EP International
Test Report on
Interlayer Bond Strength on
C55 Emulsion Prime

EXECUTIVE SUMMARY	
Title:	Comparison of Interlayer Bond Strength between C55 Emulsion Prime and MC30 Cutback Bitumen Prime
Abstract:	
Keywords:	Interlayer Bond Strength, Emulsion, Prime, Adhesion, Binder
Objective:	To evaluate application on damp or wet surfaces, to evaluate trafficability under one hour with no stripping or tyre pickup
Purpose:	To evaluate functionality for good adhesion with aggregates on granular base layers
Methodology:	Test Methods, SANS 4001 BT4, ASTM D2397, ASTM D88, ASTM D95, ASTM 6997, EN 12697, EN 12849, EN 13808
Conclusions:	The C55 Emulsion Prime demonstrates superior interlayer bond strength compared to MC30 Cutback Bitumen Prime in wet regions or when dry aggregate condition is not feasible. The C55 Emulsion prime has a rapid curing rate within 60mins of application to a wet surface making it an efficient protective layer over granular surfaces to adhere and form a good bond strength property with the asphalt layer. This superiority is evident in both the direct shear test, rolling bottle tests, penetration power test and Zeta Potential test making C55 Emulsion Prime a more reliable choice for enhancing pavement performance in accordance with environmental friendliness and climate change sequences
Limitations or Risks:	
Benefits:	Environmentally Friendly Product
Contract Context:	This document is the result of a research effort funded by EP International in terms of Order

DISCLAIMER *Considerable effort has been made to ensure the accuracy and reliability of the information contained in this report. However, neither CSIR nor any of its members can accept liability for any loss, damage or injury whatsoever resulting from the use of this information.*

APPROVAL PAGE			
Project Name	EVALUATION OF FAST TRACK EMULSION AND PG 70-10E BITUMEN		
Project No	59E2300		
Title			
Prepared for	EP INTERNATIONAL		
Authored for	CSIR	Signature	Date
By	Dr Samuel Abejide		24-07-2024
Designation	Principal Researcher		
Moderated for	CSIR	Signature	Date
By	Mr Christian Mulol		
Designation			
Approved for	CSIR	Signature	Date
By	Ms Nomsa Dlamini		
Designation	Executive Cluster Manager		

DISTRIBUTION LIST			
No	**Name**	**Designation**	**Organisation**
Master	Programme Office		
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			

CHANGE HISTORY			
Change Control No	**Date**	**Name**	**Description**
n/a			Original release.

Table of Contents

List of Figures **5**

List of Tables **6**

Preface **7**

Acknowledgements **8**

Abbreviations **9**

1. Introduction **10**

2. Objective **12**

3. Materials and Methods **13**
3.1 Materials 13
3.2 Test Methods 13
3.2.1 Direct Shear Bond Test: 13
3.2.2 Equipment and Materials 13
3.2.3 Procedure 14
3.2.3.1. Preparation of Specimens 14
3.2.3.2 Assembly of Test Specimen 15
3.2.4. Testing Procedure 15
3.2.5. Test Experimentation and Analysis 15
3.2.6. Results and Interpretation 15
3.3 Rolling Bottle Test Method: 17
3.3.1 Equipment and Materials 19
3.3.2. Procedure 19
3.3.2.1. Preparation of Aggregates 19
3.3.3. Preparation of Bitumen 19
3.3.4. Coating of Aggregates 19
3.3.5. Rolling Bottle Test Requirements 20
3.3.6. Assessment 20
3.3.7. Results and Interpretation 20
3.4 Zeta Potential Test: 21
3.5 Bituminous Penetration Power Test (EN12849): 25
3.5.1 Determination of penetration power of Bituminous emulsions 25
4. Conclusion 30
5. Recommendations 31
Appendix A 33

List of Figures

Figure 1: Global Emulsion usage by location 10
Figure 2: Global Emulsion usage by volumes 11
Figure 3: The coated aggregates before rolling bottle test 18
Figure 4: Rolling Bottle test with the C55 and MC30 Prime subjected to the mechanical rolling machine. 18
Figure 5: Visual Investigation of the adhesion of the prime products: A. C55 Emulsion Prime and B. MC30 Cut-Back Bitumen Prime 20
Figure 6: SurPASSTM 3 series (Anton Paar) 22
Figure 7: A characterization of aggregate surface chemistry pH regions (Labib et al., 2007) 23
Figure 8: Relationship between the Zeta potential and pH of the aggregates 23
Figure 9: Relationship between the Zeta potential and pH of the C55 Emulsion Prime 24
Figure 10: Relationship between the Zeta potential and pH of the MC30 Prime 24
Figure 11: A-. Sample of Silica Sand placed in pan to cool. B-. Sample of Silica Sand placed in test apparatus 25
Figure 12: Sample of Silica Sand placed in pan to cool. B. Sample of Silica Sand placed in test apparatus 26
Figure 13: MC30 Sample after test 27
Figure 14: C55 Sample after test. 27
Figure 15: Binder Sample drop test for Prime curing rate. 28
Figure 16:C55 and Distilled water (1:1): Binder penetration Sample after test. 29
Figure 17: Direct Shear Results 33
Figure 18: C55: Sample 1(50Mpa) 34
Figure 19: C55: Sample 2 (100Mpa) 34
Figure 20: C55: Sample 3 (150Mpa) 35
Figure 21: BT4 Test Results: C55 Sample 36

