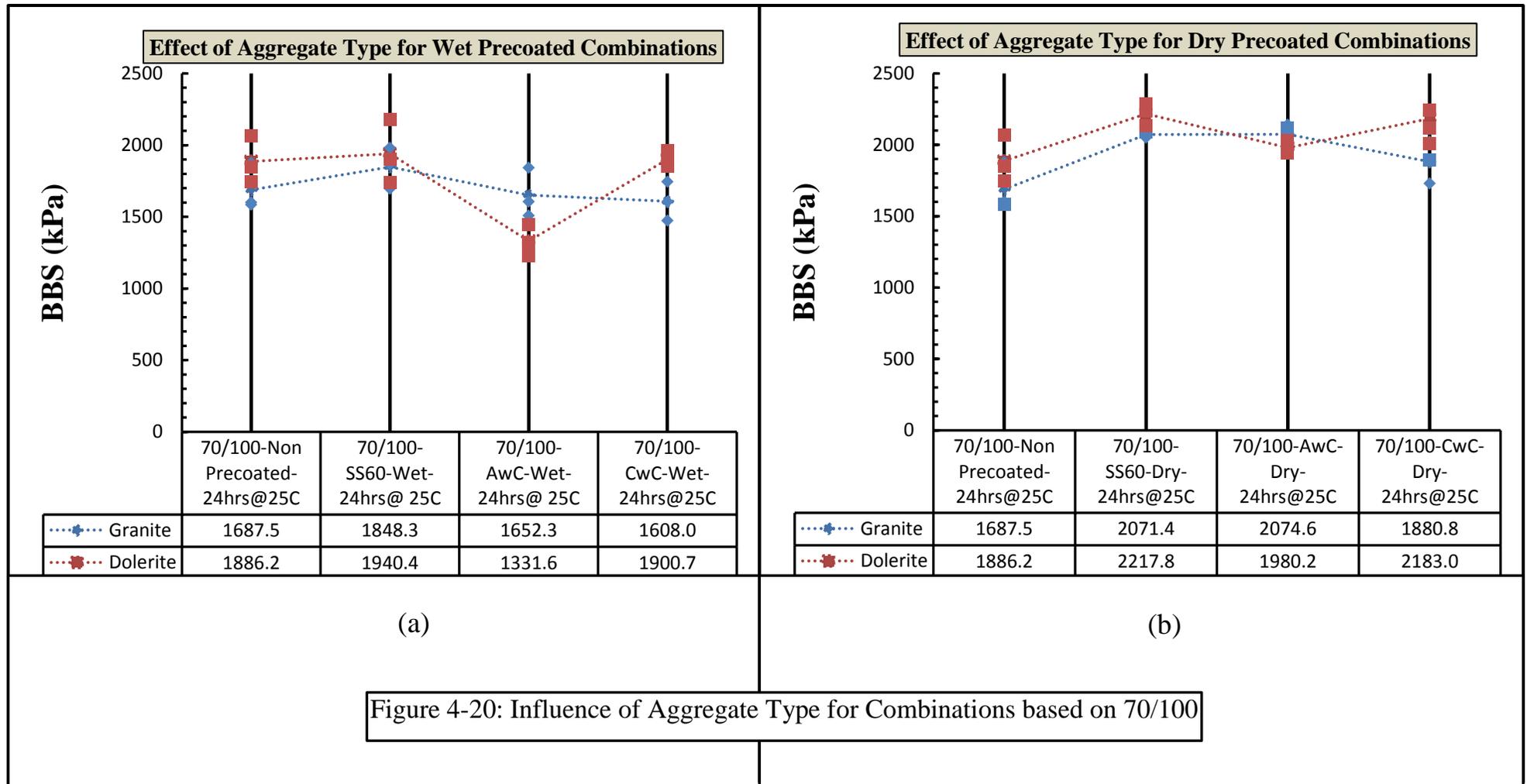
Three main categories were identified based on the bitumen binder types. In each category, the combinations were further subdivided into two subcategories based on precoat conditioning. Thus, the influence of aggregate type was analysed into six different combination sub-categories. Moreover, each sub-category has four different classes elaborated based on precoat fluid type. Prior to the discussion on aggregate type influence, the tables 4-2, 4-3 and 4-4 were first rearranged into Table 4-10. The latter summarises the data used for aggregate type influence analysis.

Table 4-10: Summary of data used for analysis of influence of the aggregate type on bitumen-aggregate bond strength

EFFECT OF AGGREGATE TYPE											
70/100											
Combinations				Granite				Dolerite			
				BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV
70/100-Non Precoated-24hrs@25C				1687.5	9.7%	776.4	1.3%	1906.1	15.0%	757.9	2.1%
70/100-SS60-Wet (15C)-24hrs@ 25C				1802.2	4.7%	750.5	0.4%	2078.7	17.2%	749.0	1.5%
70/100-SS60-Wet-24hrs@ 25C				1848.3	7.8%	783.3	3.8%	1940.4	11.4%	744.1	2.7%
70/100-SS60-Dry-24hrs@25C				2071.4	0.9%	756.2	5.0%	2217.8	3.5%	746.5	1.7%
70/100-AwC-Wet-24hrs@ 25C				1652.3	10.4%	759.2	0.3%	1331.6	8.3%	766.3	3.3%
70/100-AwC-Dry-24hrs@25C				2074.6	3.1%	748.6	2.1%	1980.2	2.4%	763.6	4.0%
70/100-CwC-Wet-24hrs@25C				1608.0	8.4%	752.4	1.6%	1900.7	3.0%	770.8	0.6%
70/100-CwC-Dry-24hrs@25C				1880.8	7.8%	749.0	1.6%	2183.0	6.5%	752.3	3.0%
S-E1											
S-E1-Non Precoated-24hrs@25C				1183.4	9.1%	761.9	4.5%	1206.0	11.1%	729.8	2.2%
S-E1-SS60-Wet (15C)-24hrs@ 25C				1374.0	4.8%	753.8	0.7%	1309.9	10.2%	764.4	1.3%
S-E1-SS60-Wet-24hrs@25C				1409.3	6.8%	761.7	1.9%	1363.2	2.6%	771.9	1.7%
S-E1-SS60-Dry-24hrs@25C				1411.1	2.0%	777.8	0.9%	1496.0	12.2%	773.9	3.7%
S-E1-AwC-Wet-24hrs@25C				972.5	9.6%	776.9	1.8%	1010.9	2.3%	759.4	2.2%
S-E1-AwC-Dry-24hrs@25C				1748.9	1.5%	753.5	1.2%	1736.3	3.6%	755.4	2.8%
S-E1-CwC-Wet-24hrs@25C				1063.3	10.0%	748.0	4.3%	1079.3	7.2%	752.3	2.0%
S-E1-CwC-Dry-24hrs@25C				1406.6	8.7%	762.0	2.1%	1713.7	9.2%	750.3	0.8%
SC-E1											
SC-E1-Non Precoated-24hrs@25C				1225.9	8.2%	749.6	1.9%	1289.4	9.1%	751.0	0.4%
SC-E1-SS60-Wet (15C)-24hrs@ 25C				808.5	14.0%	759.2	0.8%	1172.6	1.8%	753.2	0.5%
SC-E1-SS60-Wet-24hrs@25C				854.6	13.8%	755.1	1.6%	1212.3	12.8%	751.6	1.2%
SC-E1-SS60-Dry-24hrs@25C				967.5	7.6%	747.9	1.4%	918.8	8.9%	766.6	1.8%
SC-E1-AwC-Wet-24hrs@25C				876.8	5.7%	755.6	1.3%	1034.4	5.4%	741.9	0.6%
SC-E1-AwC-Dry-24hrs@25C				1063.3	17.8%	759.3	1.6%	1155.4	7.6%	768.4	1.4%
SC-E1-CwC-Wet-24hrs@25C				823.9	13.2%	766.4	0.5%	947.2	11.4%	750.0	0.7%
SC-E1-CwC-Dry-24hrs@25C				1477.9	3.9%	745.5	0.5%	1477.9	3.9%	745.5	0.5%







The analysis of Figures 4-18, 4-19 and 4-20 does not highlight any significant influence of aggregate type on the bitumen-aggregate adhesion bond strength. In order to interpret this behaviour, there is a need to step back in the literature. The literature shows that the basicity and acidity of the two types of aggregates used here are based on the amount of SiO₂ (Silica) minerals in their composition. None of these two aggregate types contain calcite minerals in its composition. Therefore, according to section 2.2.1.1 none of these aggregates is expected to develop positive charges on the surface. Both granite and dolerite are likely to develop similar charges (negative) on the surface after cleavage. The granites (acid rocks) are predominated by the feldspar minerals (orthoclase) and quartz. On the other hand, the dolerites (basic rocks) consist also of feldspar minerals (Plagioclase type) and pyroxene (Weinert, 1980). All of the minerals stated above contain some silica (SiO₂) but in a different amount.

Making reference to section 2.2.1.1 which discusses the concept of charge development on an aggregate surface, it may be reasonable to say that their charges developments upon cleavage are more or less similar. Therefore, their surface characteristics may have some similarities. Thus, based on chemical and thermodynamic (surface energy) adhesion theory, the interaction between these two types of aggregate and the bitumen binder may be more or less similar. However, for uncoated aggregate the failure mode was different, the granite favoured adhesion failure whereas dolerite favoured cohesive failure. This might indicate that the surface physical characteristics are the main contributors to adhesion. This can be shown by the results of laser scanning that illustrates dolerite as more rough and porous than granite thus improve adhesion bond and failure by cohesion.

4.3.4 Effect of Emulsion Precoating Fluid Types and Precoat conditioning

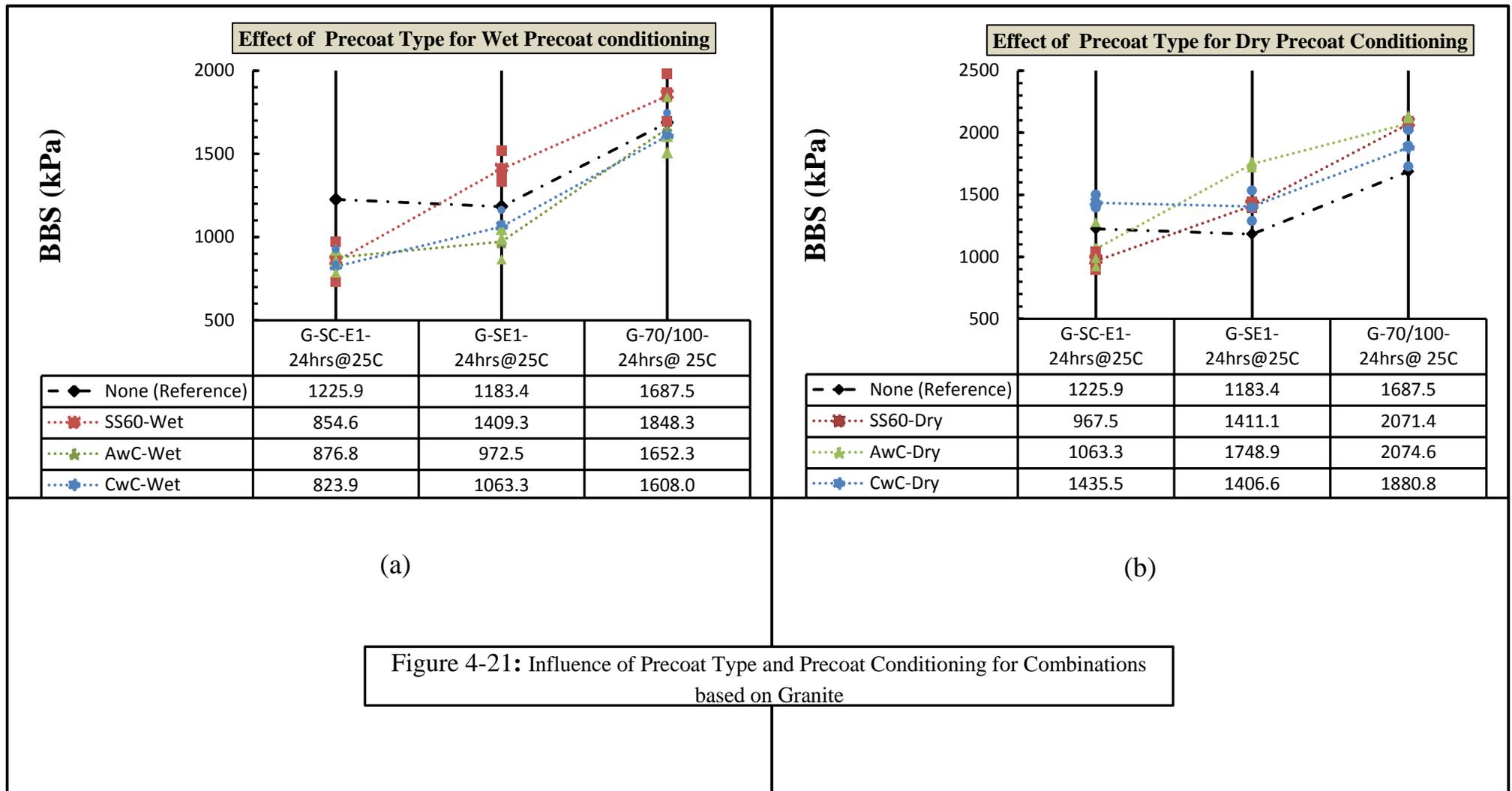
A detailed materials experiment was developed to investigate various emulsions precoating fluids performance. The experiment included three types of precoating fluids. It involved two emulsion precoat types with cutters and one emulsion precoat type without cutter.

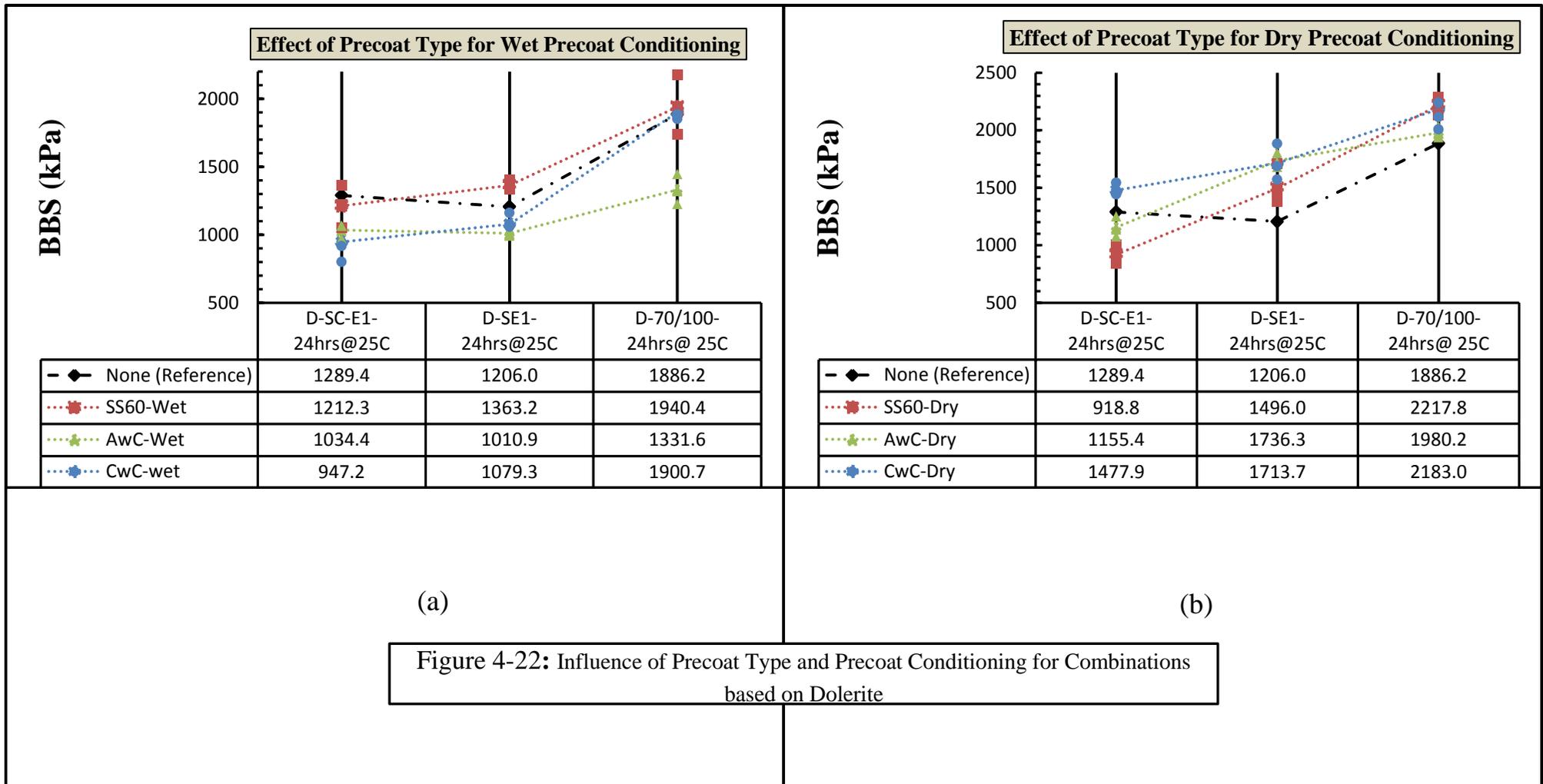
The influence of these precoats on bond strength development was investigated by subjecting them into two different curing conditions. The wet precoat curing condition and dry precoat curing conditions as discussed in sections 3.2.2, 3.3 and 3.5.2. The precoat fluids performances are evaluated relatively to the performance of the corresponding non-precoated combinations.

In order to be able to identify the precoat influence, the material combinations were grouped in two main categories based on aggregate type. Each category is subdivided in two sub-categories based on the precoat conditioning. Finally, each sub-category is divided in three classes based on bitumen binder type. Thus by varying the precoat type in each class, the effect of precoat type on bond strength development, can be easily depicted. The Table 4-11, Figure 4-21 and Figure 4-22 are the summary of the results of experiments on investigation of precoat type and precoat conditioning relatively to bitumen bond strength development.

