Development of a Standard Test Method for Determining the Bitumen Bond Strength of Emulsions - A South African Perspective

by

Andries Hendrik Greyling

Thesis presented in fulfilment of the requirements for the degree of Master of Science in Engineering in the Faculty of Civil Engineering at Stellenbosch University
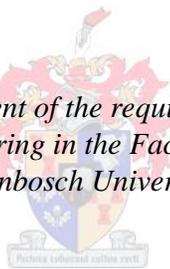
Supervisor: Professor Kim Jenkins

December 2012

Declaration

By submitting this thesis electronically, I declare that the entirety of the work contained therein is my own, original work, that I am the owner of the copyright thereof (unless to the extent explicitly otherwise stated) and that I have not previously in its entirety or in part submitted it for obtaining any qualification.

Andries Hendrik Greyling

Date: 20 November 2012

Abstract

Chip and spray surfacing seals has been widely used in South Africa as the preferred surface treatment for rural roads. The design of these seals has also experienced renewed interest and continuous development in several other countries over the past two decades. In South Africa seals are continually used as increasing attention is given to the periodic maintenance of existing surfaced roads. There is also a significant increase in the use of surfacing seals in North America as the need to develop more energy and resource efficient surfacing options becomes a priority.

Despite this growing use of surface seals, the seal design and especially the selection of binder type and grade does not always follow scientific processes. Seals are often designed based on client preference, previous experience, material availability and industry trends. With an ageing road network and limited funding to ensure timeous maintenance, the focus in South Africa is moving towards more stringent and scientific design processes.

The USA and Europe, forced by increasing traffic volumes and heightened performance demands, are moving towards performance- based specification to account for deficiencies in their current surface seal design methods. One of the major failure mechanisms of surface seals is ravelling which takes place when the binder and the aggregate bond becomes weak and are broken by the forces generated by traffic. This leads to loose aggregate on the road which in turns leads to bare surface patches and broken windscreens. To prevent and address this as part of the development of performance-based specifications, the need for a simple and inexpensive technique for evaluating bitumen and bitumen emulsion bond strength development over time, as well as binder-aggregate compatibility, was identified.

Although various tests exist for investigating adhesion between bituminous emulsions and aggregate chips most of the tests does not deliver the level of information required by the performance-based specifications. The Bitumen Bond Strength (BBS) test method was therefore developed with the aim to address some of the limitations encountered in evaluating bond strength between binders and aggregates.

The BBS test (AASHTO TP-91, 2011) was developed by the University of Wisconsin – Madison (UWM) in partnership with the University of Ancona – Italy (UAI) and the University of Stellenbosch – South Africa (US) specifically for evaluating bond strength between aggregates and hot applied binders and emulsions, respectively.

US became involved in BBS test efforts in 2008 to assist in the development and practical evaluation of the BBS test method. UAI contributed significantly to the development of the test apparatus through their work in conjunction with UWM. Due to time and resources available, the involvement of the US was limited to various discussion sessions, a study tour, the evaluation of the BBS test, and conducting a series of control tests.

By using the Bitumen Bond Strength test it was possible to evaluate the practicality and repeatability of the developed procedure and the results measured was successfully used to evaluate the bond strength development of modified and unmodified bitumen emulsions on tillite and granite aggregates. It was also possible to correlate the results achieved at the University of Stellenbosch with results from the University of Wisconsin-Madison due to the fact that testing took place at both institutions.

The development of the test and the inter-laboratory test results in essence reinforced the hypothesis that the BBS test protocol can be used to effectively evaluate bond strength of different emulsion types and aggregate types. Except for the loading rate which is a known critical influence, the emulsion type and curing intervals are both identified as the most significant other factors contributing to bond strength development.

Aggregate type is also identified as a significant factor that will influence the bond strength development. Interactions between emulsion type and curing interval are identified as the most significant interaction. A lot of further validation test on the BBS test method is still required for the test to be integrated into a performance-based specification system for surface seals.

Opsomming

"Chip and Spray" oppervlak seëls is die verkose seël tipe vir gebruik op Suid Afrikaanse plattelandse paaie. Die ontwerp van hierdie tipe seëls is ook tans besig om hernude aandag te trek in heelwat lande. In Suid Afrika word die tipe seël al meer gebruik soos die behoefte na herseël projeke op die verouderde pad netwerk groei . Daar is ook heelwat groei in die gebruik van "Chip en Spray" seëls in Noord Amerika aangesien daar n behoefte is om n meer energie en materiaal effektiewe seëls te gebruik.

Ten spyte van die groeiende gebruik van hierdie seëls het daar min wetenskaplike ontwikkeling plaasgevind in the ontwerp daarvan. Seëls word meestal ontwerp gebaseer op klient voorkeur , ondervinding, materiaal beskikbaarheid en ook industrie norms. 'n Verouderde pad netwerk dwing die Suid Afrikaanse industrie om meer deeglike en wetenskaplike ontwerp prosedures te volg.

Die VSA en Europa word deur vinnig groeiende verkeersvolumes en verhoogte kwaliteits behoeftes gedwing om prestasie gebaseerde spesifikasies te ontwikkel. Een van die hoof defekte op seëls is klipverlies wat plaasvind as die verbinding tussen die bitumen en aggregaat verswak en gebreek word deur die kragte wat deur verkeer oorgedra word. Dit lei tot los klip op die pad wat weer tot skade aan voertuie lei. Om dit te voorkom en aan te spreek het die behoefte laat ontwikkel vir n maklike en goedkoop tegniek om te meet hoe sterk die verbinding is wat ontwikkel tussen verskillende bitumen, bitumen emulsies en aggregaat monsters.

Alhoewel daar alreeds toetse bestaan wat kan toets hoe sterk die verbinding is , gee die meeste van die toetse nie die informasie wat benodig word deur die voorgestelde prestasie gedrewe spesfikasies nie. Die "Bitumen Bond Strength" (BBS) toets metode is daarom ontwikkel om die tekortkoming in die toets van die bitumen en aggregaat verdindinge aan te spreek.

Die BBS toets (AASHTO TP-91, 2011) is ontwikkel by die Universiteit van Wisconsin – Madison (UWM) in vennootskap met die Universiteit van Ancona – Italy (UAI) en die Universiteit van Stellenbosch (US) , spesifiek om die die sterkte van die verbinding wat vorm tussen aggregaat en bitumen te meet.

Die US het in 2008 betrokke geraak by die BBS toets studie om hulp te verleen met die ontwikkeling en praktiese evalueering van die BBS toets metode. As gevolg van tyd en personeel tekorte is die betrokkendheid by die US

beperk tot verskeie besprekings sessies, n studie toer, die evalueering van die BBS toets sowel as die voltooing van n uitgebreide stel toetse.

Deur die voltooing van eksperimente met die BBS toets was dit moontlik om die uitvoerbaarheid en herhaalbaarheid van die ontwikkelde prosedure te toets. Die resultate van die toetse is suksesvol gebruik om die ontwikkeling van die verbinding sterkte tussen gemodifiseerde en ongemodifiseerde bitumen emulsies en tilliet en graniet te definïeer en te evalueer. Dit was ook moontlik om die resultate van die Universiteit Stellenbosch en die Universiteit of Wisconsin-Madison suksesvol met mekaar te vergelyk aangesien toetse by beide die instansies voltooi is.

Die ontwikkeling van die toets en die inter laboratorium toets resultate het dit moontlik gemaak om die hipotese te bevestig dat die BBS toets prosedure effektief gebruik kan word om die bitumen en aggregaat verdindinge te toets en te evalueer. Behalwe vir die tempo van die lading waarteen die aftrek sterkte getoets word , is die emulsie tipe en die nabehandeling tydperk beide geidentifiseer as die mees beduidende invloede wat bydrae tot die ontwikkeling van die verbinding sterkte.

Die aggregaat tipe is ook geidentifiseer as n belangrike faktor wat die verbinding sterkte ontwikkeling sal beïnvloed. Die interaksie tussen die emulsie tipe en nabehandeling tydperk was geïdentifiseer as die mees beduidende interaksie. Daar sal wel nog heelwat eksperimente voltooi moet word met die BBS toets prosedure voordat dit volkome geïntegreer kan word as deel van n prestasie gebaseerde spesifikasie stelsel vir die ontwerp van seëls.

Acknowledgements

TABLE OF CONTENT

TABLE OF FIGURES

AH GREYLING   **December 2012**

LIST OF TABLES

Stellenbosch University http://scholar.sun.ac.za

LIST OF PHOTOS

LIST OF ADDENDUMS

GLOSSARY/ACRONYMS

ALD	Average Least Dimension'
COV	Covariance
CRS65	Cationic Rapid Set Emulsion with 65% net bitumen content
CSIR	Council for Scientific and Industrial Research
DSR	Dynamic Shear Rheometer
EVA	Ethylene-Vinyl-Acetate
FHWA	Federal Highway Administration
HMA	Hot Mix Asphalt
PATTI	Pneumatic adhesion tensile testing instrument
PG	Performance Grading
PQG	PATTI Quantum Gold
PSV	Polished Stone Value
SABITA	Southern African Bitumen Association
SABS	South African Bureau of Standards
SBR	Styrene-Butadiene-Rubber
SBS	Styrene-Butadiene-Styrene
SHRP	Strategic Highway Research Programme
SS60	Stable Grade Anionic Emulsion with 60% net bitumen content
TG	Technical Guideline
TMH	Technical Methods for Highways
UAI	University of Ancona-Italy
US	University of Stellenbosch
USA	United States of America
UWM	University of Wisconsin - Madison

1 INTRODUCTION

1.1 BACKGROUND

Chip and spray surfacing seals have been widely used in South Africa as the preferred surface treatment for rural roads. The design of these seals has also experienced renewed interest and continuous development in several other countries over the past two decades. In South Africa seals are continually used as increasing attention is given to the periodic maintenance of existing surfaced roads. There is also a significant upward trend in the use of surfacing seals in North America as the need to develop more energy and resource efficient surfacing options becomes a priority.

Despite this growing use of surface seals, the seal design and especially the selection of binder type and grade does not always follow scientific processes. Seals are often designed based on client preference, previous experience, material availability and industry trends. With an ageing road network and limited funding to ensure timeous maintenance, the focus in South Africa is moving towards more stringent and scientific design processes.

The USA and Europe, forced by increasing traffic volumes and heightened performance demands, are moving towards performance- based specification to account for deficiencies in their current surface seal design methods (Opus International Consultants, 2010). One of the major failure mechanisms of surface seals is ravelling which takes place when the binder and the aggregate bond becomes weak and are broken by the forces generated by traffic. This leads to loose aggregate on the road, which in turn, leads to bare surface patches and broken windscreens. To prevent and address this as part of the development of performance-based specifications, the need for a simple and inexpensive technique for evaluating bitumen and bitumen emulsion bond strength development over time, as well as binder-aggregate compatibility, was identified.

Although various tests exist for investigating adhesion between bituminous emulsions and aggregate chips (European Commitee for Standarization, 1999) most of the tests do not deliver the level of information required by the performance-based specifications. The Bitumen Bond Strength (BBS) test method was therefore developed with the aim to address some of the limitations encountered in evaluating bond strength between binders and aggregates.

The BBS test (AASHTO TP-91 , 2011) was developed by the University of Wisconsin – Madison (UWM) in partnership with the University of Ancona – Italy (UAI) and the University of Stellenbosch – South Africa (US) specifically for evaluating bond strength between aggregates and hot applied binders and emulsions, respectively.

US became involved in BBS test efforts in 2008 to assist in the development and practical evaluation of the BBS test method. UAI contributed significantly to the development of the test apparatus through their work in conjunction with UWM. Due to time and resources available, the involvement of the US was limited to various discussion sessions, a study tour, the evaluation of the BBS test, and conducting a series of control tests.

The developers of the BBS test foresee that the test method will be able to quantify and characterize bond strength development, adhesive properties and aggregate-binder compatibility, thereby improving the effectiveness of surface seal designs (Miller, 2010).

1.2 PROJECT DEFINITION AND OBJECTIVES

1.2.1 PROJECT DEFINITION

The project title can be defined as follows:

Development of a Standard Test Method for Determining the Bitumen Bond Strength of Emulsions- A South African Perspective.

1.2.2 RESEARCH OBJECTIVES

The research objectives of this study are to address the following identified outcomes:

1. Complete a detailed literature review to identify the factors that significantly affect the bond strength development between bituminous binders and aggregates.
2. Confirm the need for a test method that will be able to quantify and characterize bond strength development, adhesive properties and aggregate-binder compatibility.
3. Describe the writer's assistance in the development of a test method to quantify bond strength development in bitumen and emulsified bitumen binders.

4. Evaluate the practicality and repeatability of the developed Bitumen Bond Strength (BBS) Test procedure.

5. Evaluate the bond strength development of modified and unmodified bitumen emulsions on tillite and granite aggregates using the Bitumen Bond Strength Test method.

6. Correlate the results achieved at the University of Stellenbosch with results from the University of Wisconsin-Madison

1.3 STUDY LOCATION

The study took place at the University of Stellenbosch in the Western Province of South Africa and at the University of Wisconsin-Madison in the state of Wisconsin in the United States of America. *Figure 1-1* below shows locations in relation to a world map.

Figure 1-1- Study Tour Location

1.4   METHODOLOGY

The study was divided into three distinct phases:

- The first phase consisted of an extensive literature study and review on various subjects relating to surfacing seals, bitumen, aggregate and their behaviour. It investigated the factors influencing bitumen and aggregate adhesion as well as the current test methods available to evaluate adhesion. The need and initial development of the Bitumen Bond Strength test is also addressed.

- The second phase details the physical processes and actions followed to assist in the development of the Bitumen Bond Strength test. It includes the evaluation of the BBS Test procedure during its development phase by completing a number of trail tests. This phase also investigates the final procedure by completing a detailed experimental setup and an extensive set of tests.

- The third and final stage is the evaluation of the study results and includes a discussion on required future research.

1.5   THESIS OUTLINE

The thesis can be divided into five sections:

Chapter 1 gives a brief introduction of the proposed study; it defines the project definition and objectives and discusses the methodology followed to complete the study. *Chapter 2* consists of a brief introduction on the history of roads and the use of bitumen surfacing seals.

Chapters 3 to 6 focusses on road surfacing seals, their function , behaviour and the material used in seals. It discusses the design methods of seals, the modes of distress and factors that typically influence the life cycle of roads. It also looks at bitumen, bitumen emulsion and aggregates and these materials' properties and specification systems.

Chapter 7 discusses the current test methods available for testing adhesion and evaluates the PATTI Test as a possible new alternative.

Chapter 8 & 9 discusses the involvement of the University of Stellenbosch, the development of the Bitumen Bond Strength Test as well as the test results. In addition, factors that influence the bitumen bond strength development are confirmed.

Chapter 10 concludes the report by discussing how the research objects was met and proposes some recommendations for future studies.

LITERATURE STUDY:
BITUMEN &AGGREGATE ADHESION

2   AN INTRODUCTION TO ROADS AND ROAD SURFACING SEALS

2.1   A BRIEF HISTORY OF ROADS

Most people give very little thought to the roads we drive on every day. We tend to take them for granted, at least until they are in a bad condition or closed for any reason. From the earliest times, one of the strongest indicators of a society's level of development has been its road system or lack of one. Increasing populations and the introduction of towns and cities brought with it the need for communication and trade between those growing population centres. A road built in Egypt by the Pharaoh Cheops around 2500 BC is believed to be the earliest paved road on record. It consisted of a 1,000 meters long and 20 meters wide construction road that led to the site of the Great Pyramid.

Various other ancient roads were established by different civilizations and their armies but the Romans where, without a doubt the champion road builders of them all. Roman roads were masterpieces of road construction, and was designed and built with great engineering skill. At their peak, the Roman Empire maintained around 85,000 km of roads that covered almost all of Europe. Roman roads consisted of a graded soil foundation topped by four layers: a bedding of sand or mortar; rows of large, flat stones; a thin layer of gravel mixed with lime; and a thin surface of flint-like lava. Many of their original roads are still in use today, although they have been resurfaced numerous times.

Thomas Telford and John Loudon McAdam, two well-known British Engineers, initiated the modern highway construction technology in 18th century Britain. Telford, originally a stonemason, came up with a system of road building which required digging a trench, installing a foundation of heavy rock, and then surfacing with a 150mm layer of gravel. During construction, the centre of the road was raised, producing a crown that allowed water to drain off. Telfords' system was faster and less expensive than the Romans' method but it was still costly and required frequent resurfacing with gravel.

On the other hand, McAdam's system was based on the principle that a well-drained road made of suitable material does not need the stone foundation of Telford's system, but could be built directly on the subsoil. McAdam firstly placed a closely compacted 250mm- to 300mm layer of stone which had been broken to 25mm

in diameter, and which was raised in the centre to facilitate drainage. This was followed by a layer of finer grained stone that was cemented by the setting of fines of the graded stone. This process was completed in stages and it allowed the road's traffic to compact each stage.

The greatest advantages to McAdam's system were the speed of construction and low cost, and it was generally adopted throughout Europe. However, it was the lack of a firm foundation for the roadbed that was to prove the ultimate undoing of macadam roads with the advent of heavy motor vehicles, especially trucks. For that reason, on roads that had to support heavy loads, Telford's system of construction became the standard (Sponholtz, 2012).

2.2 HISTORY OF BITUMEN SURFACING SEALS

2.2.1 *REST OF THE WORLD*

The history of modern road construction and paving materials dates back to the days of the late 1800's when it became widespread in all major cities around the world. The chief paving materials of the day consisted of loose cobble stones and blocks in addition to miscellaneous wooden bricks, blocks, crushed rocks and pebbles, and at times some naturally occurring tar and cement concrete surfacing materials were also used.

As discussed in the previous section the design of John McAdam provided the most cost effective and widely used paving options at the time. Its open graded surface was unfortunately not very suitable to sustain a combination of heavy traffic and moisture and resulted in slippery and muddy road surfaces. At the start of the 20th century, roads were mostly inadequate to cater for the everyday traffic demands which were going to be challenged increasingly by the newly invented motorcar. As time went by and the motor car technology developed and vehicle velocities increased, the friction between the road surfaces and the car tyres became a critical road safety issue, especially when subjected to stresses encountered during constant acceleration, cornering and braking conditions.

In addition to numerous pavement failures experienced at the time it was also made apparent that much more durable and tougher road construction materials were called for. The answer at the time was relying in the on-going research which was being conducted to find better, cheaper and stronger road pavement materials and processes. There was a need for a road surface that could withstand the forces of everyday road traffic. Roads made from asphalt and concrete seemed to offer the most promise.

Asphalt is generally a mixture of bitumen and aggregates consisting of crushed rock whereas concrete is a mixture of cement and rock. Asphalt pathways were first paved in France in the early 1800's, but the method was not perfected until around 1829. The first use of roads constructed using asphalt and bitumen took place at around 1825, when asphalt blocks were first put down as a pavement on French streets in Paris, and the first successful great asphalt and bitumen (tar) paving application was first laid down in 1858. The first successful paved cement concrete sidewalk in turn was built in Inverness in Scotland in 1865, neither technology however progressed greatly without the added pressures which the motorcar posed on the roads infrastructure in the coming *years* (Ecopave Australia, 2009).

The history and advancement of the modern asphalt/bitumen road, as we see on our roads today, came initially from the United States. They had vast natural occurring bitumen deposits from which to draw from and the local engineers were forced to analyse the principles behind the behaviour of this promising new road engineering material.

The first real initiative came in the late 1860's, from the research work conducted by a Belgian immigrant Edward de Smedt from Columbia University in New York City. Mr De Smedt conducted his first field testing in New Jersey in 1870 and by 1872 was already producing the equivalent of modern day continuously graded road asphalt. The first practical application was followed by the laying of asphalt in Battery Park and on Fifth Avenue in New York USA in 1872 (Sponholtz, 2012).

One of the great convenient coincidences in the history of asphalt/bitumen roads was the development of the motor car which by pure chance ran on petrol. Petrol, at that time, was just another petroleum by-product which was distilled from kerosene which in turn was derived from petroleum by the fast emerging and growing petroleum refining industry. Another useful by-product that was derived in the process of refining crude oil was bitumen (Ecopave Australia, 2009).

2.2.2   *SOUTH AFRICA*

Paved Roads in South Africa can be described as fairly recent as they have been present for just over 100 years. The first new asphalt roads in South Africa that were laid in Cape Town soon after the turn of the 20th century. A Mr John Whitford Griffits arrived in Cape Town from Wales in 1902 and he started a road construction company called the *Union Granolithic and Asphalt Company in* 1908. This company surfaced Church, Longmarket, Shortmarket, Hout and Castle streets in 1910 using bitumen imported from Switzerland.

After a break in activity during the First World War, the main road system in and around Cape Town was progressively reconstructed with asphalt surfaces. Shell Oil imported Trinidad Lake Asphalt as binder and, as the City Council of Cape Town had ample funds at the time; specifications based on sand seals to penetration layers using granite aggregates became the order of the day (SABITA, 2009).

The plant used by these early road builders requires some comment. Mr Wolton Gray came to South Africa in 1924 to sell steam rollers for John Fowler of Leeds, England. The steam rolling business was slow to take off so Mr Wolton launched Fowler Tar Spraying with two fellow steam men, Mr Lowman and Mr Fishwick. They started off by hiring out the Fowler rollers and eventually went on to undertake road construction contracts. Some of their early work included the roads and wharves of Cape Town Docks for the S. A. Railways & Harbours.

The Fowler roller was unique in that it had a tar kettle under the boiler and a chain driven tar pump feeding a 2.750m (9ft) spray bar at the rear end. It came equipped with a drawn hopper which spread chips onto the freshly sprayed binder. This method is in all probability the forerunner of the Chip Seal currently used throughout South Africa.

Photo 2-1 on the next page shows a restored Fowler steam roller and *Photo 2.2* shows the manual application of a bituminous binder (SABITA, 2009).

Photo 2-1- Restored Fowler Roller (www.TractionTime.co.uk)

Photo 2-2- Early Bitumen Spray Operations (SABITA, 2009)

3   ROAD SURFACING SEALS

3.1   INTRODUCTION

The previous section gave a very brief overview of the history and background of bituminous surfacing treatments used in road construction. Asphalt, Chip and Spray and variations of the two are currently the most commonly used surfacing seals in South Africa.

Asphalt is a blend of graded aggregate, bitumen and a filler material that is typically mixed in a designated plant using a specific mix design and mix procedure. It is hot or warm applied on the road using an asphalt paver after which it is rolled to a specified density. This is the most durable but also the most expensive bituminous surfacing type and it is generally only provided on heavily trafficked rural roads, freeways, urban streets and at intersections. Asphalt and the behaviour of asphalt do not form part of this study, but it is however envisaged that some of the outcomes may be relevant for use in the asphalt industry.

The size of South Africa and the location and distribution of our road network in relation to the large centres, as well as the relatively low traffic flow on some of our roads, has led to the extensive use of chip and spray seals. A chip and spray seal typically consist of a layer of bituminous binder sprayed directly on the road followed by an even sized or graded aggregate mix that is spread over the binder before being rolled and opened to traffic (South African National Road Agency, 2007).

It is used in various combinations of binders and aggregate sizes applied in different layer combinations. It is the most used seal and re-seal method in the rural areas of South Africa. It is also the only type of surfacing seal that will be further investigated as part of study and all further references to *surfacing seal* and *seals* will include only chip and spray seals unless otherwise stated.

3.2   SURFACING SEALS FUNCTION

The Technical Recommendation for Highways Number 3 (TRH3) is the South African authority on surfacing seal design and is currently the proposed method to be used for chip and spray seal designs. It defines a good road as a structurally sound surface that has a durable, waterproof, skid-resistant and all weather dust-free surfacing to ensure that it perform optimally (functionally and structurally). These characteristics are required to

provide the road user with an acceptable level of service and to protect the structural layers of the pavement from the forces of traffic as well as from the effects of the environment

In South Africa chip and spray seals and slurries are commonly used for new construction and for the resealing of existing bituminous roads. This is mainly because they are relatively inexpensive and simple to construct. They have proved to be successful on highways, rural roads and urban streets, under light and heavy traffic conditions.

The main functions of a surfacing seal as per TRH3 can therefore be summarised as follows:

- o Provide a waterproof cover to the underlying crushed stone and natural material pavements;

- o Provide a safe all-weather, dust-free riding surface for traffic with adequate skid resistance;

- o Protect the underlying layer from the abrasive and destructive forces of traffic and the environment.

Most surfacing seals are relatively thin and have no load distribution properties. The seal itself should however have enough strength to accommodate the horizontal and vertical stresses induced by traffic (South African National Road Agency, 2007).

3.3 *SURFACING SEAL FAILURE MODES AND MECHANISMS*

3.3.1 *BACKGROUND*

The functional requirements of any road are speed, comfort and safety. These functions are almost always related with relationships that are not easily quantifiable or measurable. For this reason various functional features have been defined. These are features that can be measured and for which a standard can be set to ensure suitable levels of comfort, safety and speed. For surfaced roads these are generally the riding quality, skid resistance and surface drainage of the road.

3.3.2 *MODES OF DISTRESS*

Distress is the visible manifestation at the road pavement surface of the deterioration of the condition of the pavement with respect to either the serviceability or the structural capacity. Part of a comfortable and safe road is a road with limited signs of distress that will negatively influence the riding quality, skid resistance or surface drainage of a road. There are four major ways in which distress occurs (CSIR-TRH6, 1985). These are called the modes of distress and they can be summarised as follow:

- *Deformation* – This is the development of a change in the profile of the surface of the pavement which leads to unevenness and reduce riding quality.

- *Cracking* – This refers to cracks that appear in the surface of the pavement.

- *Disintegration of the surfacing* - This is the break-up and loss of surfacing. It can occur through spalling of the material at cracks or through the crumbling and loss of material at the surface due to the abrasive action of traffic.

- *Smoothing of Surface Texture* - This is the loss of surface texture which leads to a loss of skid resistance.

Each of these modes of distress can occur in one of several different typical manifestations. These are called the various types of distress. *Table 3-1* below summarise the modes and types of distress found on surfaced roads:

Table 3-1- Typical Modes and Types of Distress on Surfaced Roads (CSIR-TRH6, 1985)

MODE OF DISTRESS	TYPE OF DISTRESS
Deformation	Depressions
	Mounds
	Ruts
	Ridges
	Displacements
	Corrugations
	Undulations
Cracking	Transverse cracks
	Longitudinal cracks
	Block Cracks
	Map Cracks
	Crocodile Cracks
	Parabolic Cracks
	Star Cracks
	Meandering Cracks
	Multiple Cracks
Disintegration of Surfacing	Ravelling
	Potholes
	Edge Breaks
	Patches
Smoothing of surface texture	Bleeding &Polishing

When looking at these modes and types of failure and distress it is extremely important to understand and differentiate between what is in essence structural distress and what is surfacing distress. Structural distress refers to distress that affects the structural integrity and the load-bearing capacity of a pavement in either origin or consequent. Surface distress is that distress that affects only the surface texture and behaviour. (CSIR-TRH6, 1985)

As part of this study structural distresses will not be investigated further. The following sections will briefly discuss the typical surface distresses experienced under each mode of distress. The importance, effect and influence of the seal design, binder and aggregate on these distresses will be pointed out in subsequent sections.

3.3.3 *MODES OF SURFACE DISTRESS*

3.3.3.1 *DEFORMATION*

Deformation in a road is almost always related to structural issues. No distresses related only to thin chip and spray surfacing's relevant and it will therefore not be discussed further.

3.3.3.2 *CRACKING*

Cracking of the surfacing is either due to reflective cracking from problems in the underlying structure or due to the ageing of a seal. When a seal ages it becomes dry and brittle, losing much of its elasticity. This leads to various types of localised cracks on the surface. Surface cracks may lead to water ingress which in turn weakens the support layers and lead to disintegration of the surface.

3.3.3.3 *DISINTEGRATION OF THE SURFACING*

Ravelling and stone loss is one of the most important surfacing seal related distresses. Ravelling occurs when aggregate "ravel" from the surface due to loss of adhesion between the binder and aggregate. This forms depressions which may fill with moisture which may pose problems. Loose aggregate on the road may also lead to vehicle and windscreen damage. Ravelling can be due to any of the various reasons that influence the bitumen binder and aggregate adhesion. These reasons will be discussed in more detail further on in this study. *Photo 3-1* below shows a typical ravelled strip on a chip seal.

Photo 3-1- Areas showing ravelling and stone loss.

3.3.3.4 BLEEDING AND SMOOTHING OF SURFACE

Bleeding and the loss of surface texture are further surface seal specific defects. Bleeding takes place when the voids within the aggregate matrix, that should accommodate the bitumen binder, are reduced by either aggregate that embeds or punches into the base course or the rotation of stone to their least dimension. This decrease in voids, coupled with high temperatures that lead to decreased bitumen viscosity, causes the road to "bleed" bitumen.

The liquid bitumen migrating to the surface creates a sticky and shiny, black surface. The result can mean a loss of surface texture on the pavement. *Photo 3-2* below shows a section of road showing signs of bleeding. Note the darker strip in the right wheel track of the left lane.

Photo 3-2- Area showing signs of bleeding

A second cause of the smoothing of the surface may be polished aggregate. This happens when the bitumen has been worn away from the pavement surface by traffic, and the surface aggregate has lost macro texture, having been smoothed and rounded. This happens when the aggregate has insufficient resistance against polishing as defined by the Polished Stone Value (PSV) and measured according to SABS Method SM84818.

The degree of possible polishing depends on the aggregates mineral composition and crystal structure. These properties will be discussed further in *Chapter 6* of this report.

3.4   SURFACING SEAL DESIGN

3.4.1   INTRODUCTION

The TRH3- South African National Road Agency, 2007 defines a chip and spray seal as follows:

"In its simplest form a chip and spray seal consists of a coat of bituminous binder sprayed onto the road surface which is then covered with a layer of aggregate (stone or sand). The aggregate cover is applied immediately after the binder has been sprayed and then rolled to ensure close contact and thus good adhesion between the aggregate and the binder film. Rolling initiates the process of orientating the particles into a mosaic pattern and working the binder into the voids between the aggregate particles. The process is completed by the action of traffic, so that finally a dense and relatively impermeable pavement surfacing is obtained."

This method is used in various combinations to construct single seals, double seals, Cape Seals, slurry seals and sand seals. Further alternatives include inverted double seals, geotextile seals, split seals, graded aggregate seals and choked seals. *Figure 3-1* below, extracted from the TRH3, shows visuals of the two most common used seal types
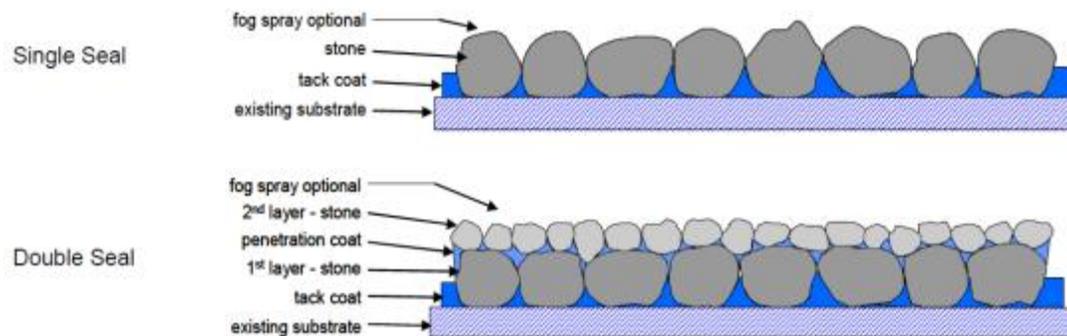
Figure 3-1- Visual Representation of a Single and Double Chip and Spray Seal (South African National Road Agency, 2007)

The very early practitioners of chip seals appear to have used a purely empirical approach to their seal designs. Sealing a pavement was considered then, as it is now in many circles, an art. Scientific developments in the field have yielded a few relevant design methods that vary across the world. The following section will aim to describe the essence and the philosophy of the majority of these methods.