List of Tables

Table 1: Interlayer Bond Shear Strength tabular standard deviation Table 1: Interlayer Bond Strength tabular summary on Standard Deviation 17

Table 2: Detailed test report and sample description for MC 30 penetration power 28

Table 3: Detailed test report and sample description for C55 penetration power 28

Table 4: Detailed test report and sample description for C55 penetration power 29

Table 5: Testing as per SANS 4001-BT4: 2014 specification requirements 38

Preface

The purpose of this test report documents the relationship between the adhesion strength and penetration power of two Bituminous prime products; C55 Emulsion Prime and MC30 Cutback Bitumen Prime. The document does not in any way disregard the performance of either of the two products, the test results only highlight the performance of each product subjected to the same test condition and environment for performance-related specifications in accordance with SANS and EN standards where applicable.

The methods are also subject to validation where necessary. This test report is based on research conducted by the CSIR (Council for Scientific and Industrial Research, Pretoria) and completed in July 2024. This report comprises test report documented by qualified laboratory technicians followed by laboratory investigations. The intention is solely to foster the need to drive the initiative of green bituminous products that are environmentally friendly.

Acknowledgements

The test procedure and results in this report were conducted by CSIR-TIE-PDC laboratory technicians and reviewed by CSIR-TIE-PDC staff members:

Dr Samuel Abejide
Mr Johan O'Connell
Ms Santa Conrad
Ms Dorathy Maleka
Mr Bonke Nomlahla
Mr David Molebalo
Mr Kgosi Molejane
Ms Keneilwe Mphathiwa
Mr Reatile Pitso

ABBREVIATIONS

ASTM	American Standard Testing Method
C55	Cationic Emulsion Prime (55% Binder Content)
MC30	Medium Curing Cut-Back Bitumen Prime
SANS	South African National Standard
KCL	Potassium Chloride
HCL	Hydrochloric Acid
NaCl	Sodium Chloride
IEP	Isoelectric Point
EN	European Testing Method

1. Introduction

The interlayer bond strength for bituminous products for either a prime or tack-coat is a critical factor in pavement performance. This property has the potential to affect the durability, stability in relation to the bond force and longevity of the pavement layers. This report developed by CSIR for EP International compares the interlayer bond strength between two prime coat products: C55 Emulsion Prime and MC30 Cutback Bitumen Prime. The C55 Emulsion Prime is a cationic based emulsion prime while the MC30 is a Cut-Back Bitumen product.

Although, bitumen emulsions have been in use for more than a decade and has over time proven to fulfil performance requirements for liquid bitumen applications, Figure 1 and 2 provides a global use for bitumen. During this period the most widely used liquid bitumen is Cut Back Bitumen with the MC30 Primer used to bind granular layers to asphalt layers. The formulations and material specification data to develop MC30 bitumen with hazardous effects has prohibited its use in many countries. Thus, global use, production and application of liquid bitumen (emulsions) has resulted to the development of various types of environmentally and economically friendly bituminous emulsion products which makes it a stable use in road construction products.