Table 4-11: Summary of the experimental results for analysis of influence of the precoat type and precoat conditioning on bitumen-aggregate bond strength

EFFECT OF PRECOAT TYPE AND PRECOAT CONDITIONING																	
GRANITE																	
Combinations	Non-Precoated				SS60(30% Diluted)-Wet				AwC-Wet				CwC-Wet				
	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	
G-SC-E1-24hrs@25C	1225.9	8.2%	749.6	1.9%	854.6	13.8%	755.1	1.6%	876.8	5.7%	755.6	1.3%	823.9	13.2%	766.4	0.5%	
G-SE1-24hrs@25C	1183.4	9.1%	761.9	4.5%	1409.3	6.8%	761.7	1.9%	972.5	9.6%	776.9	1.8%	1063.3	10.0%	748.0	4.3%	
G-70/100-24hrs@25C	1687.5	9.7%	776.4	1.3%	1848.3	7.8%	783.3	3.8%	1652.3	10.4%	759.2	0.3%	1608.0	8.4%	752.4	1.6%	
Combinations	Non-Precoated				SS60(30% Diluted)-Dry				AwC-Dry				CwC-Dry				
	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	
G-SC-E1-24hrs@25C	1225.9	8.2%	749.6	1.9%	967.5	7.6%	747.9	1.4%	1063.3	17.8%	759.3	1.6%	1435.5	4.0%	763.3	0.3%	
G-SE1-24hrs@25C	1183.4	9.1%	761.9	4.5%	1411.1	2.0%	777.8	0.9%	1748.9	1.5%	753.5	1.2%	1406.6	8.7%	762.0	2.1%	
G-70/100-24hrs@25C	1687.5	9.7%	776.4	1.3%	2071.4	0.9%	756.2	5.0%	2074.6	3.1%	748.6	2.1%	1880.8	7.8%	749.0	1.6%	
DOLERITE																	
Combinations	Non-Precoated				SS60(30% Diluted)-Wet				AwC-Wet				CwC-Wet				
	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	
D-SC-E1-24hrs@25C	1289.4	9.1%	751.0	0.4%	1212.3	12.8%	751.6	1.2%	1034.4	5.4%	741.9	0.6%	947.2	11.4%	750.0	0.7%	
D-SE1-24hrs@25C	1206.0	11.1%	729.8	2.2%	1363.2	2.6%	771.9	1.7%	1010.9	2.3%	759.4	2.2%	1079.3	7.2%	752.3	2.0%	
D-70/100-24hrs@25C	1906.1	15.0%	757.9	2.1%	1940.4	11.4%	744.1	2.7%	1331.6	8.3%	766.3	3.3%	1900.7	3.0%	770.8	0.6%	
Combinations	Non-Precoated				SS60(30% Diluted)-Dry				AwC-Dry				CwC-Dry				
	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	BBS (kPa)	BBS CoV	L.R (kPa/s)	L.R CoV	
D-SC-E1-24hrs@25C	1289.4	9.1%	751.0	0.4%	918.8	8.9%	766.6	1.8%	1155.4	7.6%	768.4	1.4%	1477.9	3.9%	745.5	0.5%	
D-SE1-24hrs@25C	1206.0	11.1%	729.8	2.2%	1496.0	12.2%	773.9	3.7%	1736.3	3.6%	755.4	2.8%	1713.7	9.2%	750.3	0.8%	
D-70/100-24hrs@25C	1906.1	15.0%	757.9	2.1%	2217.8	3.5%	746.5	1.7%	1980.2	2.4%	763.6	4.0%	2183.0	6.5%	752.3	3.0%	





In analysing Figure 4-21 (a) and Figure 4-22(a), the following initial observations can be made. In a wet conditioning of the precoat for hot applied binders, only SS60 (30% water diluted) precoat had performed better than the similar non-precoated combinations (Reference).

However, it was not the case for cold applied binder. For the latter, all the wet precoat conditioning induced a poor performance compared to the corresponding non-precoated combinations.

The interpretation of this behaviour is assigned to the presence of cutters in other precoat types except for SS60 (30% water diluted). In wet precoat conditioning, the cutters contained in anionic emulsions with cutters (AwC) and cationic emulsions with cutters were not allowed to evaporate. Up to 24hrs of curing of the binder, the cutters were still active. Thus, the cutters softened the binder, and prevented the development of appropriate bond at the interface. The observations showed that the predominating failure type in this condition was adhesive failure.

The observations of Figure 4-21 and Figure 4-22 in both wet and dry conditions show, an incompatibility between the SC-E1 and aggregate precoated with anionic emulsion fluids (SS60 and AwC). The aggregates used contain silica as has been discussed in section 4.3.3. Based on section 2.2.1.1, it can be believed that upon cleavage the negative charge will be developed on the aggregate surface, due to the presence of silica minerals. Thus, if a negatively charged emulsion is applied, there will be resultant repulsive forces of interactions between them. Thus, they result into poor adhesion bond between the negatively charged precoat fluid and the negatively charged aggregate surface.

Moreover, due to the tendency of any system to establish the thermodynamic equilibrium with lowest energy, the water contained in SC-E1 seeks to replace the precoat on the aggregate surface. This phenomenon is known as stripping. It can be argued that, stripping together with the weak bond between the aggregate and anionic precoat leads to the poor bond strength for these specific combinations. This interpretation could be supported by the type of failure observed in this type of combinations as shown in Figure 4-23. The adhesive failure was taking place between the precoat and the aggregate surface in most cases.

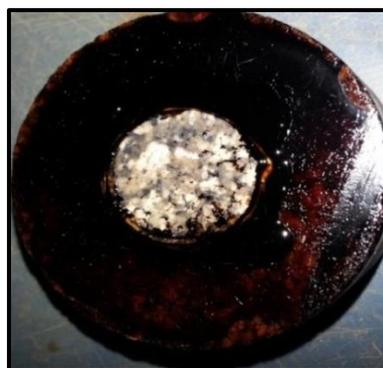


Figure 4-23: Typical Adhesive failure between SC-E1 and Aggregate precoated with anionic emulsions.

The Cationic emulsifiers generally form a strong irreversible amine-silicate bond between the Amine ($R-NH_3^+$) and the Silica (SiO_2^-) and this facilitate the compatibility between negatively charged aggregate and cationic precoat (CwC) (Louw, 2014).

In dry conditioning of the precoat for hot applied binder combinations, it is observed that all the pre-coated aggregate performed well compared to the corresponding non-pre-coated combinations. This is due to the fact that a significant proportion of the cutters were allowed to evaporate. Thus, the cutters were not strongly active and the binder developed strong bond at the interface. The good performance of AwC and CwC relative to SS60 under dry conditions can be interpreted based on the presence of adhesive agents in AwC and CwC that increases their interface bond strength.

However, a solid conclusion regarding the use of these pre-coats can be made after carrying out a cost analysis. This was not part of this research but it can be recommended for further research as it could help to fully evaluate the pre-coating benefits.

4.3.5 Effect of Curing Time

An experimental matrix was developed to investigate the influence of curing time on bond strength between SC-E1 and various pre-coats. It was intended to get an insight on the bond development rate for various material combinations under low temperature conditions. This helps to identify the materials combinations among the combinations investigated, which are likely to allow the early opening to traffic of surfacing seals in winter. It is an important aspect for both the contractor and the road users, as well as to the road authorities.

Two levels of curing time (2hrs and 24hrs) were investigated. The power curve was found to be the most fitting curve based on the R^2 value (These R^2 that show the degree of fitness were higher than those of a linear curve). Moreover, in the past, Miller (2010) carried-out an extensive investigation on the relationship between BBS values and the curing time, he found the power curve to be able to capture the relationship. In this study, there was a limitation on the number of curing time used and only two were used in the study. Based on the above mentioned reasons, the power curve was used to represent the relationship between curing time and the BBS values. The slope of the power curve connecting the strength at 2hrs and 24hrs was used to assess the bond strength development rate. In this analysis, two conditions were considered in order to analyse the performance of each combination. First, the performance of any combination was analysed relatively to the performance of the corresponding non-pre-coated combinations (It should be noted that here the non-pre-coated dolerite combinations were used as reference). Lastly, the performance of any combination was interpreted based on the minimum BBS specification value proposed by Miller (2010). Miller in his experimental testing; he has correlated the BBS and Sweep tests. Based on the existing recommended sweep test performance limit of 10% aggregate loss, the minimum BBS specification limit of 850kPa was proposed in his study. In this analysis, this proposed minimum value was compared to the results obtained. In order to facilitate the direct comparison between various combinations, two categories of graphical representation were used. Each category entails all the combinations under the same pre-coat curing conditions (wet or dry).

Table 4-12: Summary of the experimental results for analysis of influence of curing time on bitumen-aggregate bond strength for SC-E1

GRANITE			DOLERITE		
Precoat Conditioning	BBS (kPa)	Curing Time (hrs)	Precoat Conditioning	BBS (kPa)	Curing Time (hrs)
Non Precoated	251.2	2	Non Precoated	195.6	2
	224.1	2		232.2	2
	224.0	2		229.5	2
	1283.7	24		1419.2	24
	1110.3	24		1256.6	24
	1283.7	24		1192.5	24
SS60-Wet	356.9	2	SS60-Wet	432.8	2
	381.5	2		402.9	2
	367.7	2		451.7	2
	860.9	24		1053.4	24
	733.6	24		1221.4	24
	969.3	24		1362.3	24
SS60-Dry	202.4	2	SS60-Dry	435.5	2
	180.7	2		497.8	2
	224.1	2		462.6	2
	896.2	24		1004.6	24
	963.9	24		842.0	24
	1042.5	24		909.7	24
AwC-Wet	281.0	2	AwC-Wet	438.2	2
	275.6	2		340.6	2
	340.6	2		346.0	2
	362.3	2		1066.9	24
	313.5	2		1066.9	24
	245.8	2	969.3	24	
	858.2	24	AwC-Dry	644.1	2
	785.1	24		571.0	2
	923.3	24		684.8	2
	888.0	24		1137.4	24
898.9	24	1077.7		24	
907.0	24	1251.2	24		
AwC-Dry	478.8	2	CwC-Wet	484.2	2
	416.5	2		473.4	2
	389.4	2		443.6	2
	988.3	24		408.4	2
	923.3	24		397.5	2
	1278.3	24		408.4	2
CwC-Wet	289.1	2		801.3	24
	356.9	2		917.9	24
	337.9	2		871.8	24
	926.0	24		996.4	24
	709.2	24		1110.3	24
	836.6	24		985.6	24
CwC-Dry	289.1	2	CwC-Dry	554.7	2
	297.2	2		611.6	2
	362.3	2		598.1	2
	1411.1	24		1543.9	24
	1500.5	24		1446.3	24
	1394.8	24		1443.6	24

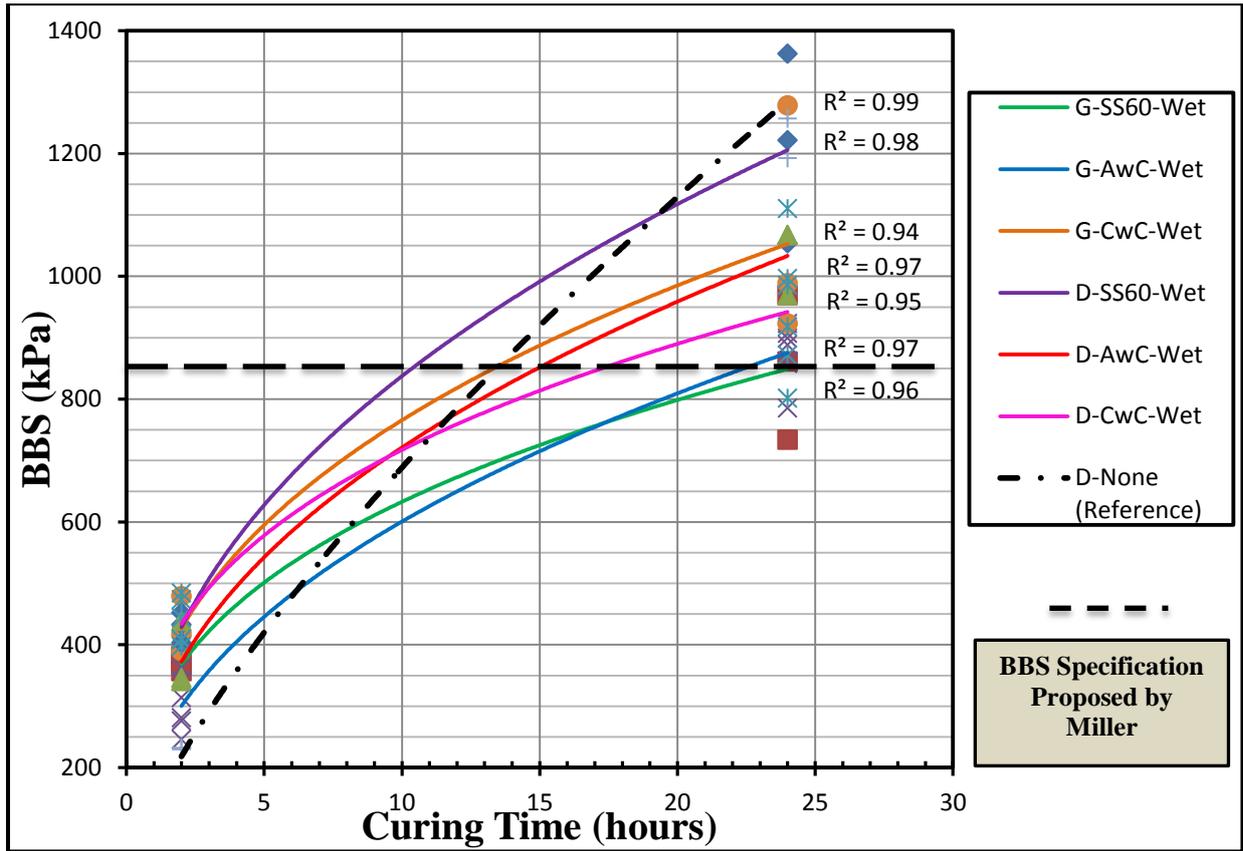


Figure 4-24: SC-E1 Bond Strength Development over time for Wet Precoated Combinations

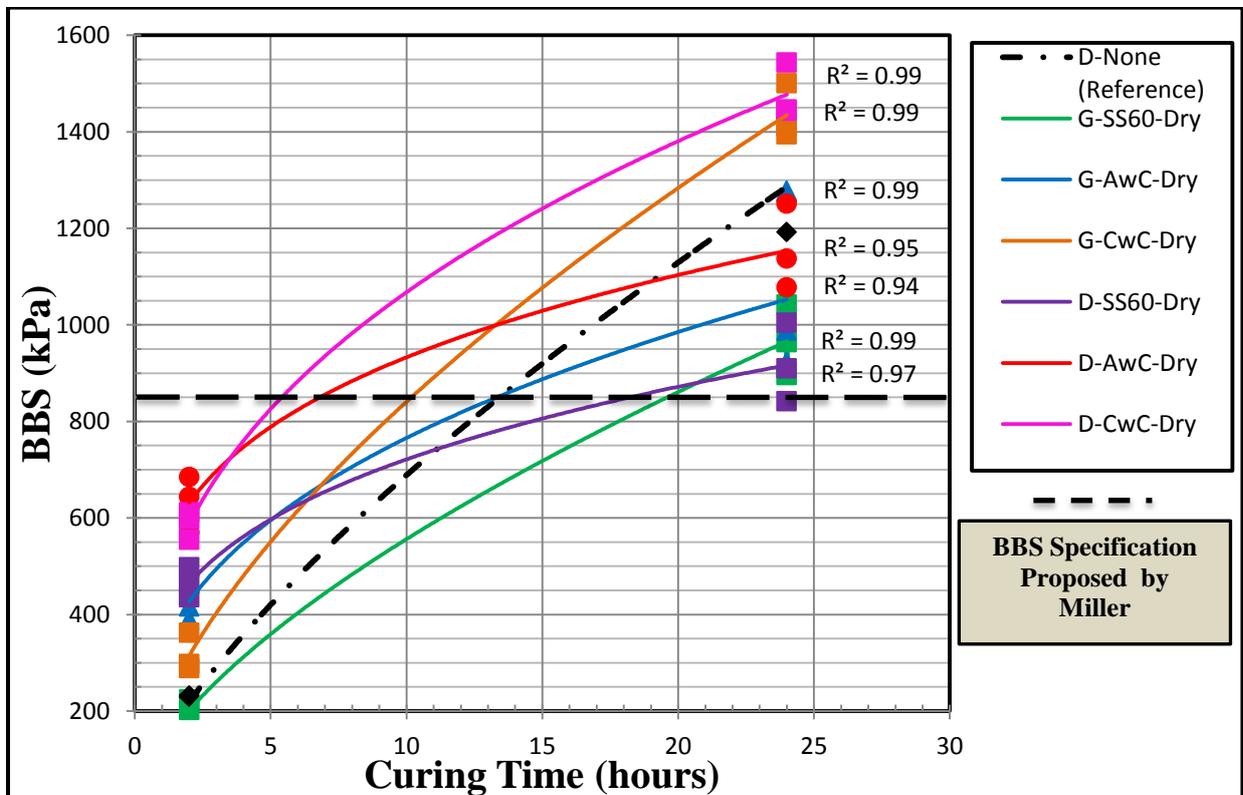


Figure 4-25: SC-E1 Bond Strength Development over time for Dry Precoated Combinations

As has been discussed previously, the analysis of performance of any combination was interpreted based on two conditions: The performance of the corresponding non-precoated dolerite combination or the minimum BBS specification limit.

The analysis based on the first condition can easily depict the following observations. In wet precoated combinations, the analysis shows that all the precoated combinations developed very good early bond strength. This is justified by the high bond strength observed for all precoated combinations relatively to the non-precoated combinations at 2hrs curing time. Unfortunately the bond strength development rate was very poor for all the precoated combinations. This is shown by the fact that after 22hrs additional curing time all the precoated combinations give lower results compared to the non-precoated combination. Therefore, there is a need to explore the extended curing time, in order to be able to draw a solid conclusion about the performance for wet conditions.

In the case of the dry precoat conditioning, all the precoated combinations show a good performance compared to the reference combination at the early stage (2hrs curing). After 22hrs additional curing time, the situation has changed. Except two precoated combinations that performed well, other four combinations performed poorly compared to the reference combination.

The CwC precoated combinations showed an exceptional good bond strength development rate. This precoat type showed promising results and there is a need to explore for further extended curing time. The trend shows that at an extended curing time, a better performance will be observed on granite than on dolerite.

The interpretation of the good performance of CwC precoated combinations can be based on molecular orientation, mechanical as well as thermodynamic theories of adhesion. Once the CwC, which is positively charged, is applied on a negatively charged aggregate surface, due to molecular orientation concept, the features presented in Figure 4-15 are developed. Then, once SC-E1 that is liquid in nature is applied, as it cures, it hardens and the mechanical theory suggests the creation of a strong interlocked network that is very hard to break. This is due to surface irregularities caused by open-up of the CwC precoat that serves as the surface roughness and thus facilitate mechanical interlock between the binder and aggregate surface irregularities. The surface energy concept (thermodynamic theory) is also thought to have a contribution through the spaces left non-precoated as a result of molecular orientation action. This may be the basis to interpret the better promise the CwC precoat is offering on granite compared to dolerite.

The analysis based on the second condition, shows that either wet or dry precoat conditioning, none has reached the minimum BBS specification limit at 2hrs curing time. However, almost all the combinations exceeded that limit at 24hrs curing time.

Based on the time taken to reach the minimum BBS specification limit, it can be easily observed that the bond strength development rate is higher for dry compared to wet conditions. For example for dolerite pre-coated with CwC (D-CwC), for wet conditions, it takes about 18hrs to develop a bond of 850kPa but under dry conditions, it takes 6hrs only, to develop the same bond strength.