3.4.2 *SOUTH AFRICAN METHOD, TRH3*

The TRH3 was published by the South African National Road Agency in 2007 as the authority on South African chip and spray seal designs. The TRH3's design process is based on a number of primary input parameters such as traffic volume, preferred new texture depth and surface hardness. Once initial results are achieved using the primary inputs, practical adjustments are made for differences in the climate, gradients, existing texture, applications temperatures, preferred aggregate spread rate, and the use of modified binders. This method may be seen as a hybrid of the United Kingdom and Australian design methodologies. It is however strongly influenced by extensive South African seal experience.

The selection of type of surfacing is usually made between single seals, double seals, Cape seals, and sand seals. The seal type decision is primarily based on the traffic level and pavement condition. The TRH3 method also measures and evaluates the surface hardness and expected penetration of aggregate by using a ball penetration test, corrected for temperature.

The grade and type of binder is selected based on expected traffic level, expected operational road surface temperature, climatic region, and aggregate condition. The required rate of binder spread is determined by using tables and charts that incorporate traffic level, expected aggregate embedment and final required texture depth for different aggregate Average Least Dimension (ALD) values.

Figure 3-2 shows the typical charts that will be used for a 13.2mm single seal. Various practical adjustments are then made to ensure the application is case specific and relevant to the specific conditions. Note the red minimum application line. This is the minimum binder required that can be practically be sprayed or that is required to accommodate and bind with the aggregate.

The aggregate spread rate varies according to the purpose of the seal and the shape and flakiness of the aggregates. The TRH3 proposes that final design spread rates should be determined on site by spreading the preferred matrix of aggregates by hand. (South African National Road Agency, 2007)

DESIGN CHART FOR SINGLE SEALS: 9mm ALD
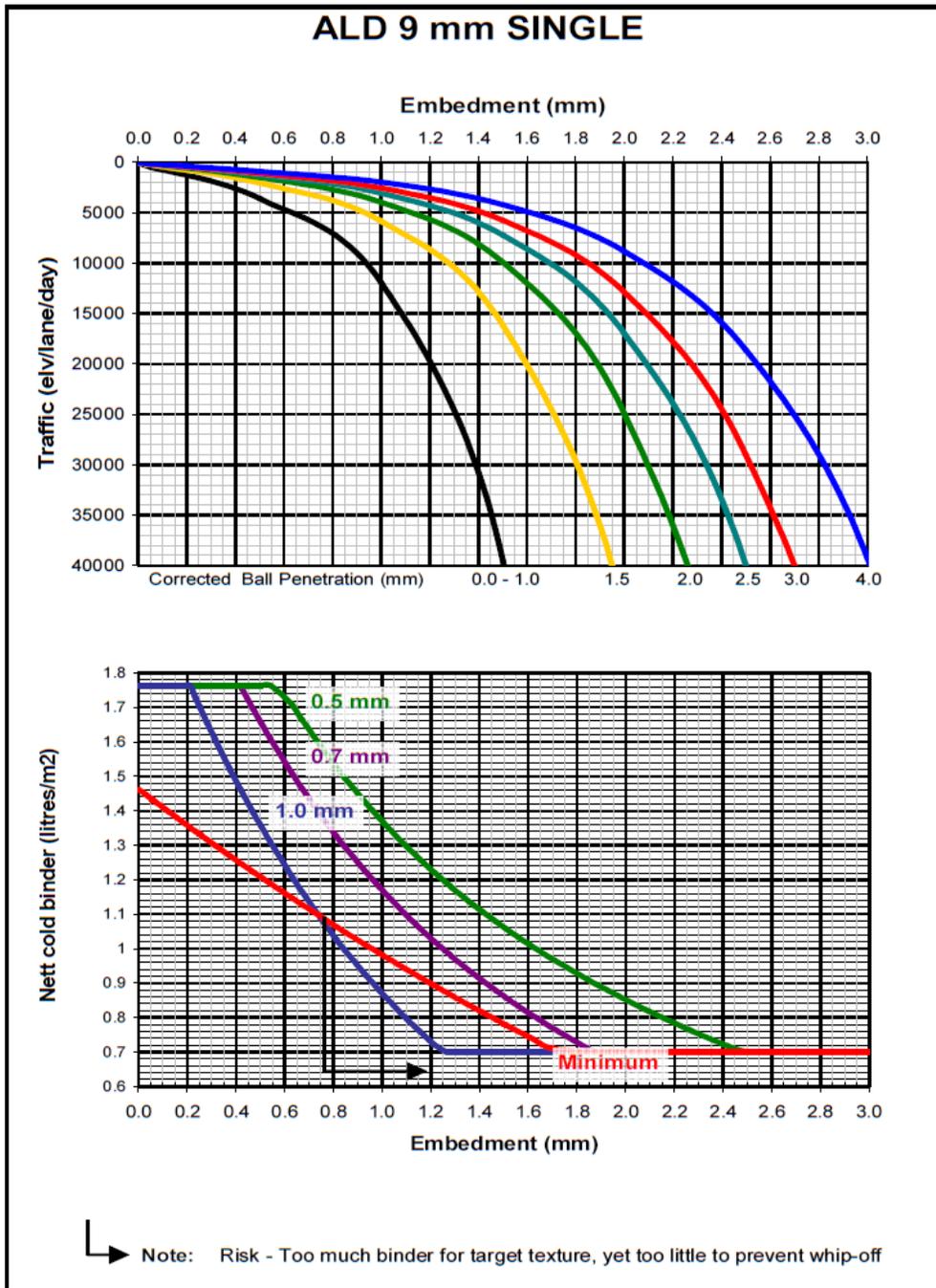
Figure 3-2- Typical Design chart for a single seal using aggregate with ALD=9mm (South African National Road Agency, 2007)

3.4.3   REST OF THE WORLD

3.4.3.1 INTRODUCTION

The TRH3 is one of the newest methods in the world and it takes a lot of inspiration from international design methods. The section below will aim to give an overview of the current and historic methods used in the rest of the world.

3.4.3.2 HANSON METHOD

In 1935 New Zealand's F.M. Hanson was the first to present a scientific approach to the design and construction of chip seals in his paper *"The bituminous surface treatment of rural highways"* (Hanson, 1935) . The principles presented by Hanson initiated a practical approach to chip sealing that remained unchallenged for the next fifty years. His design method was developed primarily for bitumen and in particular cutback bitumen and it was based on the Average Least Dimension (ALD) of the aggregate spread on the road.

Hanson calculated the ALD by manually calipering a representative aggregate sample to obtain the smallest value for ALD that represents the typical thickness of the rolled aggregate layer. He observed that when aggregate is dropped from a chip spreader onto a bituminous binder, the void remaining between the aggregate particles is approximately 50% of the ALD. (This is true in most cases for a cubical type stone.) He theorized that when the layer is rolled, this value is reduced to 30% and it is further reduced to 20% when the aggregate is embedded or compacted by traffic.

His design method involved the calculation of bituminous binder and aggregate spread rates to be applied to fill a certain percentage of the voids between aggregate particles. Hanson specified the percentage of the void space to be filled by residual binder to be between 60% and 75%, depending on the type of aggregate and traffic level (Towler & Dawson, 2008).

3.4.3.3 MCLEOD METHOD

Throughout the 1960's, Mr Norman McLeod developed a design procedure based partially on Hanson's previous work for use in the USA (McLeod, 1969). Mr McLeod's design determines the aggregate application rate based on gradation, specific gravity, shape, and a wastage factor. He provided a correction factor based on the fraction of voids.

The binder application rate depends on the aggregate gradation, absorption and shape, traffic volume, existing pavement condition, and the residual bitumen content of the binder. It should be noted that this method was developed primarily for use with emulsion binders. McLeod made it apparent that correction factors for the quantity of binder lost by absorption of aggregate and texture of existing surface are recommended. McLeod's work also gives guidelines on the appropriate type and grade of bitumen for the selected aggregate and surface temperature at time of application (McLeod, 1969).

The United States of America's Asphalt Emulsion Manufacturers Association and the Asphalt Institute have adapted this method in the form of recommendations for binder types and grades for various aggregate gradations, and correction factors to the binder application rate based on existing surface condition.

3.4.3.4 KEARBY METHOD

In 1953, Mr J.P. Kearby, an engineer with the Texas Highway Department in the USA, made one of the first efforts at designing chip seal material application rates in the United States. Kearby's design philosophy was that "*computations alone cannot produce satisfactory results and that certain existing field conditions require visual inspection and the use of judgment in the choice of quantities of asphalt and aggregate*" (Kearby, (1953)). This is still relevant and a very good design philosophy to follow.

Kearby developed a method to determine the amounts and types of bitumen and aggregate rates for chip seals. Kearby's work resulted in the development of a nomograph, shown below as *Figure 3-3*. It developed provided a bitumen application rate in volume per area. It required input data in the form of average seal thickness, % aggregate embedment, and % voids. The design methodology requires the knowledge of some physical characteristics of the aggregate, such as unit weight, bulk specific gravity, and quantity of aggregate needed to cover a specific area of roadway (Gransburg, et al., 2010).
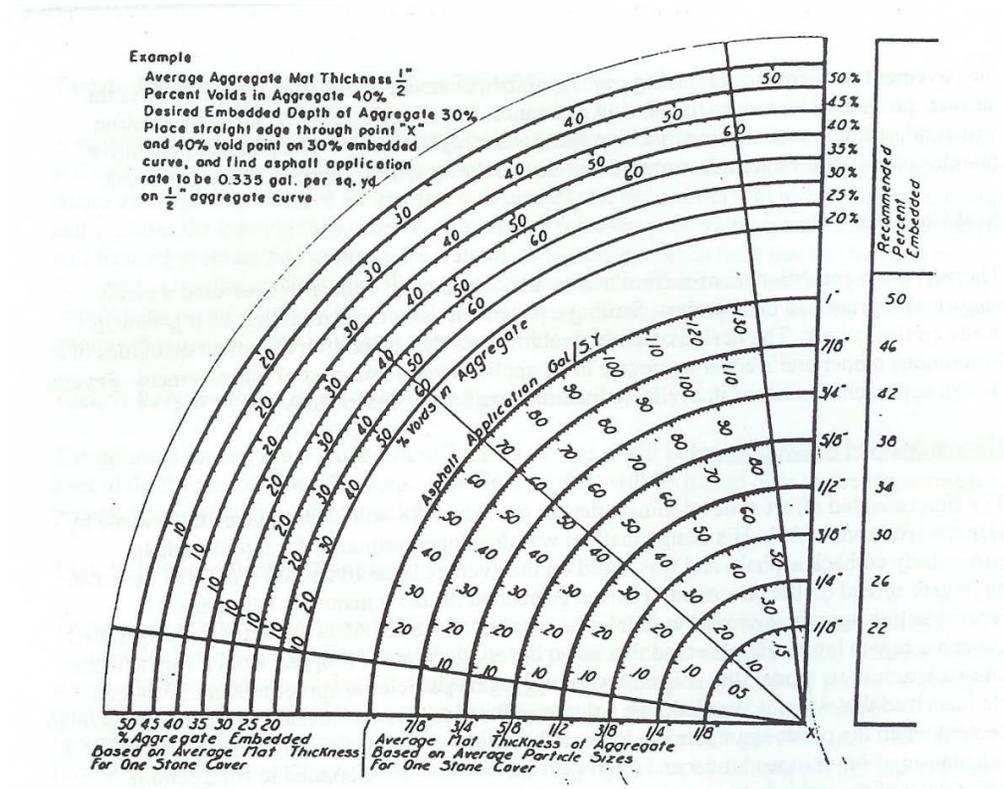
Figure 3-3- Kearby Nomograph to determine asphalt cement application rate in chip seals (Kearby, (1953))

Kearby suggested that when surface treatments are applied over existing chip sealed or tightly bonded hard base courses, the percentage of embedment should be increased for hard aggregates and reduced for soft aggregates (Gransburg, et al., 2010) .

3.4.3.5 MODIFIED KEARBY METHOD (TEXAS)

In 1974, Epps and associates proposed a change to the design monograph developed by Kearby to make it suitable for the use of synthetic aggregates in chip seals (Epps, et al., 1974). They further developed correction factors that were based on experiences and combinations that worked well in practice (Epps, et al., 1980) .

The developed binder application rate correction factors corresponded to traffic level and surface condition. Epps also suggested that consideration be given to varying the bitumen application rate both longitudinally and transversely, as reflected by the pavement surface condition. Since that time, this design approach has

been labelled as the modified Kearby method by both practitioners and researchers. Since the publication of that design procedure, the Texas Department of Transportation's Brownwood District has expanded on the bitumen application correction factors to include adjustments for truck traffic and existing surface condition.

The modified Kearby method also recommends a laboratory "board test" method to find the quantity of aggregate needed to cover a specific area of roadway. The board test is performed by placing an adequate number of chips on the required area (Gransburg, et al., 2010).

3.4.4 *ROAD NOTE 39-UK*

The United Kingdom's Transport Research Laboratory has published several editions of a comprehensive design procedure for "surface dressing" roads in the United Kingdom. The technology that makes this design procedure so advanced is the extensive use of a computer design program based on decision trees. Known as Road Note 39, this design procedure is highly advanced and uses a multitude of input parameters. Traffic level, road hardness, surface conditions, and site geometry are all critical input factors. Skid-resistance requirements and likely weather conditions are secondary inputs into the program (Roberts & Nicolls, 2008).

This procedure includes the following five steps:

1. *Selection of the type of dressing*—the selection of surface dressing (Chip Seal) is made from five treatments: single seal, texture slurry plus a single seal, racked-in seal, double seal, and sandwich seal.

2. *Selection of binder*—Binders are selected from either emulsion or cutback bitumen, specified based on viscosity. Modified binders such as polymer-modified binders are also recommended if their need and additional cost can be justified.

3. *Selection of aggregate*—The nominal size of aggregate is selected based on traffic and hardness of existing surface. Specified are 20-, 14-, 10-, 6-, and 3-mm nominal-size aggregates.

4. *Binder spread rate*—The required rate of binder spread depends on the size and shape of aggregates, nature of existing road surface, and degree of embedment of aggregate by traffic. The rate of binder spread should not vary by more than 10% from the target figure.

5. *Rate of aggregate spread*—The aggregate spread rate is determined based on a "tray test" and depends on the size, shape, and relative density of the aggregate. The basic inputs into the decision trees include selection of the type of treatment and selection of grade and type of binder based on traffic and construction season.

The aggregate type and size are selected based on skid and friction requirements, likely weather conditions, and hardness of existing surface. The resulting design application rate of binder is determined by the size and shape of aggregates, nature of existing road surface, and degree of embedment of aggregate by traffic. The resulting design application rate of aggregate spread rate depends on the size, shape, and relative density of the aggregate (Gransburg, et al., 2010).

3.4.5 *AUSTROADS SPRAYED SEAL DESIGN METHOD*

The 2004 Austroads published a *Sprayed Seal Design Manual* that provide a performance-based design method that uses an extensive list of input parameters for determining aggregate and binder application rates. Aggregate angularity, traffic volume, road geometry, ALD of aggregate, aggregate absorption, pavement absorption, and texture depth are the input variables for this method.

The main assumption of this design model is based on the original Hanson philosophy. It assumes that the aggregate in a seal is orientated approximately one layer thick and contains a percentage of air voids. Thus, filling a percentage of the voids with binder determines the binder application rate. The minimum binder application rate is determined by the percentage of voids to be filled, the total available voids, and the thickness of the seal.

The first step in the Austroads procedure is to determine a basic voids factor. Adjustments for aggregate characteristics and anticipated traffic levels are added to derive a design voids factor. That factor is then multiplied by the ALD of the aggregate to determine the basic binder application rate. This base binder application rate is then adjusted with allowances to cater to the texture and absorption of the pavement surface, the aggregate properties and road geometry. Further adjustments are made for designing as a reseal, but adjustments for surface texture and embedment are not performed (Austroads, 2004).

3.5 GENERAL

Although aggregate and binder adhesion is probably one of the most important requirement of any chip seal it is interesting to note that it is not a critical input parameter in any of the design methods. Some of the design methods propose test to ensure binder/aggregate compatibility but it is generally assumed that the selection of the binder and aggregate combination will ensure adequate adhesion.

Most of the design methods share a number of input parameters of which traffic, aggregate ALD, pavement texture and voids to be filled are the most dominant. The lack of adhesion strength as an input is attributed to the fact that there is no measurable value that relates the behaviour during the seal lifetime. This motivates the need for a test to accurately measure the bitumen aggregate bond strength and development for possible use in future design methods.

3.6 FACTORS INFLUENCING THE DESIGN & PERFORMANCE OF SEALS

3.6.1 INTRODUCTION

From the different design processes above it is clear that various factors should be considered in the design of surfacing seals. This is due to the fact that these factors directly influence the behaviour of seals. The following sections include information as presented in the original text of the TRH3 (South African National Road Agency, 2007) and will aim to detail some of the most influential factors that should be considered. *Figure 3-4* below illustrates the most relevant seal terms that is used throughout this study.
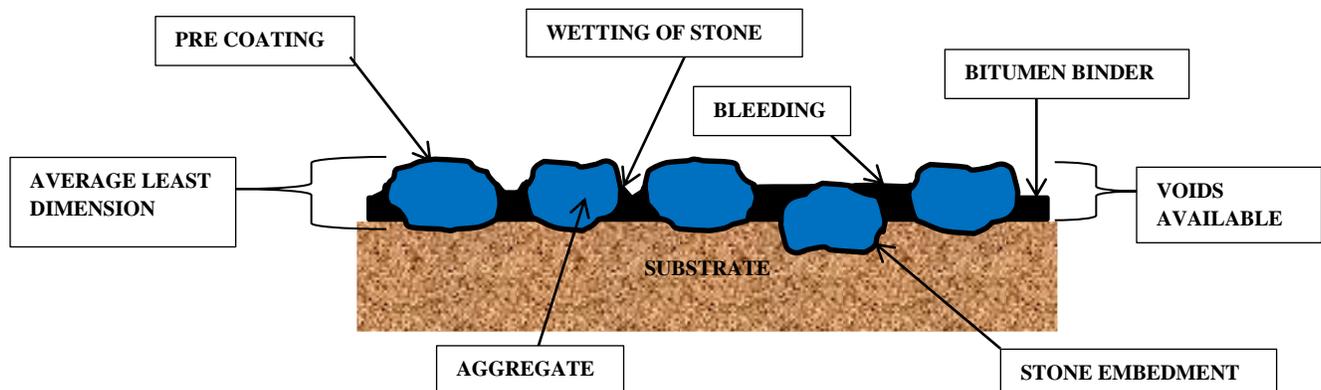
Figure 3-4 – Schematic illustration of a single seal and the terms relevant to a single seal

3.6.2   *SUBSTRATE*

Chip and spray seals are usually applied as first seals on compacted gravel or crushed stone material base material or as a reseal on varying existing surface seals. It is therefore no surprise that most important influences on the behaviour of a surfacing seal are the condition of the underlying surface and the type of material of which it is constructed. The substrate condition is one of the most important influences in determining the appropriate seal type and the type and quantity of binder to be applied as well as the size of stone to be used and the required pre-treatment.

The binder application rate will be influenced by the texture of the existing road. A new smooth pavement with low air voids will not absorb much of the binder applied to it. A dry, porous and pocked pavement surface can absorb much of the applied binder. Failure to recognize when to increase or decrease the binder application rate to account for the pavement condition can lead to excessive stone loss or bleeding.

The condition of the existing surface is generally described and measured by the visual distress discussed in *Section 3.3* above as well as by the measurement of surface texture depth and unevenness, permeability and the expected embedment of the stone. The measured values can influence the seal design as follows:

- Texture depth values and variations gives an indication of the additional binder required due to the surface texture and it may indicate the need for a texture treatment if the texture varies significantly.
- The permeability gives an indication of the need to pre-treat the existing surfacing by adding additional binder to waterproof the porous layer.
- The expected embedment of the stone gives an indication of the amount of voids in the seal that would be lost as a result of the embedment of the stone into the existing surface. In this instance, the ball penetration test provide useful information but do not take into account the softening of the existing binder by the new, sometimes very hot and oily binder applied during resealing.
- The degree and extent of cracking provides an indication of the likely reflection of such cracking through the new surfacing or of the relative brittleness and loss of flexibility of the existing surfacing which need to be made up by the new seal (South African National Road Agency, 2007).

3.6.3   *TRAFFIC*

The reason behind the existence of every road is to make it possible for various forms of traffic to travel along it safely. Traffic is also the most significant influence on the performance and behaviour of seals and the number, type, speed and combination of vehicles have a significant influence on the behaviour and performance of surfacing seals.

The traffic volume, in terms of vehicles per day, plays an important role in determining the amount of bitumen binder needed to sufficiently bond and embed the aggregate. Typically, the higher the traffic volume, the lower the binder application rate will be. The number of vehicles greatly affects embedment, wearing and polishing of the stone. This in turn reduces the voids in the seal and results in flushing and a reduction in surface texture which in turn reduces the skid resistance. It is also believed that a seal requires a minimum number of vehicles per day to keep the binder elastic and flexible (South African National Road Agency, 2007).

The forces induced by heavy axle loads forces the aggregate particles to orientate towards their ALD and to lie on their flattest side. This leads to more rapid embedment of the stone into the existing surface than in the case of light vehicle loads. Embedment in turn leads to bleeding and loss a texture required for skid resistance. The applied forces and vertical stresses induced on the surfacing can be much higher than expected due to increased tyre inflation pressures.

Speed and the rate of application of forces and stresses is another major influence in the behaviour of surfacing seals. Surfacing's under slower-moving traffic (typically less than 40km/h) generally do not perform as well as those trafficked by fast-moving vehicles. The reasons for this are the extended period of loading, horizontal stresses induced as a result of traction, particularly of heavy vehicles accelerating or breaking. Further damages include fuel and oil spillages associated with trucks and slower moving traffic.

Traffic distribution and occurrence also plays a major role in the long and short term behaviour of a seal. When traffic is concentrated in a particular wheel paths, as is typical of narrow roads and temporary deviations it will lead to accelerated embedment. High and concentrated traffic during the early life of the seal (when the binder is still soft) or during cold temperatures (when the binder is brittle) will also adversely affect the performance of the seal (South African National Road Agency, 2007).

3.6.4 *ROAD GEOMETRY*

Most design methods notes that road geometry and specifically steep gradients, sharp curves and intersection areas require specific attention during seal design and construction. Steep gradients are subjected to increased traction forces of vehicle tyres which can results in de-bonding or slippage of the surfacing. The risk of poor seal performance increases on climbs and descents because of construction difficulties at these sites. Steep gradients also cause slower vehicle speeds and that often results in flushing and bleeding.

Sharp curves cause vehicles to induce high horizontal stresses on the surfacing. These may result in ravelling and slippage of the surfacing. Vehicles on lower volume roads " cut " corners , resulting in the outer part of the road becoming dry and brittle, with consequent stone loss. Because of the camber on curves, higher loads are transferred to the inner side of curves, often resulting in fattiness. The situation may be aggravated by excess binder accumulated at these positions, as a result of run off during construction.

Breaking and acceleration, particularly of heavy vehicles at intersections, create high horizontal stresses in the surfacing which may result in the slippage, flushing and deformation of the surfacing. Fuel and oil spillages at intersection aggravate flushing and deformation (South African National Road Agency, 2007).

3.6.5 *CLIMATE AND ENVIRONMENTAL INFLUENCES*

The factors affecting the seal performance through the physical environment can be summarised as follows:

3.6.5.1 CLIMATIC CONDITIONS

The climatic conditions and other environmental influences is a major influence in the selection of the appropriate grade and type of binder to be used in a chip and spray seal. The following climatic influences should be considered carefully during the design of a seal:

- *Hot Weather:* Reduces viscosity and cohesion in binders.
- *Cold Weather:* Some binders become hard and brittle during cold weather and this will accelerate aggregate loss and cracking.
- *Uncertain Weather:* Large variations in temperatures require bitumen with specific properties.
- *UV Radiation:* Accelerates the aging of binders.
- *Humidity:* Affect the evaporation of volatiles and the breaking of emulsions , it will also accelerate ageing

3.6.5.2 TEMPERATURE

Bitumen is a temperature sensitive visco-elastic material. Very hot temperatures may lead to the softening of a binder and this in turn will decrease the viscosity. The decreased viscosity will lead to increased flow which will make the embedment of aggregate easier. Embedment will in turn lead to bleeding. Cold weather on the other hand can results in brittle, hard binders which will lead to subsequent aggregate loss or cracking. It is therefore very important that the correct and relevant binder be selected for each specific climatic area.

It is interesting that the Superpave Hot Mix Asphalt (HMA) Design System, used in the USA, determines both an expected high and a low design pavement temperature. The Superpave Binder specification and the supporting test procedures are products of the Strategic Highway Research Program (SHRP) completed in the USA during 1987-1992. It uses a bitumen performance grading system as penetration grading and viscosity grading are somewhat limited in their ability to fully characterize bitumen binder for use at varying temperature ranges. The Superpave performance grading (PG) is based on the idea that a bitumen binder's properties should be related to the conditions under which it will be used. For bitumen binders, this involves expected climatic conditions as well as ageing considerations. Performance specifications will be discussed in more detail in a subsequent section (Superpave, 2012).

3.6.6 AGGREGATES

Aggregates used in road building include both natural and processed gravels and rock. The following aggregate characteristics are listed in the TRH3 & TRH14 have an influence on the performance of surfacing seal:

- *Shape:* The shape of the aggregate influences the way it will interlock in a completed layer and this in turn influences the stability of the seal. The more angular the aggregate, the better the interlock as there is more points of contact.

- *Type, Size and Grading's***:** The size and grading influence the voids of the matrix and in turn the amount of binder it can accommodate. A single sized tone develops good interlock which increases friction and skid resistance. With large single-sized aggregates more voids is available than in small single sized aggregates. Larger aggregates therefore allow more binder to be used, resulting in a more impermeable, longer lasting seal.

- *Spread Rate of Aggregate***:** The aggregate protects the subgrade against damage and should be applied to lie shoulder to shoulder, in a single layer and in a tightly knit pattern. If the spread rate is to low it will

---

lead to excessive ultra-violet damage to the binder and in turn ravelling of the seal. If the spread rate is too high, the excess aggregate will be forced into the mat, leading to whip-off of the bonded aggregate.

- *Average Least Dimension:* The ALD is the assumed minimum thickness of the aggregate layer.

- *Crushing Strength***:** Aggregate with inadequate crushing strength will crush under the forces applied by traffic and lead to ravelling and stone loss

- *Resistance to Polishing***:** Aggregates should show resistance to polishing to ensure that friction applied by traffic does not polish the stone and lead to smoothing of the surface and in turn a loss of skid resistance.

- *Adhesion Properties***:** It is very important for the aggregate and binder to develop early and long term adhesion to ensure the aggregate is retained on the road. A loss in adhesion will lead to a loss in stone and ravelling.

- *Absorption /Porosity Properties*: Porous aggregates absorb primarily the lighter fractions of the volatiles present in the bituminous binder. This may result in the binder becoming too brittle to retain the aggregate on the road. Porous aggregate should generally be pre coated.

Aggregate and their properties will be discussed in more detail in *Chapter 6* below.

3.6.7   BITUMEN BINDERS

3.6.7.1 GENERAL

The entire service life and performance of a seal is based on good adhesion between the bitumen binder, the aggregate and the road surface and on the durability and behaviour of the binder under different climatic conditions. Adhesion is one of the vital functions of the binder. Loss in retention of the aggregate, the degree of aggregate whip-off and the durability are all related to the adhesive forces developed by the binder, which depend on the type , grade and amount of binder applied (South African National Road Agency, 2007).

A correctly selected binder must achieve two initial functions:

1. It must develop sufficient adhesive strength. It should be fluid enough once placed to allow for the placing and wetting of the stone and ten rapidly becomes harder to ensure retention of the aggregate. .

2. It must develop sufficient cohesive strength. The cohesive strength of the binder facilitates opening to the traffic and prevents the stone form being pulled out of or whipped off the surface.

The TRH3 notes that the choice of the type of bituminous binder depends on:

- *Type and purpose of seal:* Hot modified binder allow for a thicker application of binder. This enhances the ability to delay crack reflection and ensure rapid adhesion with aggregate when traffic has to be accommodated immediately after resealing. Modified binders are less sensitive to bleeding under high traffic volumes and have better elastic properties than unmodified bitumen. This enables modified binders to accommodate high deflections and low temperatures.

- *Climatic Conditions:* The climatic influences were discussed previously. The TRH3 proposed binder selection included for the different climates and traffic and is shown in *Table 3-2* below.

- *Durability of binder and long-term performance:* Penetration grade bitumen, cut-back bitumen and bitumen emulsions are durable and consequently are suitable for single and double seals. Experience in South Africa has shown that polymer modified binders are superior to conventional binders.

- *Convenience of application:* All the types of binder used in seal construction require some form of heating to bring them to a suitable spraying viscosity. Bitumen emulsions are convenient to apply, since they require less heating than other types. Some modified binders require heating up to 200°C which in turns brings special energy and safety considerations.

- *Compatibility with aggregate:* The properties of the aggregates used for seal work in South Africa vary a great deal. A binder should be selected to ensure good adhesion with a specific aggregate to be used. For example: cationic bitumen emulsions will be found to be more suitable than anionic emulsion when aggregates such as granites or quartzite's are used.

- *Traffic & Road Geometry:* Please note the influence of traffic on the selection of the type of binder as specified in *Table 3-2* taken from the TRH3. The influence of traffic and road geometry was discussed in the previous section and will not be discussed in more detail.

Table 3-2- Recommended Binders for different Traffic and Climatic conditions (South African National Road Agency, 2007)

Traffic (elv/l/d)	Winter : Dry	Summer: Dry	Winter and Rain *(c)	Summer and Rain *(c)
<10 000	(i) 80/100 pen bit + cutter * (b) (ii) MC 3000 (iii) Emulsion (80/100 pen base bitumen) (iv) Lowveld - 80/100 pen bitumen (v) Modified hot binder or emulsion *(d)	(i) 80/100 pen bitumen (ii) 65% emulsion (80/100 pen base bitumen) (iii) Highveld -80/100 pen bitumen (iv) Modified hot binder or emulsion *(d)	(i) Cationic emulsion (quick setting) (ii) MC3000 (iii) Modified hot binder *(d)	(i) 80/100 pen bitumen + 2% cutter * (a) (ii) Cationic emulsion (iii) Modified hot binder *(d)
10 000 - 20 000	(i) 80/100 pen bitumen + cutter *(b) (ii) Modified hot binder or emulsion *(d)	(i) 80/100 pen bitumen (ii) Modified hot binder or emulsion *(d)	(i) Modified hot binder or emulsion *(d)	(i) 80/100 pen bitumen + 2% cutter * (a) (ii) Cationic emulsion (iii) Modified hot binder *(d)
20 000 +	(i) Modified hot binder *(d)	(i) Modified hot binder *(d)	(i) Modified hot binder *(d)	(i) Modified hot binder *(d)

Once a binder is selected it should be able to withstand 'softening-up" at higher temperatures encountered in service and to retain the stone under the action of moving wheel loads. Softening up will happen when the used binders' softening point is lower that the operational road temperature. On the other hand, at colder temperatures, the binder should stay flexible for as long as possible to prevent reflection cracking, to accommodate road deflections and to prevent, as far as possible, the ingress of moisture into the base. The general bitumen binder properties that affect the performance of the binder on the roads can be defined as follows:

- *Binder type and physical properties:* The different types of binder such as penetration grade, cut back, emulsion and modified binder all behave differently under certain conditions and will all affect the performance of the seal in specific way.