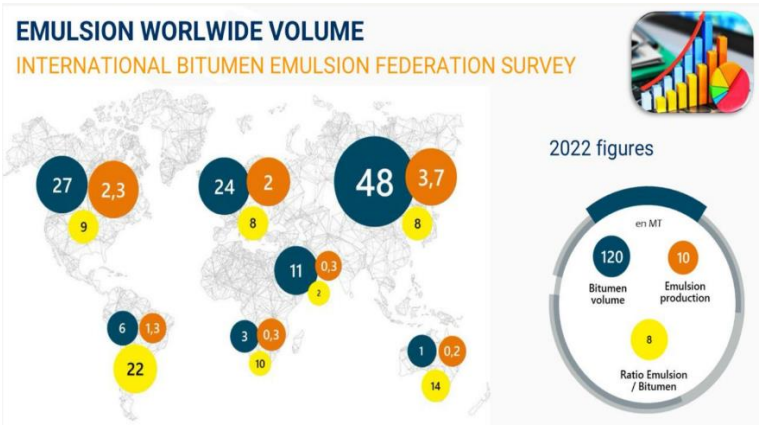


Figure 1: Global Emulsion usage by location

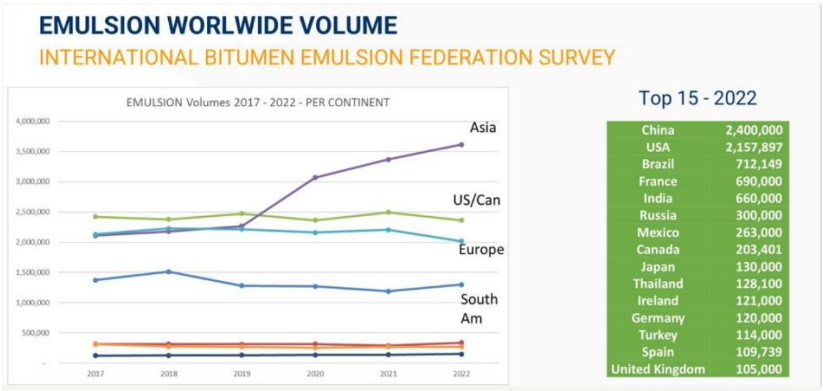


Figure 2: Global Emulsion usage by volumes

Bituminous emulsions are colloidal dispersions of (conventional or modified) bitumen droplets in an aqueous phase, composed of water and one or more anionic and cationic emulsifying agents, in addition to other additives such as latex for modified emulsions, which have the purpose of dispersing the bitumen, ensuring the emulsion is stable and guaranteeing adhesion to aggregates at room temperature. The main characteristic of bituminous emulsions is that they can be used as a binder at lower temperatures than other grades of bitumen, even at room temperature. Liquid bitumen consistency allows them to be used as a tack coat or prime coat to improve adhesion between the different layers of the road surface courses bound with aggregates layer or asphalt layer as the case may be. This is achieved during the emulsion-breaking process, where the free bitumen particles provide cohesion to the whole. Bituminous emulsions are the fundamental base that has made it possible to develop cold-mix technology for roads. Moreover, modified bituminous emulsions can be used in road surface layers that require high performance in the presence of heavy-duty traffic and adverse weather conditions, guaranteeing excellent bonding between layers and exceptional cohesion to aggregates.

2. Objective

The primary objective of this technical report is to evaluate and compare the interlayer bond strength property between C55 Emulsion Prime and MC30 Cutback Bitumen Prime under controlled laboratory conditions. However, field representation on a pilot site is essential for the localisation of this C55 Emulsion product for African and Tropical regions as well as its application over swampy or wet granular layers. The objectives are:

a). Evaluation of the Prime product application on damp or wet surfaces

b). Evaluation of Prime product to enhance performance regarding trafficability within 60 minutes without tyre pickups.

c). Evaluation for same-day paving of asphalt layer over primed surface

For this purpose, a preliminary classification of the Emulsion was conducted based on the BT4 test (SANS 4001-BT4). The results are as displayed in Appendix A. The 55% by proportion of Binder content is in accordance with EN 13808 and 56.17% in accordance with ASTM D2397.

3. Materials and Methods

3.1 Materials

1. **C55 Emulsion Prime**
    - A cationic slow-setting emulsion is used as a prime coat in pavement construction developed by EP International (Extra performance for Bitumen and Emulsions)
2. **MC30 Cutback Bitumen Prime**
    - A medium-curing cutback bitumen used as a prime coat in pavement construction layer.

3.2 Test Methods

The study employed the following tests to measure interlayer bond strength: Direct shear test and Rolling Bottle Test.

3.2.1 Direct Shear Bond Test:

The Direct Shear Test was one of the selected laboratory procedures used to evaluate the interlayer bond strength of bituminous materials. This test measures the shear strength between layers in a bituminous pavement structure, providing valuable data for assessing the performance and durability of the pavement.

- **Sample Preparation**: Asphalt specimens were prepared using a standard shear mould, with each specimen consisting of three layers with the top asphalt layer bonded to a granular G1 layer by the two prime coats (C55 emulsion Prime and MC30 Prime). It is needful to note that the two granular layers G1 (Dolerite) and the G5 layer as the subbase.
- **Testing Procedure**: The specimens were tested for direct shear strength using a shear testing apparatus, applying a constant displacement load cells rates to the test sample until failure.

3.2.2 Equipment and Materials

1. **Shear Testing Apparatus**:
    - A device capable of applying a constant displacement rate and measuring shear force until failure.
2. **Asphalt Specimen Mold**:
    - A mould to prepare cylindrical test specimens with specified dimensions (300mm Height and 150mm Diameter cylinder).
3. **Loading Frame**:
    - A loading frame equipped with a data acquisition system to record load and displacement until failure.
4. **Compaction Machine**:
    - Capable of maintaining the required compaction of granular layers G1 (Dolerite) and G5 prior to the bitumen prime application.
5. **Weighing Balance**:
    - Accurate to ±0.1 g.
6. **Bituminous Materials**:

- The bitumen samples (C55 Bitumen Emulsion Prime and MC30 Bitumen Prime) and aggregates (G1 and G5) as well as a medium grade asphalt mix to be tested.