4.3.6 Statistical Analysis

The graphical representation presented in section 4.3.2 to section 4.3.5 illustrated the influence of the variables on bitumen-aggregate system bond strength. The figures presented in the aforementioned sections are interpretative enough to illustrate the individual effect of each variable under consideration. In order to be able to refine predictions about the population represented by the tested samples, the use of inferential statistics is required.

The inferential statistical technique was used to prove whether, based on the data sample available, the effect of one or another variable on BBS is statistically significant or not. This provides a confidence for predicting the behaviour of the population represented by the sample. Moreover, the statistical technique for analysis allows evaluating the interactions between variables.

Due to the nature of the study, the univariate analysis of variance (univariate ANOVA) as discussed in section 4.1 was found to be appropriate. ANOVA is based on the concept of comparing the means of three or more independent variables (Montgomery & Runger, 2007). It yields a value that is tested to determine whether a specific independent variable has a significant influence on the dependent variable.

In this study, statistical software known as SPSS was used for the analysis of variance. In order to facilitate the interpretation of ANOVA results, the author preferred to present the main effect results and each level of interaction results separately. The tables here-after illustrate the results of the statistical analysis.

Table 4-13: Summary of the ANOVA for the main effects of various variables

Dependent Variable: F (Bitumen Bond Strength "BBS")						
Source	Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	6.128E7 ^a	8	7659597	220.508	0.000	0.906
Intercept	9.51E+07	1	9.51E+07	2.74E+03	0.000	0.937
A (Aggregate Type)	130549	1	130549	3.758	0.054	0.020
B (Precoat Type)	427528.9	2	213764.5	6.154	0.003	0.063
C (Precoat Conditioning)	2262147	1	2262147	65.124	0.000	0.262
D (Binder Type)	1.73E+07	2	8650057	249.022	0.000	0.731
E (Curing Time)	1.17E+07	1	1.17E+07	337.327	0.000	0.648
Error	6356704	183	34736.09			
Total	3.21E+08	192				
Corrected Total	6.76E+07	191				
a. R Squared = .906 (Adjusted R Squared = .902)						

Table 4-13 summarises the results of the analysis of variance for the main effects of the various variables. In the table, the following abbreviations were used:

df: Degree of Freedom: Can be defined as the number of independent pieces of information or data that contribute into the estimate of a parameter.

F: F-test statistic value: it is the ratio of the mean square between the treatment groups and the mean square within the treatment groups

Sig: Level of significance or p-value: it measures the significance of the effect of independent variables taken in isolation or in combination on the dependent variables. It is obtained from the F-value for a particular value of level of confidence and degree of freedom

The results in the table 4-13 shows that with 95% confidence level, it can be concluded that aggregate type does not have any statistical significant effect on the BBS value for the specific types of aggregate used in this study. This is illustrated by the value in the table 4-13 surrounded by a red coloured circle. Even though the trend is more or less the same, the failure modes were different, granite aggregate favoured adhesive failure whereas dolerite aggregate favoured cohesive failure. All other variables; precoat type, precoat conditioning, binder type and curing time; they have a statistical significant effect on the BBS at 95% confidence level. This statement is based on the fact that, their p-values are less than 0.05.

So as to have an insight on which variable that affects strongly the bond strength, the partial Eta squared was calculated. The partial Eta squared values as shown by the green circle in the table 4-13,

identified the binder type as the most influential factor on the bond strength for the materials under investigation. This seems to concur with the graphical representation used in section 4.3.2 to section 4.3.5 which shows highest difference in BBS values when the binder types are varied more than for any other variable.

Table 4-14: Summary of the ANOVA for the interaction effects of various variables

Dependent Variable: F (Bitumen Bond Strength "BBS")						
Source	Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Two Levels Interaction						
A * B	751143.6	2	375571.8	37.119	0.000	0.353
A * C	75.785	1	75.785	0.007	0.931	0.000
A * D	125260.1	2	62630.06	6.19	0.003	0.083
A * E	106.803	1	106.803	0.011	0.918	0.000
B * C	381999.8	2	190999.9	18.877	0.000	0.217
B * D	443387.7	4	110846.9	10.955	0.000	0.244
B * E	72249.07	2	36124.54	3.57	0.031	0.050
C * D	142033.8	2	71016.89	7.019	0.001	0.094
C * E	139861.2	1	139861.2	13.823	0.000	0.092
D * E	0	0	.	.	.	0.000
Three Levels Interaction						
A * B * C	206931.8	2	103465.9	10.226	0.000	0.131
A * B * D	275900.7	4	68975.16	6.817	0.000	0.167
A * B * E	22905.48	2	11452.74	1.132	0.325	0.016
A * C * D	131508.5	2	65754.23	6.499	0.002	0.087
A * C * E	114212.2	1	114212.2	11.288	0.001	0.077
A * D * E	0	0	.	.	.	0.000
B * C * D	547462.9	4	136865.7	13.527	0.000	0.285
B * C * E	277902.6	2	138951.3	13.733	0.000	0.168
B * D * E	0	0	.	.	.	0.000
C * D * E	0	0	.	.	.	0.000
Four and Five Levels Interaction						
A * B * C * D	496244.1	4	124061	12.261	0.000	0.265
A * B * C * E	61408.81	2	30704.4	3.035	0.051	0.043
A * B * D * E	0	0	.	.	.	0.000
A * C * D * E	0	0	.	.	.	0.000
B * C * D * E	0	0	.	.	.	0.000
A * B * C * D * E	0	0	.	.	.	0.000
Error	1376043	136	10117.97			
Total	3.21E+08	192				
Corrected Total	6.76E+07	191				
a. R Squared = .980 (Adjusted R Squared = .971)						

The results in Table 4-14 represent the summary of the analysis of variance for the interaction effects of different variables. Due to the number of variables considered, the interactions can go up to level five.

However, the results of statistical analysis have shown that the interactions for levels higher than 3 are more or less negligible.

The section of two levels interaction consists of 10 different cases. Among the ten cases of variables interaction, only seven interacting variables which are,

- Aggregate type and precoat type,
- Aggregate and binder types,
- Precoat type and precoat conditioning,
- Precoat and binder types,
- Precoat type and curing time,
- Precoat conditioning and binder type, and
- Precoat conditioning and curing time

Have significant interaction effect on BBS values. Two in ten different cases analysed which are aggregate type and precoat conditioning, and aggregate type and curing time do not show any statistical significant interaction effect, at 95% confidence level as illustrated by their p-values. These p-values are surrounded in the table by red coloured circles and are all higher than 0.05. One case in this section does not have the p-value. This is caused by the fact that, the interaction is between binder type and curing time. The curing time was investigated for only one binder type which SC-E1, the sum of squares and the degree of freedom are equal to zero. Thus, the corresponding p-values could not be calculated. A similar situation is observed for all other interaction levels when binder type interacts with curing time.

The section on the observation of the interaction of the three levels shows that the interaction between aggregate type, precoat type and curing time does not have a statistical significant effect on the bond strength. The four and five levels interaction section shows only one interaction that has statistical significant effect on the bond strength as shown in Table 4-14.

4.3.7 Results Discussion Summary

The performance of any surfacing seal depends on the adhesion bond strength developed in bitumen-aggregate system. The latter is influenced by different factors that include but not limited to binder type, aggregate surface characteristics, precoat type and conditioning. By using the Bitumen Bond Strength (BBS) testing apparatus, results of developed bond strength in various bitumen-aggregate combinations were obtained. The materials were combined and cured at low temperatures. This enabled the evaluation of adhesion bond strength for the specific combinations under the conditions similar to winter conditions. The results of evaluation of bitumen-aggregate system adhesion properties by using BBS test can be summarized as follows:

4.3.7.1 Aggregate Precoating

- The selected precoating fluids used with granite and dolerite aggregates show that their behaviours on aggregate surface depend on both their composition and conditioning. The precoat without cutters cure in a short period of time and do not show a significant dependency on curing condition. On the other hand the behaviour of precoat with some cutters shows a significant dependency on curing condition. Under wet conditions (24hours at 20°C), they were tacky. But, under dry conditions (48hours at 60°C), the tackiness was minimal.
- For the cationic emulsion with cutters (CwC) precoating fluid after 48hours at 60°C (dry curing), the residual bitumen demonstrated a special behaviour. The precoat opened-up during curing and agglomerated in some specific areas, leaving some aggregate surface areas unattended. It is thought that, this behaviour could be explained by the molecular orientation theory of adhesion discussed in 2.3.3.3.

4.3.7.2 Binder Type

- In either wet or dry precoat conditions the 70/100 and S-E1 performed better than SC-E1. This is thought to be caused by the presence of water in the emulsion that prevents the quick bond strength development. It should be pointed out that the BBS test was carried-out after 24hours curing time of the applied binder. Therefore, further investigation is recommended for extended curing time for a solid conclusion to be drawn.
- The neat bitumen (70/100) in all the tested cases developed higher bond strength than a polymer modified bitumen (S-E1). The poor bond strength of S-E1 might be caused by its high viscosity compared to 70/100. The high viscosity reduced the S-E1 wetting ability at low temperatures and thus, resulting in poor adhesion properties.

4.3.7.3 Aggregate Type

The bitumen bond strength test results have failed to show any evidence of the difference between the adhesion properties of granite (with acidic behaviour) and dolerite (with basic behaviour). It is unexpected based on the theoretical concept for basic and acidic aggregates. This might result from their mineralogical compositions that are not far different.

4.3.7.4 Precoat Type and Conditioning

- For wet precoat conditioning, all the SC-E1 and precoat aggregate combinations developed poor bond strength compared to the corresponding non-precoat combination. For both 70/100 and S-E1 only SS60 (30% water diluted) precoat aggregate combinations developed higher bond strength than non-precoat combinations. This is thought to be due to the

presence of cutters in the other precoat types, which soften the binder and prevent a strong bond strength development.

- For dry precoat conditioning, SC-E1 developed higher bond strength with CwC precoat aggregate compared to aggregate precoat with any other type of precoat or to the corresponding non-precoat combinations. This is thought to be caused by the strong bond developed between the positively charged precoat (CwC) and the negatively charged aggregate surface. In addition to this, the mechanical interlock created between SC-E1 as it cures and the valleys created on the CwC precoat aggregate surface. SS60 (30% water diluted) developed a very weak bond with SC-E1 in all cases tested. This is due to the poor bond resulting from interaction between the negatively charged emulsion (SS60) and the negatively charged aggregate surface. The stripping due to water contained in SC-E1 is also believed to weaken further the above mentioned bond. This can be explained by the surface energy (thermodynamic) theory of adhesion discussed in section 2.3.3.4.

The behaviour of S-E1 and AwC precoat aggregates suggests that S-E1 is better compatible with AwC than any other precoat type used in dry condition. But, further investigation is recommended for extended curing time in order to conclude about this statement.

4.3.7.5 Curing Time for SC-E1

- The bitumen-aggregate system adhesion bond strength develops with time. The curing time evaluation shows the bond strength development rate dependency on material combinations. The precoat type and conditioning play an important role. The wet precoat combinations show a higher strength than non-precoat combinations at 2 hours. But at 24 hours the observed bond strength is much lower than that of non-precoat combinations. This might be a result of the continuing cutting back action which retards the bond strength development. Further investigation into extended curing time is recommended to enable the solid conclusion to be drawn. Otherwise, the use of precoat fluid especially with cutters during winter period may be questioned.
- The BBS test results have shown significant variability in time taken for the proposed minimum BBS specification limit (850kPa) to be achieved depending on the material combinations. The time varies from 11 hours for dolerite SS60 precoat aggregate to 24 hours for granite SS60 precoat aggregates for wet conditions. This simulates the time that can be taken after construction for opening to traffic.
- For dry precoat conditioning, at 2 hours, all the precoat combinations have higher bond strength than the corresponding non-precoat combinations. However, after 24 hours only the CwC precoat combinations have higher bond strength than the corresponding non-precoat combinations. This shows an incompatibility between the anionic emulsions

precoated aggregates and SC-E1. This is a result of repulsion interaction between the negatively charged bitumen droplets contained in anionic emulsion and the negatively charged aggregate surface.

- For the combinations of dry precoat conditioning, the time spent to reach the proposed minimum BBS specification limit (850kPa) varies from 6hours for dolerite CwC precoated aggregate to about 20hours for granite SS60 precoated aggregate

CHAPTER 5

ANALYSIS, INTERPRETATION AND APPLICABILITY

5.1 INTRODUCTION

Though the study results were presented and discussed in the previous Chapter, the need of an in depth analysis was identified. This section intends to analyse and interpret results relatively to the application. As previously discussed, the main objective of this study is to contribute to the understanding of adhesion properties in binder-aggregate system for winter seals.

If achieved, it can reduce the risk of surfacing seals ravelling failure. The latter is one of the three dominant surfacing seals failure mechanisms as discussed in section 2.5.6. In order to understand the contribution of adhesion properties to the performance of winter surfacing seals, the conceptualisation of ravelling failure is required. The factors contributing to the resistance to ravelling need to be identified and the role of adhesion clarified.

Figure 5-1 illustrates the ravelling failure appearance on an existing road and conceptual representation of surfacing seals sollicitation leading to ravelling.

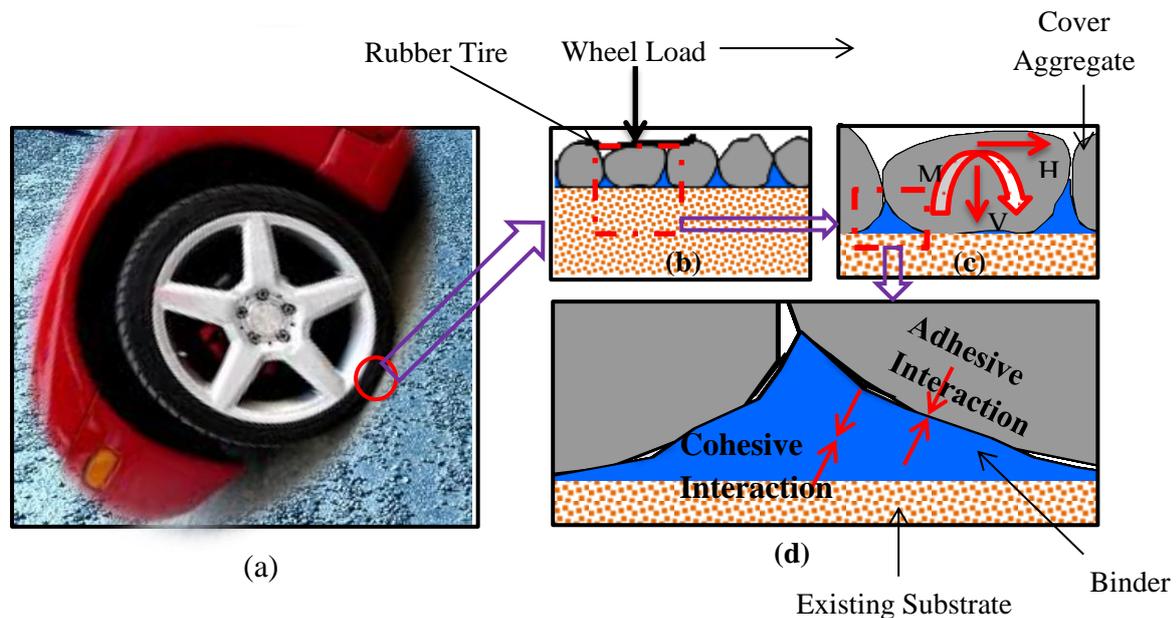


Figure 5-1: Conceptualisation of traffic forces leading to seals ravelling and bonding interactions resisting to those forces

Figure 5-1 (c) illustrates that if a specific aggregate particle is analysed under traffic action, three types of solicitation can be identified as follows: the vertical force (V) intending to induce aggregate embedment; the rolling moment (M) intending to yield the rolling-over of aggregate and the horizontal force (H). It appears that only the horizontal force and the rolling-moment contribute in ravelling failure. The surfacing seal's design concept as described in section 2.5.3 suggests the shoulder-to-shoulder packing for cover aggregates. In such a case, the resistance to ravelling depends on both stone-to-stone friction and binder-aggregate bond strength.

In practice due to a significant number of contributing factors, including but not limited to aggregate shape variability and construction practices, the aggregates are not always packed shoulder-to-shoulder. As a result, the binder-aggregate bond strength is believed to be most influential towards resistance on ravelling failure. Therefore, a number of factors related to traffic loading and speed, construction practices, environmental conditions and material characteristics influencing bond strength development contribute strongly to the resistance to seals ravelling. In this study, the emphasis was placed on the influence of material characteristics and winter environmental related factors to bond strength development as mentioned in section 2.1.

Figure 5-1 (d) illustrates that the binder-aggregate bond strength encapsulates both the adhesive bond strength and cohesive bond strength. Each of the components that is exceeded first by the traffic induced stresses causes the binder-aggregate bond failure.

The study results revealed that the material combinations in which emulsion is used as the binder, the bond failures are predominantly adhesive. On the other hand, when the binder used is a hot applied binders (S-E1 or 70/100), two options prevailed. The first option is when the precoat is under wet condition; in this case, the predominant failure mode was adhesive contrary to the dry precoating condition that yields predominantly the cohesive failure and cohesive-adhesive failure. In this study, based on the binder-aggregate adhesion bond performance, the proposed practical considerations are as follows:

5.2 BINDER SELECTION

The selection of an appropriate binder for a specific surfacing seal project depends on a number of factors including cost and binding ability. The initial binding ability reflected by the fundamental adhesion depends on the affinity of the binder with stone and the capacity to wet the aggregate surface. Initial adhesion development influences significantly the performance of winter surfacing seals, especially through ravelling prevention.

Generally, the binders with low viscosity at the time of application are believed to yield high initial adhesion bond due to increased wettability. Due to that, especially in winter, the emulsion binders have preference (Van Zyl et al., 2012). In this study, regarding the binder selection, based on the

testing results of the various cases investigated, the binder performance ranking was proposed as follows:

It is emphasised that the following binder performance ranking relates to the specific testing conditions as specified in Chapter 3.