- *Binder grade:* The correct binder grade for expected climatic, pavement and traffic conditions, both under construction and long term, must be selected to ensure optimal seal performance. Each grade of binder is characterized by its own temperature/viscosity relationship. It has an optimum range over which the bitumen can be sprayed, stored, mixed or pumped. It is essential that the viscosity be kept within the range for spray application in order to obtain optimum application and it is important for a binder to have sufficient viscosity to not soften up during operational temperatures in the road.

- *Spray Rate:* The optimal amount of bitumen is required to keep the stone on the road, (through adhesion) but to maintain enough voids to prevent bleeding. The optimum amount of binder is determined by size and shape of stone, and the volume of voids in the compacted stone layer, traffic, gradients and condition of underlying surface as per the chosen design method.

- **Viscosity at application:** Viscosity at application determines the uniformity of the application. Where bitumen is applied at too low temperature it causes streaking, due to high viscosity at the spray bar, and too high temperature it will cause binder degradation and run-off on steep gradients (South African National Road Agency, 2007).

3.6.7.2 TOWARDS PERFORMANCE SPECIFICATIONS

Considering the large amount of considerations and counter consideration mentioned in the previous sections, it is no wonder that chip seal design is still considered to be some form of art. Most of the binder selection criteria has very little to do with the short term behaviour of a seal and it is mainly based on previous experiences and the elimination of certain binder types in certain conditions as problems are experienced.

This is one of the major reasons that there is currently a global movement towards bitumen binder performance specifications and the identification of binder properties that relates to actual road performances. Various studies and discussions have identified the principles underpinning the development of a performance-based specification system. These principles include: considering binder properties related to actual road performance; testing properties that reflect emulsion residues following breaking processes; evaluating properties that have quantifiable relationships to common distresses; analysing binder properties at temperatures related to actual field climates; and recognizing that distress modes differ for chip seals and other surface treatments. (Miller, 2010) Proposed specification systems currently under development in New Zealand consider the life cycle of a chip seal from a performance perspective. *Table 3-3* shows the proposed performance grading system. It summarizes relevant phases, distress mechanisms and construction properties that must be controlled, proposed measured binder properties, and tested materials.

Through this system it may be possible to place minimum and maximum values on viscosities, bond-strength and binder ageing based on the performance of seals under different environmental and traffic conditions. It is in some way be related to the method described in the TRH3 as shown in *Table 3-3* above. It will differ that instead of a specific binder type is will include measureable binder properties related to the physical behaviour of the seal (Opus International Consultants, 2010) .To make this a reality it should be possible to measure the specific binder properties related to the seal behaviour. It is here where the bitumen & aggregate bond strength test described and developed as part of this study may help to improve binder and aggregate selection as well as to improve the measurements of the critical binder properties during the early life phases.

Table 3-3- Proposed New Zealand Performance Grading System (Opus International Consultants, 2010)

Phase	Construction Property	Binder Property	Material Tested
Construction	Chip Wetting during construction	Viscosity	Sprayed Binder
	Chip wetting and reorientation	Viscosity	Sprayed Binder/Emulsion residue
Early Life 0-5 years	Chip loss due to loss of adhesion	Bond Strength	Sprayed Binder/Emulsion residue
	Chip loss due to loss of cohesion	Bond Strength	Sprayed Binder/Emulsion residue
	Chip loss due to 'single event' cohesive failure	Bond Strength	Sprayed Binder/Emulsion residue
Mid Life 5-15 years	Binder Adhesion to tires	Viscosity	Sprayed Binder/Emulsion residue +5 years aging
End of Life 15 years +	Chip loss and cracking due to cohesive failure of binder	Cracking/Fatigue	Sprayed Binder/Emulsion residue +15 years aging
	Chip loss due to adhesive failure of binder	Bond Strength	Sprayed Binder/Emulsion residue +15 years aging

What is very important to note is that that no specification systems can accommodate poor construction practices. It therefore always assumed that under normal construction operations, chip seals will perform according to expected distress mechanisms. For example, specifications assume that chip seals will not be constructed during heavy rain, with excessively dirty aggregates, or after prolonged time intervals because these conditions will have deleterious effects on the final delivered product.

Miller (2010) described the four phases during the life cycle of a surfacing seal as shown in *Table 3-4* above in more specific detail as part of his thesis. .He noted that during the construction phase, high binder viscosities may result in inadequate chip wetting and subsequent ravelling because binders do not sufficiently coat and wet aggregates.

Bitumen emulsions overcome this deficiency due to the water based nature of the material, so this property applies only to penetration grade binder. Following the initial construction, emulsion residue's viscosity must remain sufficiently low to allow for aggregate reorientation under compaction and vehicle loading. Because all

bitumen binders are prone to water-induced stripping, chip loss due to loss of adhesion is of primary concern during the early life of seal. Chip loss can also results from ductile or cohesive binder failures resulting from stresses and strains induced by traffic loading.

Brittle binder failures may lead to chip loss when the binder becomes too stiff during prolonged periods with cold temperatures. This is especially relevant to modified binders. Mid-life distresses occur when the road bleeds due to high temperature and traffic that reduces the binder viscosity. This may lead to the binder adhering to tires which in turn will damage the seal and expose the layers underneath. Bleeding will also result in a loss of traction and skid resistance due to the loss of surface texture due to flushing.

Durability issues related to end-of-life chip loss and cracking may result from binder fatigue, so loss of bond strength between aggregates and binders has implications for long-term durability as well. Collectively, these distresses and performance properties define the effectiveness of chip seal applications (Miller, 2010).

4   BITUMEN

4.1   INTRODUCTION

The South African National Standard 4001-BT1 defines bitumen as: "*A non-crystalline solid of viscous mixture of complex hydrocarbons that possesses characteristic agglomerating properties, soften gradually when heated, is substantially soluble in trichloroethylene and is obtained from crude petroleum by refining processes.* "

Mankind has been using bitumen in a range of applications, including mummifying the dead and as a waterproofing agent, for over 5000 years. The Shell Bitumen Handbook (2003) gives some 250 uses of bitumen. Today, the main use of bitumen is in the road construction and maintenance industry.

In a chip seal the bitumen binder are used in a combination with mineral aggregates to form waterproof layers in the various combination discussed above. As a binder, bitumen is especially valuable to the engineer because it is a strong, readily adhesive, highly waterproof and durable material. Bitumen also provides some flexibility to mixtures of mineral aggregates with which it is usually combined. The strong adhesion that occurs between the bitumen and mineral aggregates enables the bitumen to act as a binder, with the mineral aggregate providing mechanical strength for the road. Bitumen is also highly resistant to the action of most acids, alkalis and salts (Shell Bitumen, 2003).

4.2   MANUFACTURE OF BITUMEN

Bitumen is manufactured during the fractional distillation of crude oil. Fractional distillation takes place in the tall steel towers usually seen at refineries. It uses the evaporation, distillation and different boiling points of all the material in crude oil to distil different fractions at various temperatures and heights within these towers.

In South Africa most bitumen used in road construction is processed at refineries in Cape Town, Durban and Sasolburg. At these refineries imported crude oils are refined to produce petrol, diesel fuel and other petroleum based products. Bitumen represents only about 2.5% of a total barrel of oil.

4.3 COMPOSITION OF BITUMEN

The arrangement of the internal structure of bitumen is largely determined by the chemical composition of the molecular types present. Bitumen is an intricate chemical mixture of molecules that are mainly hydrocarbons with a small amount of functional groups containing sulphur, nitrogen and oxygen atoms.

The precise composition varies according to the source of the crude oil from which the bitumen is distilled, modification and blowing during manufacture. It is therefore clear that the chemical composition of bitumen is extremely complex and a complete analysis of bitumen would be impractical. It is however possible to separate bitumen into two broader groups called asphaltenes and maltenes as indicated in *Figure 4-2*. The maltenes can be further sub divided into saturates, aromatics and resins.
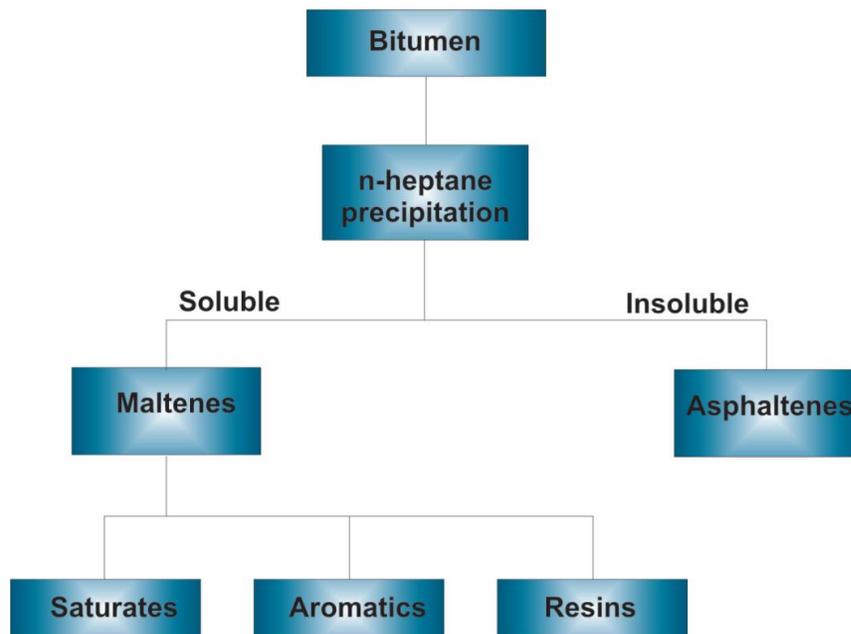
Figure 4-1- Broad Chemical Composition of Bitumen (SABITA-M2, 2007)

4.4 TYPES AND GRADES OF BITUMEN

There are six major classifications of bitumen produced by the refining and manufacturing process in South Africa.

4.4.1 PENETRATION GRADE BITUMEN'S

Penetration grade bitumen is the most widely used bitumen and is refined and blended to meet civil engineering and industrial specifications. The specifications are based on specific test properties that aim to account for different climatic conditions. Paving grade bitumen is also considered as the parent bitumen/base binder from which the other forms are produced. The table below indicates the South African Penetration Grade requirements (SANS4001-BT1, 2012).

Table 4-1- South African Penetration Grade Bitumen Requirements

1	2	3	4	5	6
Property	**Penetration grade**				**Test method**
	35/50	50/70	70/100	150/200	
	Requirements				
Penetration at 25 °C/100 g/5 s, 1/10 mm	35-50	50-70	70-100	150-200	EN 1426
Softening point (ring and ball), °C	49-59	46-56	42-51	36-43	ASTM D36[a]
Viscosity at 60 °C, Pa.s	220-400	140-250	75-150	30-60	ASTM D4402[b]
Viscosity at 135 °C, Pa.s	0,27-0,65	0,22-0,45	0,15-0,40	0,12-0,30	ASTM D4402[b]
Performance when subjected to the rolling thin film oven test:					ASTM D2872
a) mass change, % (by mass fraction), max.	0,3	0,3	0,3	0,3	ASTM D2872
b) viscosity at 60 °C, % of original, max.	300	300	300	300	ASTM D4402[b]
c) softening point (ring and ball), °C, min.	52	48	44	37	ASTM D36[a]
d) increase in softening point, °C, max.	7	7	7	7	ASTM D36[a]
e) retained penetration, % of original, min.	60	55	50	50	EN 1426
Spot test[c], % xylene, max.	30	30	30	30	AASHTO T102

[a] Using shouldered ring.
[b] Recommended apparatus is the RV viscometer, using SC 4 spindles with thermosel system.
[c] Actual values to be reported in five-unit intervals (see annex A).

4.4.2 CUTBACK BITUMEN

Cutback bitumen are in essence penetration grade bitumen blended with a small quantity of volatile solvents to reduce the viscosity for ease of handling and application, which, after the volatile solvents have evaporated, essentially reverts to the penetration-grade bitumen base (SANS4001-BT2, 2012) .Classifications include rapid curing (RC), medium curing (MC) or slow curing (SC). A cutback bitumen varies in behaviour according to the type of cutter or flux used as the diluent. White spirits is commonly used for rapid curing grades, kerosene for medium curing grades and diesel for slow curing grades.

4.4.3 *BLOWN BITUMEN*

Blown bitumen also known as Industrial bitumen or oxidised bitumen's are made by blowing air through hot paving grade bitumen. The so-called blowing process results in harder bitumen that softens at a higher temperature than that at which paving grade bitumen softens. Blown bitumen also has more rubber-like properties and their viscosities are much less affected by changes in temperature than is the case with paving grade bitumen (BP Bitumen, 2011).

4.4.4 *BITUMEN EMULSIONS*

Bitumen emulsion consists of bitumen particles dispersed in water in the form of an oil-in-water type emulsion. The bitumen is held in suspension by an emulsifying agent that also determines the charge of the bitumen emulsion. The two most common types are Cationic (+) bitumen emulsions that have a positive charge and Anionic (-) bitumen emulsion that have a negative charge.

The South African national Standards define bitumen emulsion as a *"liquid mixture in which a substantial amount of bitumen is suspended in a finely divided condition in an aqueous medium by means of one or more suitable emulsifying agents."* (SANS 309, 2004) Due to its important role in this study, bitumen emulsion will be discussed in more detail in *Chapter 5* below.

4.4.5 *MODIFIED BITUMEN AND EMULSIONS*

4.4.5.1 *INTRODUCTION*

Chapter 3 has shown that the properties of standard bitumen are not always able to meet the functional and behavioural requirement when a bituminous surfacing are subjected to severe conditions such as steep gradients, very high road surface temperatures, high traffic loading or heavily trafficked inter sections, or are used on highly flexible and cracked pavements.

Under these circumstances a definite improvement in the rheological properties of the bitumen is required and this is where the different types of modified bitumen come into play. Some of the benefits that may be derived from modification include; improved consistency, reduced temperature susceptibility, improved stiffness and cohesion, improved flexibility, resilience and toughness, **improved binder aggregate adhesion** and improved resistance to in services ageing.

4.4.5.2 MODIFICATION AND USES IN SOUTH AFRICA

Modification is achieved by mixing penetration grade bitumen with polymers, crumb rubber, aliphatic synthetic wax or naturally occurring hydrocarbons. The SABITA Manual 2 classifies polymers into "elastomers" (sometimes referred to as thermoplastic elastomers) for improving the strength and elastic properties of a binder, and "plastomers" (sometimes referred to as thermoplastic polymers) for increasing the viscosity of the bitumen. However a wide range of binder properties can be improved, the most important being:

- Improved Rheology of flow characteristics
- Resistance to permanent deformation and fatigue cracking due to increased elasticity
- Cohesion and Adhesion
- Elasticity
- Reduced Ageing and Durability (SABITA-M2, 2007).

The section below is a modified extract from the original text of (SABITA-M2, 2007) and describes the types of modified bitumen that are readily available and used in South Africa:

"

- ***Styrene-Butadiene-Rubber (SBR) latex:*** *SBR is a block co-polymer and can be classified as either linear or radial. It is available in the form of anionic or cationic latex, which makes blending with bitumen emulsion easier. It is also used to modify hot bitumen, but the water phase in the latex must first be re moved by boiling or foaming during the controlled addition of latex to the hot bitumen. SBR modified bitumen has been used extensively in South Africa as a cold applied bitumen emulsion in chip seals and micro surfacing, as well as a hot applied binder in chip seals and hot mix asphalt. The modified binder exhibits elastic properties which make it ideal for surfacing lightly cracked pavements.*

- ***Styrene-Butadiene-Styrene (SBS):*** *SBS polymers are available in powder, crumb or pellet form for modifying hot bitumen. Linear as well as radial co polymers can be used depending on the end properties sought. High shear mixers are recommended for blending high polymer content binders, particularly for industrial applications. Depending on the concentration of this polymer, it increases or improves softening point, cohesive strength, elasticity, low temperature flexibility and resistance to permanent deformation. SBS modified bitumen is used in both chip seals and hot mix asphalt applications to enhance the bitumen's all-round performance characteristics.*

- ***Ethylene-Vinyl-Acetate (EVA):*** *EVA polymers are available in pellet form and are easily dispersed in hot bitumen. EVA modified binders are thermally stable have the most pronounced effect on the binder's end properties are its molecular weight and vinyl acetate content. EVA modified bitumen is used mainly as a plastomer in hot mix asphalt to improve rut resistance.*

- ***Bitumen Rubber:*** *About 20% of rubber crumb (recycled tyres) is blended with bitumen at a mixing temperature of 170° - 210°C for a period of approximately one hour. During this time the aromatic oils in the bitumen are absorbed by the rubber particles, causing them to swell. It is much more viscous than unmodified bitumen, and is not a homogeneous binder, requiring special equipment for pumping and spraying. Bitumen rubber is widely used in South Africa in chip seals. On cracked and flexible pavements, bitumen rubber has resisted crack reflection remarkably well and, in spite of high application rates, its resistance to flushing has been clearly demonstrated. This resistance is due, amongst others, to its improved temperature susceptibility of viscosity. The carbon black contained in the rubber also acts as an anti-oxidant, thereby increasing the durability of the binders.*

- ***Synthetic Waxes:*** *Long-chain hydrocarbons produced by the Fischer-Tropsch (F-T) synthesis process are used to extend the plasticity range of bitumen. Bitumen modified with F-T wax displays unique properties in that it has a lower viscosity than unmodified bitumen above 100°C, but on cooling the viscosity is higher. This enables bitumen modified with F-T wax to be sprayed and placed at lower temperatures than using conventional bitumen. The significant increase in Ring and Ball softening point of binders modified with F-T wax renders asphalt incorporating such binders to have enhanced resistance to permanent deformation and bleeding. The Figure below shows these properties in more detail:*
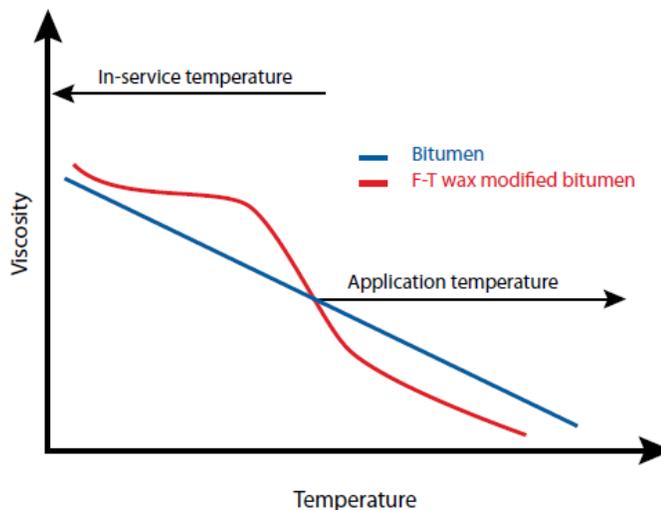
Figure 4-2- Typical effect of F-T wax on viscosity over temperature

- *Naturally occurring hydrocarbons: Naturally occurring hydrocarbons that are used for bitumen modification occur in deposits in North and South America, and are known as Gilsonite and Durasphalt respectively. These materials have high asphaltene contents, generally in excess of 70%, and are used to stiffen bitumen by raising the Softening Point and lowering the Penetration value. Asphalt mixes containing binders modified with these hydrocarbons display high resilient modulus values compared with those containing binders modified with polymers. The high resilient moduli achieved will enhance both the resistance to permanent deformation and the load spreading capability of the layers* (SABITA-M2, 2007).*"*

4.4.5.3 CLASSIFICATION OF MODIFIED BINDERS

The Asphalt Academy's Technical Guideline 1 (TG1) is currently the most important authority on modified binders used in road construction. It classifies modified binders according to the four groups that include the type of application, type of modifier, type of binder system and the level of modifications. The details of these four groups can be summarised as follows:

- *The type of application:* Seal (S) , Asphalt (A) or Crack sealant (C)
- *Type of modifier:* Elastomer (E) , Plastomer (P) , Rubber Crumb (R) of Hydrocarbon H)
- *Type of binder system:* If the product is an emulsion, the letter C would follow directly after the letter indicating the type of application.
- *Level of modification:* Usually indicates modifier content

A numerical value is used to indicate increasing softening point values. This classification system allows for a polymer-binder specification whereby the test properties for a specific class must be achieved in order to meet the specification requirements. *Table 4-2* below shows the Typical Binder Classes specified in the TG1.

Table 4-2- Modified Binder Classification System (Asphalt Academy-TG1, 2007)

Modified Binder Class	Application – Surface Seal
S – E1	Surface seal – hot applied elastomer modified
S – E2	Surface seal – hot applied elastomer modified
S – R1	Surface seal – hot applied bitumen rubber
SC – E1[1]	Surface seal – emulsion elastomer modified
SC – E2[1]	Surface seal – emulsion elastomer modified
Modified Binder Class	**Application – Premixed Asphalt**
A – E1	Hot mix asphalt – elastomer modified
A – E2	Hot mix asphalt – elastomer modified
A – P1[2]	Hot mix asphalt – plastomer modified
A – H1	Hot mix asphalt – hydrocarbon modified
A – H2[2]	Hot mix asphalt – hydrocarbon modified
A – R1	Hot mix asphalt – bitumen rubber
AC – E1	Microsurfacing – emulsion elastomer modified
AC – E2	Microsurfacing – emulsion elastomer modified
Modified Binder Class	**Application – Crack Sealant**
C – E1	Crack sealant – hot applied elastomer modified
CC – E1	Crack sealant – emulsion elastomer modified
C – R1	Crack sealant – hot applied bitumen rubber

4.4.5.4 BENEFITS OF MODIFICATIONS

The primary aim of the modification of bitumen for use in surfacing seals is to increase the resistance to bleeding at high road temperatures without compromising the properties of the seal over the rest of the prevailing temperature range. (Asphalt Academy-TG1, 2007)

The use of modified bitumen is rising as a result of increases in tyre pressures, axle loads and higher traffic volumes that in turn required improved binder performance. Improved performance can be achieved in three ways:

- An increase in the elastic component with an associated reduction in the viscous component; and

- Stiffening of the bitumen to reduce the total viscoelastic response of the layer.

- Increased adhesion and cohesion.

To explain the benefits of modification it is important to understand the improvements it makes to each of the properties of normal penetration grade bitumen. The four sections below is a combination of extracts from the original text included in (SABITA-M2, 2007) *&* (Asphalt Academy-TG1, 2007) and it will aim give a brief overview look of the influence of modification on certain behavioural characteristics.

RHEOLOGY (FLOW BEHAVIOUR)

The TG1 defines Rheology as the study of the flow and deformation behaviour of materials. Bituminous binders behave as visco-elastic materials and their behaviour is influenced by the loading time as well as the operating temperature. At high temperatures or long loading times, binders will generally behave as viscous liquids. This will lead to bleeding and permanent deformations in asphalts. At low temperatures or short loading times, binders will behave as elastic (brittle) solids, with most deformation recovered at the end of the loading period.

The rheology of unmodified binders is relatively simple, and behaviour can be predicted through the use of simple tests such as Penetration, Softening Point and Viscosity at various temperatures. The rheology of modified binders on the other hand is highly complex, and, although the results from conventional tests may indicate a significant improvement in properties, the in-service performance of these binders is not easily categorised. (Asphalt Academy-TG1, 2007)

These properties can be explained by looking at an elastomer modified binder. The figure below shows the typical effect of an elastomer on the rheological profile of penetration grade bitumen. The following interesting observations can be made:

- At high road temperatures, say 55°C - 60°C, the modified binder has a significantly higher viscosity than, the base penetration grade bitumen and is therefore much stiffer. In conjunction with the elastic nature of the polymer network, such modified binders will exhibit a significantly higher resistance to deformation and bleeding.

- At the spray temperature of 130°C-180°C the modified binder also shows significantly higher viscosity. This enable a higher application of modified binder as runoff will not be a problem.

- On the other had of the scale at lower service temperatures, the modified binder is more flexible than the unmodified bitumen and therefore would be less prone to cracking and becoming brittle. This can be seen from the higher penetration values at around and below 0°C. Increased flexibility and resistance to fatigue distress has also been shown to result from elastomer modification.
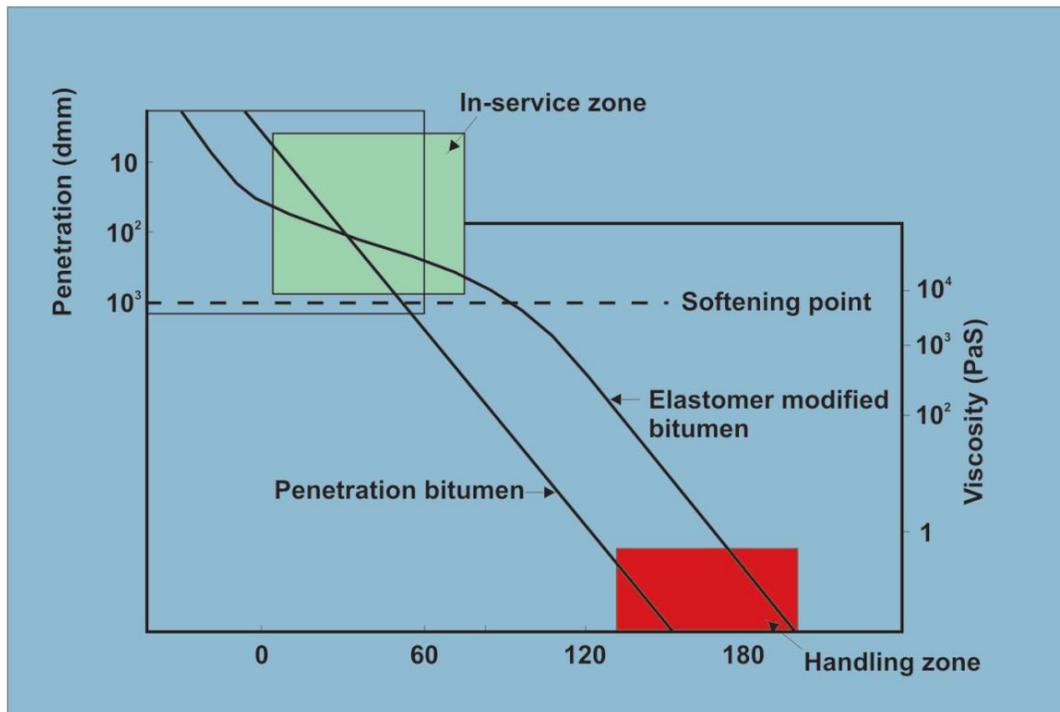
Figure 4-3- Effect of elastomers on the rheological profile of bitumen (SABITA-M2, 2007)

It can be deduced from the information included in *Figure 4-6* above that the modified binder material would offer improved rheological performance in areas where high temperature and tensile strains are likely to occur.

ELASTICITY

Elasticity can be defined as a physical property of materials which return to their original shape after the stress that caused their deformation is no longer applied. (Atanackovic & Guran, 2011) The elastic recovery of a binder is commonly used to measure the fatigue resistance of a binder or its ability to absorb large stresses without necessarily cracking or deforming.

Particular modifiers have unique elastic recovery characteristics depending on the morphology of the modifier/binder system. For example, SBS as described above forms a three-dimensional network of highly elastic, butadiene chains connecting stiff styrene domains, resulting in very high elastic recoveries. SBR has random styrene/butadiene molecules, resulting in relatively lower elastic recovery.

EVA forms a rigid three-dimensional network, imparting no elastic recovery properties to the base binder but provides a high stiffness. The elastic property of a binder is generally influenced by the type of modifier as well as the degree of modification and is determined by measuring the recovery of a sample which has been extended in a low temperature ductilometer. In general, there is a direct relationship between elastic recovery and the degree of modification". (Asphalt Academy-TG1, 2007)

COHESION

The TG1 defines cohesion as a measure of the tensile stress required to break the bond between molecules of the bituminous binder. The inherent strength, tenacity and toughness of the bituminous binders are improved by modification with thermoplastic polymers and rubber crumbs. Hence, a greater force or tensile stress is required to break the molecular bonds of modified binders and cause failure compared with a lower tensile stress required to break the bonds of conventional binders.

A force-ductility test is used to determine the cohesive strength of a modified binder and involves the elongation of a sample with the force measured at very small elongation intervals. *Figure 4-7* shows a graph of the typical profile of various modified binder types obtained during the test.

As shown in *Figure 4-7*, the maximum force is reached early in the elongation process. The elastic phase is represented by the area before the initial peak and the total area under the curve can be used to calculate toughness. This is a good indication of the energy required to extend the binder and therefore provides a good estimation of resistance to cracking.
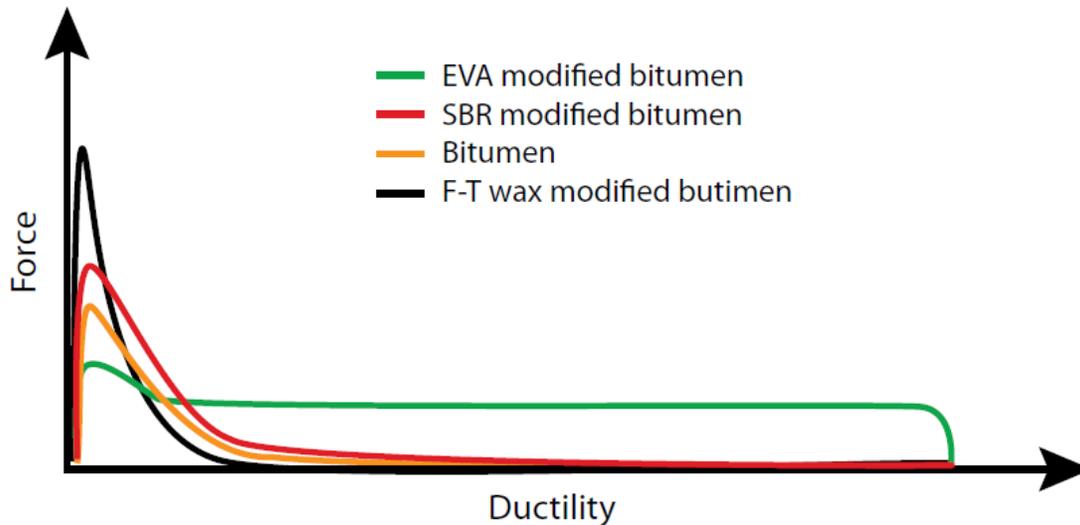
Figure 4-4- Typical force-ductility curves for various modified binders (Asphalt Academy-TG1, 2007)

The energy required to elongate elastomeric modified binders is generally significantly more than that for conventional binder. Plastomeric modified binders will impart stiffness to the bituminous binders but not necessarily improve its cohesive nature. Such modified binders may well perform in a brittle manner in tension. The cohesive properties of modified binders provides direction to design engineers on how soon after construction a seal could be opened to traffic as well as providing an assessment of the ability of the binder to withstand shear stresses imparted by heavy traffic. (Asphalt Academy-TG1, 2007)

ADHESION

The TG1 further describes and define adhesion as the measure of the stresses required to break the bonds between the bituminous binder and mineral aggregate. **Adhesion is largely dependent on the physical chemistry as well as the chemical nature of the bituminous binder and aggregate type when combined for application.** The following factors have an impact on adhesion at the stone/bitumen interface:

- The presence of dust and/or moisture which could reduce adhesion at the bitumen/aggregate interface.
- The level of modification that influences the viscosity of bitumen which in turn affects the wetting ability or time to coat the road stone with bituminous binder.
- Ambient road and air temperatures and, especially overnight temperatures.

Generally by increasing the film thickness of the binder layer the adhesion can also be improved. Temporary reduction of the viscosity by the addition of cutters during colder weather conditions also improves the adhesion properties by keeping the binders flexible. Care should however be taken in areas with hot humid climates and/or heavy traffic conditions as binders with cutter may tend to bleed.