7. **Press machine**:
   - For crushing the specimens to the record and measure the extent of penetration of the two bituminous samples.

3.2.3 Procedure

3.2.3.1. Preparation of Specimens

1. **Material Selection**:
   - Select appropriate bitumen products (C55 Emulsion Prime and MC30 Prime) and aggregate materials (G1-Dolerite and G5 - for the test.
2. **Specimen Preparation**:
   - The G1 material and G5 material was selected and weighed using the weighing balance. The optimum moisture content of the two samples were determined as (4.9% for the G1) and (6.2% for the G5).
   - The samples were mixed according to predetermined weights and height that will feed into the cylindrical mould Figure 3). The heights for each sample were predetermined to give a total testing height of all three layers equal to (300mm ± 10mm) as the test height specimen for the direct shear test.
   - The selected heights were determined by compacting the three-material samples; G1, G5 and Asphalt medium grade mix in the mould to form the cumulative effective test specimen with specified dimensions (e.g., 150 mm diameter and 300 mm height for the cylindrical specimens).

Figure 3: Direct Shear Sample Preparation for C55 and MC30

3. **Curing**:
   - During the preparation of the sample, the two bitumen samples were applied to the surface of the G1 granular layer and allowed to cure prior to the application of the asphalt layer.
   - The C55 emulsion prime specimens was applied at room temperature to the surface of the G1 material and this cured within 60mins after being applied to the wet/moist G1 granular surface, afterwards the asphalt layer was applied and compacted ready to be tested in the shear machine.

- The MC30 prime was equally applied to the surface of the G1 material at room temperature. Due to the nature of Cut-Back bitumen products, the MC30 prime was only applied to the surface G1 material after 48 hours to a dry surface. Although, this was not sufficient time for the G1 material to be dried enough to ensure a stable bond between the asphalt layer and the G1 granular layer while in the mould.

3.2.3.2 Assembly of Test Specimen

1. **Specimen Layer Assembly to Direct shear machine**:
   - Three samples each were prepared to be tested for the two products (C55 Emulsion Prime and MC30 Prime).
   - Each sample was tested on three different load cells 50Mpa, 100Mpa and 150Mpa load application rates to failure.

3.2.4. Testing Procedure

1. **Specimen Installation**:
   - The bonded specimen in the mould was placed in the mounted shear testing apparatus.
2. **Shear testing Conditioning**:
   - The specimens were conditioned to the test load cells (50Mpa, 100Mpa and 150Mpa) until failure. Afterwards the load at the top layer of the specimen was applied until failure and the maximum load at failure was recorded and documented accordingly.

3.2.5. Test Experimentation and Analysis

1. **Failure Identification**:
   - The peak shear load at which failure occurs for each of the samples on each of the three load cells (50Mpa, 100Mpa and 150Mpa) was identified and recorded.
2. **Calculation of Shear Strength**:
   - The interlayer shear strength was calculated using the report generated by the Direct shear test apparatus:
     - The tests were repeated for the C55 Sample as well as the MC30 Sample.

3.2.6. Results and Interpretation

Direct Shear Bond Test Strength: Asphalt specimens were prepared using a standard shear mold, with each specimen consisting of three layers with the top asphalt layer bonded to a granular G1 layer by the two prime coats (C55 emulsion Prime and MC30 Prime) Fig 3. The two granular layers used were G1 (Dolerite) for the base and G5 as the subbase (SANS 3001). The C55 emulsion prime specimens was applied at room temperature to the surface of the G1 material and this cured within 60mins after being applied to the wet/moist G1 granular surface, afterwards the asphalt layer was applied and compacted ready to be tested in the shear machine. The MC30 prime was applied to the surface of the G1 material at room temperature. Due to the nature of Cut-Back bitumen products, the MC30 prime was only applied to the surface G1 material after 72 hours to a dry surface. Although, this did not provide sufficient time for the G1 material to be dried enough to ensure a stable bond between the asphalt layer and the G1 granular layer while in the mold. Three samples each were prepared to be tested for the two products (C55 Emulsion Prime and

MC30 Prime). The peak shear load at which failure occurs for each of the samples on each of the three load cells (50MPa, 100MPa and 150MPa) was identified and recorded Fig 4a and 4b.