BINDER PERFORMANCE RANKING

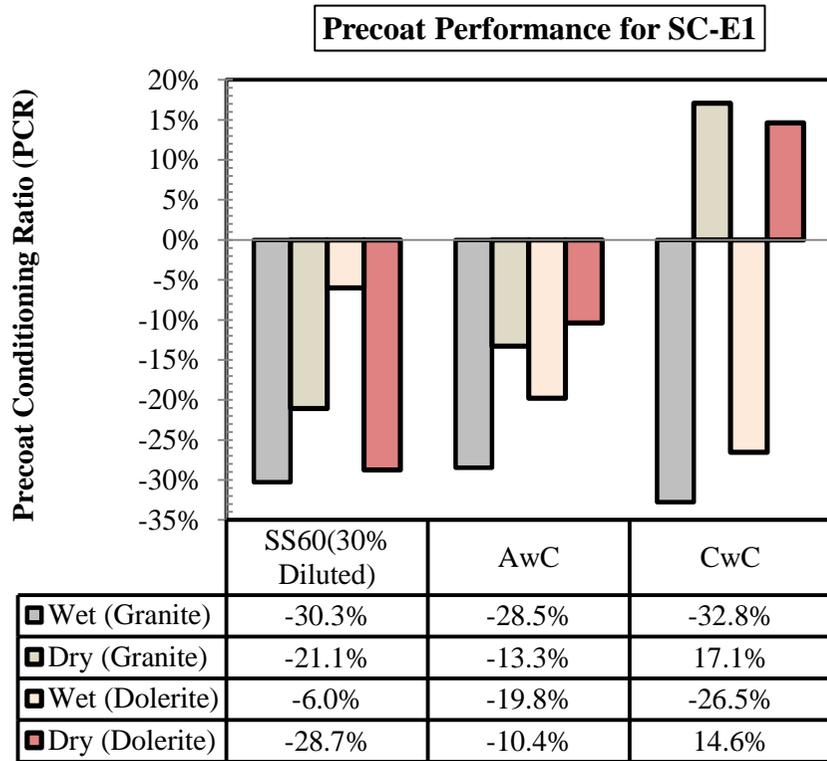
- Non-precoated aggregates combinations
70/100 > SC-E1 > S-E1
- SS60 (30% water diluted) precoated aggregates combinations
70/100 > S-E1 > SC-E1 : Wet & Dry
- Anionic emulsion with cutters precoated aggregates combinations
70/100 > S-E1 > SC-E1 : Dry
70/100 > S-E1 >= SC-E1 : Wet
- Cationic emulsion with cutters precoated aggregates combinations
70/100 > S-E1 > SC-E1 : Wet & Dry

This binder performance ranking illustrates that the performance is dependent on material combinations. However, in almost all the cases the hot applied binder performed better than the emulsion, with 70/100 as the high performer in all the cases investigated.

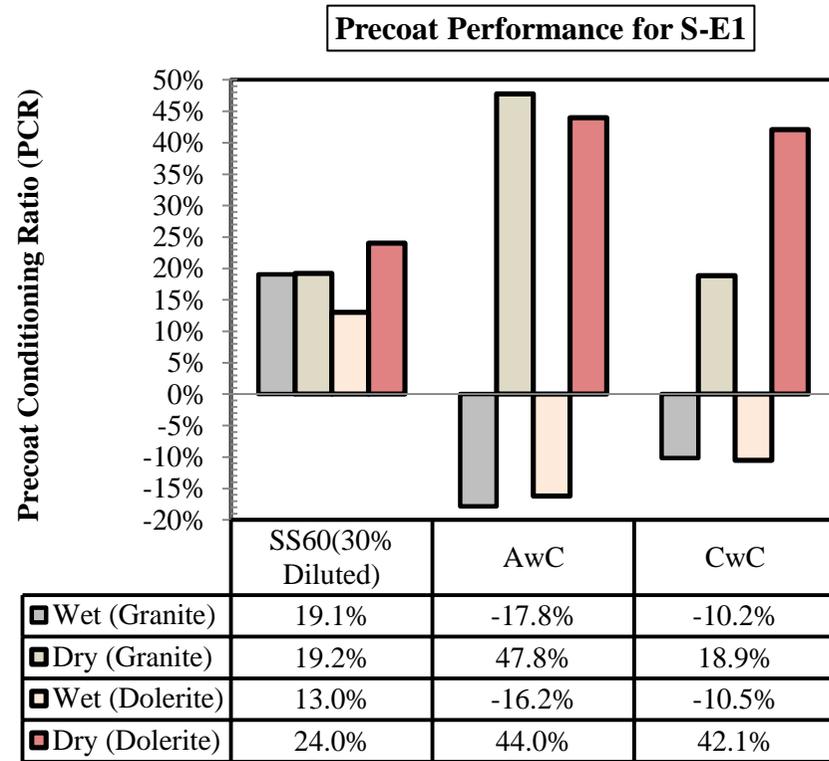
5.3 AGGREGATE AND PRECOATING PRACTICES

Aggregate precoating is one of various methods developed to enhance the binder-aggregate adhesion bond and thus, reduce the premature ravelling. Section 2.2.3.2 discusses in details the precoating practices. However, as has been mentioned by Van Zyl, (2012) field practitioners still have some controversial opinions regarding whether precoating could be still used in wet conditions during sealing. Another practical point of concern is the fact that, the precoat conditioning and type should depend on the type of the binder (material compatibility).

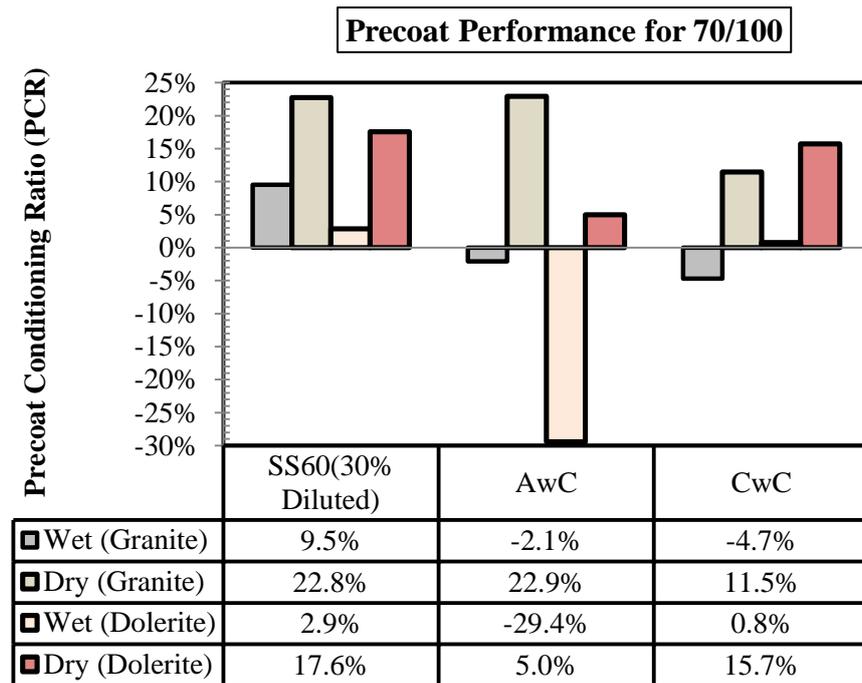
In an effort to contribute to the understanding as well as to provide an insight on the issues mentioned above, in this study, various precoat types in both wet and dry conditions were explored. Different binder types were also the subject of concern in this study. The results of the study are presented in section 4.3.4. Nevertheless, in order to present the practical implications of the precoat conditioning, the ratio named “Precoat Conditioning Ratio (PCR)” is used here. The precoat conditioning ratio shows how the precoat type and condition affect the bond strength relatively to the similar non-precoated combination. In the following figures, the line showing 0% on the graph represents the non-precoated combination. Therefore, the combination results projecting towards negative side means that the specific combination resulted in the bond strength less than the corresponding non-precoated combination for the specified percentage. If it is projecting towards the other way around, it reflects that precoating has improved the bond strength to that specified percentage.



(a)



(b)



(C)

Figure 5-2: Graphical representation of Precoat Conditioning Ratio for (a) SC-E1 binder; (b) S-E1 binder, (c) 70/100 binder

The results presented above show, that the precoating condition is a factor, which needs consideration during winter sealing. The reduction in bond strength of 32.8% was observed due to the use of wet precoat. The benefit of using dry precoat approximates 50% increment in bond strength relatively to the similar non-precoated combination as illustrated on S-E1 with AwC dry as precoat for granite (figure 5-2 (b)). The results also alert that dry precoating is not always beneficial to the bond strength development. This is illustrated in figure 5-2 (a), in which the dry precoating caused the bond strength reduction ranging between 10.4% and 13.3% for AwC and between 21.1% and 28.7% for SS60. Nevertheless, in general the results suggest that the dry precoat should be preferred than wet precoat, especially for the precoat with some cutter contents.

Furthermore, another interesting finding clarified by these results, is the fact that the type of the binder, type and condition of the precoat combinations is an important factor to be considered in winter sealing. Figure 5-2 (a) shows that when the binder available is SC-E1, the use of anionic emulsion with or without cutters as precoat has always undermined the bond strength development, regardless whether the precoat was wet or dry. The comparison between Figure 5-2 (a), (b), and (c) illustrates clearly that the bond strength development depends on the binder-precoat types combinations. Based on the results obtained it can be recommended that, the use of anionic precoat when the binder available is SC-E1 should be avoided. Currently, the precoating specifications are being more accurately stipulated and in order to be more conclusive, this needs to be simulated through extended research.

5.4 ADHESION BOND DEVELOPMENT AND OPENING TO TRAFFIC

Freshly constructed surfacing seals are usually restricted to uncontrolled traffic for a certain period after construction. This allows the surfacing seals to develop appropriate cohesive and adhesive bonds that can withstand the traffic stresses. The traffic restriction time is usually estimated based on experience. Experienced field personnel using visual observation and engineering judgement of the surfacing seals bond strength, they decide subjectively whether the freshly constructed seals can be opened to uncontrolled traffic or not (Shuler, 2011; Pretorius, 2013).

Due to a significant number of factors contributing to bond strength development rate including but not limited to cover stone type and moisture content, binder spray rate, stone spread rate, binder type and grade and weather condition, the fore-mentioned judgement becomes difficult and not always accurate (Shuler, 2011).

Failure to estimate accurately the time to opening for traffic may result in premature ravelling (chip loss), vehicle's windscreen damage or the road user's unnecessary delays and complains. In an effort to reduce the subjectivity associated with estimation of time for opening to traffic, some research projects were carried out in the past. A number of laboratory and on site bond strength testing methods have been developed. Among others, there is a method developed by Shuler (2011) that predicts the time to opening based on monitoring the moisture content of the surfacing seal and the on-site pull-out test described in South African technical guideline one (TG1), which is based on the force required to dislodge stone from the surfacing seal.

In this investigation BBS test results was used to contribute to the reduction in subjectivity in the determination of time to opening to traffic. During BBS test development, Miller (2010) has validated the BBS test using the sweep test. Miller developed an experimental design in which, he investigated the correlation between the BBS and the sweep test. Based on sweep test specification of retaining at least 90% of cover aggregate of the laboratory prepared seal, he has proposed the minimum BBS specification limit as 850kPa.

This section through the figure below illustrates an objective estimation of the time required for opening to traffic based on the above-mentioned Miller's BBS minimum specification limit of 850kPa and the results of material testing of this study.

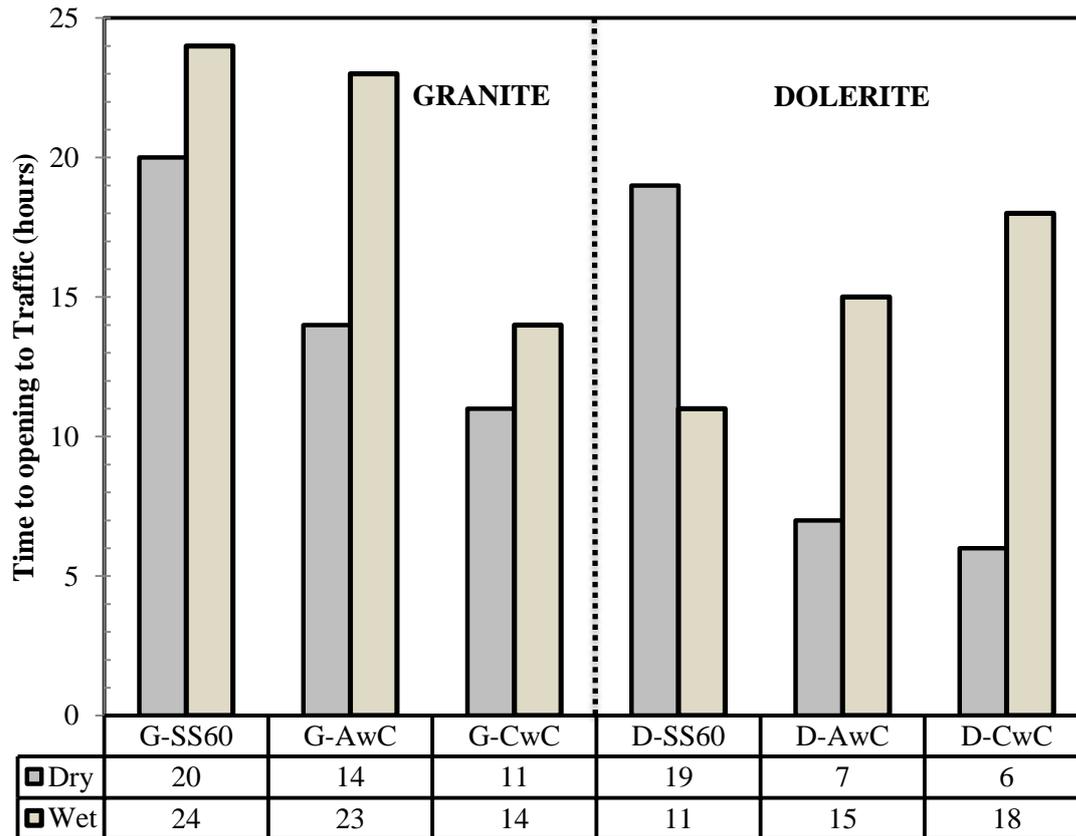


Figure 5-3: Time to opening for traffic after surfacing seals construction for SC-E1 based on $BBS_{min} > 850kPa$

Figure 5-3 illustrates the minimum time required for opening to traffic, proposed based on the BBS minimum specification limit (850kPa). This graphical illustration is not intended to fix the time required for the surfacing seals to develop appropriate bond strength before opening to traffic. Rather it can help to visualise that the time required depends on the material combinations used during surfacing seals construction and strongly on the type and condition of the precoat. The combination based on dolerite performed well with the time required ranging between 6 hours and 19 hours whereas the granite based combinations required the time ranging between 11 hours and 24 hours for the uncontrolled traffic to be released.

Moreover, the figure illustrates also that the conditioning of the precoat plays an important role on the rate at which the binder-aggregate system develops the bond. The figure shows also that, the BBS test is a promising testing method that can contribute in the reduction of subjectivity in the determination of time required before opening to traffic. For the solid conclusion about the time required for opening to traffic on these specific material combinations, this study recommends further exploration on more extended curing time to know what should happen after 24hours. Furthermore, it may be also interesting if more intermediate curing times are included as they may yield a more accurate fitting curve as well as to compare the BBS test results with the above mentioned on-field test.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 INTRODUCTION

The study on evaluation of adhesion properties in bitumen-aggregate systems for winter seals consisted of various components. The latter are the critical review of literature intended to understand and document the factors influencing bitumen-aggregate adhesion properties, the study research methodology and experimental design as well as the study results analysis and interpretation. This concluding Chapter summarises the salient observations and conclusions made in the above-mentioned study components. Moreover, this Chapter documents the recommendations for further studies that could result in supporting and reinforcing ideas to the conclusions drawn in this study.

6.2 CONCLUSIONS

To conclude this study, the objectives elaborated in Chapter 1 were re-visited and reconciled with the research findings interpretation.

6.2.1 Factors Influencing Adhesion Bond Strength Development

- i. According to the literature reviewed for the interest of this study, the factors influencing the bond strength are categorised in material properties, environmental conditions, traffic, and construction techniques related factors. The most influential environmental and material related factors include but are not limited to temperature, rainfall (moisture), humidity, aggregate mineralogy and physico-chemical characteristics, precoating type and conditioning, binder type and binder properties.
- ii. The results of the statistical analysis provided by this study point out the following significant influential factors: precoat type, binder type, precoat conditioning and curing time. Among these factors, the binder type was found to be most influential. Moreover, the influence of interaction between some factors on either 2, 3 or 4 levels was found to be statistically significant on bond strength development. At the interaction of level two, the most influential interaction was found to be aggregate type with precoat type. At level three, the precoat type, conditioning and binder type interaction was the most influential.

6.2.2 Compatibility between Acidic and Basic Aggregates with various types of Binder

In evaluation of the compatibility of acid and basic aggregates with various types of binder, the following conclusions were drawn:

- i. Based on the BBS trend, no statistical significant difference in compatibility was found between basic and acid aggregates with a given specific binder type for the materials used in this study. However, in regards of failure modes, granite aggregate favoured adhesion failure and dolerite favoured cohesive failure.
- ii. For a specific aggregate type, variation of binder type resulted in statistically significant different BBS results. The hot applied binder (70/100 and S-E1) performed better than emulsion binder (SC-E1). In all the cases, the 70/100 was the highest performer. For more information section 4.3.7.2 may be helpful. The general ranking of binder performance was:
70/100 > S-E1 > SC-E1

It is noted that, in some few specific conditions, this ranking was not respected, for more clarification about this; section 5.2 may serve as a reference.

6.2.3 Precoat Type and Conditioning

- i. In winter sealing, the precoat type and conditioning are factors that need attention. The use of wet precoats resulted in reduction of bond strength ranging from 2.1% to 32.8%. The dry precoating has benefited the bond strength up to around 50%. However, in some cases the dry precoat has resulted in reduction of bond strength of around 28.7%. Generally, the results suggest that, the dry precoat condition should be preferred over wet conditioned precoat, especially for the precoat containing some cutters.
- ii. The type of the binder, type and condition of the precoat combinations need also special attention in winter seals. It was found that the bond strength depends strongly on these combinations. For example, the behaviour of S-E1 and AwC precoated aggregates suggests that S-E1 is compatible with AwC more than any other precoat type used in dry condition. However, further investigation is recommended for extended curing time in order to draw a solid conclusion about this statement.
- iii. For wet precoat conditioning, all the SC-E1 and precoated aggregate combinations developed a poor bond strength compared to the corresponding non-precoated combination. For both 70/100 and S-E1 only SS60 (30% water diluted) precoated aggregate combinations developed higher bond strength than non-precoated combinations.
- iv. In dry precoat conditioning, SC-E1 developed higher bond strength with CwC precoated aggregate than any other precoats and even than non-precoated combinations. SS60 (30% water diluted) developed very weak bond with SC-E1 in all cases tested.

This is due to the poor bond resulting from interaction between the negatively charged emulsion (SS60) and the negatively charged aggregate surface. The stripping due to water contained in SC-E1 is also believed to weaken further the above mentioned bond. The results suggest that SC-E1 is not compatible with anionic precoat.

- v. The rate, at which the bond strength develops, affects the appropriate time for opening to traffic. This was found to be strongly dependent on the type and condition of the precoat as well as on material combinations. The time required for opening to traffic ranging between 6 hours and 19 hours were observed for dolerite combinations. Similarly, the time ranging between 11 hours and 24 hours were observed for granite combinations. This can reduce the subjectivity during estimation of time to opening, however, these time values relate to the testing conditions and material combinations adopted in this study.