It will be discussed in more detail in *Chapter 6* but it should also be noted **that different types of aggregates will exhibit different adhesion behaviour depending on the chemical nature of the parent rock in terms of its hydrophilic (water-attractive) or oleo phobic (oil-repelling) nature.** Bitumen is by nature oleophilic (oil-attracting) or hydrophobic (water-repelling).

Therefore, based on the inherent character of the aggregate it may or may not react (form chemical, charge-related bonds) with water but the presence of water will have a negative influence on the adhesion properties and it will repel the bitumen. Acidic aggregates are more hydrophilic than basic aggregates. Acidic aggregates will therefore have poor adhesion properties in the presence of water. Cationic spray grade emulsion overcomes this tendency when the free electrons on the aggregate form physical/electrical bonds with the positively charged bitumen and SBR latex droplets as shown if *Figure 4-8*. (Asphalt Academy-TG1, 2007)
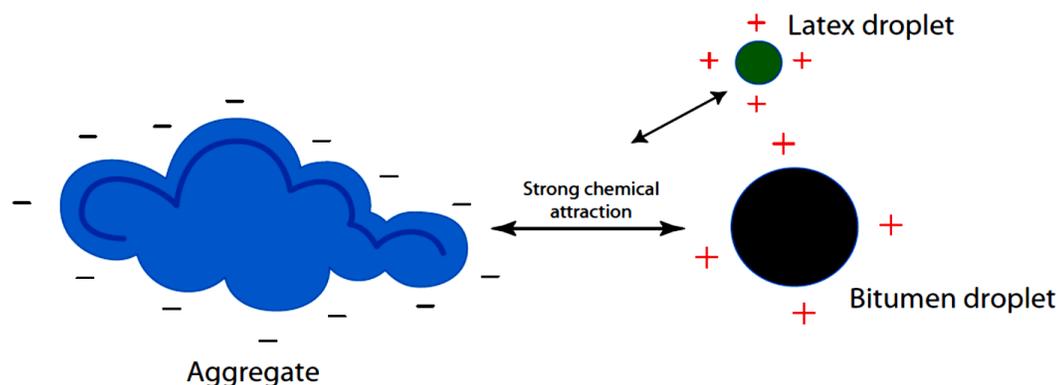
Figure 4-5- Adhesion characteristics of Cationic latex modified emulsions (Asphalt Academy-TG1, 2007)

5 BITUMEN EMULSIONS

5.1 BACKGROUND

In a paper by Kobus Louw published in 2004 he described the origin of bitumen emulsion as follows:

"In 1922, two British scientists patented their invention of bitumen emulsion, which was further improved the following year when a new patent was filed in France. The use of bitumen emulsions rapidly spread throughout the world and by 1928 the first emulsion factory in South Africa, was erected in Bellville by the company Colas. The first emulsions manufactured were all anionic in character and prepared from the soaps formed by neutralising long chain fatty acids such as tall oil, oleic acid, stearic acid and napthenic acids. Due to the neutralisation of the fatty acids by sodium or potassium hydroxide, anionic emulsions are alkaline in character and impart a negative charge to the dispersed bitumen droplets. It was long realised that the use of anionic emulsions presented adhesion problems with certain difficult aggregates such as quartzite's and granites. The development of cationic emulsifiers, based on long chain amines, largely overcame these problems during the late 1950's. The long chain amines are neutralised with mineral acids such as hydrochloric, sulphuric and sometimes phosphoric acid, and the resultant acidic emulsifier imparts a positive character to the bitumen emulsion (Louw, et al., 2004)."

5.2 DESCRIPTION

Bitumen is difficult to work with at ambient temperatures since it is a highly viscous material under these conditions. It can, however, be transformed into a workable state by either applying heat (hot mixes), by blending with petroleum solvents (cutback mixes) or by emulsification with a surfactant in water to form bitumen emulsion (Gorman, et al., 2004).

An emulsion is a dispersion of small droplets of one liquid in another liquid. Typical examples include such everyday products as milk, butter, mayonnaise, and cosmetic creams. Emulsions can be formed by any two immiscible liquids, but in most emulsions one of the phases is water.

Oil-in-water emulsions are those in which the continuous phase is water and the dispersed (droplet) phase is an "oily" liquid. Water-in-oil "inverted" emulsions are those in which the continuous phase is oil and the disperse phase is water. Emulsions can also have more complex structures.

Bitumen emulsions are therefore dispersions of bitumen in water. It is manufactured by processing hot bitumen, water and emulsifier in a high speed colloid mill that disperses the bitumen in the water in the form of small droplets. These droplets or particles of bitumen are normally in the 5-10 micrometre size range but may be even smaller.

The emulsifier assists in forming and maintaining the dispersion of fine droplets of bitumen. Bitumen emulsions normally comprise between 30% and 80% bitumen by volume and have a lower viscosity compared to the bitumen from which they are produced. This makes emulsion workable at ambient temperatures.

Emulsions are brown liquids with consistencies from that of milk to double cream, which depend mostly on the bitumen content and the particle size *(Gorman, et al., 2004).* The figure below shows a micrograph of a bitumen emulsion:
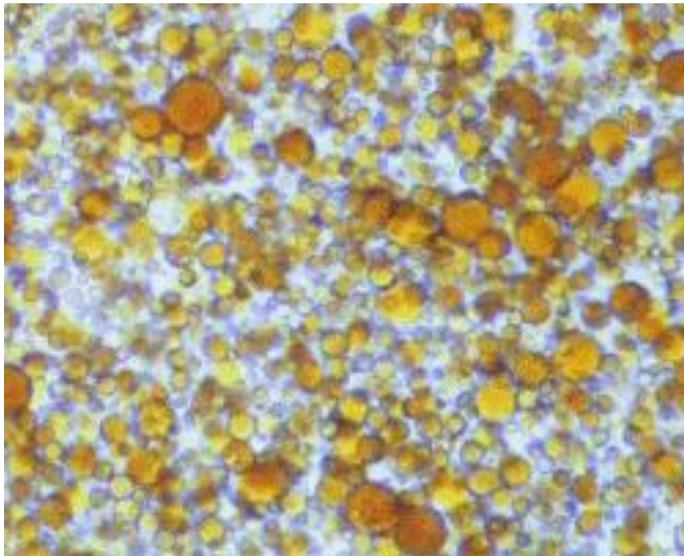
Figure 5-1- Micrograph of Bitumen Emulsion (James, 2006)

Bitumen emulsions are generally available in either Cationic or Anionic Emulsions. The terms cationic and anionic is derived from the electrical charges on the bitumen globules. In an anionic emulsion the bitumen particles are negatively charged. In a cationic emulsion the bitumen particles are positively charged. Cationic emulsions are more widely used as they have superior adhesive properties to a range of mineral aggregates (Gransburg, et al., 2010).

5.3 MANUFACTURING OF EMULSIONS

Emulsions are manufactured by mixing hot bitumen with water containing emulsifying agents and applying mechanical energy sufficient to break up the bitumen into droplets. The mechanical energy is applied using a colloidal mill as shown in *Figure 5-2* below. A colloidal mill consists of a high speed rotor revolving at 1000-6000 rpm in a stator. The clearance between the rotor and stator can usually be adjusted between 0.25 and 0.5 µm.

Figure 5-2- Colloidal Mill (Jaixing Mide Machinery Company, 2012)

The emulsion manufacturing process is shown in a schematic from the TG2 below:
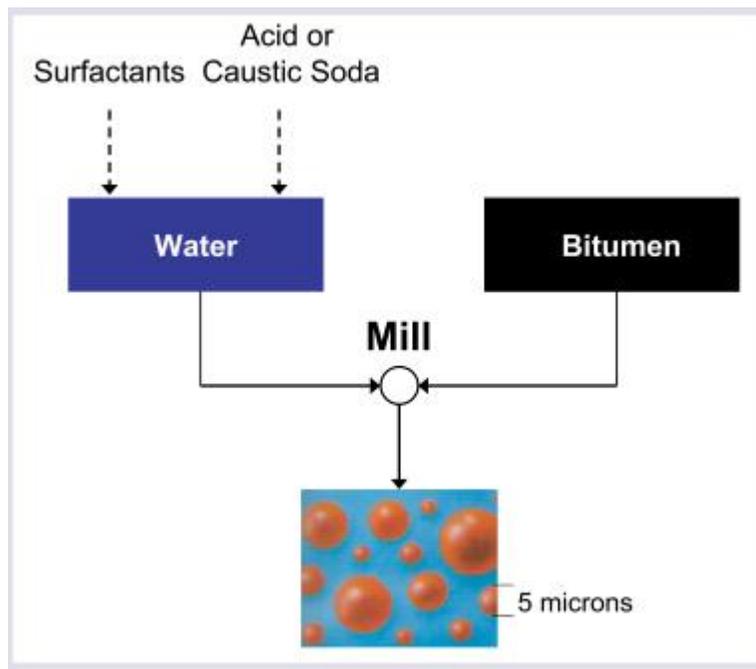
Figure 5-3- Bitumen Emulsion Manufacturing Process (Asphalt Academy-TG2, 2009)

The water phase is prepared whereby the emulsifier chemicals are dissolved in heated water and either mixed (soaped) with alkalis in the case of an ionic emulsion, or acidified with inorganic acids for cationic emulsions. The temperature of the bitumen phase should be strictly controlled to ensure that its viscosity is within the appropriate range for emulsification, and to prevent boiling of the emulsion on exit from the colloid mill.

The base bitumen normally used is 70/100 penetration grade. The stability of the emulsion is determined primarily by the type and quantity of the emulsifying agent used. Mixing any anionic emulsion with any cationic emulsion in any proportion will cause the mixture to "break", i.e. separate into water and bitumen, almost immediately.

5.4 TYPES AND CLASSIFICATION OF EMULSIONS

Bitumen emulsions are classified according to the sign of the charge on the emulsified bitumen droplets and according to their reactivity and rate of setting. Cationic emulsions have droplets which carry a positive charge (+). Anionic emulsions have negatively charged droplets (-).

Rapid-setting (RS) emulsions set quickly in contact with clean aggregates of low-surface area, such as the aggregates used in chip seals. Medium-setting (MS) emulsions set sufficiently less quickly that they can be mixed with aggregates of low surface area, such as those used in open-graded asphalt mixes. Slow Setting (SS) emulsions will mix with reactive aggregates of high surface area.

In the USA emulsions are named according to the *ASTM D2397 - 05, 2012* and *ASTM D977-12, 2012* standards. Cationic Rapid Set, Cationic Medium Set and Cationic Slow Set emulsions are denoted by the codes CRS, CMS, and CSS, whereas anionic emulsions are just called Rapid Set , Medium Set , and Slow Set, followed by numbers and text indicating the emulsion viscosity and residue properties.

In South Africa the *SANS 309, 2004* and *SANS 548, 2003* specifications are relevant. These standard divide both anionic and cationic into three usage specific categories as defined below:

- **Pre-mix type** emulsions is emulsion with sufficient stability to allow mixing with certain types of aggregate before breaking of the emulsion occurs. (Medium Set)
- **Spray type** emulsions is characterized by rapid breaking of the emulsion on application, and is normally unsuitable for mixing with crushed aggregate. (Rapid Set)

- **Stable-mix type** emulsions have sufficient mechanical and chemical stability for all purposes involving mixing with stone chippings, natural gravels, and soil. This includes aggregates containing large proportions of fines or chemically active materials such as cement or hydrated lime. (Slow Set)

The tables below indicate the specification requirements of Anionic and Cationic Emulsions.

Table 5-1- South African Anionic Bitumen Emulsion Specification (SANS 309, 2004)

1	2	3	4
	Requirement		Test method
Property	Pre-mix type and spray type	Stable-mix type	
Viscosity at 50 °C, Saybolt Furol seconds	20-50	–	ASTM D244
Binder content*, % (m/m)	60-62	60-62	*
esidue on sieving, g/100 mL, max.	0,25	0,25	See 6.4
Sedimentation after 60 complete rotations	Nil	Nil	See 6.5
* By difference from water content determined in accordance with method ASTM D244.			

Table 5-2- South African Cationic Bitumen Emulsion Specification (SANS 548, 2003)

1	2	3	4	5	6	7	8
	Requirement						Test method
Property	Spray type			Pre-mix type		Stable-mix type*	
	Grade						
	60	65	70	60	65	60	
Viscosity at 50 °C, Saybolt Furol seconds	20-50	51-200	51-400	20-50	51-200	50 max.	ASTM D 244
Binder content, %(m/m)	60-63	65-68	70-73	60-63	65-68	60-63	+
Fluxing agent content, # %(m/m) of binder	5 max.	5 max.	5 max.	5-10	5-10	Nil	ASTM D 244
Residue on sieving, g/100 mL, max.	0,25	0,25	0,25	0,25	0,25	0,25	See 6.2
Particle charge							See 6.3 ASTM D 244
a) Modified procedure	Positive	Positive	Positive	Positive	Positive	—	
b) Standard procedure	—	—	—	—	—	Positive	
Binder deposit on the cathode after 30 min, g, min.	1,0	1,0	1,0	—	—	—	See 6.3
Sedimentation after 60 complete rotations	Nil	Nil	Nil	Nil	Nil	Nil	See 6.1
Coagulation value when mixed with standard silica flour, %(m/m), max.	—	—	—	—	—	2,0	See 6.5

* A purchaser should ensure that the proposed stable-mix emulsion is suitable for use with the aggregate that he intends to use for a specific project.

+ By difference from water content determined in accordance with method ASTM D 244.

In ASTM D 244 "Fluxing agent" is referred to as "oil distillate".

5.5 BITUMEN EMULSION PROPERTIES

The emulsification of bitumen into bitumen emulsions may decrease the viscosity but the addition of emulsifiers gives emulsion certain specific properties that control their behaviour. The section below will have a look at the chemical properties and their influence on the breaking mechanism of emulsions.

5.5.1 CHEMICAL PROPERTIES

The emulsifiers used in bitumen emulsion can be classified as surface active agents or surfactants. Surfactants have non-polar lipophilic (oil-loving) and polar hydrophilic (water-loving) portions in the same molecule. These molecules concentrate at the interface between the water and bitumen and are orientated with the polar group in the water and the nonpolar parts of the molecule in the oil. The figure below shows a visual representation of these molecules. (James, 2006)
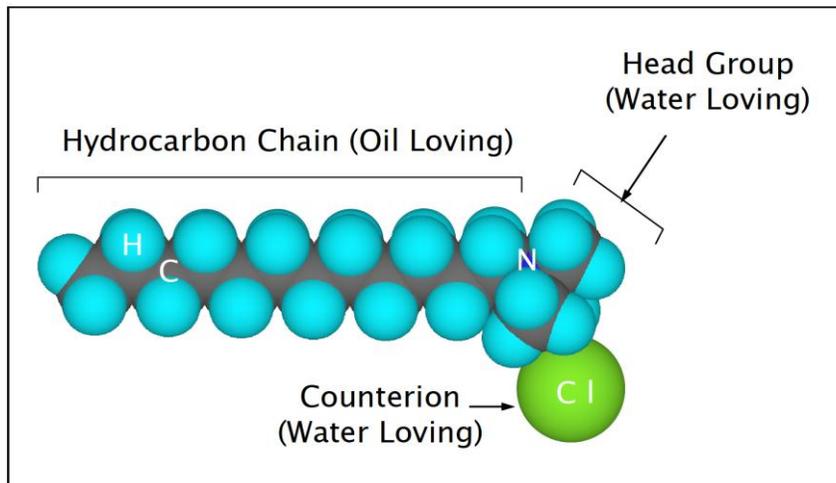
Figure 5-4- Cationic Emulsifier Molecule (James, 2006)

The orientation of the molecules reduces the energy required to emulsify the bitumen and prevents coalescence of the droplets once formed. The choice and concentration of emulsifier also largely determines the charge on the bitumen droplet and the reactivity of the emulsion produced (Boussad & Martin, 1996). From *Figure 5-4* above it can be seen that a typical emulsifier has a hydrophilic "head" group and lipophilic (hydrophobic) hydrocarbon "tail" comprising 12 to 18 carbon atoms. This hydrocarbon tail is represented by "R" in chemical formulas. It is most often derived from natural fats and oils, tall oil, wood resins, or lignin.

Emulsifiers can be classified into anionic, cationic, and non-ionic types depending on the charge their head groups adopt in water, although this charge may also depend on pH (James, 2006) .*Table 5-3* below details the chemistry of bitumen emulsifiers.

Table 5-3- Chemistry of Bitumen Emulsifiers (James, 2002)

Lipophilic Portion (Hydrophobic)	Head Group	Counter Ion	Head Group Charge at pH=2 (Acidic)	Head Group Charge at pH=11 (Alkaline)
Tallowalkyl-	$[-NH_2CH_2CH_2CH_2NH_3]^{2+}$	$2\ Cl^-$	Positive	Neutral
Tallowalkyl-	$[-N(CH_3)_3]^+$	Cl^-	Positive	Positive
Nonylphenyl-	$[-O(CH_2CH_2O)_{100}H]$	None	Neutral	Non Ionic
Tall Oil -	$[-COO]^-$	Na^+	Neutral	Negative
Alkylbenzene	$[-SO_3]^-$	Na^+	Negative	Negative

Cationic emulsifiers are ammonium compounds that contain positively charged nitrogen (N) atoms in their head group; anionic emulsifiers typically contain negatively charged oxygen (O) atoms. The two Figures below shows the typical chemical formulas of Anionic and Cationic emulsions. Note the head and tail groups as described above:

Figure 5-5- Anionic Emulsifier (Bickford, 2001)

Figure 5-6- Cationic Emulsifier (Bickford, 2001)

Several studies have shown that even non-ionic emulsifiers produce emulsions whose droplets have a small negative charge in water and non-ionic emulsifiers are often used in slow-setting bitumen emulsions. In the case of bitumen emulsions, the bitumen itself contains surface active species which can also concentrate at the interface. The size and sign of the charge on the bitumen droplets can be measured and is expressed as the "zeta potential" of the droplet (Wates, 1993).

The zeta potential is strongly pH-dependent both because of the pH dependence of the charge on the emulsifier and also because polar components of the bitumen itself may ionize. Zeta potential measurements show that the *charge on the bitumen droplets becomes more negative as the pH rises.* As the concentration of the emulsifier increases, the particle size of the emulsion is reduced. Slow Setting emulsions, which contain higher concentrations of emulsifier, generally have smaller particle size than Rapid Setting grades (Rabiot & B. G. Koenders, 1999).

Emulsifiers are often supplied in a water-insoluble form to the emulsion producer and need to be neutralized with acid or alkali by the emulsion manufacturer to generate the anionic or cationic water-soluble form used to prepare the soap solution. The choice of the acid or alkali and the final pH of the emulsion influence the emulsion properties. Hydrochloric acid and occasionally phosphoric acid are the acids used, and sodium and potassium hydroxide are the most common alkalis. Cationic emulsions are usually acid, and anionic emulsions are typically alkaline. *Table 5-4* below shows some typical emulsion recipes.

Table 5-4- Typical Emulsion Recipes (James, 2006)

Cationic Rapid Setting (CRS65)		Cationic Slow Setting (CSS60)		Anionic Rapid Setting (RS65)		Anionic Slow Setting (SS60)	
Bitumen	65	Bitumen	60	Bitumen	65	Bitumen	60
Tallowdiamine	0.2	Tallow diquaternary ammonium chloride	0.6	Tall Oil	0.3	Ethoxylated nonyl phenol	0.5
Hydrochloric acid, 35%	0.15			Sodium hydroxide	0.2	Lignins	0.5
Soap pH	1.5-2.5	Soap pH	3-7	Soap pH	11-12	Soap pH	10-12
Water	To 100	Water	To 100	Water	To 100	Water	To 100

Increasing the emulsifier concentration decreases the reactivity of the emulsion. Medium Set emulsions are generally formulated with the same emulsifiers as Rapid Set grades but at higher concentration (0.4%–0.8%). The emulsion producer can adapt the emulsion recipe to cope with reactive aggregates or high temperatures, generally by increasing the emulsifier concentration or blending emulsifiers of lower reactivity (James, 2006).

5.5.2 *BREAKING OF EMULSION*

Emulsified bitumen must revert to a continuous bitumen film in order to act as a binder in a surfacing seal or other application. This typically involves flocculation and coalescence of the bitumen droplets and removal of the water from the applied emulsion. There has been a lot of research on the specific mechanisms that is responsible for the breaking of bitumen emulsions (James, 2002) .Consensus has been reached that breaking takes place due to one or more of the following mechanisms:

- Adsorption of free emulsifier on aggregate.
- Rise in pH caused by aggregate or cement.
- Loss of water.
- Adsorption of bitumen particles on aggregate.

The figure below is a visual representation of the possible stages of setting of a cationic emulsion
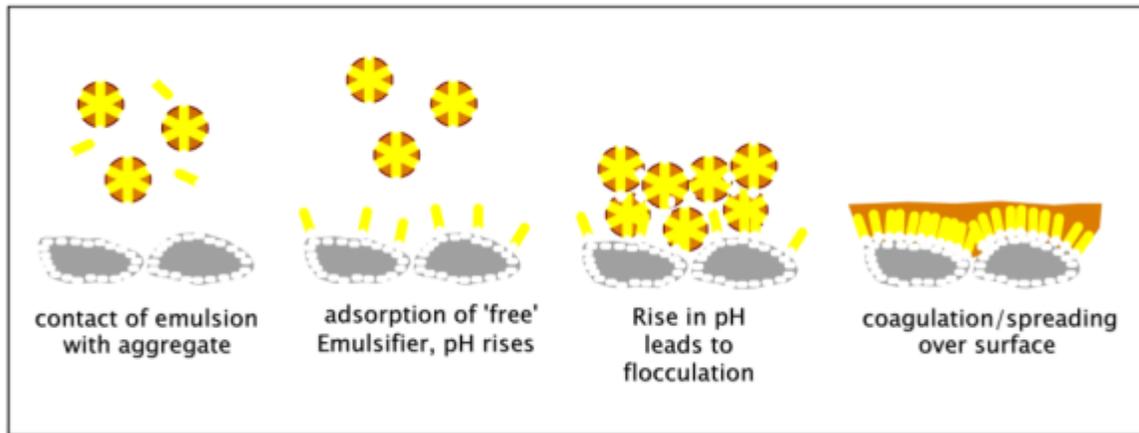
*Figure 5-7- Possible stages in the setting of cationic emulsion (**James, 2002**)*

The process above is also described in the *Transport Research Circular Number E-C102* (James, 2006) as follows:

 "

- *Free emulsifier adsorbs onto the (oppositely charged) mineral surface, which neutralizes some charge on the surface while at the same time making the surface somewhat lipophilic. Too high a free emulsifier concentration in relation to the surface area of the aggregate can actually reverse the charge on the minerals and so inhibit the setting of the emulsion.*

- *Minerals neutralize acids in the emulsion, causing loss of charge on the emulsion droplets, leading first to flocculation of the asphalt droplets and then to a slower coalescence of the droplets.*

- *The coalescence of the droplets results in a continuous film of binder, with some minute water droplets initially being trapped in the coalesced layer. Water is adsorbed by the mineral, as well as evaporates from the system.*

- *Droplets in contact with the mineral spread on the surface, especially that surface made lipophilic by adsorbed emulsifier, eventually displacing the water film on the aggregate surface."*

The speed of the breaking, setting and curing processes depends on the reactivity of the emulsion, the reactivity of the aggregate and environmental factors, such as temperature, humidity, wind speed, and mechanical action. The setting or breaking of a bitumen emulsion can therefore be divided into four distinct phases as per James (2006) and are depicted in *Figure 5-8* on the next page.
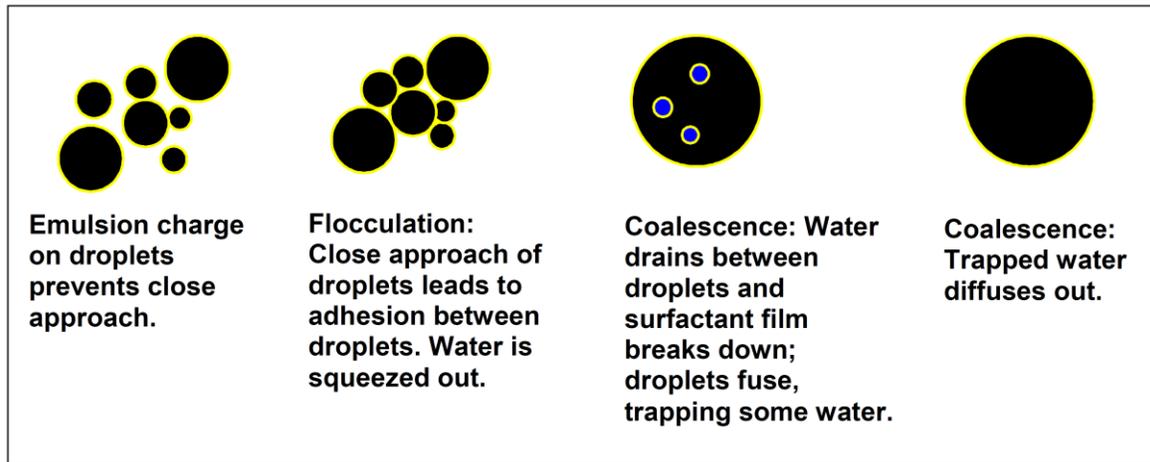
Figure 5-8- Typical stages in the breaking of emulsions (James, 2006)

- In the first phase the emulsion is stable and the distance between the dispersed particles is sufficient to prevent contact or possible coalescence.
- In the second phase there is a close approach between the droplets which can be caused by destabilisation of the emulsifier phase, evaporation of the water phase or mechanical action. This phase is called the flocculation phase.
- In the third phase the close proximity of the bitumen droplets, cause them to fuse and flow together.
- The final phase is the coalescence phase where the last of the water is fused out and the bitumen residue is in its final form.

Each of these phases is influenced by different chemical and environmental factors. Important factors in the breaking process are as follows:

- Changes in pH caused by reaction of the aggregate with acids in the emulsion
- Adsorption of free emulsifier onto the aggregate surface
- The flocculation of the emulsion droplets with the fines.
- Evaporation of water increases emulsifier content which leads to flocculation
- Mechanical action, such as compaction or traffic, may squeeze the droplets together, promoting coalescence and squeezing water out of the coalesced film (Walter, 2002).

5.5.3 ADHESION

Aggregates take up a characteristic surface charge in water which depends on the nature of the minerals, the pH, and the presence of soluble salts. So-called "acid" aggregates high in silica tend to take up a negative charge. The Figure below is a simplified visual representation of the reaction of Cationic and Anionic Emulsion on "Acid" aggregates. From this is can be seen that Cationic emulsion will form a strong tight bond with "Acid" aggregates as the positive charges of the emulsion will be strongly attracted by the negative charge of the "Acid" aggregate.
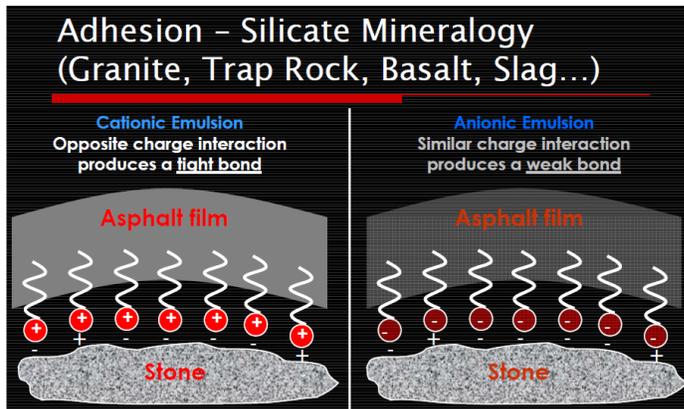
Figure 5-9- Emulsion reaction with Acid Rock Types (Bickford, 2001)

Other, none "Acid" aggregates like carbonates, and fillers, like cement, may neutralize the acid in cationic emulsions causing the pH to rise and the emulsion to be destabilized. The figure below is a simplified visual representation of the reaction of Cationic and Anionic Emulsion on "Basic" aggregates.
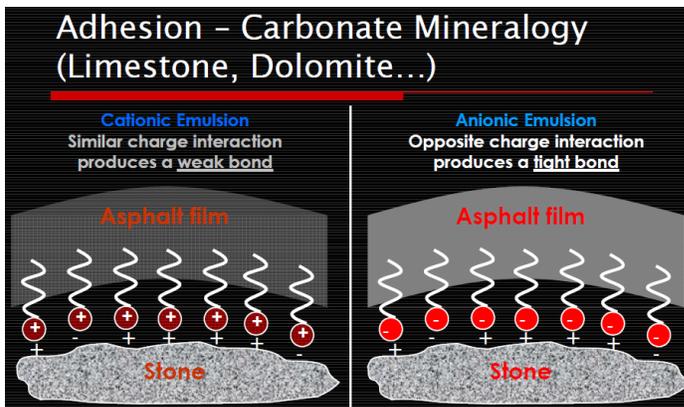
Figure 5-10- Emulsion reaction with Acid Rock Types (Bickford, 2001)

From the figures above it can be concluded that Cationic Emulsion will form stronger bonds with Acidic rocks than Anionic Emulsion would. This will be tested and confirmed in subsequent Chapters of this report.

6   AGGREGATES

6.1.1   INTRODUCTION

The section above briefly touched on acid and basic rocks and it is clear that there is a significant influence on bitumen and bitumen emulsions behaviour when they come into contact with different aggregates. The term aggregate generally refers to crushed rock but other materials may also be used. Aggregates are the second major component of any chip and spray seals as it bonds with the bitumen binder to form a strong, durable and waterproof surface that must fulfil the following six major functions:

- The texture, angularity of a chipped matrix of aggregate has a specific texture that provides a skid resistance surface.
- Rock aggregate has a natural resistance to abrasion and polishing that is expected on a road due to moving wheel loads and traction forces.
- The resistance to crushing also makes the transfers the wheel loads to the underlying pavement structures possible.
- The chipped aggregate matrix also provides a strong skeletal structure to accommodate the elastic and impermeable bituminous binder.
- This skeleton structure needs to have sufficient strength and voids to prevent the binder from flushing to the surface under loading.
- Aggregate also protect the bitumen binder from the harmful ultra-violet rays of the sun (Milne, 2004).

6.1.2   TYPES OF AGGREGATE

Rock and crushed rock aggregate types used in road construction have historically been classified as either "Acid" or "Basic" rocks. The geology and chemistry of rocks is unfortunately not that simple. Rocks are generally classified by mineral and chemical composition, by the texture of the constituent particles and by the processes that formed them. These indicators separate rocks into three major rock types: igneous, sedimentary, and metamorphic.

Igneous rocks are formed when molten magma cools and are divided into two main categories: plutonic rock and volcanic.

Plutonic rock or intrusive rocks result when magma cools and crystallizes slowly within the Earth's crust (e.g. granite), while volcanic or extrusive rocks result from magma reaching the surface either as lava or fragmental ejected rock (e.g. pumice and basalt)

Sedimentary rocks are formed by deposition of clastic sediments, organic matter, or chemical precipitates (evaporates), followed by compaction of the particulate matter and cementation during diagenesis. Sedimentary rocks form at or near the Earth's surface. Typical distribution and examples include:

- Mud rocks comprise 65% (mudstone, shale and siltstone)
- Sandstones 20 to 25%
- And Carbonate rocks 10 to 15% (limestone and dolerite)

Metamorphic rocks are formed by subjecting any rock type (including previously formed metamorphic rock) to different temperature and pressure conditions than those in which the original rock was formed. These temperatures and pressures are always higher than those at the Earth's surface and must be sufficiently high so as to change the original minerals into other mineral types or else into other forms of the same minerals (e.g. by recrystallization) (Blatt & Tracy, 1996) . Examples of Metamorphic rock include Quartzite and Hornfels.