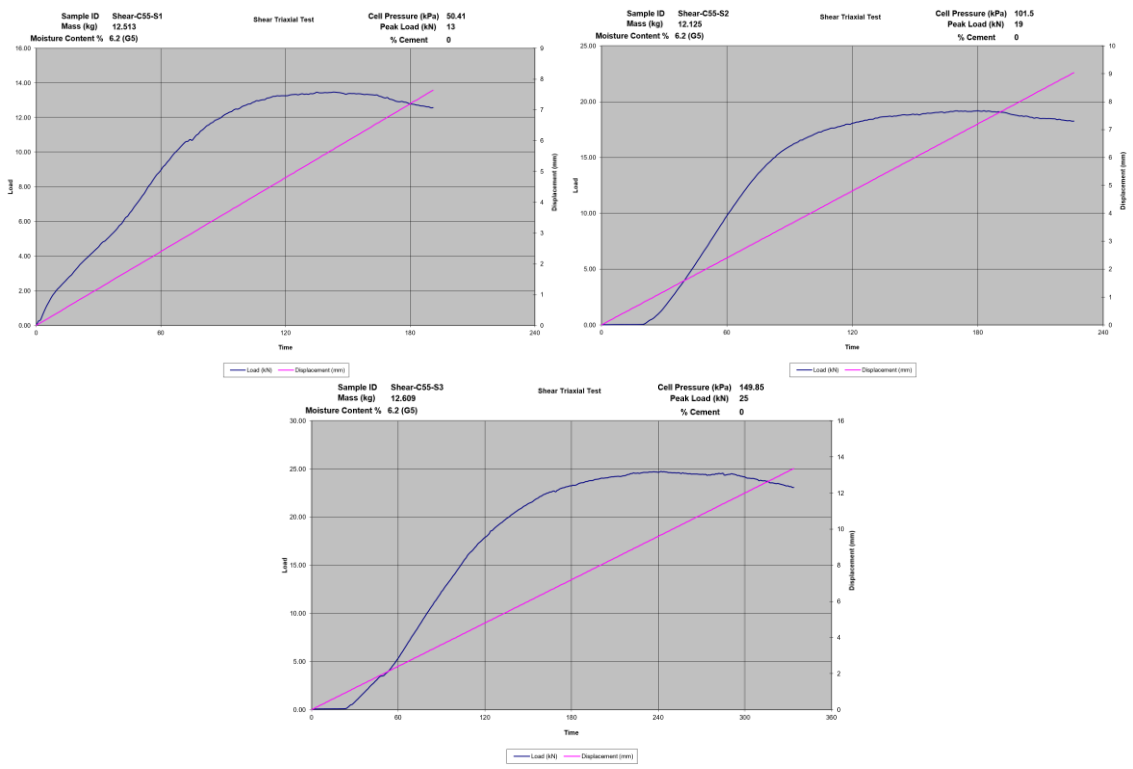
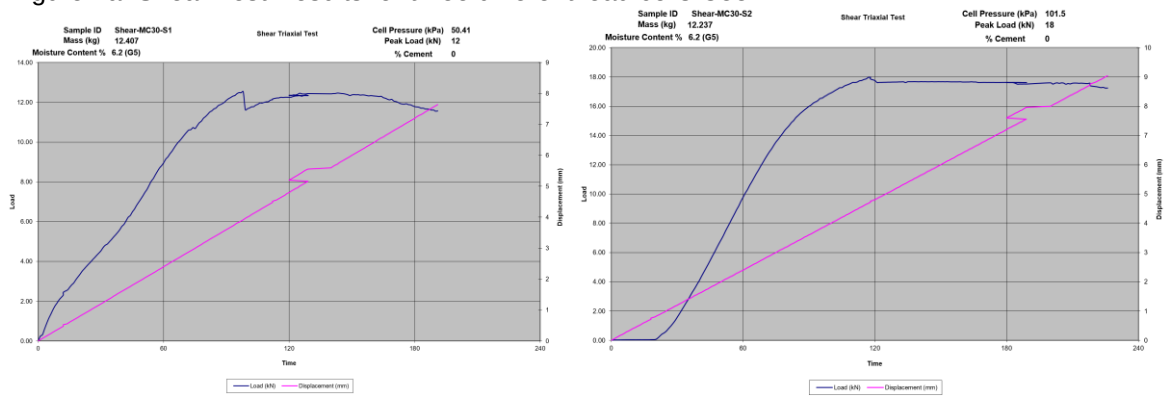
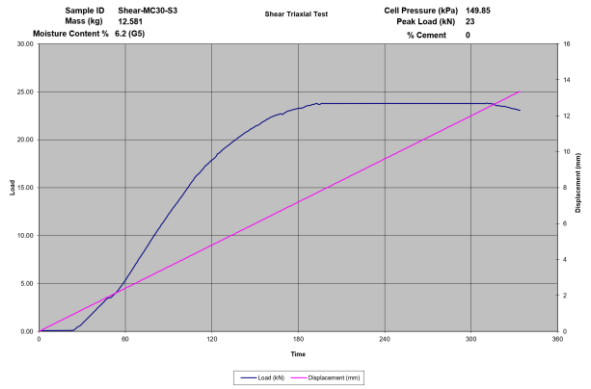


Figure 4a: Shear Test Results for three different load cells C55





Shear Failure Peak Load			
	C55	MC30	
Sample 1	13	12	
Sample 2	19	18	
Sample 3	25	23	

Figure 4b: Shear Test Results for three different load cells MC30 and Comparison

Each sample was tested on three different load cells 50MPa, 100MPa and 150MPa load application rates to failure. The shear failure for each of the comparative specimens are as presented in Fig 4b.