6.2.4 Repeatability of Bitumen Bond Strength Test

As a general concept, the repeatability of any measurement system is assessed on one item measured several times by the same operator. This is applicable on non-destructive test only. It is thus, understood that for the destructive test like BBS, the repeatability is strongly influenced by the consistence in sample preparation and testing procedure. In this section the conclusion regarding the sample preparation, testing procedure and repeatability are as follows:

- i. Aggregate substrate's faces preparation prior to the application of the precoat is one of the key factors that determine the consistency in adhesion test results. It was realized that non-spirit levelled aggregate faces, regardless of their smoothness, provide inconsistency in the adhesion test results. It is recommended to check for the flatness of each of the aggregate substrates to be used in testing, by using a spirit level
- ii. Due to the viscoelastic nature of bitumen binder, the loading rate has a considerable influence on the BBS results to be obtained on a specific test. Therefore, effort should be made to keep consistency in loading rate for a given set of tests. For this to be achieved, care should be taken during pressure supply to the BBS machine because the sensitivity of the loading rate knob valve seems to be related to the supplied pressure. During the testing phase, the operator is recommended to identify the position of the loading rate regulator and the corresponding pressure range that can optimise the consistence of the targeted loading rate.
- iii. The Gage R&R study: ANOVA method revealed that bitumen bond strength (BBS) test method is repeatable but with caution as has been discussed in section 4.2.2. Proposed recommendations on improvement of BBS testing method are provided in the subsequent sections.

6.3 RECOMMENDATIONS FOR FURTHER STUDIES

The following are suggested recommendations for further studies that could assist in substantiating the analysis and conclusions made in this study:

- i. The BBS loading rate was found to be one of the factors influencing the bond strength results. Failure to control it may mislead the adhesion properties characterisation. The study found that the sensitivity of the loading rate regulator and thus the consistence of the testing loading rate to depend strongly on pressure supplied from the compressor to the BBS machine. The loading rate regulator can occupy various position and the level of influence of the pressure supply to the loading rate as explained previously is dependent on the position of the regulator. Thus, the development of pressure supply-BBS loading rate relationship for a full range of possible position of the loading rate regulator is recommended. This could contribute significantly to the repeatability of BBS test and the accuracy of bond strength characterisation.
- ii. A study on modification of the BBS loading rate operating principal may be an alternative to improve the consistency of BBS results. It is recommended that the pressure supply feedback controlled loading rate regulator development can be investigated for its applicability on BBS. For example by using proportional-integral-derivative (PID) loading rate controller.
- iii. To carry-out a set of BBS tests on a specific pre-determined loading rate was found to be practically impossible. However, due to viscoelastic properties of bitumen binder, the inconsistency in loading rate makes comparison of results very difficult. The further studies on development of BBS loading rate versus BBS value relationship or evaluation of the influence of variation of loading rate to the BBS value using a controlled system such as DSR tensile test could play significant role in enhancing BBS results comparison.
- iv. The sample preparation contributes significantly in the BBS results. During sample preparation, the roughness of aggregates core is assumed to be uniform after lapping with 1000 grit silicon carbide. It is believed that the uniformity of roughness is not ensured due to variability in mineralogical composition of aggregates; thus, roughness measurement is recommended for all the aggregate to be used in testing. Moreover, the degree of roughness that optimises the adhesion bond strength needs investigation.
- v. The study did not show any significant statistical difference in BBS development between granite (acid rock) and dolerite (basic rock). In order to be able to draw a solid conclusion about this, there is a need to investigate further various types of aggregates. It is strongly recommended to involve basic aggregates that contain carbonate minerals such as dolomite.

- vi. At 24 hours curing time, the hot applied binder (70/100 and S-E1) performed better than emulsion (SC-E1). In order to draw a concrete conclusion, there is a need to investigate the bond strength at an extended curing time (more than 24hours) to check whether the ranking will remain in same direction or not.
- vii. The precoat conditioning was found to be more influential on the bond strength development rate. Future research on extended curing time for the precoat is recommended. This could assist in the clarification on the curing time required on the field for wet and for dry precoat conditioning. Moreover, it can assist in identification of the appropriate time for opening to traffic after construction in winter sealing. Furthermore, this could involve correlation of BBS results to some on-site adhesion tests such as pull out test to validate the BBS capability to determine the time to opening to traffic.
- viii. In order to have more confidence of the BBS results, it is recommended that the reproducibility analysis of BBS test should be carried-out to support the conclusion drawn from repeatability analysis.
- ix. The precoat used in this study are emulsion of Colcote STM (Colas's bitumen based precoat). The trial to compare the present results with the past studies results have failed due to some variables that were different. An objective comparative study between the commonly known bitumen based precoat and the present results is recommended.

REFERENCES

- AASHTO. 2011. AASHTO TP 91-11-Standard Method of Test for Determining Asphalt Binder Bond Strength by Means of the Asphalt Bond Strength (ABS) Test. s.l: AASHTO.
- Abraham. S & Jenkins. J. K. 2012. Influence of precoating on the adhesion of surfacing seals. Unpublished Bachelor of Engineering thesis. Stellenbosch: Stellenbosch University.
- Airey, G.D. 2011. Adhesion and Moisture Damage Characterisation of Asphalt Mixtures. Granada: Nottingham Transportation Engineering Centre.
- Airey, G.D. 1997. Rheological characteristics of polymer modified and aged bitumen. PhD dissertation. UK: University of Nottingham.
- AKZO NOBEL. 2014. *Adhesion promoters for bitumen*. [Online]. Available: http://www.e-asphalt.com/ingles/akzo/adhesion_data/akzoadhesion.pdf [15/06/2014]
- Anderson, David A., Donald W. Christensen, Hussain U. Bahia, Raj Dongre, M. G. Sharma, Charles E. Antle, et al. 1994. Binder Characterization and Evaluation, Volume 3: Physical Characterization. Washington, DC: Strategic Highway Research Program, National Research Council.
- APRG. 1996. APRG Technical Note 6: Sprayed Sealing-Aggregate Precoating. Australia: Australian Roads Research Board.
- Asphalt Academy, (ed.). 2009. *TG2: Bitumen stabilized materials*. A guideline for the design and construction of bitumen emulsion and foamed bitumen stabilized materials. Second Edition. Pretoria: Asphalt Academy.
- Asphalt Academy, (ed.). 2007. *TG1: The use of modified binders in road construction*. Second. Pretoria: Asphalt Academy C/O CSIR Built Environment
- Bagampadde, U., Isacson, U. & Kiggundu, B. 2003. Fundamentals of Stripping in Bituminous Pavements-State of the Art. Sweden: KTH, Highway and Railway Engineering.
- Bahia, H., Jenkins, K. and Hanz, A. 2008. Performance Grading of Bitumen Emulsions for Sprayed Seals. Paper presented at first International Sprayed Sealing Conference, Adelaide, South Australia, Australia.
- Bickford, A. 2001. Basic Emulsion Chemistry. Paper presented at AEMA Asphalt Emulsion Technologies Workshop. s.l: Asphalt innovations.
- Butt, H., Graf, K. & Kappl, M. 2003. *Physics and chemistry of interfaces*. Weinheim: WILEY-VCH Verlag GmbH & Co. KGaA.
- Committee of State Roads Authorities(ed.). 1986. *Technical methods for highways. TMH1: Standard methods for testing road construction materials*. Vol. 2 Pretoria: Department of Transport

- Copeland, A.R., Youtcheff, J. & Shenoy, A. 2007. Moisture Sensitivity of Modified Asphalt Binders: Factors Influencing Bond Strength. *Transportation Research Record: Journal of the Transportation Research Board*, 1998(1).
- Coyne, L.D. 1988. Evaluation of Polymer Modified Chip Seal Coats. *J. Asphalt Paving Technol*, 57
- CSRA. 1994. Draft TRH7: The use of bitumen emulsion in the construction and maintenance of roads. Pretoria-South Africa: Department of transport
- Curtis, C.W., Ensley, K. & Epps, J. 1993. Fundamental properties of asphalt-aggregate interactions including adhesion and absorption-Report No. SHRP-A-341. Washington D.C: Strategic Highway Research Program, National Research Council.
- Das, S.K. 2004. Evaluation of Asphalt-Aggregate Bond and Stripping Potential. Master of Science Thesis. Texas: Texas Technical University.
- Douglas, G. & Keith, M.B. 2002. Measurement System Analysis and Destructive Testing. *Six Sigma Forum Magazine*. August: 16.
- Gast, R.G. 1977. Surface and colloid chemistry. Minerals in soils environments. Madison, Wisconsin: Soil Science Society of America.
- Gransberg, D.D. & James, D.M. 2005. *Chip seal best practices*. Washington D.C: Transportation Research Board
- Greyling, A.H. 2012. Development of a standard test method for determining the bitumen bond strength of emulsions: A south african perspective-Master's thesis. Stellenbosch: Stellenbosch University.
- Hanz, A., Bahia, H., Kanitpong, K. & Wen, H. 2007. Test Method to Determine Aggregate/Asphalt Adhesion Properties and Potential Moisture Damage. Report WHRP 07-02. Wisconsin. University of Wisconsin-Madison.
- Hefer, A.W., Bhasin, A. & Little, D.N. 2006. Bitumen Surface Energy Characterization using a Contact Angle Approach. *Journal of Materials in Civil Engineering*, 18(6).
- Hefer, A.W. 2004. Adhesion in bitumen-aggregate systems and quantification of the effects of water on the adhesive bond-PhD dissertation. Texas: Texas A&M University.
- Hicks, R.G. 1991. *Moisture damage in asphalt concrete*. Washington D.C: Transportation Research Board
- Holtrop, W. 2008. Sprayed Sealing Practice in Australia. *Road & Transport Research: A Journal of Australian and New Zealand Research and Practice*, 17(4).
- Huurman, M., Scarpas, T., Kasbergen, C. and Milne, T. 2003. Development of a Structural FEM for Road Surfacing Seals. Paper presented at Proceedings of the 2003 International Conference on Computational and Experimental Engineering and Sciences (ICCES), Greece.

- James, A. 2006. *Overview of Asphalt Emulsion. Asphalt Emulsion Technology*. Washington D.C.: Transport Research Board.
- James.A. 2014. *Asphalt emulsions (chemistry and concepts)*. [Online]. Available: http://www.e-asphalt.com/ingles/akzo/technical_paper/AEMA99-chemistry.pdf [26/07/2014].
- Jamieson, I., Moulthrop, J. & Jones, D. 1995. SHRP Results on Binder-Aggregate Adhesion and Resistance to Stripping. *THE ASPHALT YEARBOOK 1995*,
- Jenkins, K.J., Van de Ven, M., Greyling, A., Constable, B., Abrahams, S. and Stander, A. 2013. Binder Adhesion Testing using the Bitumen Bond Strength Test: State of the Art. Paper presented at AAPA International Flexible Pavements Conference, 15th, 2013, Brisbane: Queensland, Australia.
- Jenkins.K.J and Van de Ven. MFC. 2013. Revision of South African Pavement Design Method- Draft Synthesis Report on the Binder Adhesion Testing using the Bitumen Bond Strength (BBS) Towards the Development of a Mechanistic Seal Model: Reporting on B1, B4-1(i),B4-1(Ii), B2 & B5. SANRAL Report. Stellenbosch-South Africa.
- Jeon, Y.W., and Curtis, W.C., (1990). A literature review of the adsorption of asphalt functionalities on aggregate surfaces. Strategic Highway Research Program (Report SHRP-A/IR-90-014). Washington, D.C.: National Research Council
- Jones IV, D.R. & Kennedy, T.W. 1991. The Asphalt Model: Results of the SHRP Asphalt Research Program. Transportation Research Board VTI Rapport A, 372. Texas: University of Texas.
- Jordan, W.S. and Isaac, L. 2010. Applicability of Modified Vialit Adhesion Test for Seal Treatment Specifications. Paper presented at Transportation Research Board 89th Annual Meeting.
- Kandhal, P.S. & Motter, J.B. 1991. *Criteria for accepting precoated aggregates for seal coats and surface treatments*. Washington D.C.: Transport Research Board.
- Karrasahin, M, Aktas, B., and Gurer C. 2011. Determining of Precoated Aggregate Performance on Chip Seals using Vialit Test. *Transportation Research Board 90th Annual Meeting*.
- Kashaya, A.A. 2013. Surface run-off behaviour of bitumen emulsions used for the construction of seals. Master of Engineering Thesis. Stellenbosch: Stellenbosch University.
- Khattak, M.J., Baladi, G.Y. & Drzal, L.T. 2007. Low Temperature Binder-Aggregate Adhesion and Mechanistic Characteristics of Polymer Modified Asphalt Mixtures. *Journal of Materials in Civil Engineering*, 19(5).
- Kiggundu, B.M. & Roberts, F.L. 1988. Stripping in HMA mixtures: State-of-the-art and critical review of test methods-NCAT Report 88-02. Alabama: National centre for Asphalt Technology, Auburn University
- Lesueur, D. 2009. The Colloidal Structure of Bitumen: Consequences on the Rheology and on the Mechanisms of Bitumen Modification. *Advances in Colloid and Interface Science*, 145(1).

- Louw, K. 2004. USE OF BITUMINOUS BASED PRECOATING FLUIDS FOR SURFACE DRESSING. Paper presented at Proceedings of the 8th Conference on Asphalt Pavements for Southern Africa (CAPSA'04). Sun City, South Africa. 12-16 SEPTEMBER, 2004.
- Louw, K., Rossmann, D. and Cupido, D. 2004. THE VIALIT ADHESION TEST: IS IT AN APPROPRIATE TEST TO PREDICT LOW TEMPERATURE BINDER/AGGREGATE FAILURE? Paper presented at Proceedings of the 8th Conference on Asphalt Pavements for Southern Africa (CAPSA'04).
- Louw, K., Spence, K. and Kuun, P. 2004. The use of Bitumen Emulsions as a Cost Effective Solution for Constructing Seals during Winter. Paper presented at Proceedings of the 8th Conference on Asphalt Pavements for Southern Africa (CAPSA'04).Sun City-South Africa.
- Louw, K. 2014. Emulsion Precoats, e-mail to Twagirimana, E (Online), 29th August. Available e-mail: 18119425@sun.ac.za
- Lyttleton, D. & Traxler, R. 1948. Flow Properties of Asphalt Emulsions. *Industrial & Engineering Chemistry*, 40(11).
- Merrill, W.W., Pocius, A.V., Thakker, B.V. & Tirrell, M. 1991. Direct Measurement of Molecular Level Adhesion Forces between Biaxially Oriented Solid Polymer Films. *Langmuir*, 7(9).
- Mertens, E. and Wright, J. 1959. Cationic Asphalt Emulsions: How they Differ from Conventional Emulsions in Theory and Practice. Paper presented at Highway Research Board Proceedings,38, 386-397
- Miller.T. 2010. Development of bond strength test for improved characterization of asphalt emulsion. Master's Thesis. Wisconsin: University of Wisconsin.
- Milne, T. & Jenkins, K.J. 2005. Towards Modelling Road Surfacing Seal Performance: Performance Testing and Mechanistic Behavioural Model: Technical Paper. *Journal of the South African Institution of Civil Engineering= Joernaal van die Suid-Afrikaanse Instituut van Siviele Ingenieurswese*, 47(3).
- Milne, T. & Visser, A.T. 2011. Bitumen Rubber Seal Behaviour Assessment. Paper presented at 30th annual Southern African Transport Conference.
- Mitchell, J.K.(ed.). 1993. *Fundamentals of soil behaviour*. Vol. Second Edition. New York: John Wiley & Sons, Inc.
- Montgomery, D.C & Runger, G.C.2007. *Applied Statistics and Probability for Engineers*. USA: John Wiley & Sons, Inc.
- Packham, D.E. 2006. *Handbook of adhesion*. England: John Wiley & Sons
- Petersen, J.C. & Plancher, H. 1998. Model Studies and Interpretive Review of the Competitive Adsorption and Water Displacement of Petroleum Asphalt Chemical Functionalities on Mineral Aggregate Surfaces. *Petroleum Science and Technology*, 16(1-2).

- Petersen, J. C, Robertson, R. E, Branthaver, J. F, Harnsberger, P, Duvall J. J, Kim, S. S, Anderson, D. A, Christiansen, D. W and Bahia, H. U. 1994. Binder Characterization and Evaluation. Volume 1. (Report SHRP-A-367). Washington D.C. :National Research Council.
- Plancher, H., Dorrence, S. and Petersen, J. 1977. Identification of Chemical Types in Asphalts Strongly Adsorbed at the Asphalt-Aggregate Interface and their Relative Displacement by Water. Paper presented at Association of Asphalt Paving Technologists Proceedings .Vol.46.
- Pocius, A. 1997. *Adhesion and Adhesives Technology*. Cincinnati, Ohio: Hanser/Gardner Publications, Inc.
- Pretorius, M.W. 2013. Adhesion of Bitumen Test Using a BBS Test. Unpublished Bachelor of Engineering Thesis. Stellenbosch: Stellenbosch University.
- Raushan. 2011. *Measurement system analysis*. [Online]. Available: <http://raushan-advanceinnovationgroup.blogspot.com/2011/10/measurement-system-analysis.html> [2014]
- Read, J. & Whiteoak, D.(eds.) 2003. *The shell bitumen handbook*. Vol. Fifth Edition UK: Thomas Telford Publishing
- Roberts, F.L., Kandhal, P.S., Brown, E.R., Lee, D. & Kennedy, T.W. 1996. *Hot Mix Asphalt Materials, Mixture Design and Construction*. National Asphalt Pavement Association Research and Education Foundation. Lanham, Maryland.
- Ross, D., Mirsalis, J., Loo, B. & Rhee, S. 1991. Fundamentals of the Asphalt aggregate Bond. Progress Reports (SHRP-87-AIIR-11):(a) January,(b) May,(c) June,. California: SRI International.
- SABITA. 2012. SANRAL Initiates a Project to Maximise Seal Work through Out the Year. *Asphalt News, August*: 10-12.
- Sabita(ed.). 2007. *Bituminous binders for road construction and maintenance*. Manual 2. Vol. 4th Edition Howard Place: Sabita.
- Sabita. 2006. Interim Guidelines for Primes and Stone Precoating Fluids. Manual 26. Howard Place: Sabita.
- Salou, M., Siffert, B. & Jada, A. 1998. Study of the Stability of Bitumen Emulsions by Application of DLVO Theory. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 142(1).
- SANRAL. 2013. South African pavement engineering manual (SAPEM) Vol. 1, in *Chapter 3: Materials testing*, South Africa: South African National Roads Agency Limited (SANRAL). 42
- SANRAL. 2007. Technical Recommendations for Highways (TRH3): Design and Construction of Surfacing Seals. Pretoria: South African National Roads Agency Ltd.
- Schramm, G. & Haake, G. 1994. *A practical approach to rheology and rheometry*. Germany: Haake Karlsruhe GmbH.