The table below was extracted from *"The natural road construction materials of South Africa"* (Weinert, 1980) and it shows the characteristic combinations of rock forming materials in rocks and well as examples of typical rocks in each of the rock group.

Stellenbosch University http://scholar.sun.ac.za

Table 6-1- Characteristic combinations of rock forming materials in rocks (Weinert, 1980)

Major Rock Group	Sub Group	Characteristic Minerals	Typical Rock
Igneous	Acid	Quartz, Orthoclase, Mica or amphibole	**Granite**
	Intermediate	Orthoclase, amphibole	Syenite
		Plagioclase, Amphibole	Diorite
	Basic	Plagioclase, Amphibole	Norite,Dolerite
	Ultra-Basic	Pyroxene,Oliviae	Pyroxenite,Peridotite
Sedimentary	Clastic	Quartz	Sandstone
		Quartz, Orthoclase	Arkose
		Quartz and incidental others	Conglomerate
		Clay mineral , some quartz	Shale
		Quartz, clay mineral and incidental others	**Tillite, Greywacke, volcanic ejecta**
	Chemical Precipices	Calcite	Limestone
		Dolomite	Dolomite
		Opal and/or chalcedony	Chert
		Various Salts	Gypsum and other salt deposits
	Organic	No Minerals	Coal, oil
Metamorphic	Subdivisions are complicated and of little relevance to the engineering properties of rock	Quartz, Orthoclase (occasionally mica)	Gneiss
		Quartz, Muscovite (occasionally biotite)	Mica Schist
		Amphibole	Amphibolite
		Quartz	Quartzite
		Calcite	Marble
		Amorphous silica or quartz and various others	Hornfels

The classification of aggregate as acid and basic in the road construction industry was originally done in the TRH7 published in 1972. Weinert explained that the basis for the TRH7 classification was not always correct due to the fact that original classification was based on the assumption by earlier petrologists the silica (SiO_2) is the radical of silicic acid (Weinert, 1980).

Weinert proposed the chemical concept that a rock (or aggregate) is acid if the acidic compounds, SiO_2 and CO_2 comprise more than 60 percentage mass as determined in a chemical analysis. The table below shows this classification for some of the better known and used aggregates

Table 6-2- Rock Classification System (Weinert, 1980)

ROCK NAME	ALTERNATIVE NAME (OLD TRH7)	% BY MASS OF SUM OF SiO_2+CO_2	CLASSIFICATION
Quartz	Quartzite	100	ACIDIC
Quartzitic sandstone	Sandstone	94.6	
Vein quartz	Quartzite	64.26	
Sparagmite	Tillite	80.9	
Arkose	Sandstone	75.9	
Granite	**Granite**	**70.18**	
Greywacke	**Tillite**	65.8	
Shale	Hornfels	**60.7**	
Syenite	Syenite	60.19	
Andesite	Andesite	59.59	SLIGHTLY ACIDIC
Quartz basalt	Basalt	55.46	
Quartz	Norite	54.39	
Magnesite	Dolomite	52.38	
Trachy-dolerite	Dolerite	49.2	BASIC
Basalt	Basalt	49.06	
Gabbro	Norite	48.27	
Dolomite	Dolomite	47.82	
Limestone Marble	Marble	46.77	
Basanite Basalt	Basalt	44.64	
Calcite Marble	Marble	44	
Nephelinitc Dolerite	Dolerite	41.17	

The TRH14 published in 1985, also states that bitumen as a rule does not adhere well to the "acidic" rocks referred to above. Weinert concluded that the strength of adhesion of bitumen to aggregate depends, as far as the stone is concerned, on its surface texture and not due to its Acidity. He reasoned that the more unevenly textured the surface, the stronger the adhesion is, and the smoother the surface the weaker the adhesion (Weinert, 1980). It is assumed that he did not include Bitumen Emulsions in this theory as it has been shown that the acidity of aggregate influences the breaking of emulsion types (Louw, et al., 2004).

As noted above, the quantity of silica (SiO_2) or carbon dioxide (CO_3) and whether the rock is 'acid' or 'basic', is of little importance. All rocks are weakly negatively charged and there is no noticeable difference in regard to the total charge between the different types of rock. Weinert goes on to make the following very interesting statement.

"Considering the number of charged i.e. ionized, atoms in a rock, just more than 30 per cent of the atoms in a rock are anions, all being oxygen ions and just more than 20 per cent are cations. These values are very much the same for all types of rock. In general, therefore, all rocks have a weak negative charge and there is no justification for explaining the different adhesive properties of binders through a difference between 'acid' and 'basic' rocks."

The section below is based on extracts from the original text of Weinert book *(Weinert, 1980).*

"The chemical composition of rocks has, however, an indirect influence on the surface texture which is obtained after crushing. If only a limited number of chemical components make up the bulk of the mass of a rock, the number of different types of mineral in such a rock is small and the individual minerals may reach a relatively large size. In most cases, silica (SiO_2) is the most prevalent material component in rocks. In this case the properties of the rock may be superior to such a degree that the other component has little or no influence.

The resulting rock is of that type which is commonly called 'acid', e.g. granite but also quartzite, and which is normally characterized by rather large minerals, particularly the orthoclase feldspar in the case of granite. On crushing, such a rock produces many smooth faces or smooth areas within such faces. If silica becomes so pronounced that it is the only or nearly the only component of the rock, as in vein quartz, the extremely strong bond between the quartz minerals causes a fracture, as developed during crushing, to pass right through the minerals and smooth surfaces are again the result. If much or most of the rock consists of amorphous silica and its bond with other minerals, particularly quartz, is sufficiently strong, e.g. in quartzite or hornfels, smooth crushing faces are again produced.

Also in cases where another component is present in above average quantity, e.g. potassium in syenite, large individual minerals (orthoclase feldspar in the case of syenite) develop and crushing leads to smooth, flat faces. There is no such tendency to produce flat, smooth crushing faces in 'basic' rocks which contain large sized minerals only in exceptional cases. These differences in the type of crushing face formed in the different types of rock have probably led to the impression that 'acid' rocks cause more problems with adhesion than do 'basic' ones."

Stellenbosch University http://scholar.sun.ac.za

Road building experience has shown, no matter what the reasons, that that not all rock types adhere equally well to bitumen and there is definite difference in the affinity of acidic and alkaline rocks to bitumen and bitumen emulsions. This can in part be explained by the interaction of emulsion and aggregates as discussed in the previous section. This is also confirmed by the following extract from a paper by Kobus Louw published in 2004.

"Basic aggregates such as limestones are slightly soluble in water, raising the pH of the water phase which will destabilise the emulsifier system of a cationic emulsion, resulting in the initiation of coagulation. During the dissociating of limestone, calcium ions are also released into the water phase. When anionic emulsions are used with limestone aggregates, the calcium ions can destabilise the anionic emulsifier system, promoting the on-set of coagulation. (Louw, et al., 2004)"

6.1.3 *GENERAL*

Based on the information above it is clear that aggregates crushed from different rocks will show varying behaviour in combination with different binders. These influences include the mineralogy of the rocks, whether it is "Acid" or "Basic" and the final surface texture of the crushed aggregates. These influences will be discussed and evaluated in *Chapter 9* below.

It must be noted that various types of both acid and basic aggregates are used in South Africa to construct seals. The major influence on the type of aggregate used is usually the aggregate availability. Aggregate sources are generally situated near large cities and town centres or in strategic positions in rural areas. Due to the vast distances of the South African road network, coupled with the cost involved of hauling material over long distances, the closest available source is usually used. It is almost always easier and more economical to change the binder than it is to change the aggregate sources.

During the testing phase of this project the properties of granite and tillite will be investigated in more detail.

7 TEST METHODS AND EQUIPMENT

7.1 INTRODUCTION

The information discussed in the preceding section showed how important it is in the early life of a chip seal for the bitumen binder and aggregate to develop adequate adhesion. This is critical to ensure that no premature failure takes place due to adhesion-related problems. The need for a laboratory test capable of measuring the required information is therefore critical. As part of his thesis Mr Timothy Miller described the need as follows:

"A number of different types of test have been developed to compare combination of bitumen, aggregate and water. While some tests measure performance properties, it is clear from the literature review that existing test methods do not capture the full spectrum of chip seal performance over the entire chip seal life cycle. Common tests related to emulsion composition; consistency and material stability assess binder production but do not address field performance. Therefore, the focus of this investigation is on the test methods and equipment needed to evaluate performance properties, as this is seen as the greatest obstacle to instituting performance-based specification systems" (Miller, 2010)

The section below will evaluate the current adhesion tests available as well as comment on their suitability to test the bitumen-aggregate adhesion bond strength development.

7.2 ADHESION TESTS

The Shell Bitumen Handbook places adhesion testing into a number of categories. There may be several different tests within each type but, in most cases, the individual tests of one type differ in detail rather than in principle (Shell Bitumen, 2003) .These categories can be summarised as follows:

- Static immersion & Dynamic immersion tests
- Chemical immersion tests
- Immersion Mechanical & Immersion Trafficking tests
- Coating Tests
- Absorption tests
- Impact tests
- Pull –off Tests

7.2.1 STATIC IMMERSION TESTS

This is the simplest type of test and consists of aggregate being coated with bitumen that is then immersed in water. The fundamental problem with this method is its subjective nature and the fact that it is completely operator dependant and has poor repeatability. It must also be noted that in some cases, an aggregate with good laboratory performance may perform poorly on some occasions and those with poor static immersion test results may perform satisfactory in practise. An example of a static immersion test is *the British Standard EN 13614:2011, Bitumen and bituminous binders. Determination of adhesively of bituminous emulsions by water immersion test.*

7.2.2 DYNAMIC IMMERSION TESTS

This type of test is very similar to the static immersion test with the only difference being that the sample is agitated mechanically by shaking or kneading. The degree of stripping is again estimated visually and repeatability is very poor and results are operator dependant. An example of a dynamic immersion test is the British Standard *EN 12274-7:2005 Slurry surfacing. Test methods. Shaking abrasion test.*

7.2.3 CHEMICAL IMMERSION TESTS

In this type of test, aggregate coated in bitumen is boiled in solutions containing various concentration of sodium carbonate. The strength of sodium carbonate solution in which stripping is first observed is used as a measure of adhesion. However the artificial condition of the test is unlikely to predict the likely performance on the road. An example of a chemical immersion test is the *TMH 1 - Method B11 -The determination of Adhesion of Bituminous Binder to Stone Aggregate by means of the Chemical Immersion test* also known as the Riedel & Weber test.

7.2.4 MECHANICAL AND IMMERSION TRAFFICKING TESTS

Immersion mechanical tests and Immersion trafficking tests is mostly relevant to Asphalt and include test like the *Retained Marshall Stability Test*, the *Retained stiffness test*, retained *Cantabro test* and the *Immersion wheel tracking* test. These tests are not relevant for chip seals and will therefore not be discussed in more detail.

7.2.5 COATING TESTS

This type of testing aims to assess the adhesion between aggregate and bitumen in the presence of water. In the *Immersion Tray Test* from the British Road Note 39 (Roberts & Nicolls, 2008), aggregate chips are applied to a tray of bitumen covered with a thin layer of water. By careful examination of the chippings, it may be possible to determine whether surface active agents improve adhesion under wet conditions.

7.2.6 ADSORPTION TESTS

As part of the Strategic Highways Research Program (SHRP) in the USA, the *Net Absorption Test* method was developed to evaluate moisture damage in asphalts (Curtis, et al., 1993) .This test is an extremely complicated test and it requires a (not so readily available) spectrophotometer to perform measurement. It is not a commonly used test in South Africa and will not be discussed in further detail.

7.2.7 IMPACT TEST

The Vialit pendulum Test and the Vialit Plate Test is generally the only two test available to measure bitumen adhesion with impact tests. Both these methods are easily adapted to measure a wide range of conditions. The test are relevant in situations where aggregate is in direct contact with traffic stresses and it is therefore ideal for test related to chip seals.

The Vialit Pendulum Test is shown in the picture below. The procedure involves placing a thin film of binder between two cubes and measuring the energy required to remove the upper block.
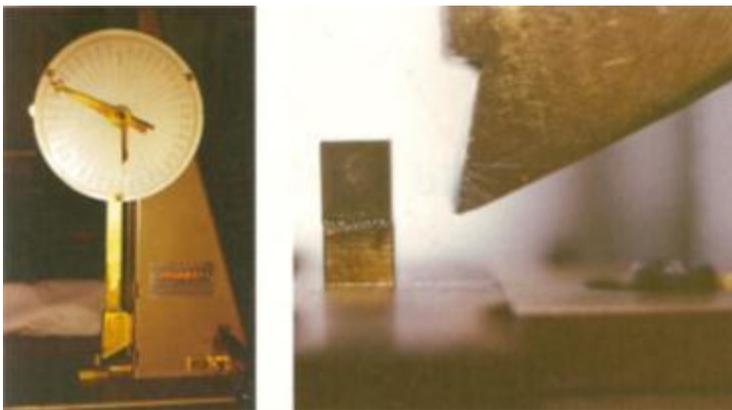
Figure 7-1- Vialit Pendulum test from the Shell Bitumen Handbook (Shell Bitumen, 2003)

For the Vialit Plate Test, aggregate particles are pressed onto a steel plate of bitumen. The plate is the turned upside down and a steel ball is dropped onto the reserve side. The impact of the ball may cause detachment of the aggregate particles depending on the test conditions. The number of detached aggregate chips vs. the number of impacts can be used as an indication of performance. Visual inspection of the detached aggregate can usually determine the type of failure. In South Africa a modified version of the Vialit Plate test is used as per Method MB-7 of the TG1 (Asphalt Academy-TG1, 2007)

7.2.8   *PULL -OFF TESTS*

The Shell Bitumen Handbook list two pull-off tests used to asses bitumen adhesion. The first is the Instron pull-off test which uses an Instron Tensile apparatus to extract aggregate test specimens from containers of bitumen under controlled laboratory conditions. The second is the limpet pull-off test that was developed to measure, the bond strength between the aggregate of a chip seal and the base course. The test consists of a 50mm diameter steel plate that is fixed to the road surface and the maximum load to achieve pull off is achieved.

The forum of European National Highway Research Laboratories in the BiTVal Phase 1 Report published in 2004 (Forum of European National Highway Research Laboratories, 2004) notes the pneumatic adhesion tensile testing instrument (PATTI) as a possible adhesion tester. This method was originally developed to test the pull-off strength of a coating on rigid substrates such as metal, concrete or wood as per ASTM D 4541-02 and was used as early as the middle 1990's to evaluate the adhesive loss of bitumen binder-aggregate systems exposed to water (Youtcheff & Aurilio, 1999).

The sketch on the next page shows a schematic cross section of the original PATTI setup consisting of a piston connected to a pull out stub. To perform a test, air pressure is transmitted to the piston which is placed over the pull stub and screwed on the reaction plate. The air pressure induces an airtight seal formed between the piston gasket and the aggregate surface. When the pressure in the piston exceeds the cohesive strength of the binder or the adhesive strength of the binder-aggregate interface, a failure occurs. The pressure at failure is recorded and converted into the pull-off tensile strength (kPa).
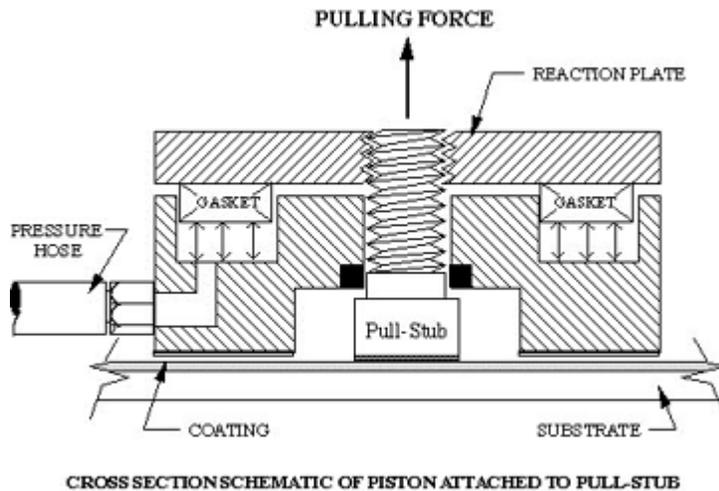
Figure 7-2- Cross section schematic of PATTI piston attached to a pull stub (Youtcheff & Aurilio, 1999)

The BiTVal report notes the following concerns related to the original Patti Test:

"

- *Results could be significantly improved by including a temperature controlling system and a standard methodology for detecting the type of failure of the specimens.*
- *Attention needs to be paid to examining the entire soak-time/pull-off strength curve while evaluating binder.*
- *Little attention is given to the preparation and characterization of substrates other that glass.*
- *Questions concerning the surface chemistry or roughness of substrates remain unanswered. "*

In South Africa two types of pull-out test is specified in the (Asphalt Academy-TG1, 2007)

- Method MB-8: Pull out test method for surfacing aggregate
- Method MB-9: Pliers test for assessment of adhesion properties

Both these tests are extremely complicated and difficult to complete and the results are almost entirely operator dependant. The test has low repeatability and is therefore not often used in practise.

7.3   PROPOSED TEST METHOD

7.3.1   *PATTI TEST*

The bitumen industry first utilized the Pneumatic Adhesive Tensile Testing Instrument, or PATTI, in the late 1990's to evaluate adhesive loss of binder-aggregate systems exposed to moisture conditioning. (Youtcheff & Aurilio, 1999) *&* (Kanitpong & Bahia, 2003) . Initial tests identified several significant effects, including variations in preparing the test assembly (operator dependence), binder film thickness, and curing and testing temperatures.

Recent generations of the PATTI, notably the PATTI Quantum Gold (PQG), address some of these shortcomings while ensuring compliance with surface seal industry requirements (Miller, 2010). Further modifications and the development of a new Bitumen Bond Strength (BBS) Testing procedure (AASHTO TP-91 , 2011) made the testing of the bitumen aggregate bond a reality.

Early generations of the PATTI consisted of a pressure hose, adhesion tester, piston, reaction plate and a metal pull-out stub. *Figure 7-3* below shows a typical assembly.
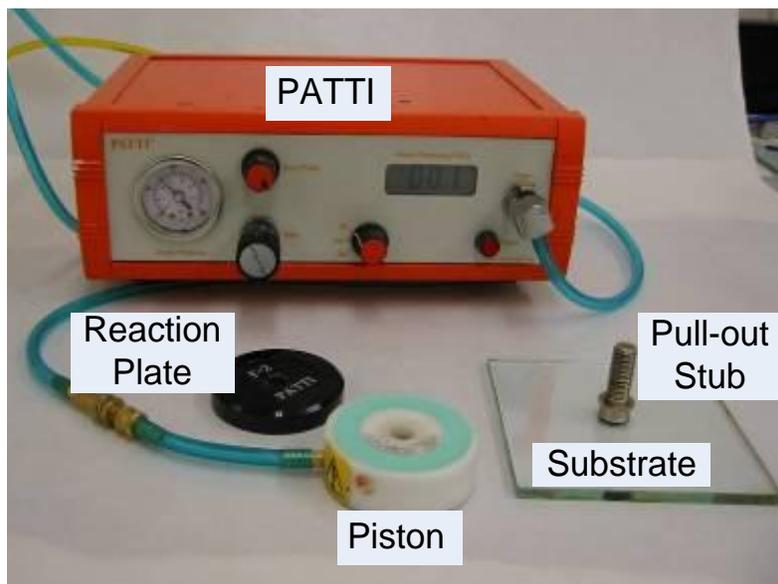
Figure 7-3- Original PATTI assembly.

Stellenbosch University http://scholar.sun.ac.za

The original test procedure consisted of the following steps:

- A hot bitumen sample is applied to a glass substrate and allowed to cure for a fixed time interval.

- A metal pull-out stub is applied to the bitumen sample and allowed to set for a given time interval.

- After placing the piston over the pull-out stub, the reaction plate is fixed to the stub.

- The pressure hose introduces compressed air to the piston, resulting in an upward force on the specimen and eventual failure of the binder. Failure occurs when the applied pressure exceeds the cohesive strength of the binder or the adhesive strength of the binder-aggregate interface.

- The pressure at failure is recorded and the procedure is repeated for other test specimens. (Youtcheff & Aurilio, 1999)

While the original test procedure did yield quantitative information related to bond strength characteristics and failure behavior, research at University of Wisconsin-Madison (UWM) and University of Ancona – Italy (UAI) identified several factors influencing the effectiveness of the test method. Some of these factors include:

- The binder film thickness between the stub and substrate could not be controlled easily.

- Because the original pull-out stub measured only 12.7 mm in diameter, the stub geometry limited the measurement of smaller tensile strengths. Recent modifications to the pull-out stub design at UWM and UAI improved the geometry by nearly doubling the stub diameter.

- The device did not record pressure over time, making the calculation of loading rate difficult. Rather, the PATTI reported only the real-time applied load but not within a computer-based graphical user interface.

- The loading rate varied and could not be set easily. While the PATTI is equipped with a rate control dial, the dial did not effectively control the loading rate or report the real-time loading rate.

- Initial tests were performed on glass substrates, hardly a suitable surface seal material.

7.3.2   *INITIAL TEST METHOD LIMITATIONS & MODIFICATIONS*

The original PATTI test had limitations which were addressed in various ways. Improvements to the pull-out stub design, loading rate control and substrate preparation procedures represented significant advancements from the original PATTI test to its current form as the BBS test method. Binder film thickness is effectively controlled in the BBS test with an improved stub design. Loading rate is effectively controlled with new functions of the PATTI Quantum Gold. Substrate surface characteristics are controlled with improved substrate preparation procedures. Each of these improvements will be discussed in more detail in the sections below.

DEVELOPMENT, TESTING AND EVALUATION OF RESULTS

8   DEVELOPMENT OF THE BITUMEN BOND STRENGTH TEST

8.1   INTRODUCTION

Throughout the literature review the study focused on chip seals, their behaviour and especially the different factors that influence the bitumen binder and aggregate adhesion. The literature review also aimed to motivate the need for an adhesion test that will be capable of measuring the physical tensile strength required to break the bitumen and aggregate bond. The section below will aim to summarise the role that the University of Stellenbosch played in the development of the Bitumen Bond Strength Test procedure, using the PATTI Quantum Gold testing equipment, as well as to practically confirm some of the influences identified during the literature review.

8.2   UNIVERSITY OF STELLENBOSCH INVOLVEMENT

The University of Stellenbosch (US) was requested by the University of Wisconsin Madison (UWM) in 2008 to assist the Asphalt Research Consortium (ARC) with the Federal Highway Administration (FHWA) Seal research project , and in particular to form part of the development of the proposed pull-off test. At this stage the need for an Adhesion Test was already identified and the UWM was in the process to search for suitable equipment. The US brief was to assist the UWM in the following:

- Assist in identifying and evaluating suitable equipment.
- Assist in developing a testing protocol.
- Evaluate the test for practicality and repeatability once complete.
- To assist with inputs gained through experience on chip seals.

During the period from 2008-2010 the US completed these tasks and played an important role in the research and process that led to the final submission and approval of the *Standard Method of Test for Determining Asphalt Binder Bond Strength by Means of the Asphalt Bond Strength (ABS) AASHTO TP 91-2011* as included in *Addendum 1* for information purposes.

This was achieved by the following actions:

- A study tour was undertaken to UWM by Professor Kim Jenkins and the author during August and September 2008. A report was completed by the author as part of this study and it is included as *Addendum 2* for information purposes.

- Following the successful study tour a Research Group was established in January 2009 consisting of representatives from the US and UWM.

- The research group completed parallel literature reviews and tests and combined their knowledge and experiences in monthly conference calls during 2009. The minutes of these conference calls as well as reports completed during this phase are in included as *Addendum 3* for information purposes.

- Mr Bryce Constable completed his Final Year Thesis for completion of his B.Eng. degree in 2009. He completed the majority of the first round testing and was mentored by the Author.

- The Author, with the assistance of Mr Constable, completed a second round of testing to evaluate the Bitumen Bond Strength Test procedure. The results are included as *Addendum 4* for reference.

- Mr Timothy Miller completing his Master Thesis titled *Development of Bond Strength Test for Improved Characterization of Asphalt Emulsions* in 2010. This was based on the work done at the UWM and US.

- The results of Millers' study were accepted for presentation at the *2nd International Spray Sealing Conference in Australia*. In 2010. It was included in the proceeding as follows:

  o *The Development of a Test Method for determining Emulsion Bond Strength using the Bitumen Bond Strength (BBS) Test by Timothy Miller , Andre Greyling , Prof Hussain Bahia & Prof Kim Jenkins*

  o The paper is included as *Addendum 5* for information purposes.

- Further work done by the Author lead to the acceptance of the study as a paper at the *10th Conference on Asphalt Pavements for Southern Africa in 2011* – It was published under the following title:

  o *The Development of a Test Method for determining Emulsion Bond Strength using the Bitumen Bond Strength (BBS) Test- A South African Perspective by Andre Greyling, Timothy Miller, Prof Kim Jenkins & Prof Hussain Bahia*

  o The paper is included as *Addendum 6* for information purposes.

The results of the testing completed during 2009 & 2010, as well as test performed by Mr Miller, forms the basis of the test results discussed in the sections below.

8.3   MODIFICATION TO ORIGINAL PATTI TEST

8.3.1   INTRODUCTION

The previous section touched briefly on the development of the original PATTI test into the formal Bitumen Bond Strength Test (BBS) procedure. To make this possible various shortcomings had to be addressed. The section below will aim to give a brief overview of these actions:

8.3.2   FILM THICKNESS

Previous research completed at the UWM, identified film thickness as a critical parameter in investigating pull-off behavior. (Youtcheff & Aurilio, 1999) (Miller, 2010)**.** Varying the binder film thickness will lead to variation in adhesion strength.   Early experimentation by Youtcheff and Aurilio to evaluate moisture sensitivity utilized glass beads of 200 μm diameter mixed with bituminous binder to control film thickness. This had obvious shortcomings and was only used as a temporary solution.

With input from UAI, Kanitpong and Bahia further modified the pull-out stubs to better control binder film thickness (Canestrari, et al., 2010). They proposed using a smooth-surface aluminum stub and two metal support blocks to replace the glass beads. *Photo 8-1* shows the modified stubs with metal support blocks, and *Photo 8-2* depicts a later iteration of the pull-out stubs with aluminum frame supports.
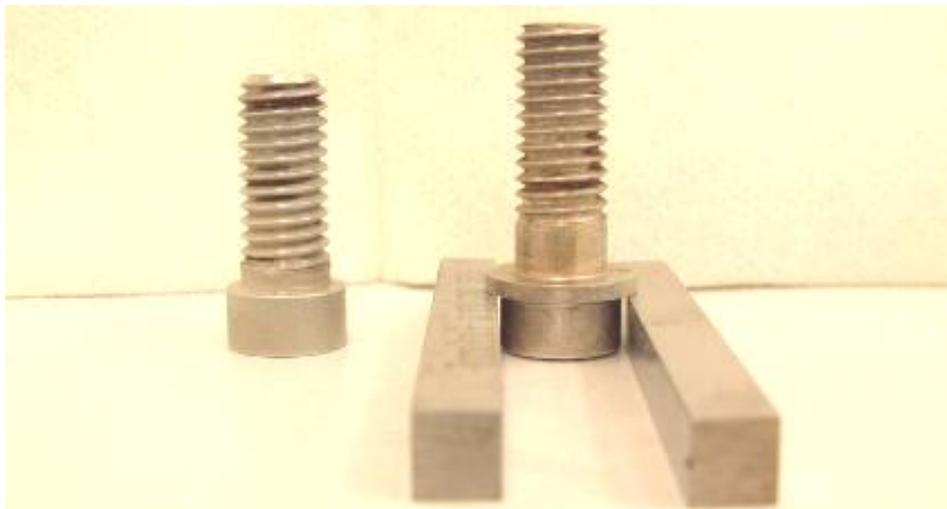
Photo 8-1- Modified pull-out stubs with metal support blocks (Kanitpong & Bahia, 2003)

In early rounds of experimentation, it became clear that film thickness was still not adequately controlled with the pull-out stub support system shown in *Photo 8-2. Photo 8-3* shows an improved pull-out stub designed at UWM in conjunction with UAI.

Improvements to the original stub include an increase in stub diameter to 22 mm to increase the contact area and the addition of circumferential support edges to limit the vertical position of the stub surface and therefore the film thickness to 0.08mm. The Perimetrical channels in the stub edge allow excess binder to flow out from beneath the stub surface.

Photo 8-2- Modified pull-out stub with aluminum frame supports.

Photo 8-3- Modified BBS test pull-out stubs.

8.3.3   LOADING RATE

Bitumen's viscoelastic nature necessitates effective loading rate control for the consistent evaluation of the pull-off tensile strength.   Research by Meng confirmed that load control is critical for consistent pull-off tensile test results (Meng, 2010). Early versions of PATTI were found to inadequately control loading rate so at the beginning of 2009, SEMicro launched the PATTI Quantum Gold© (PQG) test instrument that incorporated user feedback into the revised design, including improved loading rate control. The ability to control the loading rate with a graduated rate control dial further improves consistency. The PQG comes equipped with LabView© software and effectively captures load over time, allowing for calculation of the loading rate.

8.3.4   SUBSTRATE & SURFACE ROUGHNESS

As glass is only relevant as a control subgrade it was necessary to find a way to test the bitumen binder applied directly on aggregate as in practice. This was made possible by the improved substrate preparation procedures that involved the use of aggregate substrates that are actually used in practice. However, crushed aggregates commonly used in surface seals are not suitable substrate materials due to variations in in shape, surface roughness and texture. A procedure developed at UWM to prepare aggregate plates involves cutting large rocks into flat plates (Miller, 2010). The aggregate plates and disks are then lapped with a silicon carbide compound to achieve a consistent surface texture. While aggregate plates and disks may not fully capture aggregate surface characteristics, they represent substantial improvements over glass plates, which are still used as control surfaces. The smooth aggregate surfaces are seen and treated as a worst case scenario.

8.4   TEST APPARATUS AND SETUP

8.4.1   INTRODUCTION

The culmination of the various research and test led to the establishment of the proposed Bitumen Bond Strength Test procedure (AASHTO TP-91 , 2011). The final procedure is attached in *Addendum 1*. The section below will aim to describe the proposed procedure on a step by step basis using the PQG with the modifications discussed in the sections above.

8.4.2   *STEP 1 – AGGREGATE PREPARATION*

The first requirement of the BBS Test is solid and flat aggregate subgrade samples of suitable size to be functional for testing. These samples are prepared by cutting pieces of large rock into flat plate for use as aggregate testing samples. The UWM originally proposed that a lapidary rock saw must be used to cut these aggregate plates. This type of saw is preferred over other, quicker methods, as it produces rock plates with smoother/parallel faces. (Constable, 2009) This is due to a screw-driven mechanism which slowly feeds the rock into the saw blade and can take about 45 minutes to complete one cut. The lapidary rock saw and screw-driven mechanism can be seen in *Photo 8-4 & 8-5* below:

Photo 8-4- Rock Slab Saw (Miller, 2010)

Photo 8-5- Rock Slab Saw and Screw Driven Mechanism. (Miller, 2010)

Due to the specialist nature of this equipment there were unfortunately no rock slab saw available at the University of Stellenbosch or in the greater Cape Town area. Initially rock samples where saw cut using a concrete saw but the aggregate samples were too large and uneven due to the manual feed of these saws. Please note the saw marks on the sawn slabs in *Photo 8.9* below. The problem was solved at the US by sawing smaller geological 'cores' using a standard lab concrete saw. These cores are easier to saw and therefore give more uniform and smooth surface finishes.