The Table 1.0 provides a tabulated summary of the average shear strength and standard deviation of the specimens tested. The test results indicate that C55 Emulsion Prime had superior shear strength and was more consistent than MC 30 Cutback Bitumen.

Table 1: Interlayer Bond Strength tabular summary on Standard Deviation

Prime Coat	Average Shear Strength (MPa)	Standard Deviation (MPa)
C55 Emulsion Prime	1.25	0.15
MC30 Cutback Bitumen Prime	1.10	0.18

Failure Mode: This shear test presents a summary of the recorded failure modes on the two different bituminous prime materials with recorded maximum failure loads on the direct shear test machine. The Direct Shear Test provided essential data on the interlayer bond strength of bituminous materials, helping to assess the effectiveness of different bonding agents and bitumen types in pavement applications while load was applied at the top. Though this method does not give a precise indication or relationship on the bond performance. It was then necessary to conduct further test that provided direct indication to the adhesion property developed between the Bituminous products and the aggregates. Three other tests were carried out. Rolling Bottle test, Zeta Potential and Penetration Power test (EN12849).

3.3 Rolling Bottle Test Method:

For the purpose of correlating the nature of adhesion developed between the two bituminous interlayer bond products, the rolling bottle test procedure was further recommended to provide a verification of the results of interlayer bond strength on the two products.

The Rolling Bottle Test also formed one of the selected laboratory procedures used to evaluate the adhesive properties and bond strength of bitumen to aggregate. This test simulates the interaction between bitumen and aggregate under dynamic conditions, providing insight into the effectiveness of the bitumen as a bonding agent.

Two samples of 194g of aggregate passing 14mm, retained on 10mm were washed and preheated for 1 hour at 163°C. 6g of C55 and MC30 were also heated at 163°C separately. The aggregate and C55 were

mixed thoroughly and reheated at 163°C. The MC30 and the other aggregate samples were also mixed thoroughly and reheated at 163°C for 15 minutes (Figure 3)

Figure 3: The coated aggregates before rolling bottle test

- ***Sample Preparation:*** Similar to the shear bond test, the two interlayer bond samples were compared using the rolling bottle test to correlate and validate the readings from the direct shear apparatus.

  The two mixtures were then placed in two separate 1 litre glass jars filled with distilled water and allowed to cool for 24 hours. After cooling, the two glass bottle samples were placed in a rolling machine and allowed to roll for 96 hours (refer to Figure 4).

Figure 4: Rolling Bottle test with the C55 and MC30 Prime subjected to the mechanical rolling machine.

- ***Testing Procedure:*** The specimens were subjected to an aggregate conditioning dynamic rolling to determine the interaction and adhesion property between the two bituminous samples (C55 Emulsion Prime and the MC30 Prime).

3.3.1 Equipment and Materials

1. **Rolling Bottles Jars**:
   - Glass bottles with a capacity of 500 ml.
2. **Aggregates**:
   - Clean, dry aggregates (G1 – Dolerite) with specified gradation.
3. **Bitumen**:
   - The bitumen samples to be tested (C55 Bitumen Emulsion and MC30 Cutback Bitumen Prime).
4. **Thermostatically Controlled Water Bath**:
   - Capable of maintaining the required test temperature (typically 60°C).
5. **Mechanical Roller**:
   - To rotate the bottles at a constant speed (approximately 60 rpm).
6. **Thermometer**:
   - Accurate to ±1°C.
7. **Weighing Balance**:
   - Accurate to ±0.1 g.
8. **Drying Oven**:
   - For drying the aggregates.
9. **Rolling Bottle Adhesion Testing Report documentation**:
   - For visual inspection or further quantitative analysis.

3.3.2. Procedure

3.3.2.1. Preparation of Aggregates

1. **Drying**:
   - Dry the aggregates in an oven at 110°C until a constant weight is achieved.
   - The aggregate sample G1 (Dolerite) was allowed to cool to room temperature.
2. **Weighing**:
   - A specified amount by weight of aggregates was taken and the sample weight was recorded.

3.3.3. Preparation of Bitumen

1. **Conditioning and Coating**:
   - The two bituminous prime samples (C55 and MC30) were conditioned over the aggregate. The conditioning process was taken to ensure proper coating of the aggregates.

3.3.4. Coating of Aggregates

1. **Coating**:
   - The heated bitumen was added to the dried aggregates in a suitable container.

- This was then mixed thoroughly to ensure a uniform coating of bitumen on the aggregate particles.

2. **Cooling**:
   - The coated aggregates were allowed to cool to room temperature.