- Senadheera, S., Tock, Wm. R., Hossain M. S, Yazgan, B and Das, S. 2006. A testing and evaluation protocol to assess seal coat binder-aggregate compatibility-Research report FHWA/TX-06/0-4362-1. Texas: Texas Tech University, Center for Multidisciplinary Research in Transportation
- Senadheera, S. and Yazgan, B. 2008. Incorporating Construction and Performance Conditions to Develop a Testing Protocol to Select Seal Coat Aggregate-Binder Combinations. Paper presented at International Sprayed Sealing Conference, 1st, 2008, Adelaide, South Australia, Australia.
- Shuler, S. 2011. *Manual for emulsion-based chip seals for pavement preservation*. Washington D.C: Transportation Research Board.
- Shuler, S. 2011. When to safely broom or remove traffic control on fresh emulsified asphalt chip seals. Paper submitted for presentation and publication at the transportation Research Board 90th Annual Meeting. Washington D.C.
- South Africa Government Online. 2014. *About SA-geography and climate*. [Online]. Available: <http://www.gov.za/aboutsa/geography.htm> [13/07/2014]
- Stander, A.H. & Jenkins.K.J. 2011. Bitumen bond strength (BBS) test using the pneumatic adhesive tensile strength instrument (PATTI). Unpublished Bachelor of Engineering Thesis. Stellenbosch: Stellenbosch University.
- Tarrer, A. & Wagh, V. 1991. The effect of the physical and chemical characteristics of the aggregate on bonding- Report No. SHRP-A/UIR-91-507. Washington, D.C: Strategic Highway Research Program, National Research Council.
- Thelen, E. 1958. Surface Energy and Adhesion Properties in Asphalt-Aggregate Systems, *HRB*, Bulletin.192. Washington D.C: Highway Research Board.
- Towler, J. and Dawson, J. 2008. History of Chipsealing in New Zealand–Hanson to P/17. Paper presented at 1st Sprayed Sealing Conference–Cost Effective High Performance Surfacing. Adelaide: Australian Roads Research Board Group Ltd.
- Transit NZ, RCA, Roding NZ. 2005. *Typical chipseal performance, Chipsealing in new zealand*. Wellington, New Zealand: Transit New Zealand, Road Controlling Authorities, Roding New Zealand
- Twagira, E.M. 2010. Influence of durability properties on performance of bitumen stabilised materials. PhD dissertation. Stellenbosch: Stellenbosch University.
- Van de Ven, M. and Jenkins,K.J. 2013. Pavement Materials-II: Modelling of Binder Behaviour. Unpublished Class notes. Stellenbosch: Stellenbosch University.
- Van Zyl, G., O’Connell, J. and Paige-Green, P. 2012. Maximising Seal Work Throughout the Year, Phase 1 Report. Unpublished Report.

- Venugopalan, S. N. 2014. *States of matter*. [Online]. Available:
<http://www.chemmaster.co.in/showchapter.php?id=5&id2=76&title=States%20of%20Matter>
[May 31, 2014]
- Vickey, N.A. 2007. Development of an advanced adhesion test for polymer interfaces. Master of Science Thesis. Maryland : University of Maryland.
- Weinert, H.H. 1980. *The natural road construction materials of southern africa*. Pretoria: National Institute for Transport and Road Research
- Yildirim, Y. 2007. Polymer Modified Asphalt Binders. *Construction and Building Materials*, 21(1).
- Yusoff, N.I.M., Shaw, M.T. & Airey, G.D. 2011. Modelling the Linear Viscoelastic Rheological Properties of Bituminous Binders. *Construction and Building Materials*, 25(5).
- Zoghi, M., Ebrahimpour, A. & Pothukutchi, V. 2010. Performance Evaluation of Chip Seals in Idaho, Final Report. Idaho: Idaho State University.

APPENDIX

A. PRECOAT APPLICATION RATE

Surfacing seals aggregates precoating process is described in details in section 2.2.3. In the aforementioned section, the table 2-1 illustrates typical precoating fluid application rates frequently used in South Africa. From the table, it is obvious that in South African context, the precoat application rates are specified in terms of volume of precoats to be applied to unit volume of cover aggregates (litres/m³).

Due to the nature of BBS test and in an effort to simulate the application rate typically used on the field, the conversion of application rate into litres/m² was deemed to be necessary. In order to achieve this conversion, the aggregate size to be used for sealing was considered as 9.5mm. According to table 2-1, the corresponding precoat application rate range is 12-17 litres/m³. The application rate of 15litres/m³ was adopted in this study.

In an effort to estimate the surface area of aggregates contained in a specific mass of aggregates, Edwards has proposed the surface area factors based on the aggregate's size. The proposed surface area factors are illustrated in Table A-1. Edwards' surface area factors are based on the assumption that, the aggregates have a sphere shape and smooth sides.

Table A-1: Surface Area Factors proposed by Edwards (Roberts et al., 1996)

Sieve Size #	>#4	#4	#8	#16	#30	#50	#100	#200
Diameter (mm)		4.75	2.36	1.18	0.60	0.30	0.15	0.075
Surface Area (m ² /kg)	0.41	0.41	0.82	1.64	2.87	6.14	12.29	32.77
Surface Area (ft ² /lb)	2	2	4	8	14	30	60	160

Later, Hveem using the above-mentioned factors has developed a method for estimating the surface area of aggregates based on aggregate blend gradation. For a specific aggregate blend, the surface area of aggregates per unit mass is the summation of the products of the surface area factors times the percent material passing each sieve size (Roberts et al., 1996). This is expressed as shown below:

$$SA = \sum SF_i * P_i$$

With: SA: Surface Area (m²/kg)

SF_i: Surface Factor for Sieve i

P_i: Percent passing sieve i, in decimal form

In this study, a single size cover aggregates of 9.5mm was adopted as mentioned previously. This implies that P=1 and SF =0.41 (From Table A-1).

Thus, SA=0.41*1=**0.41m²/kg**

In order to determine the surface area of aggregates per cubic metres of aggregates blend, it was assumed that the bulk density of stone is 2650kg/m³.

Surface Area per Volume= SA*Density= 0.41*2650= **1086.5m²/m³**

Application rate = 15litres/m³

Application rate per m² = 15*1/1086.5 = **0.013806 litres/m²**

The aggregate stones to be precoat have cylindrical shape with diameter D, (D=60mm). The precoat is applied on the top face only. Thus, the surface area to be precoat (SA_p) per stone is calculated as follows:

$$SA_p = (\pi * D^2) / 4 = (3.14 * 60^2) / 4 = \mathbf{0.002826m^2}$$

Volume of precoat per stone = 0.013806 * 0.002826 = 3.90152 * 10⁻⁵ litres = **0.040 ml/stone**

The precoat application rate above was elaborated based on the normal bitumen based precoat such as Colcote. The normal precoat are typically consisted of about 50% kerosene and 50% residual bitumen. However, the precoat fluids used in this study have different percentage of residual bitumen. Thus, adjustment of application rate was necessary.

- Adjustment for SS60 (30% water diluted) precoat

As aforementioned in section 3.5.2, the residual bitumen of this type of precoat was determined to be 46.2%.

Thus, its adjusted application rate = 0.040 + (0.040* (50-46.2)/50) = **0.043 ml/stone**

By converting this application rate in mm³ and SA_p in mm², the thickness of the precoat on the stone may be determined as **0.015mm**

- Adjustment for anionic emulsion with cutters (AwC) and cationic emulsion with cutters (CwC) precoats

The residual bitumen of this type of precoats were determined to be 27.5% as mentioned in section 3.5.2.

Thus, its adjusted application rate = $0.040 + (0.040 * (50 - 27.5) / 50) = \mathbf{0.058 \text{ ml/stone}}$

By converting this application rate in mm^3 and SA_p in mm^2 , the thickness of the precoat on the stone may be determined as **0.020mm**.

The values calculated above are the minimum application rates based on the specifications. However, in the laboratory, there is a need to control the uniformity of stone precoating. This was intended to ensure that the results obtained in testing are comparable. In order to make the stone precoating uniformity control possible, the precoat thickness of 0.1mm was adopted for SS60 (30% water diluted) throughout this study. This resulted in a precoat volume of **0.28 ml** for SS60 precoats. On the other hand, having in mind that, the residual binder content are different for different precoats, the thickness of the AwC and CwC precoats was determined using the ratio between the thicknesses obtained in the previous calculations ($0.015/0.020 = 0.74$). The thickness of 0.13mm was found to be appropriate for AwC and CwC. This is equivalent to approximately **0.38ml** of AwC and CwC.

For quality control purposes, as has been mentioned in section 4.3.1, the weight of the specimens were recorded before and after precoating, to ensure whether the precoating process could not interfere with interpretation of results. The records made through-out the testing phase are presented below:

Table A-2: Precoat Application Control for Cationic emulsion with Cutters (CwC)

Test Identification Number	Precoat Type	Condition of Precoat	Weight Before Precoat	Weight After Precoat	Weight of Precoat	Volume of Precoat
D1-CwC-70-100-24hrs-Dry	CwC	Dry	153.1	153.54	0.44	0.41
D4-9-CwC-70-100-24hrs-Dry	CwC	Dry	151.66	152.04	0.38	0.37
D3-CwC-70-100-24hrs-Dry	CwC	Dry	108.46	108.83	0.37	0.35
D4-CwC-70-100-24hrs-Dry	CwC	Dry	149.86	150.27	0.41	0.39
D5-D4-CwC-70-100-24hrs-Dry	CwC	Dry	149.71	150.09	0.38	0.36
D6-CwC-70-100-24hrs-Dry	CwC	Dry	176.59	176.99	0.40	0.38
D1-D18-CwC-S-E1-24hrs-Dry	CwC	Dry	135.87	136.29	0.42	0.40
D2-5-CwC-S-E1-24hrs-Dry	CwC	Dry	130.67	131.08	0.41	0.39
D3-8-CwC-S-E1-24hrs-Dry	CwC	Dry	128.92	129.26	0.34	0.33
D1-D3-CwC-SC-E1-2hrs-Dry	CwC	Dry	153.31	153.66	0.35	0.33
D2-D5-CwC-SC-E1-2hrs-Dry	CwC	Dry	154.29	154.67	0.38	0.36
D3-D1-CwC-SC-E1-2hrs-Dry	CwC	Dry	108.69	109.11	0.42	0.40
D1-16-CwC-SC-E1-24hrs-Dry	CwC	Dry	130.81	131.24	0.43	0.41
D2-15-CwC-SC-E1-24hrs-Dry	CwC	Dry	151.91	152.32	0.41	0.39
D3-19-CwC-SC-E1-24hrs-Dry	CwC	Dry	136.01	136.46	0.45	0.42
D1-D6-CwC-70-100-24hrs-Wet	CwC	Wet	176.54	176.93	0.39	0.37
D2-D3-CwC-70-100-24hrs-Wet	CwC	Wet	108.6	109.05	0.45	0.42
D3-D6(AB)-CwC-70-100-24hrs-Wet	CwC	Wet	185.27	185.68	0.41	0.39
D5-D19-CwC-S-E1-24hrs-Wet	CwC	Wet	135.11	135.54	0.43	0.41
D2-CwC-S-E1-24hrs-Wet	CwC	Wet	153.97	154.36	0.39	0.37
D3-CwC-S-E1-24hrs-Wet	CwC	Wet	108.43	108.83	0.40	0.38
D4-CwC-S-E1-24hrs-Wet	CwC	Wet	149.85	150.26	0.41	0.39
D5-CwC-S-E1-24hrs-Wet	CwC	Wet	116.73	117.16	0.43	0.41
D6-CwC-S-E1-24hrs-Wet	CwC	Wet	176.54	176.89	0.35	0.33
D1-18-CwC-SC-E1-2hrs-Wet	CwC	Wet	135.06	135.44	0.38	0.36
D2-12-CwC-SC-E1-2hrs-Wet	CwC	Wet	152.68	153.04	0.36	0.34
D3-13-CwC-SC-E1-2hrs-Wet	CwC	Wet	150.96	151.36	0.40	0.38
D4-21-CwC-SC-E1-2hrs-Wet	CwC	Wet	161.05	161.49	0.44	0.42
D5-D4-CwC-SC-E1-2hrs-Wet	CwC	Wet	149.84	150.26	0.42	0.40
D6-CwC-SC-E1-2hrs-Wet	CwC	Wet	83.91	84.31	0.40	0.38
D1-12-CwC-SC-E1-24hrs-Wet	CwC	Wet	152.72	153.16	0.44	0.41
D2-SU-D1-CwC-SC-E1-24hrs-Wet	CwC	Wet	151.61	152.04	0.43	0.41
D3-13-CwC-SC-E1-24hrs-Wet	CwC	Wet	150.99	151.36	0.37	0.35
D4-14-CwC-SC-E1-24hrs-Wet	CwC	Wet	114.86	115.27	0.41	0.39
D5-20-CwC-SC-E1-24hrs-Wet	CwC	Wet	165.3	165.69	0.39	0.37
D6-SU-D2-CwC-SC-E1-24hrs-Wet	CwC	Wet	154.08	154.51	0.43	0.40
G1-CwC-70-100-24hrs-Dry	CwC	Dry	165.8	166.18	0.38	0.36
G2-CwC-70-100-24hrs-Dry	CwC	Dry	134.75	135.16	0.41	0.39
G4-2-CwC-70-100-24hrs-Dry	CwC	Dry	125.28	125.68	0.40	0.38

G1-CwC-S-E1-24hrs-Dry	CwC	Dry	83.9	84.32	0.42	0.40	
G2-CwC-S-E1-24hrs-Dry	CwC	Dry	124.86	125.25	0.39	0.37	
G3-CwC-S-E1-24hrs-Dry	CwC	Dry	132.14	132.52	0.38	0.36	
G1-G5-CwC-SC-E1-2hrs-Dry	AwC	Wet	150.14	150.52	0.38	0.36	
G2-G1-CwC-SC-E1-2hrs-Dry	AwC	Wet	116.98	117.38	0.40	0.38	
G3-11-CwC-SC-E1-2hrs-Dry	AwC	Wet	176.81	177.21	0.40	0.38	
G1-12-CwC-SC-E1-24hrs-Dry	CwC	Dry	125.27	125.66	0.39	0.37	
G2-14-CwC-SC-E1-24hrs-Dry	CwC	Dry	110.66	111.08	0.42	0.40	
G3-18-CwC-SC-E1-24hrs-Dry	CwC	Dry	160.39	160.82	0.43	0.41	
G1-CwC-70-100-24hrs-Wet	CwC	Wet	165.82	166.20	0.38	0.36	
G2-CwC-70-100-24hrs-Wet	CwC	Wet	134.77	135.16	0.39	0.37	
G3-CwC-70-100-24hrs-Wet	CwC	Wet	138.87	139.29	0.42	0.40	
G4-G1-CwC-S-E1-24hrs-Wet	CwC	Wet	165.84	166.26	0.42	0.40	
G2-CwC-S-E1-24hrs-Wet	CwC	Wet	124.87	125.28	0.41	0.39	
G6-CwC-S-E1-24hrs-Wet	CwC	Wet	132.15	132.59	0.44	0.42	
G1-11-CwC-SC-E1-2hrs-Wet	CwC	Wet	126.18	126.58	0.40	0.38	
G2-20-CwC-SC-E1-2hrs-Wet	CwC	Wet	118.5	118.89	0.39	0.37	
G3-21-CwC-SC-E1-2hrs-Wet	CwC	Wet	130.9	131.32	0.42	0.40	
G1-17-CwC-SC-E1-24hrs-Wet	CwC	Wet	135	135.42	0.42	0.40	
G2-15-CwC-SC-E1-24hrs-Wet	CwC	Wet	124.44	124.95	0.51	0.49	
G3-16-CwC-SC-E1-24hrs-Wet	CwC	Wet	134.93	135.43	0.50	0.48	
					Average		0.39
					IQR		0.035
					CoV		7.7%

Note:

IQR: Interquartile Range

CoV: Coefficient of Variation

Table A-3: Precoat Application Control for Anionic emulsion with Cutters (AwC)

Test Identification Number	Precoat Type	Condition of Precoat	Weight Before Precoat	Weight After Precoat	Weight of Precoat	Volume of Precoat
D1-AwC-70-100-24hrs-Dry	AwC	Dry	153.56	153.98	0.42	0.40
D3-AwC-70-100-24hrs-Dry	AwC	Dry	109	109.41	0.41	0.39
D4-AwC-70-100-24hrs-Dry	AwC	Dry	150.41	150.90	0.49	0.47
D6-6-AwC-S-E1-24hrs-Dry	AwC	Dry	150.8	151.22	0.42	0.40
D7-D5-AwC-S-E1-24hrs-Dry	AwC	Dry	117.01	117.44	0.43	0.41
D8-3-AwC-S-E1-24hrs-Dry	AwC	Dry	114.66	115.06	0.40	0.38
D1-SU-D2-AwC-SC-E1-2hrs-Dry	AwC	Dry	149.93	150.36	0.43	0.41
D2-SU-D1-AwC-SC-E1-2hrs-Dry	AwC	Dry	128.98	129.37	0.39	0.37
D3-18-AwC-SC-E1-2hrs-Dry	AwC	Dry	135.18	135.63	0.45	0.43
D1-12-AwC-SC-E1-24hrs-Dry	AwC	Dry	152.68	153.09	0.41	0.39
D2-13-AwC-SC-E1-24hrs-Dry	AwC	Dry	150.96	151.38	0.42	0.40
D3-14-AwC-SC-E1-24hrs-Dry	AwC	Dry	114.86	115.29	0.43	0.41
D1-AwC-70-100-24hrs-Wet	AwC	Wet	153.31	153.70	0.39	0.37
D2-AwC-70-100-24hrs-Wet	AwC	Wet	154.29	154.70	0.41	0.39
D3-AwC-70-100-24hrs-Wet	AwC	Wet	108.69	109.15	0.46	0.44
D1-AwC-S-E1-24hrs-Wet	AwC	Wet	150.14	150.50	0.36	0.35
D2-AwC-S-E1-24hrs-Wet	AwC	Wet	116.98	117.37	0.39	0.37
D3-AwC-S-E1-24hrs-Wet	AwC	Wet	176.81	177.21	0.40	0.38
D1-19-AwC-SC-E1-2hrs-Wet	AwC	Wet	136.03	136.45	0.42	0.39
D2-D4-AwC-SC-E1-2hrs-Wet	AwC	Wet	149.91	150.30	0.39	0.37
D1-14-AwC-SC-E1-2hrs-Wet	AwC	Wet	153.35	153.75	0.40	0.38
D1-22-AwC-SC-E1-24hrs-Wet	AwC	Wet	142.82	143.23	0.41	0.39
D2-21-AwC-SC-E1-24hrs-Wet	AwC	Wet	161.35	161.79	0.44	0.42
D3-17-AwC-SC-E1-24hrs-Wet	AwC	Wet	185.3	185.70	0.40	0.38
					0.00	
G1-AwC-70-100-24hrs-Dry	AwC	Dry	165.88	166.29	0.41	0.39
G2-AwC-70-100-24hrs-Dry	AwC	Dry	134.8	135.20	0.40	0.38
G3-AwC-70-100-24hrs-Dry	AwC	Dry	138.92	139.34	0.42	0.40
G4-AwC-70-100-24hrs-Dry	AwC	Dry	83.94	84.37	0.43	0.41
G5-AwC-70-100-24hrs-Dry	AwC	Dry	124.91	125.32	0.41	0.39
G6-AwC-70-100-24hrs-Dry	AwC	Dry	132.2	132.60	0.40	0.38
G1-G2-AwC-S-E1-24hrs-Dry	AwC	Dry	134.78	135.17	0.39	0.37
G2-G3-AwC-S-E1-24hrs-Dry	AwC	Dry	138.87	139.32	0.45	0.42
G3-1-AwC-S-E1-24hrs-Dry	AwC	Dry	126.18	126.58	0.40	0.38
G1-G2-AwC-SC-E1-2hrs-Dry	AwC	Dry	134.77	135.16	0.39	0.37
G2-G3-AwC-SC-E1-2hrs-Dry	AwC	Dry	138.84	139.33	0.49	0.47
G3-14-AwC-SC-E1-2hrs-Dry	AwC	Dry	110.66	111.07	0.41	0.39
G1-15-AwC-SC-E1-24hrs-Dry	AwC	Dry	124.44	124.83	0.39	0.37
G2-17-AwC-SC-E1-24hrs-Dry	AwC	Dry	135	135.41	0.41	0.39