8.4.3   *STEP 2- AGGREGATE LAPPING*

Once the cores/plates have been cut it requires a form of smoothing/lapping to ensure a uniform surface roughness over various aggregates. Samples were lapped on both sides using a 280 Grit Silicon Carbide material. Future research should include for the measurement of the surface texture using a laser profilometer. For the sake of this study it was assumed that the smoothed lapped surface where all equal in texture. The lapping machine and grit may be seen in *Photo 8-6* and *Photo 8-7* respectively.

Photo 8-6- Lapping Machine (Constable, 2009)

Photo 8-7- Ceramic Grit 280 (Constable, 2009)

8.4.4   *STEP 3- CLEANING OF AGGREGATE*

Once plates have been lapped, they should be cleaned to remove any fine particles embedded within the plate surface. To clean the samples an ultrasonic cleaner was used. It is specified that the plates should be immersed in distilled/clean water within the ultrasonic cleaner for 60 minutes at a temperature of 60°C. Thereafter the plates can be hand dried. An ultrasonic cleaner is shown in *Photo 8-8* below.

Photo 8-8- Ultrasonic cleaner

8.4.5   *STEP 4 – HEATING OF EMULSION*

Once the rock samples are complete and dried the required emulsion is placed in a plastic or another non-metallic container and preheated in a forced-draft oven to an application temperature of 60°C. The emulsion should be covered to ensure that a dry top layer/skin does not form. The oven should always be pre heated and the sampled should not be heated for longer than one hour to avoid premature breaking.

8.4.6   *STEP 5 HEATING OF AGGREGATE*

At the same time as heating the emulsion, the aggregate samples can be heated in a second forced-draft oven to a desired application temperature of 25°C. A laser thermometer should be used to ensure that the plate surface temperature is heated accurate enough. The required specification is 25°C +/- 2°C. Two rock plates showing saw marks and two cores are shown in *Photo 8-9* below:

Photo 8-9- Granite and Tillite prepared aggregate samples

8.4.7   *STEP 6-EMULSION ON AGGREGATE SAMPLE*

Silicone moulds are pre-prepared and should measure 50mm x 50mm (or at least 50mm in diameter to cover the PATTI base), with a 20mm diameter hole in the centre (for emulsion and stub application) and a thickness of at least 0.8 mm. After preheating the plates and emulsion, the silicone moulds are placed on the aggregate

surface and liquid emulsion is placed into the silicone moulds with the use of a syringe or medicine dropper. The exact amount of emulsion is not critical as the film thickness will be controlled at 0.8mm by the modified stub.
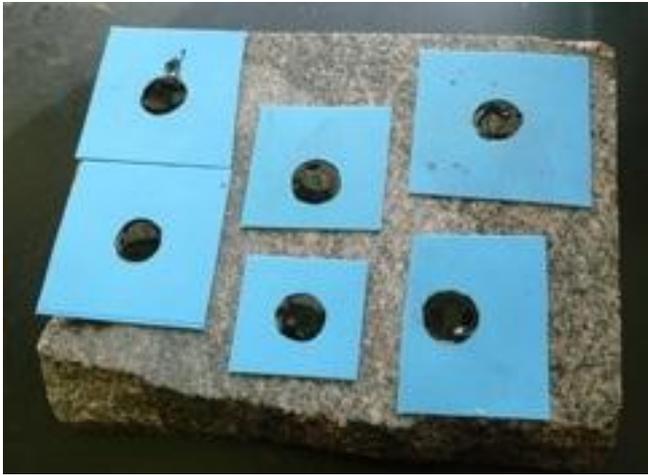
Photo 8-10- Bitumen emulsion poured into silicone moulds

8.4.8   *STEP 7 – CURING OF SPECIMEN*

The aggregate plate/silicone mold/emulsion combination *(Photo 8-10)* is placed into an environmental chamber or forced draft oven and allowed to cure under controlled conditions for specific curing intervals. During this time, the modified stainless steel pull-out stubs are preheated to a temperature of 60°C in a forced-draft oven. These aggregate plate/emulsion samples shall be tested at intervals of 2, 6 and 24 hours.

Photo 8-11- Environmental Chamber

8.4.9 *STEP 8 -APPLYING THE PULL-OUT STUB*

At each interval, 2, 6 and 24 hours, the necessary samples are removed from the forced-draft oven and the silicone molds encircling the emulsion are removed from the aggregate plate. The preheated pull-out stubs are then firmly placed on the cured emulsion ensuring that all four rims of the stub are touching the plate surface. Please note the gaps in the edges allowing excess binder to escape. The modified pull-out stubs, as described previously, can be seen in *Photo 8-3* above:

8.4.10 *STEP 9 -SAMPLE PREPARATION*

Once the pull-out stubs have been applied, the aggregate plate/emulsion/stub assembly is placed back into a forced-draft oven at a temperature of 25°C for approximately one and a half hours. This additional time allows the closed sample to acclimate to testing conditions. One can see the aggregate plate/emulsion/stub assembly in *Photo 8.12* below;

Photo 8-12- Core Aggregate samples in Forced Draft Oven (Constable, 2009)

8.4.11 *STEP 10 -TESTING*

After 1 hour the samples are removed from the oven and the excess emulsion on the plate surface is removed with a flat knife so that the emulsion along the edges of the stub does not influence the testing results. The PATTI Quantum Gold device is then used to test these samples. Small metal supports are used as a base for the pneumatic pressure ring around the stub. Special care should be taken to not disturb emulsion/stub bond the while affixing the pneumatic pull of ring.

The photo below shows the PATTI Quantum Gold device set up and ready for a test:

Photo 8-13- PATTI Quantum Gold with testing assembly (Constable, 2009)

The PATTI unit is linked to a computer via a data cable where it is connected with LabView Software© The results are displayed visually and the data is stored to a text file. Compressed air or CO_2 canisters are used to deliver the required pressure force. A gauge on the front of the equipment indicates the supply pressure. The loading rate can be adjusted using a dial mounted on top of the testing unit. A top view of the PATTI unit and loading rate dial can be seen in the two photos below:

Photo 8-14- Top View of PATTI Quantum Gold

AH GREYLING

December 2012

Photo 8-15- Loading Rate dial

The PATTI Quantum Gold software package allows the user to select various stub designs and other testing parameters. A real-time display of the test (time vs. tensile strength), compliance with ASTM standards and deliverance of spread sheet outputs are all featured options. (Constable, 2009) A screen view of the software interface can be seen in *Figure 8-1*. The test results as well as the screenshot report are saved and can be accessed at any time for further calculations and record purposes
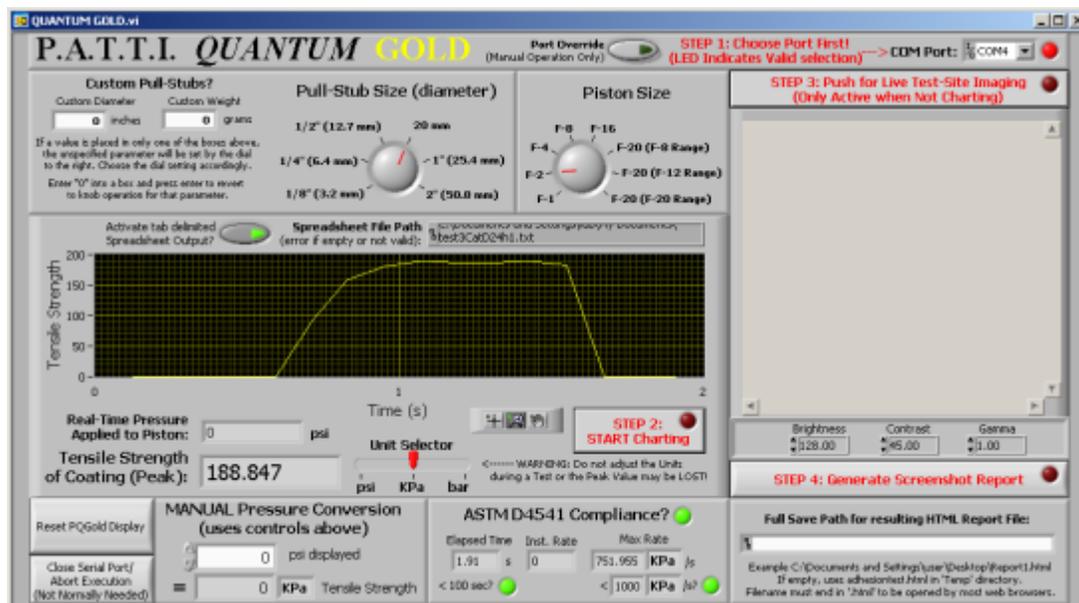
Figure 8-1- PATTI Quantum Gold user interface

8.5 GENERAL COMMENTS

As part of the evaluation of the testing procedure the following valuable lessons were learned and the following advice and comments should be noted:

- Plates have to be perfectly smooth and flat otherwise emulsion soaked underneath the silicone moulds.

- Not all newly manufactured stubs had smooth threads and this required some manual force to screw in the pressure ring. This led to stubs losing adhesion before the tests could take place. Special care should be taken that the pressure ring and the pull out stub screw in perfectly.

- The loading rate dial was difficult to control and very sensitive to adjustment. It was later discovered that the dial of the PQG used at the US was malfunctioning. After much trial and error it was possible to achieve application rates of between 500 – 1000 kPa/s.

- Without a rock plate saw it is better to use geological cores and saw them into disks.

9   EXPERIMENTAL SETUP

9.1   *INTRODUCTION*

Over 200 tests where completed over a two year period at the University of Stellenbosch. Mr Bryce Constable completed a first set of test in 2009 for the completion of his thesis. These tests were done at a curing temperature of 25°C and not at 30°C as the formal procedure was not finalised yet. A second more detailed test set was completed in 2010 at 30°C by Mr Constable under supervision of the Author. This made it possible to correlate the results for the US with the UWM.

The section below will discuss these experimental setups and results in more detail.

9.2   *MATERIALS*

9.2.1   *EMULSIONS*

The following four emulsion types where evaluated during the testing phase:

1. Cationic Rapid Set Emulsion with 65% Bitumen content (CRS65)
2. Anionic Slow Set Emulsion with 60% Bitumen content (SS60)
3. Cationic Rapid Set Emulsion with 65% Bitumen and Modified with 3% Latex (CRS65+3%)
4. Anionic Rapid Slow Set Emulsion with 60% Bitumen and Modified with 3% Latex (SS60+3%)

This combination of emulsion made it possible to evaluate Anionic vs. Cationic as well as Modified vs. Unmodified emulsion adhesion development. The Emulsion used where supplied courtesy of COLAS and TOSAS South Africa.

9.2.2   *AGGREGATES*

Two sets of aggregate samples from quarries previously used for chip seal construction where used for testing. Granite and Tillite aggregate cores where supplied by the South African National Road Agency –Western Region. Granite is an Acidic Igneous Rock and Tillite is a slightly less Acidic but slightly more porous Sedimentary rock type.

9.3 EXPECTED RESULTS

Based on the theoretical information gathered in the literature review section the following test results can be expected:

1. Adhesion will increase with curing time for all combinations of variables.
2. Cationic Emulsion will develop stronger adhesion than Anionic emulsion due to the fact that the positive charged cationic emulsion forms stronger bonds with the negatively charged acidic rock types used.
3. The stable grade Anionic Emulsion will show slower adhesion development as the stable grade emulsion is expected to have a longer breaking process.
4. The rapid setting Cationic Emulsion will develop strength earlier than the Anionic Emulsion as it is expected from a rapid setting emulsion.
5. The modification of the Emulsion with Latex should lead to increased adhesion as it is expected from modified binders.
6. Granite is slightly more acidic and coarser than tillite and will therefore develop stronger adhesion with the cationic emulsions.
7. Tillite is slightly less acidic and more porous that granite and should therefore develop stronger adhesion with the anionic emulsion than with granite.

If the BBS test procedure is suitable for testing adhesion development it will confirm the expected results above.

9.4 EXPERIMENTAL SETUP

As part of the test procedure evaluation a total of 198 tests where completed during eleven test sets shown in *Table 9-1* below. Each test set was prepared to consist of the following combinations:

- One emulsion type , either CRS65 , CRS65+3% Latex, SS60 or SS60+3% Latex
- Two aggregate types, Granite of Tillite
- Three curing times , 2,6 and 24 hours
- At least three stubs for each combination. (In most cases four to five stubs was used to ensure that the test yielded three valid results per combination.)

During the different test sets the curing temperature as well as the pull of rate was varied to evaluate the influence of the variability. The results of the various combinations is shown and discussed in the section below.

Table 9-1- Experimental Setup and Test Sets completed.

Note	Tests Set Number	Emulsion	Aggregate	Curing Time (Hours)	Curing Temp	Pull Off Rate (kPa)
5 SETS of CRS65	1	CRS65	Granite	2,6& 24 hours	25°C	300
	2	CRS65	Granite	2,6& 24 hours	25°C	300
	3	CRS65	Granite	2,6& 24 hours	30°C	700
	4	CRS65	Granite	2,6& 24 hours	30°C	800
	5	CRS65	Granite	2,6& 24 hours	30°C	900
	1	CRS65	Tillite	2,6& 24 hours	25°C	300
	2	CRS65	Tillite	2,6& 24 hours	25°C	300
	3	CRS65	Tillite	2,6& 24 hours	30°C	700
	4	CRS65	Tillite	2,6& 24 hours	30°C	800
	5	CRS65	Tillite	2,6& 24 hours	30°C	900
2 SETS of CRS65+3 % Latex	6	CRS65+3%Latex	Granite	2,6& 24 hours	25°C	400
	7	CRS65+3%Latex	Granite	2,6& 24 hours	30°C	950
	6	CRS65+3%Latex	Tillite	2,6& 24 hours	25°C	400
	7	CRS65+3%Latex	Tillite	2,6& 24 hours	30°C	950
2 SETS of SS60	8	SS60	Granite	2,6& 24 hours	30°C	900
	9	SS60	Granite	2,6& 24 hours	30°C	700
	8	SS60	Tillite	2,6& 24 hours	30°C	900
	9	SS60	Tillite	2,6& 24 hours	30°C	700
2 SETS of SS60+3% Latex	10	SS60+3%Latex	Granite	2,6& 24 hours	30°C	800
	11	SS60+3%Latex	Granite	2,6& 24 hours	30°C	700
	10	SS60+3%Latex	Tillite	2,6& 24 hours	30°C	800
	11	SS60+3%Latex	Tillite	2,6& 24 hours	30°C	700

9.5 TEST RESULTS- EMULSIONS

9.5.1 CATIONIC 65% EMULSION (CRS65)

The results for the first five sets *(Set 1-5)* of the Cationic Rapid Set Emulsions that were tested are shown in *Figure 9-1* below. Please note that the average values of at least three results per set were used for graphical representation purposes:
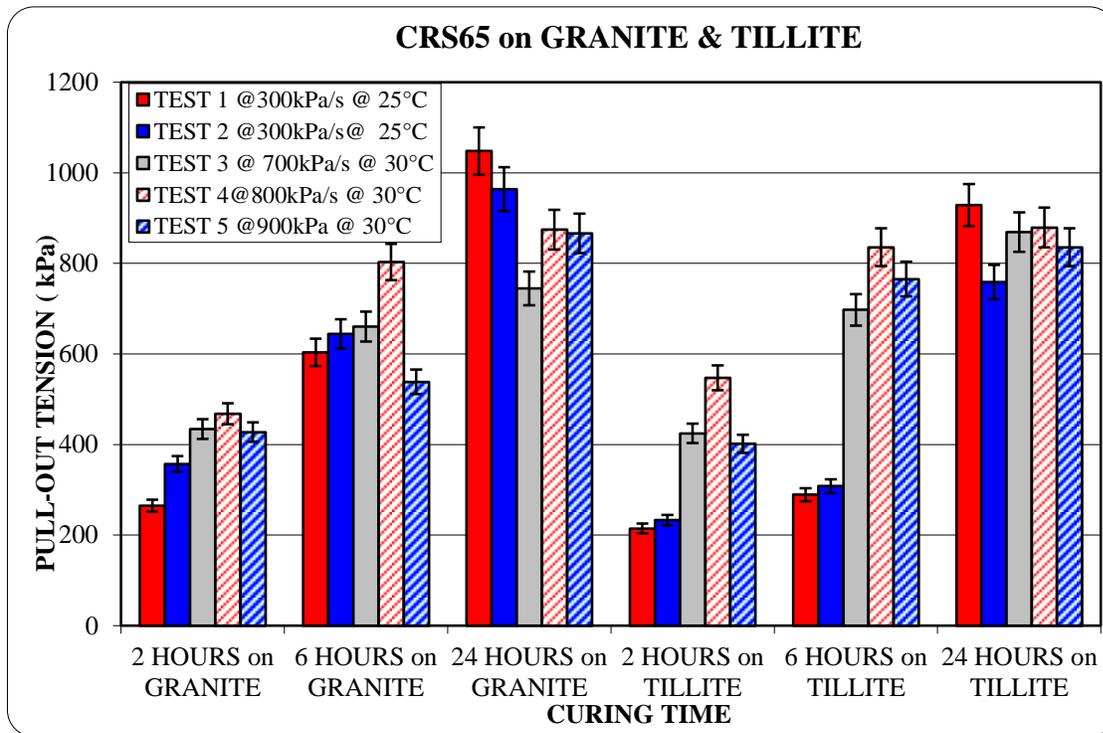
Figure 9-1- CRS65 Test Results on Granite & Tillite

The following information can be deduced from *Figure 9-1* above:

- Adhesion increased with time based on the higher results achieved after 2, 6 and 24 hours for both granite and tillite.

- The results after 2 and 6 hours shows lower results for the two sets cured at 25°C and tested at 300kPa/s on both granite and tillite

- The results after 24 hours shows higher results at the 25°C curing temperature and 300kPa/s pull of rate for the granite but results on tillite show limited variation.

The influences of the various factors will be discussed in consequent sections. *Table 9-2* below shows a statistical analysis of the results of test Set 5. The first section shows the pull of rate in kPa/s and the second section shows the results. Note the Covariance values.

Positive Covariance values indicates that higher than average values of one variable tend to be paired with higher than average values of the other variable. Negative Covariance indicates that higher than average values of one variable tend to be paired with lower than average values of the other variable. Zero Covariance indicates that two random variables are independent. However, a covariance of zero does not necessarily mean that the variables are independent. A nonlinear relationship can exist that still will result in a covariance value of zero. Covariance values of less than 10% usually indicate good repeatability.

Table 9-2- Covariance Analysis of CRS Test Set 5 on Granite

CRS65 on GRANITE TEST SET 5							
Granite		**Rep**	**Rate (kPa/s)**		**St Dev.**	**St Error**	**COV**
CRS65	2H	1	869.92	867.67	6.33	3.66	0.73%
		2	860.52				
		3	872.57				
CRS65	6H	1	1092.62	1125.19	28.97	16.73	2.57%
		2	1134.89				
		3	1148.07				
CRS65	24H	1	909.09	930.80	48.05	27.74	5.16%
		2	985.87				
		3	897.44				
Granite		**Rep**	**Tension (kPa)**		**StDev**	**St Error**	**COV**
CRS65	2H	1	427.33	424.62	4.69	2.71	1.11%
		2	427.33				
		3	419.20				
CRS65	6H	1	511.34	533.02	19.54	11.28	3.67%
		2	549.29				
		3	538.45				
CRS65	24H	1	866.36	919.66	46.65	26.93	5.07%
		2	939.53				
		3	953.08				

Table 9-3- Covariance Analysis of CRS Test Set 5 on Tillite

CRS65 on TILLITE TEST SET 5							
Tillite		**Rep**	**Rate (kPa/s)**		**St Dev**	**St Error**	**COV**
CRS65	2H	1	855.19	830.52	22.84	13.18	2.75%
		2	810.13				
		3	826.24				
CRS65	6H	1	925.23	973.35	46.00	26.56	4.73%
		2	977.95				
		3	1016.87				
CRS65	24H	1	860.75	918.46	50.15	28.95	5.46%
		2	951.41				
		3	943.21				
Tillite		**Rep**	**Tension (kPa)**		**StDev**	**St Error**	**COV**
CRS65	2H	1	400.23	406.55	15.88	9.17	3.91%
		2	394.81				
		3	424.62				
CRS65	6H	1	766.09	765.19	77.24	44.60	10.09%
		2	687.50				
		3	841.97				
CRS65	24H	1	820.29	835.65	57.13	32.98	6.84%
		2	787.77				
		3	898.88				

9.5.2 *ANIONIC 60% EMULSION (SS60)*

The average results for the three samples of each of the two test sets *(Set 8&9)* completed using Stable Grade Emulsion is shown in *Figure 9-2* below:
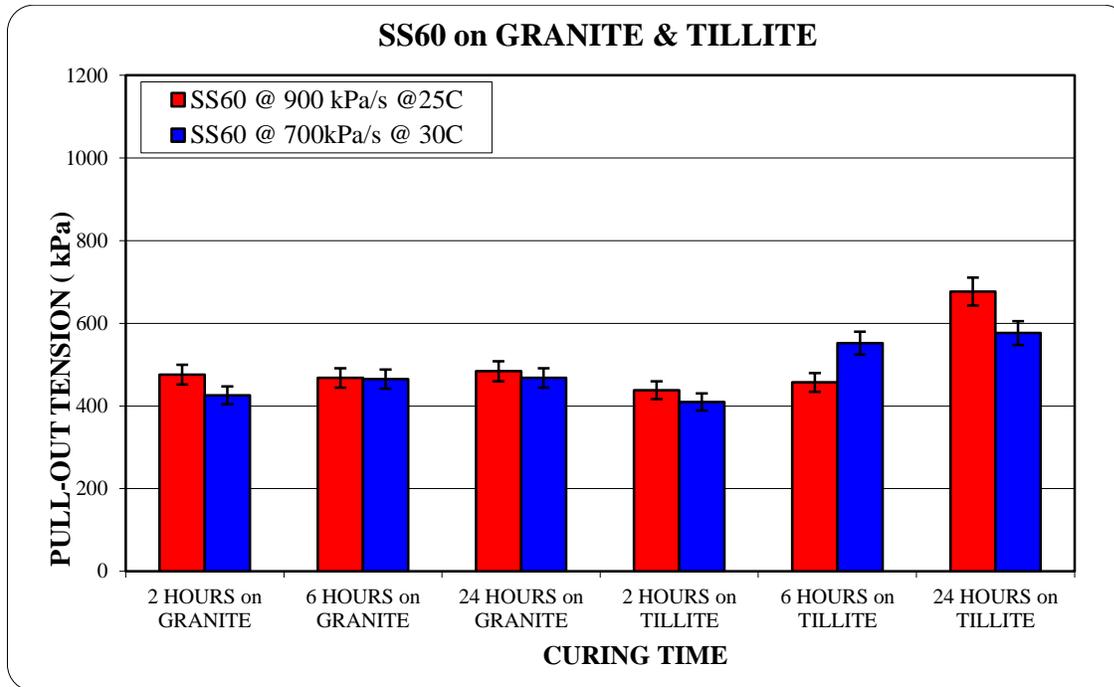

Figure 9-2- SS60 Test Results on Granite & Tillite

The following information can be deduced from the figure above:

- No adhesion increase over time can be seen for the SS60/Granite combination.
- Limited adhesion increase over time can be seen for the SS60/Tillite combination.
- Higher pull off rate at lower temperatures deliver slightly higher results and this indicate that the test results are more susceptible to pull off rate than curing temperature.

Please note the detailed results of test Set 8 completed in 2009 below. It is interesting to note that the tables show that the majority of the COV values are smaller than 10% in the 2 and 6 hour ranges. For the longer (24 hour) curing temperature the COV shows a much higher variability in the bond strength (tension) results.

Table 9-3- Covariance Analysis of SS60 Test Set 8 on Granite

SS60 on GRANITE- TEST SET 8 (2009)						
Granite		**Rep**	**Rate (kPa/s)**	**St Dev**	**St Error**	**COV**
SS60	2H	1	839.45	20.75	11.98	2.53%
		2	823.72			
		3	798.32			
SS60	6H	1	784.61	7.70	4.44	0.98%
		2	789.18			
		3	774.16			
SS60	24H	1	696.53	29.18	16.85	4.23%
		2	658.42			
		3	715.76			
Granite		**Rep**	**Tension (kPa)**	**StDev**	**St Error**	**COV**
SS60	2H	1	497.79	13.37	7.72	2.77%
		2	476.11			
		3	473.40			
SS60	6H	1	470.69	5.64	3.26	1.21%
		2	467.98			
		3	459.85			
SS60	24H	1	495.08	106.95	61.75	19.60%
		2	473.40			
		3	668.53			

Table 9-4- Covariance Analysis of SS60 Test Set 8 on Tillite

SS60 on TILLITE- TEST SET 8 (2009)						
Tillite		**Rep**	**Rate (kPa/s)**	**St Dev**	**St Error**	**COV**
SS60	2H	1	947.85	33.32	19.24	3.54%
		2	905.31			
		3	971.01			
SS60	6H	1	770.90	7.68	4.43	1.01%
		2	760.48			
		3	755.91			
SS60	24H	1	702.66	86.13	49.73	11.53%
		2	692.34			
		3	846.41			
Tillite		**Rep**	**Tension (kPa)**	**StDev**	**St Error**	**COV**
SS60	2H	1	443.59	8.28	4.78	1.86%
		2	438.17			
		3	454.43			
SS60	6H	1	457.14	9.77	5.64	2.12%
		2	451.72			
		3	470.69			
SS60	24H	1	524.89	51.49	29.73	9.06%
		2	625.17			
		3	554.71			

9.5.3   *CATIONIC 65% +3% LATEX MODIFIED EMULSION*

The average results for the three samples of each of the two test sets (*Set 6&7)* completed using Cationic Emulsion Modified with 3% Latex shown in *Figure 9-3* below:
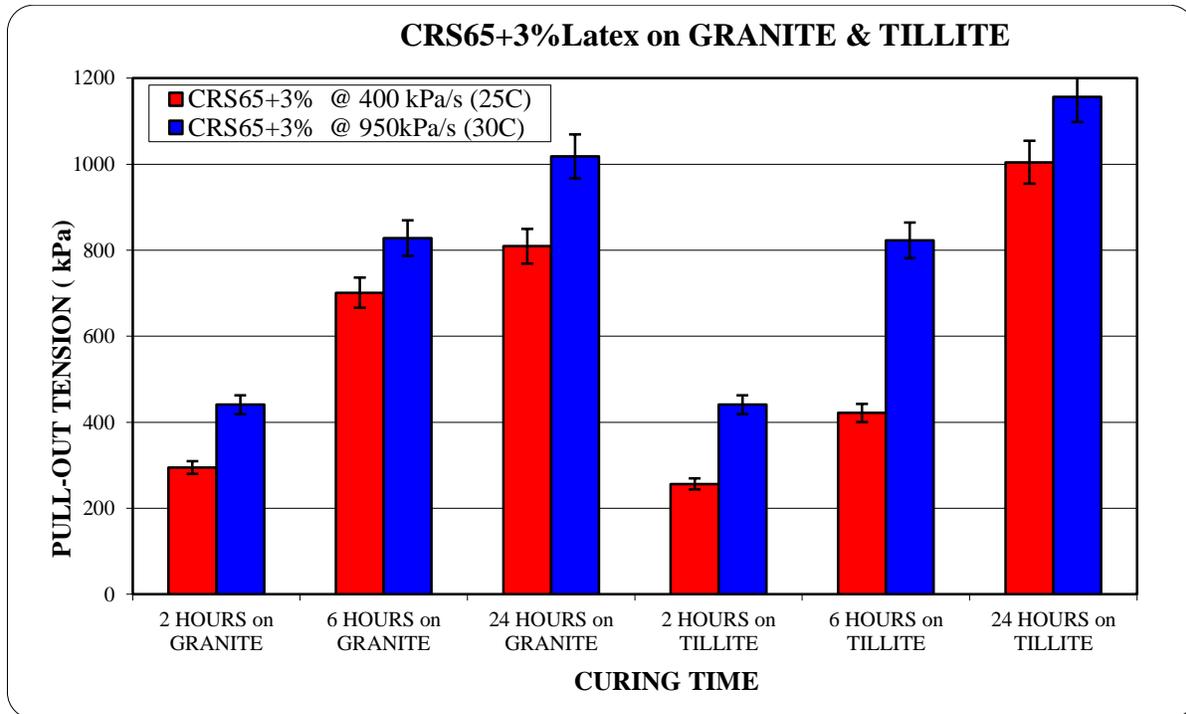

Figure 9-3- CRS65+3% Latex - Test Results on Granite & Tillite

The following information can be deduced from the figure above:

- A clear adhesion increase over time can be seen for the CRS65+3% Latex /Granite combination.
- A clear adhesion increase over time can be seen for the CRS65+3% Latex /Tillite combination.
- Higher pull off rate at higher temperatures deliver slightly higher results.
- It again seems that the test results are more susceptible to the pull off rate than curing temperature.
- There is little difference in adhesion development between Granite and Tillite with Tillite showing marginally higher results after 24 hours.

Please note the detailed results of test Set 7 completed in 2010 below. Please again note the higher Variance in both rate of applied pull off tension as well as the results after 24 hours.

Table 9-5- Covariance Analysis of CRS65+3% Latex Test Set 7 on Granite

CRS65+3% LATEX on GRANITE – TEST SET 7						
Granite		Rep	Rate (kPa/s)	St Dev	St Error	COV
CRS65+3% LATEX	2H	1	617.82	68.30	39.43	9.80%
		2	738.91			
		3	733.10			
CRS65+3% LATEX	6H	1	1010.33	73.82	42.62	7.75%
		2	977.60			
		3	869.28			
CRS65+3% LATEX	24H	1	974.32	15.06	8.69	1.57%
		2	944.46			
		3	962.75			
Granite		Rep	Tension (kPa)	StDev	St Error	COV
CRS65+3% LATEX	2H	1	443.59	4.14	2.39	0.94%
		2	438.17			
		3	435.46			
CRS65+3% LATEX	6H	1	836.55	13.91	8.03	1.69%
		2	809.45			
		3	828.42			
CRS65+3% LATEX	24H	1	806.74	169.88	98.08	17.17%
		2	1018.13			
		3	1142.79			

Note: The "Rate (kPa/s)" and "Tension (kPa)" mean values appear centered in the 2H/6H/24H groups: 696.61, 952.40, 960.51 (Rate) and 439.08, 824.81, 989.22 (Tension).

Table 9-6- Covariance Analysis of CRS65+3% Latex Test Set 7 on Tillite

CRS65+3% LATEX on TILLITE-– TEST SET 7						
Tillite		Rep	Rate (kPa/s)	St Dev	St Error	COV
CRS65+3% LATEX	2H	1	957.38	40.16	23.19	4.25%
		2	899.60			
		3	976.80			
CRS65+3% LATEX	6H	1	967.78	17.69	10.21	1.82%
		2	993.96			
		3	960.28			
CRS65+3% LATEX	24H	1	742.96	79.90	46.13	9.57%
		2	881.36			
		3	881.36			
Tillite		Rep	Tension (kPa)	StDev	St Error	COV
CRS65+3% LATEX	2H	1	449.01	12.42	7.17	2.78%
		2	432.75			
		3	457.14			
CRS65+3% LATEX	6H	1	801.32	60.44	34.89	7.14%
		2	823.00			
		3	915.14			
CRS65+3% LATEX	24H	1	974.77	104.83	60.52	9.57%
		2	1156.34			
		3	1156.34			

Note: The "Rate (kPa/s)" and "Tension (kPa)" mean values appear centered in the 2H/6H/24H groups: 944.59, 974.01, 835.22 (Rate) and 446.30, 846.49, 1095.81 (Tension).