3.3.5. Rolling Bottle Test Requirements

1. **Filling Bottles with necessary materials**:
   - A specified amount (mass by weight) of coated aggregates was placed into each rolling bottle.
   - A specified volume of distilled water (typically 200 ml) was added to the bottles.
2. **Sealing**:
   - The bottles were sealed tightly to prevent leakage during the test.
3. **Conditioning**:
   - The bottles were conditioned in a thermostatically controlled water bath set at 60°C for a specified duration (typically 24 hours) to condition the samples.
4. **Rolling**:
   - After conditioning of the samples, the bottles were placed on the mechanical roller.
   - The bottles were then rotated at a constant speed of 60 rpm for a specified duration (of 72 hours).

3.3.6. Assessment

1. **Visual Inspection**:
   - After the rolling period, the bottles were carefully opened, and loose material was collected. See Figure 5
   - The coated G1 aggregate sample was inspected for signs of stripping or loss of bitumen adhesion during the mechanical rolling stage.

3.3.7. Results and Interpretation

A  B. 

Figure 5: Visual Investigation of the adhesion of the prime products: A. C55 Emulsion Prime and B. MC30 Cut-Back Bitumen Prime

- **Visual Inspection**:
    - A Record of the observations regarding the condition of the bitumen coating on the aggregates was done to account for signs of stripping and loss of adhesion between the binder and the aggregate.

It was observed that the C55 had a surface coating of 64% on the overall sample. The MC30 was observed to have a surface coating of 4%. The MC30 sample was also observed to have aggregate clumps. The emulsion was stripped from the aggregate surfaces and clumped together (Figure 4). The C55 has a higher propensity for adhering (binding) to the aggregate sample when compared to the MC30. The Rolling Bottle Test provides valuable information on the adhesive properties of bitumen to aggregates under dynamic conditions. By assessing the degree of stripping of the Bitumen products on the aggregates based on bond strength, this test method provided a detailed basis for evaluating bitumen-aggregate bonding in the laboratory and also field performance (anti-stripping).

3.4 Zeta Potential Test:

Furthermore, it was essential to correlate the nature of adhesion developed between the two bituminous interlayer bond products, the Zeta-Potential test procedure was further recommended to provide a verification of the results of adhesion of the Bituminous product to aggregates. Zeta potential is one of the key indicators of the electrostatic interactions between particles in a suspension. This was used to assess the adhesion properties of the C55 and MC30 Prime on aggregate samples, this was essential to reflect the stability and behaviour of the bitumen-aggregate interface. The measurement of zeta potential provided understanding the compatibility and bonding efficiency between the bitumen products and aggregates, which is also a crucial factor to determine the durability of asphalt pavements.

Zeta potential provides an indication of the electrical potential on surfaces at the slipping plane. This plane is usually the interface which separates mobile fluids from fluids that remain attached to the surface of another medium in this case bitumen prime (C55 and MC30) and aggregate medium. Zeta potential is a scientific term for electrokinetic potential (Ref) in colloidal dispersions. In the colloidal chemistry literature, it is usually denoted using the Greek letter zeta (ζ), hence ζ-potential. The usual units are volts (V) or, more commonly, millivolts (mV). The zeta potential is seen as the electric potential in the interfacial double layer (DL) at the location of the slipping plane of the aggregate relative to a point in the bulk fluid away from the interface. In other words, zeta potential is the potential difference between the dispersion medium and the stationary layer of fluid attached to the dispersed particle. The zeta potential for macroscopic solid surfaces of aggregates and bitumen binder (C55 and MC30) at different pH levels were measured using the SurPASSTM 3 device. The SurPASSTM3 device for zeta potential analysis of macroscopic solid surfaces is shown in Figure 6. SurPASSTM3 series was used to analyze the samples in this study.

Figure 6: SurPASSTM 3 series (Anton Paar)

The zeta potential definition by Luxbacher, 2014 is the charging behaviour at a solid-liquid interface that can be generated by either acid-base reactions of functional groups and/or the adsorption of ions. Usually, it is observed that the zeta potential is determined by the measurement of an electrokinetic effect generated by a tangential flow of liquid across the solid surface in this case the aggregate material (G1-Dolerite), the method is referred to as the streaming potential technique.