G3-19-AwC-SC-E1-24hrs-Dry	AwC	Dry	138.49	138.89	0.40	0.38	
G1-G5-AwC-70-100-24hrs-Wet	AwC	Wet	124.88	125.28	0.40	0.38	
G2-G4-AwC-70-100-24hrs-Wet	AwC	Wet	83.93	84.34	0.41	0.39	
G3-G17-AwC-70-100-24hrs-Wet	AwC	Wet	127.91	128.36	0.45	0.43	
G1-AwC-S-E1-24hrs-Wet	AwC	Wet	165.85	166.27	0.42	0.40	
G2-AwC-S-E1-24hrs-Wet	AwC	Wet	134.78	135.19	0.41	0.39	
G3-AwC-S-E1-24hrs-Wet	AwC	Wet	138.88	139.33	0.45	0.43	
G4-AwC-S-E1-24hrs-Wet	AwC	Wet	83.91	84.32	0.41	0.39	
G5-AwC-S-E1-24hrs-Wet	AwC	Wet	124.89	125.28	0.39	0.38	
G6-AwC-S-E1-24hrs-Wet	AwC	Wet	132.18	132.62	0.44	0.42	
G1-12-AwC-SC-E1-2hrs-Wet	AwC	Wet	125.22	125.62	0.40	0.38	
G2-G1-AwC-SC-E1-2hrs-Wet	AwC	Wet	165.7	166.03	0.33	0.32	
G3-G2-AwC-SC-E1-2hrs-Wet	AwC	Wet	134.75	135.17	0.42	0.39	
G4-11-AwC-SC-E1-2hrs-Wet	AwC	Wet	126.14	126.56	0.42	0.40	
G5-G5-AwC-SC-E1-2hrs-Wet	AwC	Wet	124.85	125.31	0.46	0.44	
G6-18-AwC-SC-E1-2hrs-Wet	AwC	Wet	160.36	160.75	0.39	0.37	
G1-SU-G5-AwC-SC-E1-24hrs-Wet	AwC	Wet	124.82	125.21	0.39	0.37	
G2-SU-G6-AwC-SC-E1-24hrs-Wet	AwC	Wet	132.11	132.58	0.47	0.45	
G3-19-AwC-SC-E1-24hrs-Wet	AwC	Wet	138.49	138.89	0.40	0.38	
G4-20-AwC-SC-E1-24hrs-Wet	AwC	Wet	118.49	118.91	0.42	0.40	
G5-21-AwC-SC-E1-24hrs-Wet	AwC	Wet	130.9	131.34	0.44	0.41	
G6-13-AwC-SC-E1-24hrs-Wet	AwC	Wet	127.89	128.37	0.48	0.45	
					Average		0.40
					IQR		0.035
					CoV		7.0%

Table A-4: Precoat Application Control for SS60, 30% water diluted

Test Identification Number	Precoat Type	Condition of Precoat	Weight Before Precoat	Weight After Precoat	Weight of Precoat	Volume of Precoat
D1-D2-SS60-70-100-24hrs-Dry	SS60	Dry	152.79	153.05	0.26	0.25
D2-D24-SS60-70-100-24hrs-Dry	SS60	Dry	115.16	115.45	0.29	0.28
D3-D16-2-SS60-70-100-24hrs-Dry	SS60	Dry	152.09	152.40	0.31	0.29
D1-D22-SS60-S-E1-24hrs-Dry	SS60	Dry	129.53	129.80	0.27	0.26
D2-D23-SS60-SE-1-24hrs-Dry	SS60	Dry	165.44	165.76	0.32	0.31
D3-D14-SS60-S-E1-24hrs-Dry	SS60	Dry	130.95	131.26	0.31	0.29
D1-SU-D1-SS60-SC-E1-2hrs-Dry	SS60	Dry	151.71	152.01	0.30	0.29
D2-SU-D2-SS60-SC-E1-2hrs-Dry	SS60	Dry	154.13	154.44	0.31	0.30
D3-17-SS60-SC-E1-2hrs-Dry	SS60	Dry	185.28	185.56	0.28	0.27
D1-D1-SS60-SC-E1-24hrs-Dry	SS60	Dry	153.21	153.51	0.30	0.29
D2-9-SS60-SC-E1-24hrs-Dry	SS60	Dry	151.6	151.94	0.34	0.32
D3-22-SS60-SC-E1-24hrs-Dry	SS60	Dry	142.78	143.05	0.27	0.26
D1-D24-SS60-70-100-24hrs-Wet	SS60	Wet	114.92	115.20	0.28	0.26
D2-D2-SS60-70-100-24hrs-Wet	SS60	Wet	152.46	152.74	0.28	0.27
D3-D14-SS60-70-100-24hrs-Wet	SS60	Wet	130.95	131.28	0.33	0.31
D1-D22-SS60-SE-1-24hrs-Wet	SS60	Wet	129.21	129.52	0.31	0.29
D2-D16-SS60-SE-1-24hrs-Wet	SS60	Wet	151.75	152.07	0.32	0.31
D3-D12-SS60-SE-1-24hrs-Wet	SS60	Wet	153.86	154.14	0.28	0.27
D1-D1-SS60-SC-E1-2hrs-wet	SS60	Wet	153.29	153.58	0.29	0.28
D2-D2-SS60-SC-E1-2hrs-wet	SS60	Wet	149.85	150.15	0.30	0.29
D3-D3-SS60-SC-E1-2hrs-wet	SS60	Wet	108.54	108.81	0.27	0.26
D1-15-SS60-SC-E1-24hrs-Wet	SS60	Wet	151.91	152.21	0.30	0.29
D2-16-SS60-SC-E1-24hrs-Wet	SS60	Wet	130.81	131.10	0.29	0.27
D3-19-SS60-SC-E1-24hrs-Wet	SS60	Wet	136.03	136.34	0.31	0.29
D1-D4-SS60-70-100-24hrs-Wet(15C	SS60	Wet	149.89	150.21	0.32	0.31
D2-11-SS60-70-100-24hrs-Wet(15C	SS60	Wet	128.95	129.24	0.29	0.28

D3-12-SS60-70-100-24hrs-Wet(15C)	SS60	Wet	152.75	153.07	0.32	0.30
D1-14-SS60-S-E1-24hrs-Wet(15C)	SS60	Wet	114.81	115.10	0.29	0.27
D2-18-SS60-S-E1-24hrs-Wet(15C)	SS60	Wet	135.08	135.43	0.35	0.33
D3-21-SS60-S-E1-24hrs-Wet(15C)	SS60	Wet	161.18	161.48	0.30	0.29
D1-SU-D1-SS60-SC-E1-2hrs-Wet(15)	SS60	Wet	151.71	152.01	0.30	0.29
D2-SU-D2-SS60-SC-E1-2hrs-Wet(15)	SS60	Wet	154.13	154.53	0.40	0.38
D3-D1-SS60-SC-E1-2hrs-Wet(15C)	SS60	Wet	153.29	153.60	0.31	0.30
D1-SS60-SC-E1-24hrs-Wet(15C)	SS60	Wet	108.53	108.82	0.29	0.27
D2-D5-SS60-SC-E1-24hrs-Wet(15C)	SS60	Wet	116.95	117.26	0.31	0.30
D3-11-SS60-SC-E1-24hrs-Wet(15C)	SS60	Wet	128.91	129.22	0.31	0.29
G1-SS60-70-100-24hrs-Dry	SS60	Dry	165.85	166.14	0.29	0.28
G2-G18-SS60-70-100-24hrs-Dry	SS60	Dry	130.93	131.27	0.34	0.32
G3-G15-SS60-70-100-24hrs-Dry	SS60	Dry	110.72	111.00	0.28	0.26
G1-G17-SS60-S-E1-24hrs-Dry	SS60	Dry	127.94	128.26	0.32	0.30
G2-G6-2-SS60-S-E1-24hrs-Dry	SS60	Dry	125.28	125.64	0.36	0.34
G3-G14-SS60-S-E1-24hrs-Dry	SS60	Dry	124.53	124.86	0.33	0.32
G1-16-SS60-SC-E1-2hrs-Dry	SS60	Dry	134.96	135.24	0.28	0.27
G2-SU-G5-SS60-SC-E1-2hrs-Dry	SS60	Dry	124.87	125.17	0.30	0.28
G3-20-SS60-SC-E1-2hrs-Dry	SS60	Dry	118.52	118.82	0.30	0.29
G1-16-SS60-SC-E1-24hrs-Dry	SS60	Dry	134.83	135.15	0.32	0.30
G2-G3-SS60-SC-E1-24hrs-Dry	SS60	Dry	138.74	139.03	0.29	0.28
G3-18-SS60-SC-E1-24hrs-Dry	SS60	Dry	127.87	128.18	0.31	0.29
G1-G18-SS60-70-100-24hrs-Wet	SS60	Wet	130.93	131.29	0.36	0.34
G2-G13-SS60-70-100-24hrs-Wet	SS60	Wet	126.2	126.52	0.32	0.30
G3-G17-SS60-70-100-24hrs-Wet	SS60	Wet	127.94	128.35	0.41	0.39
G1-G15-SS60-SE-1-24hrs-Wet	SS60	Wet	110.7	110.98	0.28	0.27
G2-G14-SS60-SE-1-24hrs-Wet	SS60	Wet	124.5	124.79	0.29	0.28
G3-G12-SS60-SE-1-24hrs-Wet	SS60	Wet	135.07	135.38	0.31	0.30
G1-G1-SS60-SC-E1-2hrs-wet	SS60	Wet	165.8	166.14	0.34	0.32

G2-G2-SS60-SC-E1-2hrs-wet	SS60	Wet	134.76	135.08	0.32	0.30
G3-G3-SS60-SC-E1-2hrs-wet	SS60	Wet	138.84	139.08	0.24	0.23
G1-G5-SS60-SC-E1-24hrs-Wet	SS60	Wet	124.85	125.15	0.30	0.28
G2-12-SS60-SC-E1-24hrs-Wet	SS60	Wet	125.22	125.54	0.32	0.30
G3-G3-SS60-SC-E1-24hrs-Wet	SS60	Wet	138.84	139.17	0.33	0.31
G1-G2-SS60-70-100-24hrs-Wet(15C)	SS60	Wet	134.79	135.11	0.32	0.30
G2-17-SS60-70-100-24hrs-Wet(15C)	SS60	Wet	135.07	135.39	0.32	0.31
G3-18-SS60-70-100-24hrs-Wet(15C)	SS60	Wet	160.4	160.70	0.30	0.28
G1-19-SS60-S-E1-24hrs-Wet(15C)	SS60	Wet	138.5	138.86	0.36	0.35
G2-20-SS60-S-E1-24hrs-Wet(15C)	SS60	Wet	118.51	118.81	0.30	0.29
G3-SS60-S-E1-24hrs-Wet(15C)	SS60	Wet	138.88	139.20	0.32	0.30
G1-G4-SS60-SC-E1-2hrs-Wet(15C)	SS60	Wet	83.93	84.28	0.35	0.33
G2-14-SS60-SC-E1-2hrs-Wet(15C)	SS60	Wet	110.66	110.96	0.30	0.28
G3-SU-G5-SS60-SC-E1-2hrs-Wet(15)	SS60	Wet	124.87	125.18	0.31	0.29
G1-G2-SS60-SC-E1-24hrs-Wet(15C)	SS60	Wet	134.75	135.05	0.30	0.29
G2-16-SS60-SC-E1-24hrs-Wet(15C)	SS60	Wet	134.95	135.25	0.30	0.28
G3-18-SS60-SC-E1-24hrs-Wet(15C)	SS60	Wet	160.37	160.69	0.32	0.31
					Average	0.29
					IQR	0.027
					CoV	9.1%

B. DETAILED RESULTS FOR BBS TESTS

BBS RESULTS FOR 70/100									
Combinations	BBS (kPa)	Loading Rate (kPa/s)	Time to Break (sec)	Aver.B BS (kPa)	BBS CoV	Aver. L.R (kPa/s)	L.R. CoV	Av. T.B (sec)	T.B CoV
G1-nonprecoated-70-100-24hrs	1877.2	777.5	2.7	1687.5	9.7%	776.4	1.3%	2.4	11.3%
G2-nonprecoated-70-100-24hrs	1584.5	785.7	2.2						
G3-nonprecoated-70-100-24hrs	1600.8	765.9	2.2						
G1-G2-SS60-70-100-24hrs-Wet(15C	1890.8	749.1	2.7	1802.2	4.7%	750.5	0.4%	2.6	4.5%
G2-17-SS60-70-100-24hrs-Wet(15C	1795.9	748.0	2.6						
G3-18-SS60-70-100-24hrs-Wet(15C	1720.0	754.3	2.5						
G1-G18-SS60-70-100-24hrs-Wet	1869.1	749.3	3.0	1848.3	7.8%	783.3	3.8%	2.8	10.7%
G2-G13-SS60-70-100-24hrs-Wet	1980.2	797.4	2.9						
G3-G17-SS60-70-100-24hrs-Wet	1695.6	803.1	2.5						
G1-SS60-70-100-24hrs-Dry	2075.1	778.5	3.0	2071.4	0.9%	756.2	5.0%	3.1	6.5%
G2-G18-SS60-70-100-24hrs-Dry	2088.6	713.0	3.4						
G3-G15-SS60-70-100-24hrs-Dry	2050.7	777.1	3.0						
G1-G5-AwC-70-100-24hrs-Wet	1508.6	756.4	2.2	1652.3	10.4%	759.2	0.3%	2.4	10.1%
G2-G4-AwC-70-100-24hrs-Wet	1842.0	759.6	2.7						
G3-G17-AwC-70-100-24hrs-Wet	1606.2	761.6	2.4						
G1-AwC-70-100-24hrs-Dry	2140.1	761.1	3.0	2074.6	3.1%	748.6	2.1%	3.0	2.5%
G2-AwC-70-100-24hrs-Dry	2115.7	738.5	3.0						
G3-AwC-70-100-24hrs-Dry	2132.0	773.6	2.9						
G4-AwC-70-100-24hrs-Dry	2058.8	744.0	3.0						
G5-AwC-70-100-24hrs-Dry	1993.7	731.4	3.1						
G6-AwC-70-100-24hrs-Dry	2007.3	742.8	3.0						
G1-CwC-70-100-24hrs-Wet	1606.2	763.9	2.6	1608.0	8.4%	752.4	1.6%	2.4	11.5%
G2-CwC-70-100-24hrs-Wet	1473.4	753.6	2.1						
G3-CwC-70-100-24hrs-Wet	1744.4	739.6	2.5						
G1-CwC-70-100-24hrs-Dry	1728.2	762.0	2.3	1880.8	7.8%	749.0	1.6%	2.6	12.5%
G2-CwC-70-100-24hrs-Dry	1893.5	747.5	2.6						
G4-2-CwC-70-100-24hrs-Dry	2020.8	737.7	3.0						
D1-nonprecoated-70-100-24hrs	2066.9	774.7	2.9	1886.2	8.7%	757.9	2.1%	2.7	8.8%
D2-nonprecoated-70-100-24hrs	1847.4	743.9	2.7						
D3-nonprecoated-70-100-24hrs	1744.4	755.1	2.4						
D1-D4-SS60-70-100-24hrs-	2096.7	759.9	3.1	2078.7	17.2%	749.0	1.5%	3.2	20.6%

Wet(15C									
D2-11-SS60-70-100-24hrs-Wet(15C	2427.4	737.5	3.9						
D3-12-SS60-70-100-24hrs-Wet(15C	1711.9	749.5	2.6						
D1-D24-SS60-70-100-24hrs-Wet	1741.4	729.3	2.7	1940.4	11.4%	744.1	2.7%	2.9	12.8%
D2-D2-SS60-70-100-24hrs-Wet	1901.6	766.8	2.8						
D3-D14-SS60-70-100-24hrs-Wet	2178.0	736.3	3.4						
D1-D2-SS60-70-100-24hrs-Dry	2234.9	756.6	3.4	2217.8	3.5%	746.5	1.7%	3.4	4.2%
D2-D24-SS60-70-100-24hrs-Dry	2132.0	750.7	3.4						
D3-D16-2-SS60-70-100-24hrs-Dry	2286.4	732.3	3.6						
D1-AwC-70-100-24hrs-Wet	1446.3	766.6	2.1	1331.6	8.3%	777.2	5.5%	1.9	13.5%
D2-AwC-70-100-24hrs-Wet	1226.8	824.0	1.6						
D3-AwC-70-100-24hrs-Wet	1321.7	741.0	2.1						
D1-AwC-70-100-24hrs-Dry	1939.5	749.7	2.7	1980.2	2.4%	763.6	4.0%	2.8	6.2%
D3-AwC-70-100-24hrs-Dry	2031.7	798.6	2.8						
D4-AwC-70-100-24hrs-Dry	1969.4	742.5	3.0						
D1-D6-CwC-70-100-24hrs-Wet	1852.8	769.5	2.6	1900.7	3.0%	770.8	0.6%	2.7	4.4%
D2-D3-CwC-70-100-24hrs-Wet	1885.3	775.6	2.7						
D3-D6(AB)-CwC-70-100-24hrs-Wet	1963.9	767.2	2.9						
D1-CwC-70-100-24hrs-Dry	2007.3	763.2	2.7	2183.0	6.5%	752.3	3.0%	3.1	13.6%
D4-9-CwC-70-100-24hrs-Dry	2240.4	736.6	3.3						
D3-CwC-70-100-24hrs-Dry	2115.7	766.5	2.9						
D4-CwC-70-100-24hrs-Dry	2294.6	754.4	3.4						
D5-D4-CwC-70-100-24hrs-Dry	2373.2	715.9	3.8						
D6-CwC-70-100-24hrs-Dry	2066.9	777.1	2.8						