9.5.4   *ANIONIC% +3% LATEX MODIFIED EMULSION*

The average results for the three samples of each of the two test sets *(Set 10&11)* completed using Anionic Slow Set Emulsion Modified with 3% Latex shown in *Figure 9-4* below:
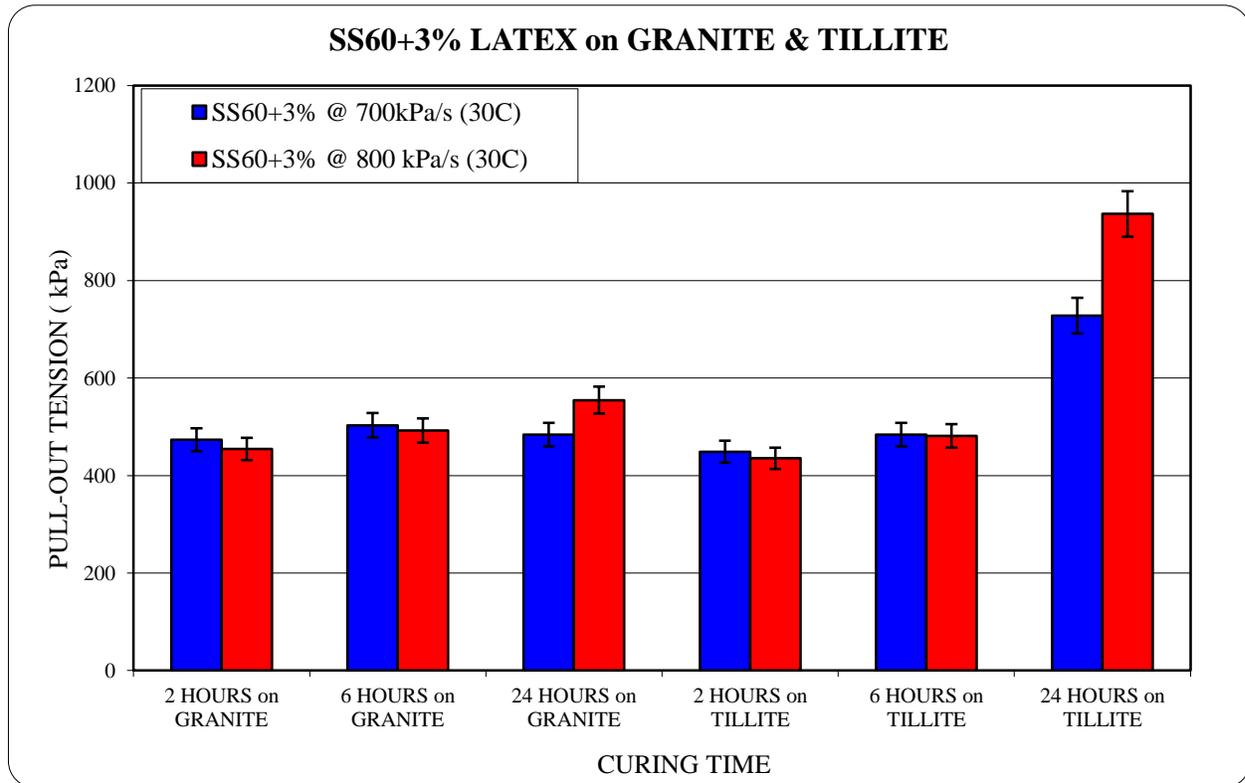

Figure 9-4- SS60+3% Latex - Test Results on Granite & Tillite

The following information can be deduced from the figure above:

- There is no clear increase in adhesion development of the first 24 hours with the SS60+3% Latex /Granite combination. This is expected from a Slow Setting Anionic Emulsion on an Acidic aggregate.

- The SS60+3% Latex /Tillite combination show limited initial adhesion but increased adhesion after 24 hours. This can be attributed to the increased porosity of the sedimentary tillite aggregate.

- There is limited sensitivity between a pull off rate of 700kPa/s and 800kPa/s.

- The figure also shows the need for the investigation of longer curing times.

Please note the detailed results of test Set 10 completed in 2010 on the next page.

Table 9-7- Covariance Analysis of SS60+3% Latex Test Set 10 on Granite

SS60+3% LATEX on GRANITE-TEST SET 10							
Granite		**Rep**	**Rate (kPa/s)**		**St Dev**	**St Error**	**COV**
SS60+3% LATEX	2H	1	665.96	665.78	7.71	4.45	1.16%
		2	673.40				
		3	657.98				
SS60+3% LATEX	6H	1	627.38	609.93	16.47	9.51	2.70%
		2	607.75				
		3	594.65				
SS60+3% LATEX	24H	1	1015.17	993.63	25.67	14.82	2.58%
		2	1000.50				
		3	965.22				
Granite		**Rep**	**Tension (kPa)**		**StDev**	**St Error**	**COV**
SS60+3% LATEX	2H	1	478.82	472.50	6.82	3.94	1.44%
		2	473.40				
		3	465.27				
SS60+3% LATEX	6H	1	519.47	505.92	12.42	7.17	2.45%
		2	503.21				
		3	495.08				
SS60+3% LATEX	24H	1	476.11	477.92	5.64	3.26	1.18%
		2	484.24				
		3	473.40				

Table 9-8- Covariance Analysis of SS60+3% Latex Test Set 10 on Tillite

SS60+3% LATEX on TILLITE-TEST SET 10							
Tillite		**Rep**	**Rate (kPa/s)**		**St Dev**	**St Error**	**COV**
SS60+3% LATEX	2H	1	755.91	750.67	4.54	2.62	0.61%
		2	748.05				
		3	748.05				
SS60+3% LATEX	6H	1	816.60	807.01	9.17	5.30	1.14%
		2	806.10				
		3	798.32				
SS60+3% LATEX	24H	1	941.11	965.36	23.76	13.72	2.46%
		2	966.38				
		3	988.60				
Tillite		**Rep**	**Tension (kPa)**		**StDev**	**St Error**	**COV**
SS60+3% LATEX	2H	1	457.14	449.92	6.82	3.94	1.52%
		2	449.01				
		3	443.59				
SS60+3% LATEX	6H	1	486.95	484.24	2.71	1.56	0.56%
		2	484.24				
		3	481.53				
SS60+3% LATEX	24H	1	676.66	717.31	52.76	30.46	7.36%
		2	776.93				
		3	698.34				

9.6 CONFIRMING SIGNIFICANT FACTORS INFLUENCING BOND STRENGTH DEVELOPMENT

Miller did some sterling work investigated factors critical to bond strength development (Miller, 2010). The factors investigated included various substrate types, moisture condition, surface roughness, loading rate, curing temperature and curing humidity. An analysis of variance (ANOVA) for his screening experiments is shown in *Table 9-9* below.

His experiments, completed with a combination of data form UWM and the US identified that the loading rate, the curing temperature and humidity, and the substrate type has significant influence on the bond strength development. The results indicate that the loading rate is the most significantly influence on the pull-out tension response. Other factors investigated included substrate and binder type as well as curing variables related to temperature, humidity and time (Miller, 2010).

Table 9-9- Analysis of variance for the factor screening experiment. (Miller, 2010)

Source	DF	Seq. SS	Adj. SS	Adj. MS	F	P
Substrate	1	1051.6	1051.6	1051.6	8.2	0.006
Moisture	1	0.5	0.5	0.5	0.00	0.952
Roughness	1	90.9	90.9	90.9	0.7	0.404
Loading Rate	**1**	**31926.2**	**31926.2**	**31926.2**	**248.5**	**0.000**
Temperature	1	1967.0	1967.0	1967.0	15.3	0.000
Humidity	1	1105.3	1105.3	1105.3	8.6	0.005
Error	57	7322.4	7322.4	128.5		
Total	63	43463.8				
S = 11.3342		R-Sq = 83.15%		R-Sq (adj) = 81.38%		

9.6.1 *LOADING RATE EXPERIMENT*

Loading rate was further identified as a factor that significantly influencing the pull-out tension response. This is primarily due to the visco-elastic properties of bitumen discussed previously. Miller did extensive testing and proved that a power law model adequately captures the relationship between loading rate and pull-out tension. The results in *Figure 9-5* show that loading rates between 690-1030 kPa/s appear to exhibit a linear relationship above pull-out tension values of 690 kPa.

Loading rates exceeding 2700 kPa/s lead to increasing variability in both the pull-out tension values and loading rate. Therefore, loading rates exceeding 2700 kPa/s should be avoided to minimize experimental error in order to obtain valid results.
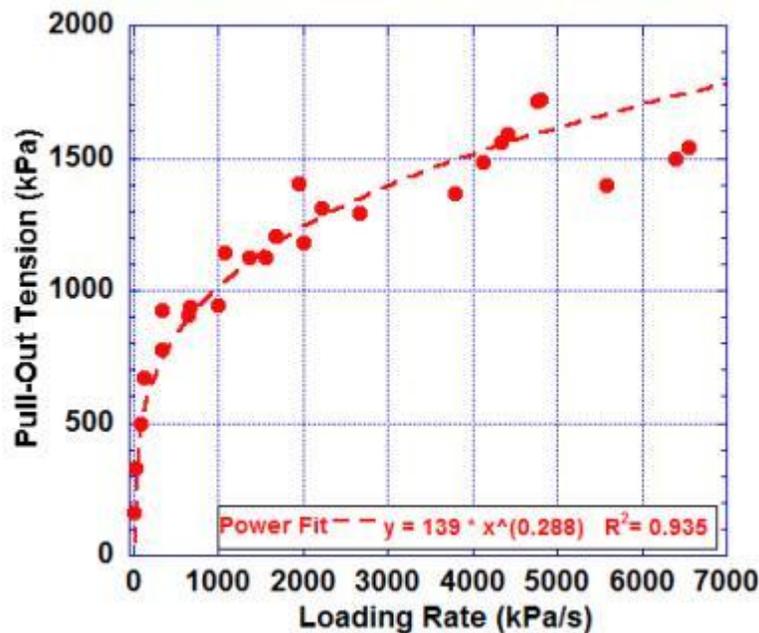

Figure 9-5- Loading rate and pull-out tension are described by a power law model. (Miller, 2010)

Some of the test done by the US confirmed these results. *Figure 9-6* shows the influence of loading rates on two different aggregates and one emulsion combinations. The figure clearly indicates that there is an increase in the pull out tension as the rate of application increases. The results are however limited to the 400 kPa/s to 950 kPa/s range.
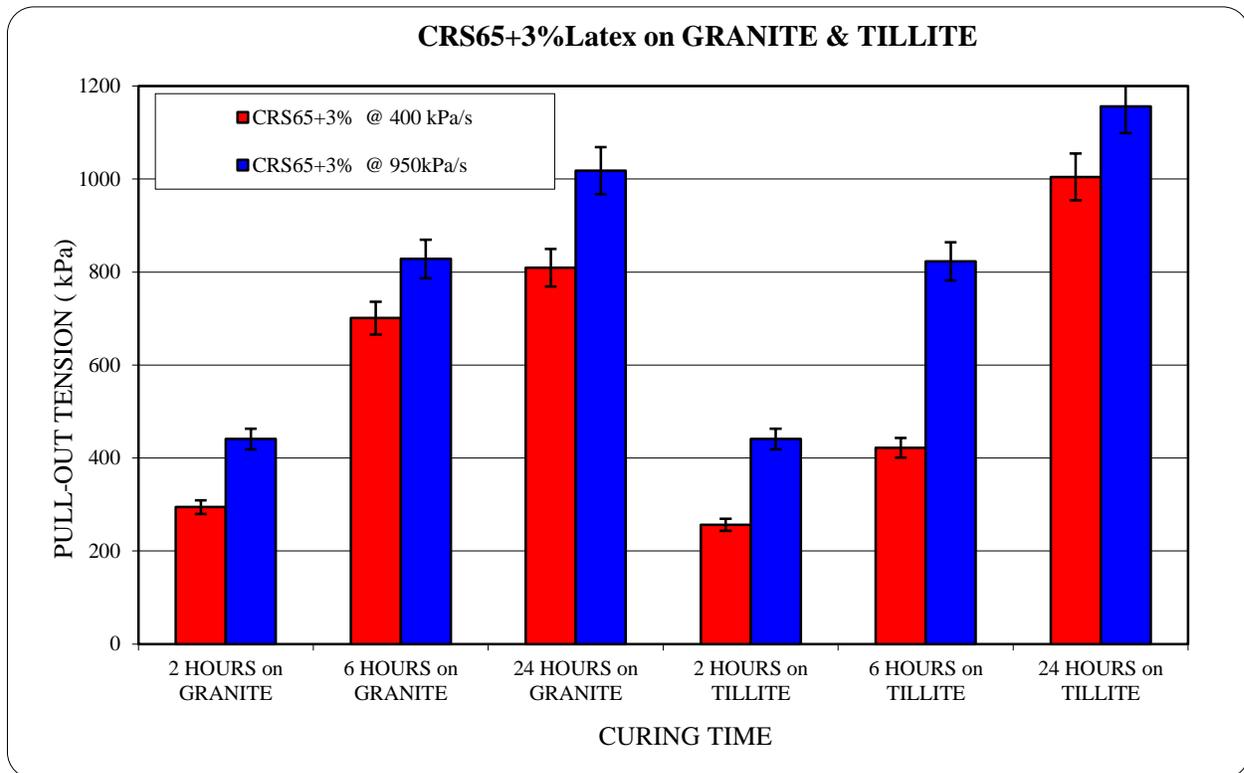
Figure 9-6- Loading rate and pull-out tension results US

9.6.2 *CURING CONDITIONS EXPERIMENT*

It is expected that the effect of curing temperature and humidity will play a very important role in the bond strength development between bitumen and aggregate. Miller investigated the effects of curing temperature and humidity on pull-out tension (Miller, 2010) and confirmed the influences.

In his experiment, the loading rate was fixed at approximately 700 kPa/s for four curing conditions. Samples were cured in an environmental chamber at the prescribed curing intervals of 2, 6 and 24 hours. The experiment used a cationic rapid-setting emulsion with high viscosity (CRS-2), granite and limestone substrates, and the following curing conditions:

- Samples cured at 35 °C and 30 percent relative humidity.
- Samples cured at 35 °C and 70 percent relative humidity.
- Samples cured at 15 °C and 30 percent relative humidity.
- Samples cured at 15 °C and 70 percent relative humidity.

An ANOVA for the curing conditions experiment, shown in *Table 9-10*, indicates that aggregate type, curing temperature, and curing interval are statistically significant main effects at a 95% confidence level, while temperature-curing interval and humidity-curing interval interactive effects are also statistically significant at this confidence level. Other important results suggest that:

- Significant strength gains were observed between two and six hours at the 35 °C temperature level.
- Granite outperformed limestone in three of four curing conditions after six hours.
- **Humidity did not significantly affect the pull-out tension response.**
- Samples tested at 35 °C and 30 percent relative humidity performed better than samples tested at other curing conditions.
- Samples exhibited only slight differences in the pull-out tension response after 24 hours of curing.

Table 9-10-Analysis of variance for the curing conditions experiment. (Miller, 2010)

Source	DF	Seq. SS	Adj. SS	Adj. MS	F	P
Substrate	2	2862.9	2862.9	1431.4	16.44	0.000
Curing Temperature (°C)	1	2079.2	2079.2	2079.2	23.88	0.000
Curing Humidity (% RH)	1	60.6	60.6	60.6	0.70	0.417
Curing Interval	2	113689.5	113689.5	56844.8	652.96	0.000
Substrate-Temperature	2	247.0	247.0	123.5	1.42	0.271
Substrate-Humidity	2	559.2	559.2	279.6	3.21	0.067
Substrate-Interval	4	838.9	838.9	209.7	2.41	0.092
Temperature-Humidity	1	54.4	54.4	54.4	0.63	0.441
Temperature-Interval	2	3695.7	3695.7	1847.8	21.23	0.000
Humidity-Interval	2	1354.8	1354.8	677.4	7.78	0.004
Error	16	1392.9	1392.9	87.1		
Total	35	126835.1				
S = 9.33046		**R-Sq = 98.9%**		**R-Sq (adj) = 97.6%**		

9.6.3 *EMULSION TYPE EXPERIMENT*

Miller also developed a detailed materials experiment to investigate a variety of emulsion and substrate combinations (Miller, 2010) The experiment included five emulsion types and three substrate types at four curing intervals. *Figure 9-7* and *Figure 9-8* show results from this experiment.

Two initial observations can be made in *Figure 9-7*: that both cationic rapid-setting emulsions (CRS-2 lab and CRS-2 field) perform better than polymer modified cationic rapid-setting emulsions (CRS-2P) and high-float anionic rapid setting (HFRS-2) emulsions; and that all emulsion types exhibit sharp increases in pull-out tension initially, with relative gains in tensile strength diminishing over time. A power law model adequately characterizes the relationship between curing interval and pull-out tension. In *Figure 9-8*, glass plates yield a near-perfect correlation using a power law model, with solid aggregate plates and chip substrates also yielding very strong relationships.
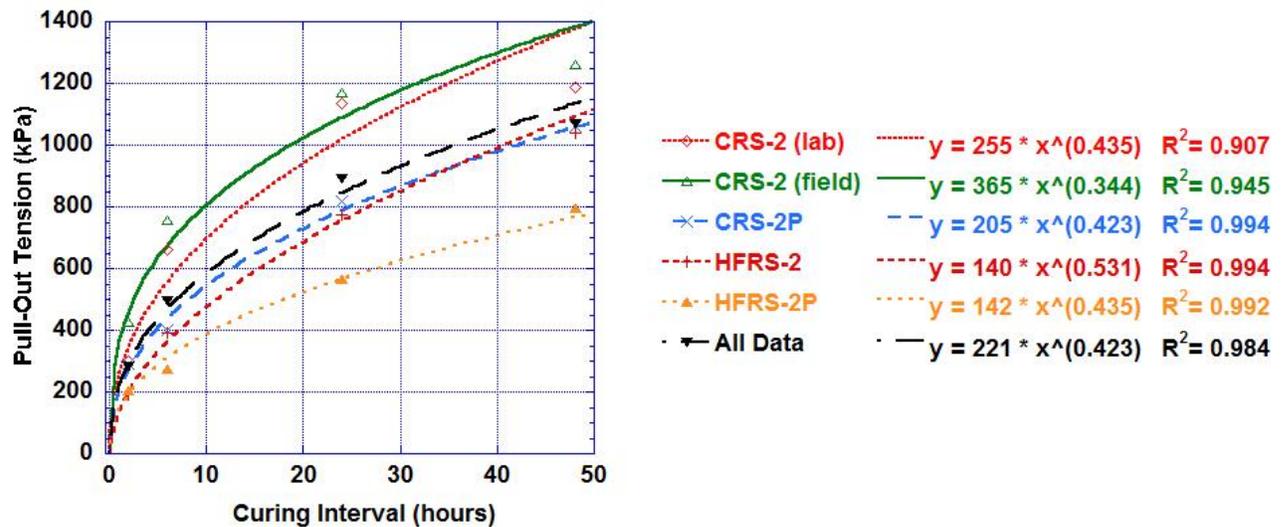

Figure 9-7- Pull-out tension values differ for various emulsion types at a range of curing intervals (Miller, 2010)
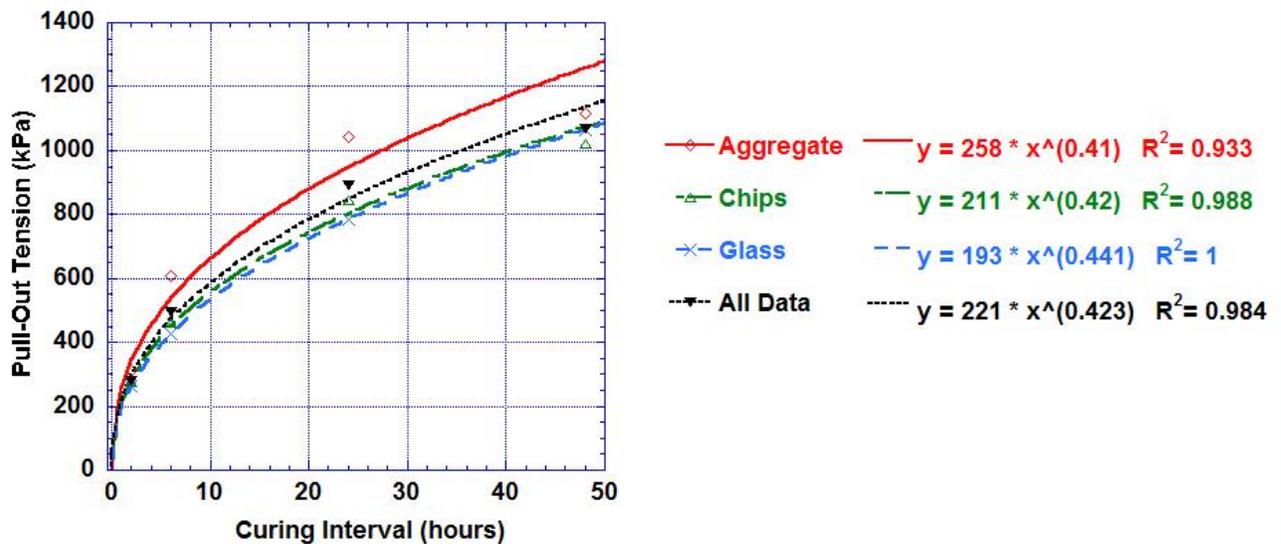

Figure 9-8- Pull-out tension values differ for various substrate types at a range of curing intervals (Miller, 2010)

The US conducted test on granite and tillite aggregates using various modified and unmodified emulsion combinations. The results of these tests correlate well with the results from UWM and are shown in the *Figure 9-9* below:
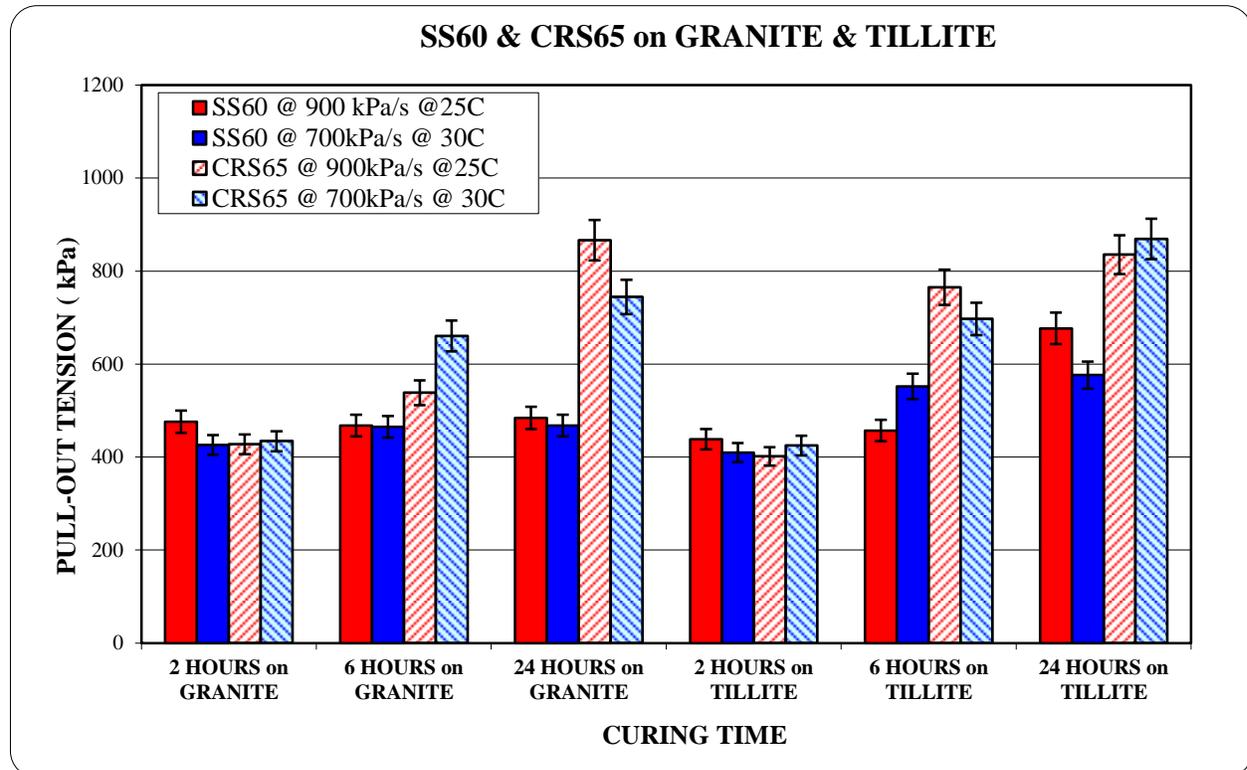

Figure 9-9- Pull Out Tension Values for SS60& CRS65 on Granite & Tillite

Figure 9-9 shows a combination of test results for CRS65 and SS60 bitumen emulsions on granite and tillite aggregates for different loading rates. The following points of interest are noted:

- The CRS65-granite combination outperforms the CRS65-tillite combination at all curing intervals. The pull-out tension appears to increase linearly with curing time.

- SS60 results show very little strength increase over 24 hours, except on tillite at 24 hours. The result is expected from a slow setting emulsion applied on acidic aggregates such as granite and tillite. These aggregates contain silica and have a strong negative charge in the presence of water. This negative charge attracts positively charged cationic bitumen particles, leading to destabilization of the surfactant system and subsequent coagulation of the bitumen particles. This breaking mechanism is absent when anionic emulsions are used with acidic aggregates (Louw, et al., 2004)

- Tests conducted at 900 kPa/s show slight increases in pull–out tensions values over those tested at 700 kPa/s, thereby confirming that higher loading rates result in higher pull-out tension values.


104
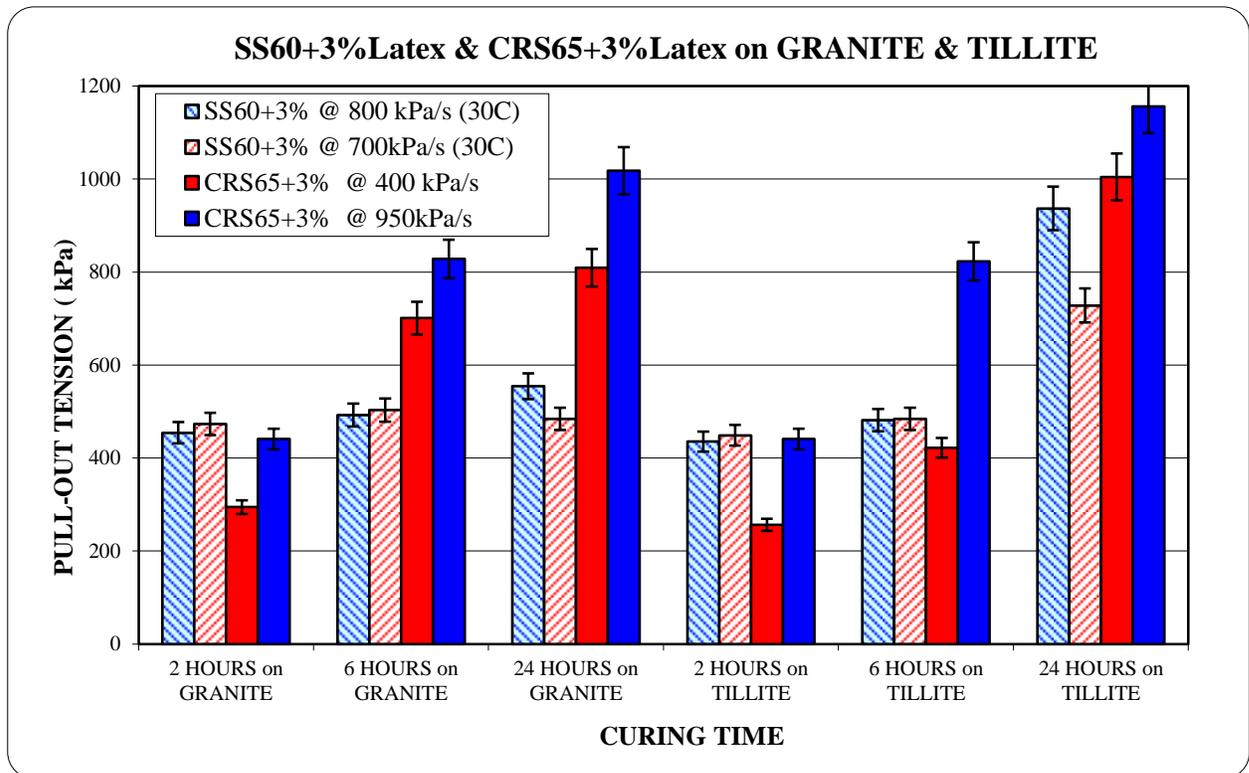
Figure 9-10- Pull Out Tension Values for SS60+3% Latex CRS65+3% Latex on Granite & Tillite

Figure 9-10 shows a combination of test results for CRS65 + 3% latex and SS60 + 3% latex on granite and tillite aggregate substrates. The following points of interest are noted:

- Tests performed at 950 kPa/s show higher results and confirm the findings of Miller that higher loading rates contribute to higher pull-out tension values.

- After 2 hours curing time, results exhibit minimal differences in the results on all four emulsion/aggregate combinations.

- After 6 hours the results for CRS65 + 3% latex/granite show higher tensile strength values than SS60 + 3% latex on granite.

- After 6 hours, there are limited differences in the results of the CRS65 + 3% latex/tillite and SS60 + 3% latex/ tillite.

- After 24 hours, the CRS65/granite combination shows significantly higher strength values than the SS60-granite combination but smaller values that the CRS65 + 3% latex/tillite and SS60+ 3 % latex/tillite combinations.

- The modified emulsions performs better that the unmodified emulsions after 24 hours.

- The CRS65 + 3% latex/tillite and SS60 + 3% latex/tillite combinations values relate well and seem to be superior to granite.


Figure 9-11- Pull Out Tension Values for CRS65& CRS65+3% Latex on Granite & Tillite

Figure 9-11 shows a combination of test results for CRS65 & CRS65 + 3% on granite and tillite aggregate substrates. The following points of interest are noted:

- Tests performed at 950 kPa/s show higher results and confirm the findings of Miller that higher loading rates contribute to higher pull-out tension values.

- The unmodified cationic emulsion (CRS65) outperforms the modified emulsion in after two and six hours on the tillite aggregate

- After 2 hours curing time, results exhibit minimal differences in the results on all four emulsion/aggregate combinations.

- After 6 hours the results for CRS65 + 3% latex/granite show higher tensile strength values than unmodified CRS65 on granite.

- After 6 hours, the CRS65 on tillite shows higher results than the CRS65 + 3% on tillite.

- After 24 hours, the CRS65-granite combination shows lower strength values than the CRS65+3% Latex/granite combination but smaller values that the CRS65-tillite and CRS65+ 3 % latex/tillite combinations.