BBS RESULTS FOR S-E1									
Combinations	BBS (kPa)	Loading Rate (kPa/s)	Time to Break (sec)	Aver. BBS (kPa)	BBS CoV	Aver.L.R (kPa/s)	L.R. CoV	Av. T.B (sec)	T.B CoV
G1-nonprecoated-SE-1-24hrs	1308.1	795.3	1.7	1183.4	9.1%	761.9	4.5%	1.8	9.9%
G2-nonprecoated-S-E1-24hrs	1113.0	726.2	1.6						
G3-nonprecoated-S-E1-24hrs	1129.2	764.2	2.0						
G1-19-SS60-S-E1-24hrs-Wet(15C)	1443.6	751.8	2.1	1374.0	4.8%	753.8	0.7%	2.0	6.3%
G2-20-SS60-S-E1-24hrs-Wet(15C)	1367.7	759.9	2.0						
G3-SS60-S-E1-24hrs-Wet(15C)	1310.8	749.8	1.9						
G1-G15-SS60-SE-1-24hrs-Wet	1516.8	763.8	2.1	1409.3	6.8%	761.7	1.9%	2.2	6.2%
G2-G14-SS60-SE-1-24hrs-Wet	1378.6	774.9	2.3						
G3-G12-SS60-SE-1-24hrs-Wet	1332.5	746.4	2.1						
G1-G17-SS60-S-E1-24hrs-Dry	1443.6	781.3	2.2	1411.1	2.0%	777.8	0.9%	2.2	8.4%
G2-G6-2-SS60-S-E1-24hrs-Dry	1397.5	769.8	2.3						
G3-G14-SS60-S-E1-24hrs-Dry	1392.1	782.5	2.0						
G1-AwC-S-E1-24hrs-Wet	991.0	766.1	1.2	972.5	9.6%	776.9	1.8%	1.3	10.7%
G2-AwC-S-E1-24hrs-Wet	866.4	775.5	1.3						
G3-AwC-S-E1-24hrs-Wet	1047.9	801.1	1.5						
G4-AwC-S-E1-24hrs-Wet	877.2	763.8	1.4						
G5-AwC-S-E1-24hrs-Wet	950.4	784.2	1.2						
G6-AwC-S-E1-24hrs-Wet	1102.1	770.5	1.4						
G1-G2-AwC-S-E1-24hrs-Dry	1720.0	742.8	2.6	1748.9	1.5%	753.5	1.2%	2.6	2.5%
G2-G3-AwC-S-E1-24hrs-Dry	1768.8	759.0	2.6						
G3-1-AwC-S-E1-24hrs-Dry	1758.0	758.6	2.5						
G4-G1-CwC-S-E1-24hrs-Wet	1077.7	783.4	1.7	1063.3	10.0%	748.0	4.3%	1.5	8.9%
G2-CwC-S-E1-24hrs-Wet	950.4	739.7	1.4						
G6-CwC-S-E1-24hrs-Wet	1161.8	720.8	1.6						
G1-CwC-S-E1-24hrs-Dry	1533.0	744.9	2.1	1406.6	8.7%	762.0	2.1%	1.8	13.1%
G2-CwC-S-E1-24hrs-Dry	1397.5	764.3	1.7						
G3-CwC-S-E1-24hrs-Dry	1289.1	776.7	1.6						
D1-nonprecoated-SE1-24hrs	1359.6	741.5	2.1	1206.0	11.1%	729.8	2.2%	1.9	9.2%
D2-nonprecoated-SE1-24hrs	1142.8	736.9	1.8						
D3-nonprecoated-SE1-24hrs	1115.7	711.2	1.9						
D1-14-SS60-S-E1-24hrs-Wet(15C)	1370.4	759.3	2.0	1309.9	10.2%	764.4	1.3%	1.9	10.9%
D2-18-SS60-S-E1-24hrs-Wet(15C)	1403.0	757.9	2.0						
D3-21-SS60-S-E1-24hrs-Wet(15C)	1156.3	776.0	1.7						
D1-D22-SS60-SE-1-24hrs-Wet	1351.5	784.7	1.9	1363.2	2.6%	771.9	1.7%	2.0	6.3%
D2-D16-SS60-SE-1-24hrs-Wet	1403.0	758.2	2.1						
D3-D12-SS60-SE-1-24hrs-Wet	1335.2	772.8	1.9						

D1-D22-SS60-S-E1-24hrs-Dry	1378.6	763.4	2.1	1496.0	12.2%	773.9	3.7%	2.2	12.5%
D2-D23-SS60-SE-1-24hrs-Dry	1403.0	806.1	2.0						
D3-D14-SS60-S-E1-24hrs-Dry	1706.5	752.2	2.5						
D1-AwC-S-E1-24hrs-Wet	1037.1	777.8	1.5	1010.9	2.3%	759.4	2.2%	1.6	12.2%
D2-AwC-S-E1-24hrs-Wet	1001.9	754.2	1.9						
D3-AwC-S-E1-24hrs-Wet	993.7	746.1	1.5						
D6-6-AwC-S-E1-24hrs-Dry	1804.0	732.9	2.7	1736.3	3.6%	755.4	2.8%	2.6	5.2%
D7-D5-AwC-S-E1-24hrs-Dry	1722.7	774.0	2.5						
D8-3-AwC-S-E1-24hrs-Dry	1682.1	759.2	2.5						
D5-D19-CwC-S-E1-24hrs-Wet	1058.8	753.1	1.7	1079.3	7.2%	752.3	2.5%	1.9	14.5%
D2-CwC-S-E1-24hrs-Wet	1161.8	765.1	1.8						
D3-CwC-S-E1-24hrs-Wet	1020.8	728.3	1.7						
D4-CwC-S-E1-24hrs-Wet	1018.1	754.8	2.2						
D5-CwC-S-E1-24hrs-Wet	1192.5	770.1	2.2						
D6-CwC-S-E1-24hrs-Wet	1023.5	742.3	1.6						
D1-D18-CwC-S-E1-24hrs-Dry	1571.0	743.5	2.4	1713.7	9.2%	750.3	0.8%	2.6	9.6%
D2-5-CwC-S-E1-24hrs-Dry	1882.6	754.4	2.9						
D3-8-CwC-S-E1-24hrs-Dry	1687.5	752.9	2.5						

BBS RESULTS FOR SC-E1									
Combinations	BBS (kPa)	Loading Rate (kPa/s)	Time to Break (sec)	Aver.B BS (kPa)	BBS CoV	Aver.L.R (kPa/s)	L.R. CoV	Av. T.B (sec)	T.B CoV
G1-G11-nonprecoated-SC-E1-2hrs	251.2	784.9	0.5	233.1	6.7%	783.0	2.1%	0.5	12.9%
G2-G12-nonprecoated-SC-E1-2hrs	224.1	798.4	0.6						
G3-G13-nonprecoated-SC-E1-2hrs	224.0	765.6	0.5						
G1-G14-nonprecoated-SC-E1-24hrs	1283.7	739.6	1.9	1225.9	8.2%	749.6	1.9%	1.9	9.2%
G2-SU-G5-non precoated-SC-E1-24	1110.3	743.2	1.7						
G3-G16-nonprecoated-SC-E1-24hrs	1283.7	766.1	2.0						
G1-G4-SS60-SC-E1-2hrs-Wet(15C)	457.1	758.0	0.8	477.0	10.8%	767.6	1.3%	0.8	14.3%
G2-14-SS60-SC-E1-2hrs-Wet(15C)	535.7	777.2	1.0						
G3-SU-G5-SS60-SC-E1-2hrs-Wet(15	438.2	767.5	0.7						
G1-G2-SS60-SC-E1-24hrs-Wet(15C)	858.2	764.3	1.3	808.5	14.0%	759.2	0.8%	1.2	11.1%
G2-16-SS60-SC-E1-24hrs-Wet(15C)	679.4	752.6	1.1						
G3-18-SS60-SC-E1-24hrs-Wet(15C)	888.0	760.8	1.3						
G1-G1-SS60-SC-E1-2hrs-wet	356.9	736.5	0.7	368.7	3.3%	744.6	1.0%	0.7	8.9%
G2-G2-SS60-SC-E1-2hrs-wet	381.5	745.3	0.7						
G3-G3-SS60-SC-E1-2hrs-wet	367.7	752.1	0.8						
G1-G5-SS60-SC-E1-24hrs-Wet	860.9	768.9	1.2	854.6	13.8%	755.1	1.6%	1.2	9.7%
G2-12-SS60-SC-E1-24hrs-Wet	733.6	749.9	1.1						
G3-G3-SS60-SC-E1-24hrs-Wet	969.3	746.6	1.3						
G1-16-SS60-SC-E1-2hrs-Dry	202.4	774.3	0.4	202.4	10.7%	751.5	2.6%	0.4	17.2%
G2-SU-G5-SS60-SC-E1-2hrs-Dry	180.7	740.1	0.4						
G3-20-SS60-SC-E1-2hrs-Dry	224.1	740.1	0.5						
G1-16-SS60-SC-E1-24hrs-Dry	896.2	742.3	1.5	967.5	7.6%	747.9	1.4%	1.5	4.5%
G2-G3-SS60-SC-E1-24hrs-Dry	963.9	741.6	1.4						
G3-18-SS60-SC-E1-24hrs-Dry	1042.5	759.8	1.4						
G1-12-AwC-SC-E1-2hrs-Wet	281.0	766.2	0.6	303.1	14.4%	751.4	1.6%	0.6	0.2%
G2-G1-AwC-SC-E1-2hrs-Wet	275.6	735.1	0.6						
G3-G2-AwC-SC-E1-2hrs-Wet	340.6	744.7	0.6						
G4-11-AwC-SC-E1-2hrs-Wet	362.3	762.9	0.6						

G5-G5-AwC-SC-E1-2hrs-Wet	313.5	744.7	0.6						
G6-18-AwC-SC-E1-2hrs-Wet	245.8	754.7	0.6						
G1-SU-G5-AwC-SC-E1-24hrs-Wet	858.2	753.2	1.3	876.8	5.7%	755.6	1.3%	1.4	4.8%
G2-SU-G6-AwC-SC-E1-24hrs-Wet	785.1	749.8	1.3						
G3-19-AwC-SC-E1-24hrs-Wet	923.3	760.2	1.4						
G4-20-AwC-SC-E1-24hrs-Wet	888.0	757.7	1.4						
G5-21-AwC-SC-E1-24hrs-Wet	898.9	770.1	1.3						
G6-13-AwC-SC-E1-24hrs-Wet	907.0	742.4	1.4						
G1-G2-AwC-SC-E1-2hrs-Dry	478.8	764.2	0.8	428.2	10.7%	755.0	1.7%	0.8	9.2%
G2-G3-AwC-SC-E1-2hrs-Dry	416.5	760.6	0.7						
G3-14-AwC-SC-E1-2hrs-Dry	389.4	740.1	0.7						
G1-15-AwC-SC-E1-24hrs-Dry	988.3	772.3	1.7	1063.3	17.8%	759.3	1.6%	1.7	4.0%
G2-17-AwC-SC-E1-24hrs-Dry	923.3	749.1	1.7						
G3-19-AwC-SC-E1-24hrs-Dry	1278.3	756.5	1.8						
G1-11-CwC-SC-E1-2hrs-Wet	289.1	758.0	0.6	328.0	10.7%	748.2	1.1%	0.6	0.1%
G2-20-CwC-SC-E1-2hrs-Wet	356.9	744.3	0.6						
G3-21-CwC-SC-E1-2hrs-Wet	337.9	742.2	0.6						
G1-17-CwC-SC-E1-24hrs-Wet	926.0	765.5	1.4	823.9	13.2%	766.4	0.5%	1.3	9.1%
G2-15-CwC-SC-E1-24hrs-Wet	709.2	763.2	1.2						
G3-16-CwC-SC-E1-24hrs-Wet	836.6	770.6	1.3						
G1-G5-CwC-SC-E1-2hrs-Dry	289.1	797.1	0.6	316.2	12.7%	771.9	3.0%	0.6	10.9%
G2-G1-CwC-SC-E1-2hrs-Dry	297.2	767.1	0.6						
G3-11-CwC-SC-E1-2hrs-Dry	362.3	751.5	0.7						
G1-12-CwC-SC-E1-24hrs-Dry	1411.1	766.1	2.0	1435.5	4.0%	763.3	0.3%	2.1	5.5%
G2-14-CwC-SC-E1-24hrs-Dry	1500.5	761.5	2.3						
G3-18-CwC-SC-E1-24hrs-Dry	1394.8	762.4	2.1						
D1-D12-nonprecoated-SC-E1-2hrs	195.6	722.7	0.5	219.1	9.3%	763.0	4.6%	0.6	12.6%
D2-D13-nonprecoated-SC-E1-2hrs	232.2	789.1	0.6						
D3-nonprecoated-SC-E1-2hrs	229.5	777.2	0.6						
D1-D14-nonprecoated-SC-E1-24hrs	1419.2	754.2	2.0	1289.4	9.1%	751.0	0.4%	2.0	3.5%
D2-D15-nonprecoated-SC-E1-24hrs	1256.6	749.4	2.0						
D3-D16-nonprecoated-SC-E1-24hrs	1192.5	749.5	2.1						
D1-SU-D1-SS60-SC-E1-2hrs-Wet(15	505.9	756.1	0.8	499.6	9.0%	758.0	0.6%	0.8	8.6%
D2-SU-D2-SS60-SC-E1-2hrs-Wet(15	451.7	762.9	0.7						

D3-D1-SS60-SC-E1-2hrs-Wet(15C)	541.2	755.1	0.8						
D1-SS60-SC-E1-24hrs-Wet(15C)	1172.7	751.5	1.7	1172.6	1.8%	753.2	0.5%	1.7	3.9%
D2-D5-SS60-SC-E1-24hrs-Wet(15C)	1150.9	750.8	1.7						
D3-11-SS60-SC-E1-24hrs-Wet(15C)	1194.3	757.5	1.8						
D1-D1-SS60-SC-E1-2hrs-wet	432.8	743.4	0.8	429.1	5.7%	752.8	1.1%	0.8	0.1%
D2-D2-SS60-SC-E1-2hrs-wet	402.9	755.0	0.8						
D3-D3-SS60-SC-E1-2hrs-wet	451.7	759.9	0.8						
D1-15-SS60-SC-E1-24hrs-Wet	1053.4	744.1	1.7	1212.3	12.8%	751.6	1.2%	1.8	6.7%
D2-16-SS60-SC-E1-24hrs-Wet	1221.4	761.8	1.8						
D3-19-SS60-SC-E1-24hrs-Wet	1362.3	749.0	1.9						
D1-SU-D1-SS60-SC-E1-2hrs-Dry	435.5	753.8	0.8	465.3	6.7%	752.9	0.8%	0.8	0.0%
D2-SU-D2-SS60-SC-E1-2hrs-Dry	497.8	758.3	0.8						
D3-17-SS60-SC-E1-2hrs-Dry	462.6	746.6	0.8						
D1-D1-SS60-SC-E1-24hrs-Dry	1004.6	767.5	1.5	918.8	8.9%	766.6	1.8%	1.5	4.9%
D2-9-SS60-SC-E1-24hrs-Dry	842.0	780.1	1.4						
D3-22-SS60-SC-E1-24hrs-Dry	909.7	752.1	1.5						
D1-19-AwC-SC-E1-2hrs-Wet	438.2	772.0	0.8	374.9	14.6%	766.0	1.4%	0.8	9.2%
D2-D4-AwC-SC-E1-2hrs-Wet	340.6	753.8	0.7						
D1-14-AwC-SC-E1-2hrs-Wet	346.0	772.0	0.7						
D1-22-AwC-SC-E1-24hrs-Wet	1066.9	746.2	1.7	1034.4	5.4%	741.9	0.6%	1.6	8.6%
D2-21-AwC-SC-E1-24hrs-Wet	1066.9	742.1	1.7						
D3-17-AwC-SC-E1-24hrs-Wet	969.3	737.4	1.4						
D1-SU-D2-AwC-SC-E1-2hrs-Dry	644.1	759.0	1.1	633.3	9.1%	749.2	1.4%	1.0	6.6%
D2-SU-D1-AwC-SC-E1-2hrs-Dry	571.0	750.9	1.0						
D3-18-AwC-SC-E1-2hrs-Dry	684.8	737.7	1.1						
D1-12-AwC-SC-E1-24hrs-Dry	1137.4	768.6	1.4	1155.4	7.6%	768.4	1.4%	1.6	17.4%
D2-13-AwC-SC-E1-24hrs-Dry	1077.7	778.9	1.4						
D3-14-AwC-SC-E1-24hrs-Dry	1251.2	757.7	1.9						
D1-18-CwC-SC-E1-2hrs-Wet	484.2	771.7	0.8	435.9	8.5%	772.1	1.0%	0.8	8.2%
D2-12-CwC-SC-E1-2hrs-Wet	473.4	776.6	0.8						
D3-13-CwC-SC-E1-2hrs-Wet	443.6	776.6	0.7						
D4-21-CwC-SC-E1-2hrs-Wet	408.4	781.1	0.7						
D5-D4-CwC-SC-E1-2hrs-Wet	397.5	764.2	0.7						

D6-CwC-SC-E1-2hrs-Wet	408.4	762.6	0.7						
D1-12-CwC-SC-E1-24hrs-Wet	801.3	758.0	1.3	947.2	11.4%	750.0	0.7%	1.5	9.8%
D2-SU-D1-CwC-SC-E1-24hrs-Wet	917.9	748.9	1.4						
D3-13-CwC-SC-E1-24hrs-Wet	871.8	748.8	1.3						
D4-14-CwC-SC-E1-24hrs-Wet	996.4	753.4	1.5						
D5-20-CwC-SC-E1-24hrs-Wet	1110.3	743.9	1.7						
D6-SU-D2-CwC-SC-E1-24hrs-Wet	985.6	746.7	1.5						
D1-D3-CwC-SC-E1-2hrs-Dry	554.7	759.7	1.0	588.1	5.1%	749.5	1.4%	1.0	0.1%
D2-D5-CwC-SC-E1-2hrs-Dry	611.6	750.6	1.0						
D3-D1-CwC-SC-E1-2hrs-Dry	598.1	738.1	0.9						
D1-16-CwC-SC-E1-24hrs-Dry	1543.9	747.3	2.3	1477.9	3.9%	745.5	0.5%	2.2	3.1%
D2-15-CwC-SC-E1-24hrs-Dry	1446.3	748.1	2.1						
D3-19-CwC-SC-E1-24hrs-Dry	1443.6	741.3	2.1						