- The modified emulsions perform better that the unmodified emulsions after 24 hours.
- The CRS65 + 3% latex/tillite and CRS65 /tillite combinations values relate well and seem to be superior to granite.
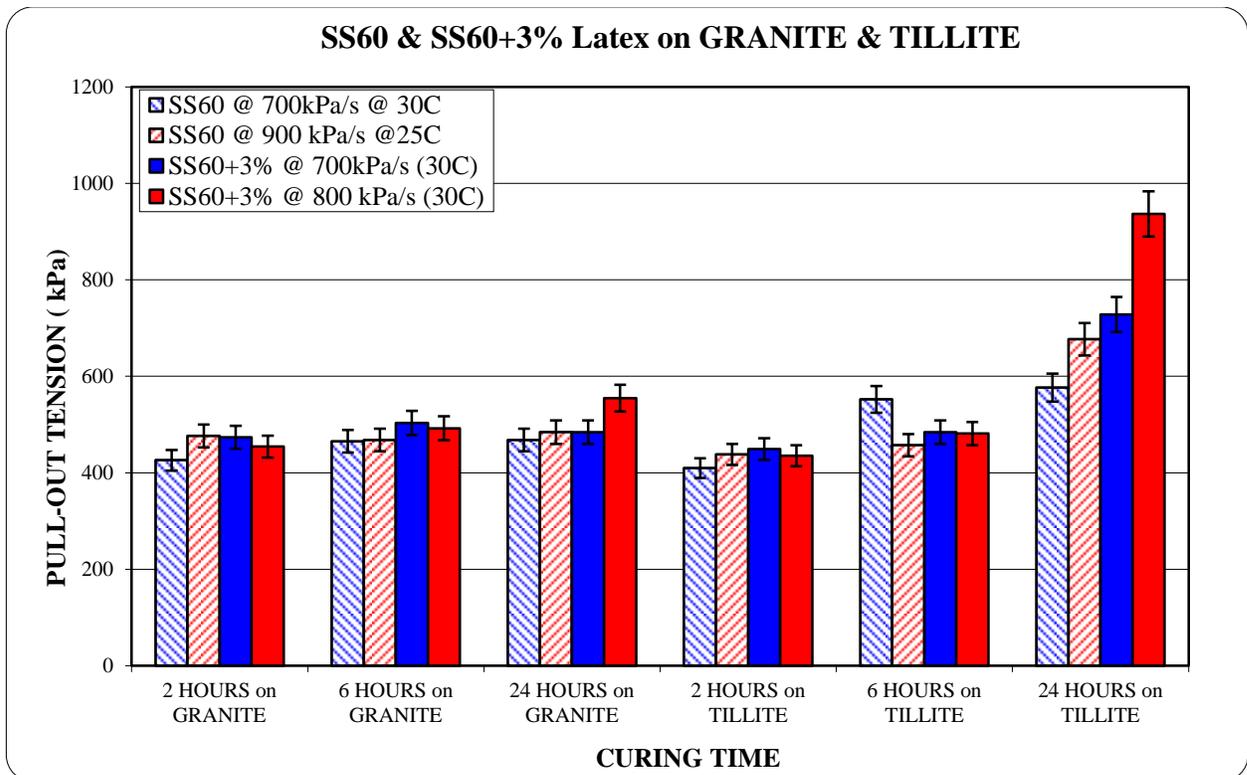

Figure 9-12- Pull Out Tension Values for SS60& SS60+3% Latex on Granite & Tillite

Figure 9-12 shows a combination of test results for SS60 & SS60 + 3% on granite and tillite aggregate substrates. The following points of interest are noted:

- There is only a slight increase after 2, 6 and 24 hours on the SS60 Granite combination. This is expected from an Anionic Emulsion on an Acidic aggregate.
- After 2 hour the Granite and SS60 & SS60+3% Latex combination shows slightly higher values that the SS60 & SS60+3% Latex and Tillite combinations.
- After 6 hour the modified and unmodified emulsion shows comparable results on both granite and tillite.
- After 24 hours the SS60 & SS60+3% Latex shows much higher result on tillite than on granite.

9.7 CORRELATIONS OF BBS TEST RESULTS

In order to validate the results of the BBS test procedure, Miller correlated the BBS test results to two other commonly used test procedures. To understand and quantify aggregate retention, Miller correlated BBS test results to results obtained using the ASTM D7000 Sweep test procedure (Miller, 2010; ASTM D7000, 2008).

In the sweep test, bitumen is applied at a fixed application rate to a felt disk before aggregate chips are applied and compacted. The test apparatus brushes the sample for 1 minute in an attempt to simulate the mechanical brooming action experienced by newly-constructed chip seals. The response variable considered in the sweep test is percent aggregate loss.

9.7.1.1 SWEEP TEST

Establishing a correlation between BBS test results and sweep test results entailed comparing the pull-out tensile strength values obtained using the BBS test to aggregate loss measured using the sweep test at identical curing conditions. Given the existing recommended sweep test performance limit of 10 percent aggregate loss, Miller devised a test correlation between BBS and sweep test results. Sweep test samples prepared with granite and limestone aggregates and CRS-2 and CRS-2P emulsions are compared to BBS test results for similar material combinations, as shown in *Figure 9-13*.

At 2 hours curing, neither the sweep test samples nor the BBS test samples exhibit good performance in terms of aggregate retention or pull-out tension as indicated by high aggregate loss in the sweep test and low pull-out tension values in the BBS test. At 6 hours curing, cohesion and adhesion become more evident with improved aggregate retention and pull-out tension values. After 24 hours curing, samples exhibit greater performance in both tests. As with other experiments, a power law model appears to adequately characterize the relationship between BBS results and sweep test results, with $R^2 > 0.995$. Based on this relationship, a minimum BBS specification limit of 850 kPa may be suggested, a limit that signifies when the binder has gained sufficient bond strength to achieve less than 10 percent aggregate loss as measured by the sweep test.

During the various University Stellenbosch tests only the CRS65 and both the Modified Emulsion, cured for 24 hours, achieved the 850 kPa threshold on both granite and tillite. This is based on curing temperatures of 30°C. In practice road temperatures will in some areas exceed 60°C and a chip seal using emulsion as binder

will usually only be opened after 24 hours. The values are therefore perceived reasonable and a good starting point for further research.
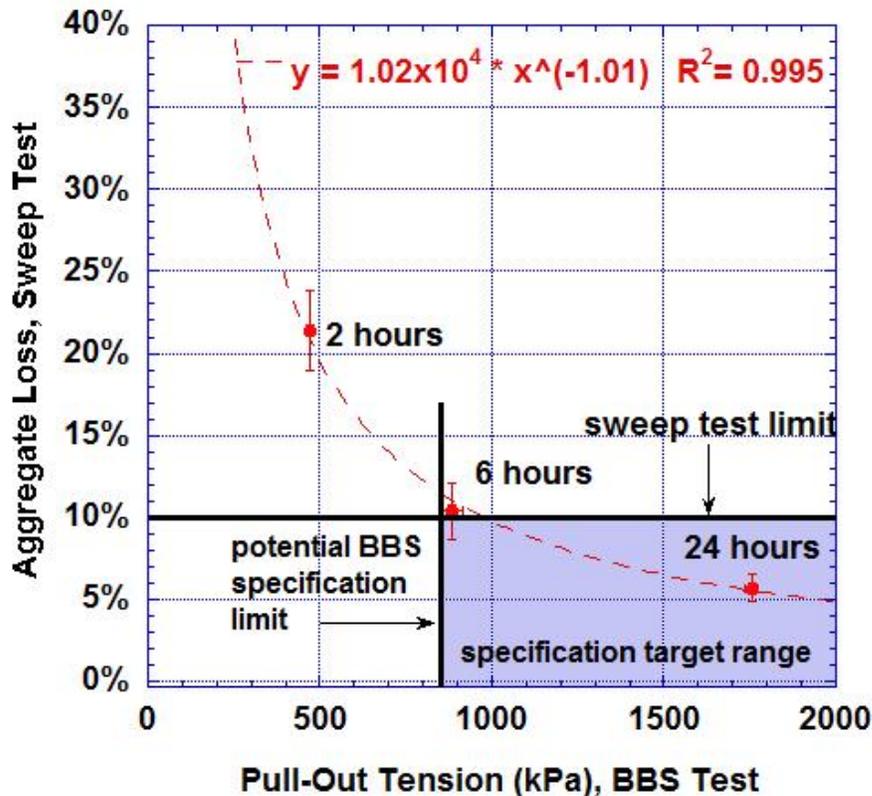

Figure 9-13- When sweep test results are compared to BBS test results, a potential BBS specification limit may be proposed at 850 kPa to define a specification target range (Miller,2010)

9.7.1.2 DYNAMIC SHEAR RHEOMETER (DSR) STRAIN SWEEP

Strain sweep procedures developed for the dynamic shear rheometer (DSR) were also compared to BBS results (Miller, 2010). DSR strain sweep results were considered for two evaluation criteria. Test results were analyzed in the linear range (G* / sin δ at 12 percent strain) and non-linear range (G* / sin δ at 40 percent strain). Emulsion residues cured on granite and limestone substrates demonstrated the effect of curing temperature on strength gain, particularly at early curing intervals. None of the samples attained the full strength of the neat base binder, indicating the presence of water in the emulsion even after 24 hours of curing.

BBS test results are correlated with emulsion residue properties in *Figure 9-14*. Results demonstrate a strong correlation between pull-out tensile strength and resistance to deformation (G* / sin δ) of the emulsion residue. The effect of curing time is also seen in comparing the results, with binder stiffness and bond

strength increasing with time. DSR strain sweep results suggest that the BBS test is capturing a fundamental engineering property in bond strength. The BBS-DSR correlation reinforces the notion that bond strength development can be quantified as the emulsion breaks and begins to release water.
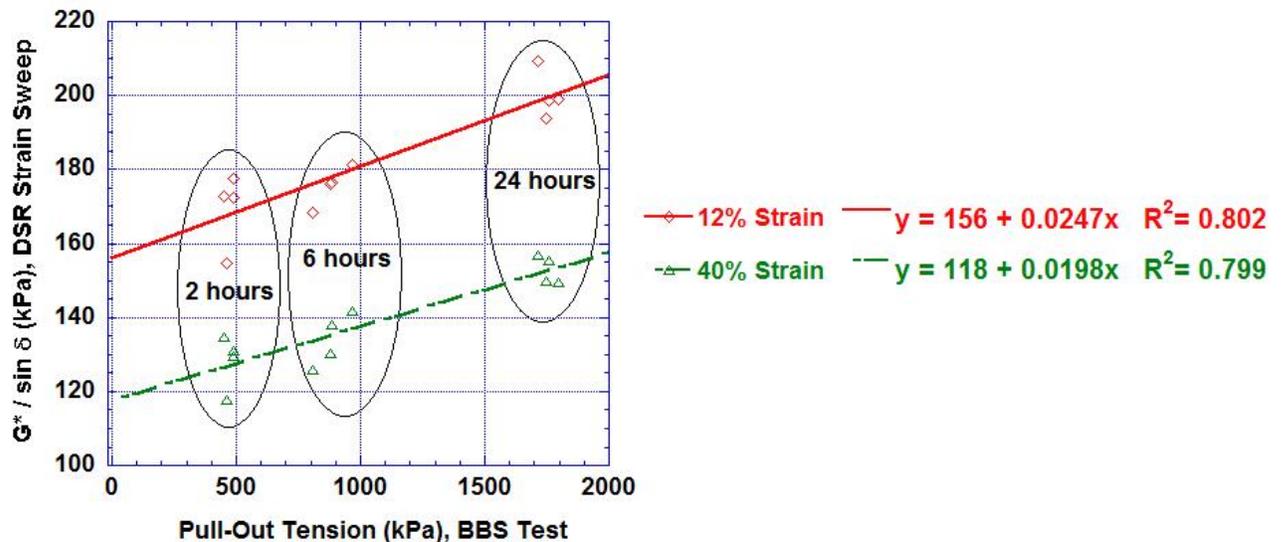

Figure 9-14- BBS test results compare well to DSR strain sweep results at two strain levels.

9.8   INTER-LABORATORY EVALUATION OF BBS TEST METHOD

Based on the results achieved at the University of Stellenbosch and at UWM it was possible to validate the BBS Test method. The South African evaluation utilized an identical experimental setup (e.g. PQG testing instrument and modified pull-out stubs) for a different set of emulsified binders and substrate types.

Substrate preparation procedures differed slightly due to differences in available substrate preparation equipment. UWM tests utilized large aggregate plates that accommodated four pull-out stubs, while US tests utilized aggregate slices from cored rock samples that accommodated only a single pull-out stub.

Figure 9-15 and *Figure 9-16* depict results for different emulsion types and substrate types, respectively. These findings corroborate the findings of Miller and research personnel at UWM that 1) bond strength development can be quantified over time; 2) the BBS test method can characterize bond strength development for different emulsion types; and 3) that advantageous combinations of aggregate-binder can be identified using the BBS test method.
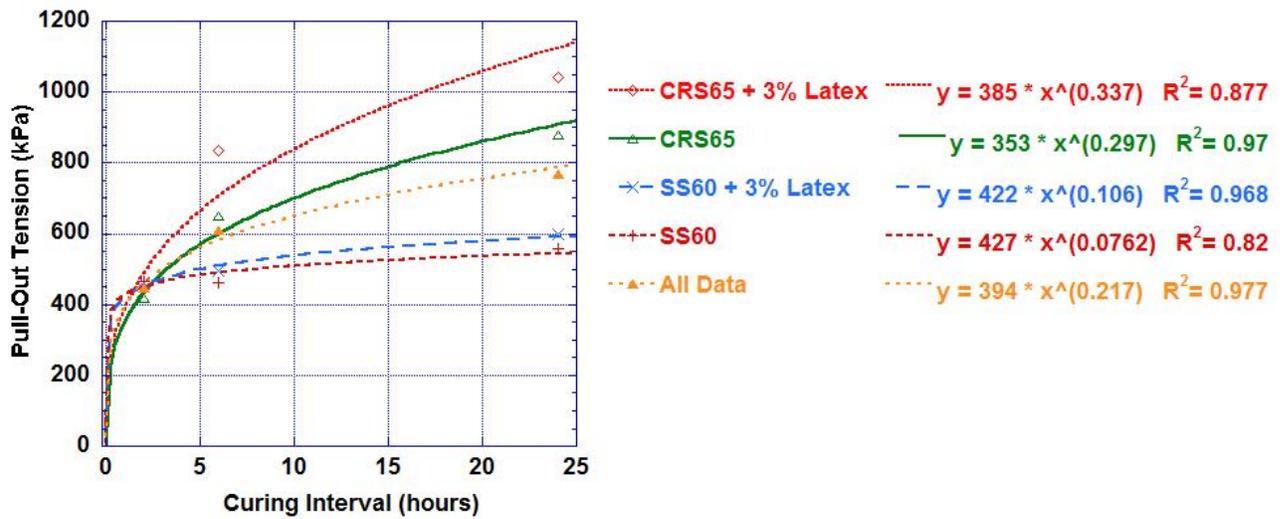
Figure 9-15- Pull-out tension values differ for various emulsion types at a range of curing intervals.
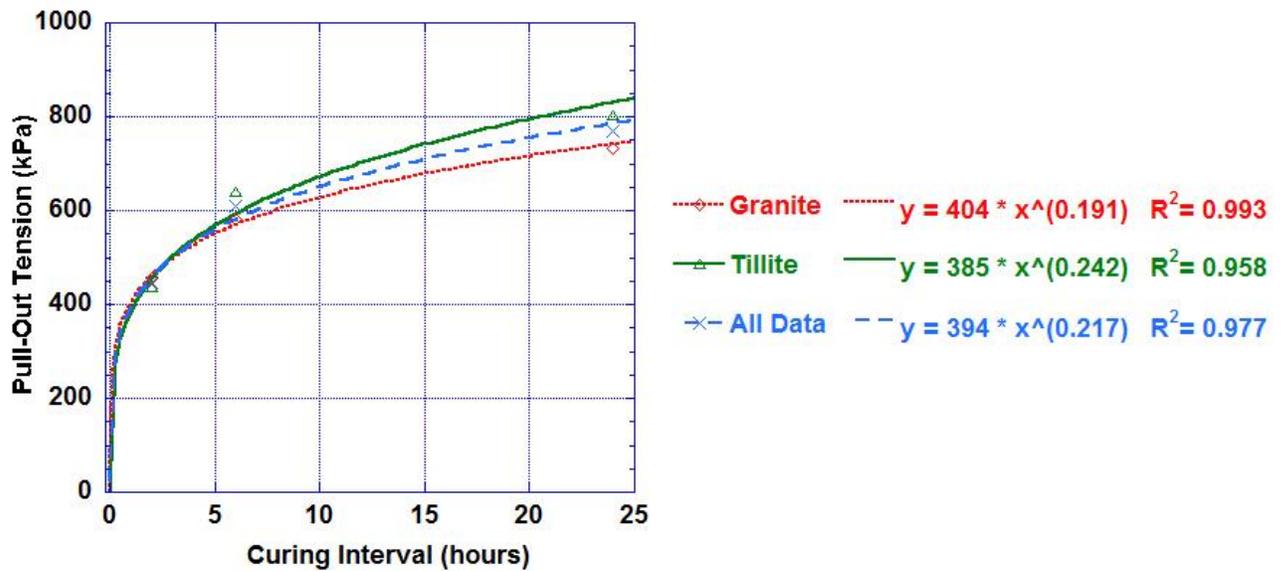
*Figure 9-16- Pull-out tension values differ for various substrate types at a range of curing intervals.*

Table 9-11 displays experimental results from the inter-laboratory testing. Values of the coefficient of variation (COV) are typically less than 10 percent, indicating good repeatability for three replicates in spite of a different substrate preparation procedure.

Table 9-11-BBS test results for materials tested at the University of Stellenbosch.

TEST SETUP			Number	Tensile Strength (kPa)			
Emulsion	Aggregate	Curing Time		Average	St Dev	St Error	COV
CRS65	Granite	2 Hours	16	424.62	4.69	2.71	1.11%
		6 Hours	15	533.02	19.54	11.28	3.67%
		24 Hours	15	919.66	46.65	26.93	5.07%
	Tillite	2 Hours	16	406.55	15.88	9.17	3.91%
		6 Hours	15	765.19	77.24	44.60	10.09%
		24 Hours	15	835.65	57.13	32.98	6.84%
SS60	Granite	2 Hours	7	482.44	13.37	7.72	2.77%
		6 Hours	6	466.18	5.64	3.26	1.21%
		24 Hours	7	545.67	106.95	61.75	19.60%
	Tillite	2 Hours	7	445.40	8.28	4.78	1.86%
		6 Hours	6	459.85	9.77	5.64	2.12%
		24 Hours	7	568.26	51.49	29.73	9.06%
CRS65+ 3% LATEX	Granite	2 Hours	8	439.08	4.14	2.39	0.94%
		6 Hours	6	824.81	13.91	8.03	1.69%
		24 Hours	6	989.22	169.88	98.08	17.17%
	Tillite	2 Hours	8	446.30	12.42	7.17	2.78%
		6 Hours	6	846.49	60.44	34.89	7.14%
		24 Hours	6	1095.81	104.83	60.52	9.57%
SS60+ 3% LATEX	Granite	2 Hours	7	472.50	6.82	3.94	1.44%
		6 Hours	3	505.92	12.42	7.17	2.45%
		24 Hours	2	477.92	5.64	3.26	1.18%
	Tillite	2 Hours	7	449.92	6.82	3.94	1.52%
		6 Hours	3	484.24	2.71	1.56	0.56%
		24 Hours	3	717.31	52.76	30.46	7.36%

10 CONCLUSION & RECOMMENDATIONS

10.1 CONCLUSION

The aim of this study was to fulfil the six study objectives defined in Chapter 1 of this report. The literature review clearly identified the factors that significantly affect the bond strength development between bituminous binders and aggregates. Through the literature review as well as through the requirements developed by the move towards performance specifications it was possible to confirm the need for a test method that will be able to quantify and characterize bond strength development, adhesive properties and aggregate-binder compatibility.

By using the Bitumen Bond Strength test it was possible to evaluate the practicality and repeatability of the developed procedure and the results measured was successfully used to evaluate the bond strength development of modified and unmodified bitumen emulsions on tillite and granite aggregates. It was also possible to correlate the results achieved at the University of Stellenbosch with results from the University of Wisconsin-Madison due to the fact that testing took place at both institutions.

The development of the test and the inter-laboratory test results in essence reinforced the hypothesis that the BBS test protocol can be used to effectively evaluate bond strength of different emulsion types and aggregate types. Except for the loading rate which is a known critical influence, the emulsion type and curing intervals are both identified as the most significant other factors contributing to bond strength development. All these factors were confirmed as part of the test completed during this study.

Aggregate type is also identified as a significant factor that will influence the bond strength development. Interactions between emulsion type and curing interval are identified as the most significant interaction. Although this study delivered a good set of initial results, a lot of further validation test on the BBS test method is still required for the test to be integrated into a performance-based specification system for surface seals.

10.2 RECOMMENDATION FOR FUTURE STUDIES

The development of the Bitumen Bond Strength Test has supplied the opportunity to conduct tests and research in a wide new field. The following areas should be investigated in more detail to answer some of the question raised during this study:

- Test should be conducted using a broader range of emulsions and aggregates. The results yielded will ultimately add to the testing database and well as ensure that the method is developed. .

- Test regimes that include bitumen in its modified and unmodified state should be completed. This information can be used for the evaluation of bond strength development in regular bitumen chip seal.

- Further tests should also be done to test the influences of curing times longer than 24 hours. This may show more insight into the nature of the specific breaking action and adhesion development relationship between different aggregates and emulsions.

- Further studies should also look to address a wider range of curing temperatures and humidity to investigate the tensile strength sensitivity to these factors. The influence of subzero temperatures in the first 48 hours may deliver some interesting results.

- There should be more studies done to see if the effects of pre coating fluids on the bitumen and aggregate bond can be quantified.

- More correlation test should be done to confirm the proposed critical pull-off value of 850 kPa or any more suitable variations on this value.

_

11 BIBLIOGRAPHY

A Epps, C. G. R. B., 2001. *A Performance-Graded Binder Specification for Surface Treatments.* s.l.:Texas Department of Transportation.

AASHTO TP-91 , 2011. *AASHTO TP-91- Standard Method of Test for Determining Asphalt Binder Bond Strength by Means of the Asphalt Bond Strength (ABS),* s.l.: AASHTO.

Asphalt Academy-TG1, 2007. *Technical Guideline 1 : The use of Modified Bituminous Binders in Road Construction,* Pretoria: Asphalt Academy.

Asphalt Academy-TG2, 2009. *Technical Guideline 2- Bitumen Stabilised Materials, A Guideline for the Design and Construction of Bitumen Emulsions and Foamed Bitumen Stabilised Materials,* Pretoria: Asphalt Academy.

ASTM D2397 - 05, 2012. *ASTM D2397 - 05-Standard Specification for Cationic Emulsified Asphalt,* USA: ASTM.

ASTM D4541-09, 2009. *ASTM D4541-09-Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers,* West Conshohocken: American Society of Testing and Materials.

ASTM D977-12, 2012. *ASTM D977-12-Standard Specification for Emulsified Asphalt,* USA: ASTM.

Atanackovic, T. M. & Guran, A., 2011. *Theory of elasticity for scientists and engineers.* Boston: Birkhäuser.

Austroads, 2004. *Spray Seal Design Method,* Australia: Austrpads.

Bickford, A., 2001. *Basic Emulsion Chemistry - AEMA Asphalt Emulsion Technoloogies Wokshop, Presentation.* s.l.:Asphalt Innovations.

Boussad, N. & Martin, T., 1996. *Emulsifier Content in Water Phase and Particle Size Distribution: Two Key Parameters for teh Management of bituminous Emulsion Performance.* s.l., s.n.

BP Bitumen, 2011. *BP Bitumen.* [Online]
Available at: http://www.bp.com/sectiongenericarticle.do?categoryId=9027521&contentId=7050109
[Accessed 03 20 2011].

Canestrari, F. et al., 2010. Adhesive and Cohesive Properties of Asphalt-Aggregate Systems Subjected to Moisture Damage. Journal of Road Materials and Pavement Design(11-32,2010).

Constable, B. T. L., 2009. *V10- Final Year Thesis - The evaluation of the Bitumen Bond Strength Test,* Stellenbosch: s.n.

CSIR-TRH6, 1985. *TRH6-Nomenclature and methods for Describing the Condition of Asphalt Pavements,* Pretoria: National Instiure of Transport and Road Research.

Curtis, C., Ensley, E. & Epps, J., 1993. *Fundamental properties of asphalt-aggregate interactions including adhesion and adsorption, Strategic Highway Research Program, Report SHRP-A-341,* Washington DC: National Research Council .

Ecopave Australia, 2009. *The History of Bitumen Asphalt Concrete Roads.* [Online]
Available at: http://www.ecopave.com.au/bio_bitumen_asphalt_concrete_research_ecopave_australia_013.htm

Epps, J., Chaffin, C. & Hill, A., 1980. *Field Evaluation of a Chip seal Design Method, Research Report 214-23,* s.l.: Texas Transportation Institute.

Epps, J., Galloway, B. & Brown, M., 1974. *Synthetic Aggregate Chip seal Research Report 83-2F,* s.l.: Texas Transportation Institute.

Eurobitume, 2012. *Eurobitume.* [Online]
[Accessed 2012].

European Commitee for Standarization, 1999. *Bitumen and bituminous binders, Determination of cohesion of binders for surface dressings, prEN 13588,* Brussels: CEN.

Forum of European National Highway Research Laboratories, 2004. *Analysis of Available Data for Validation of Bitumen Tests, Report on Phase 1 of teh BiTVal Project,* Brussels: Forum of European National Highway Research Laboratories.

Gorman, J., Crawford, R. & Harding , I., 2004. Bitumen Emulsions in road construction - A Review. Issue March 2004.

Gransburg, D., Zaman, M. & Aktes, B., 2010. *Analysis of Aggregates and Binders used for the ODOT Chip Seal Program,* Oklahoma City: Oklahoma Department of Transportation.

H Bahia, J. M. R. V. T. M. C. D. R. M., 2010. *Evaluation of the Bitumen Bond Strength (BBS) Test for Moisture Damage Charaterization.* Madision: University of Wisconsin.

Hanson, F., 1935. *The bitumitominous surface treatment of rurual highways.* s.l., s.n., pp. 89-178.

Jaixing Mide Machinery Company, 2012. *Jaixing Mide Machinery Company.* [Online]
Available at: http://www.cnmide.com/english/down.asp

James, A., 2002. *Asphalt Emulsion (Chemistry and Concepts).* s.l.:Akzo Nobel Chemicals.

James, A., 2006. Overview of Asphalt Emulsion. *Transport Research Circulary Number E-C102, Asphalt Emulsion Technology,* pp. 1-15.

Kanitpong, K. & Bahia, H., 2003. Role of Adhesion and Thin Film Tackiness of Asphalt Binders in Moisture Damage of HMA. *Asphalt Paving Technology,* Volume 72, pp. 502-528.

Kearby, J., (1953). Tests and Theories on Penetration Surfaces. *Highway Research Board*, Volume 32.

Louw, K., Spence, K. & Kuun, P., 2004. *The use of bitumen emulsion as a cost effective solution for constructing seals during winter..* Proceedings of the 8th Conference on Asphalt Pavements for Southern Africa (CAPSA'04), s.n.

McLeod, N., 1969. *Basic principles for the design and construction of seal coats and surface treatments,* Minnesota, USA.: Association of asphalt paving technologists.

Meng, J., 2010. *Affinity of Asphalt to Mineral Aggregate : Pull-Off Test Evaluation - Master of Science Thesis,* Madison: University of Wisconsin.

Miller, T., 2010. *Development of Bond Strength Test for Improved Characterization of Asphalt Emulsion- Master of Science Thesis,* Madison: University of Wisconsin-Madison.

Miller, T., 2010. *Development of Bond Strength Test for Improved Characterization of Asphalt Emulsion- Master of Science Thesis,* Madison: University of Wisconsin-Madison.

Milne, T. I., 2004. *Towards a performance related seal design method for bitumen and modified road seal binders, Dissertation presented for the Degree of Doctor of Philosophy,* Stellenbosch: University of Stellenbosch.

National Cooperative Highway Research Program, 2005. *NCHRP Synthesis 342-Chip Seal Best Practices,* Washington, DC 20001: National Cooperative Highway Research Program.

Opus International Consultants, 2010. *Performance based bitumen specification, discussion paper,* New Zealand: s.n.

Opus International Consultants, 2010. *Performance Based Bitumen Specifications.* New Zealand: s.n.

Oxford University Press, 2011. *Oxford Dictionaries.* [Online]
Available at: http://oxforddictionaries.com/
[Accessed 24 September 2011].

Rabiot, D. & B. G. Koenders, a. M. A., 1999. *A Quality Concept for Emulsion Grade Bitumens..* Washington, s.n.

Roberts, C. & Nicolls, J., 2008. *Road Note 39-Design guide for road surface dresssing,* Berkshire: IHS for TRL.

Rodríguez-Valverde, M. Á., Cabrerizo-Vílchez, M. Á., Páez-Dueñas, A. & Hidalgo-Álvarez., R., 2002. *Kinetic Model of Bitumen Emulsion Breaking.* Lyon, France,, 3rd World Congress on Emulsions.

SABITA, 2009. Research and reminiscences from roadmakers- New Graham Ross publication paves the way for future historic research. *asphaltNEWS*, 23(3).

SABITA-M2, 2007. *Manual 2 - Bituminous binders for road Bituminous binders for road,* Howard Place: SABITA.

SANS 309, 2004. *SANS 309:2004-Anionic bitumen road emulsions,* Pretoria: South African Buro of Standards.

SANS 548, 2003. *SANS 548:2003- Cationic bitumen road emulsions,* Pretoria: SABS.

SANS4001-BT1, 2012. *SANS4001-BT1,Civil Engineering Test Methods - Part BT1 - Penetration Grade Bitumen,* Pretoria: South African Buro of Standards.

SANS4001-BT2, 2012. *SANS4001-BT2 , Civil Engineering Test Methods - Part BT2 - Cutback Bitumens,* Pretoria: South African Buro of Standards.

Shell Bitumen, 2003. *The Shell Bitumen Handbook.* 5th ed. London: Shell Bitumen.

South African National Road Agency, 2007. *Technical Recommendations for Highways 3 (TRH3-2007) - Design and Construction of Surfacing Seals,* Pretoria: South African National Road Agency.

Sponholtz, S., 2012. *A Brief History of Road Building.* [Online]
Available at: http://www.triplenine.org/articles/roadbuilding.asp

Superpave, 2012. *Pavement Interactive.* [Online]
Available at: http://www.pavementinteractive.org/article/superpave-performance-grading/

Texas Department of Transportation, May 2010. *Seal Coat and Surface Treatment Manual,* s.l.: Texas Department of Transportation.

Towler, J. & Dawson, J., 2008. *History of Chipsealing in New Zealand - Hanson to P/17.* Adelaide, ARRB Group Ltd and Authors 2008.

Verlhac, P., Verzaro, F., Calderon., F. L. & J. J. Potti, a. B. E., 2002. *Characterisation of Bituminous Emulsions: Particle Size Distribution and Amount of Residual Emulsifier..* Lyon, France, s.n.

Walter, J. a. D. D., 2002. *Coalescence of Quick Set Surface Dressing PMB Emulsions.* Lyon, France,, 3rd World Congress on Emulsions,.

Wates, J. a. A. J., 1993. *Zeta Potential Measurements of Bitumen Emulsions and Road Aggregates..* Paris, s.n.

Weinert, H., 1980. *The Natural Road Construction Materials of South Africa.* Pretoria: Academia.

Wikipedia - Rock Geology, 1996. *Petrology.* [Online]
[Accessed Chisholm, Hugh, ed. (1911). "Petrology". Encyclopædia Britannica (11th ed.). Cambridge University Press.].

Youtcheff, J. & Aurilio, V., 1999. *Moisture sentivity testing of bitumen using a pneumatic adhesion test,* Luxembourg: Eurobitume Workshop.

ADDENDUM 1- FINAL DRAFT OF BBS TEST PROCEDURE - LATER ACCEPTED AS

(AASHTO TP-91 , 2011)

ADDENDUM 2- UWM STUDY TOUR FEEDBACK REPORT

ADDENDUM 3- MINUTES OF STUDY GROUP CONFERENCE CALLS AND REPORTS

COMPLETED DURING THIS PHASE

ADDENDUM 4- BBS TEST RESULTS OF TESTS COMPLETED IN 2010

ADDENDUM 5- PAPER PRESENTED AT 2ND INTERNATIONAL SPRAY SEALING

CONFERENCE, AUCKLAND, AUSTRALIA, 2010

ADDENDUM 6 – PAPER PRESENTED AT 10TH CONFERENCE ON ASPHALT PAVEMENT FOR SOUTHERN AFRICA, 2011