The surface potential (bond strength on aggregates) was determined as a function of pH in a 0.001 Potassium chloride (KCl) electrolyte solution. The varying of the solution pH was done by addition of 0.05 mol/l of sodium hydroxide (NaOH) solution prepared by dissolving a 0.5 ± 0.1 g of NaOH in 250 ml of deionised or ultra-pure water. A 0.05 mol/l of hydrochloric acid (HCl) solution was prepared by mixing 1.21 moles of HCl of 32% purity in 250 ml of deionised or ultra-pure water through the instrument automatic titration unit. Measurements for zeta potential were carried out at each pH point. Streaming potential measurements were conducted using a cylindrical cell filled with an aggregate sample that has been mounted between support disks and filters (with 25 µm mesh) on both sides of the granular sample plug. An electrolyte solution was passed through the aggregate sample. The aggregate samples were cautiously washed with sodium chloride (NaCl) electrolyte solution to exclude particle sizes less than 25 µm before conducting the test. The permeability index was monitored and adjusted to fit a range of 85 – 115 to acquire accurate zeta-potential readings. A pressure range of 100 mbar – 400 mbar was then applied between both ends of the aggregate sample plug. The bitumen sample was measured using a gap cell and followed a similar procedure as used for aggregates samples, with the same electrolyte solution. The equipment pH electrode was calibrated at three buffer standard solutions of known pH value (pH 4, pH 7 and pH 10). The conductivity electrode was calibrated using a 0.1 mol/l KCl solution or a conductivity standard solution. Electrokinetic techniques was used to determine the measurement of the zeta potential of the aggregate particles and the Bituminous Primes as a function of the pH of the electrolyte. A representation of the zeta potential vs pH curve with three separate pH regions that characterize the aggregate surface chemistry under different aqueous conditions is shown in Figure 7.

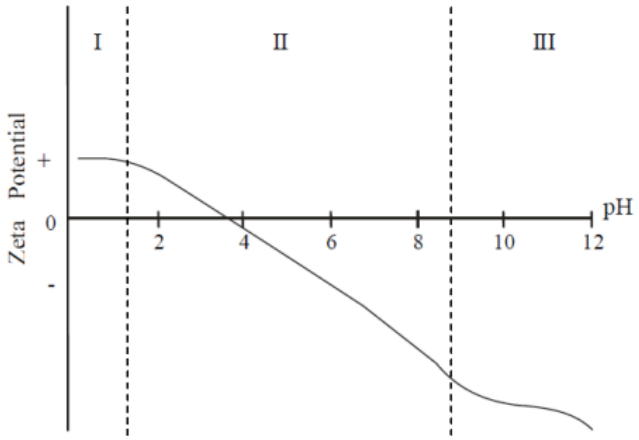


Figure 7: A characterization of aggregate surface chemistry pH regions (Labib et al., 2007)

The representation of the test results indicates that a relationship is established on the rate of adhesion between aggregates and binders by correlating its behaviour with fundamental relationship between zeta potential, isoelectric point and acid base protons transfer properties with increasing pH levels. For this test, the zeta potential was performed on the aggregate and bitumen samples at various pH levels. The relationship between the zeta potential and pH of the aggregates is shown in Figure 8.

Aggregates	
pH	**Zeta Potential (mV)**
3,46	-4,73
3,85	-4,54
4,66	-4,99
5,68	-5,48
6,69	-5,95
7,55	-6,37
8,31	-6,49
9,05	-6,02
9,63	-5,91
10,69	-14,00

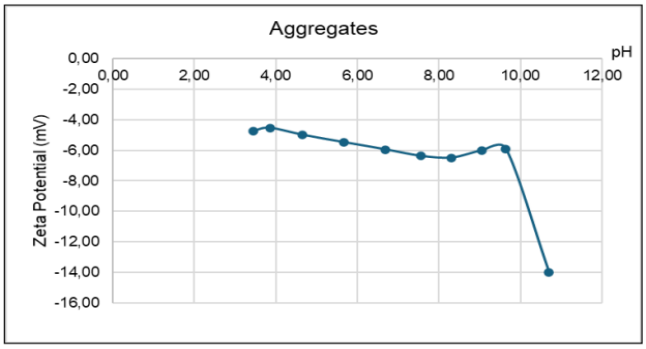


Figure 8: Relationship between the Zeta potential and pH of the aggregates

C55	
pH	**Zeta potential (mV)**
3,33	4,55
4,15	0,39
5,17	-4,08
6,27	-6,83
7,23	-9,80
7,99	-12,24
8,91	-14,02
9,98	-15,20
10,97	-16,75

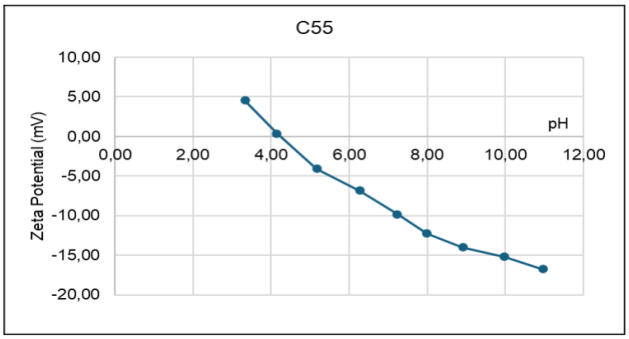


Figure 9: Relationship between the Zeta potential and pH of the C55 Emulsion Prime

MC30	
pH	**Zeta Potential (mV)**
3,50	-2,34
4,24	-2,10
5,26	-10,86
6,35	-15,44
7,33	-20,14
8,18	-20,46
9,13	-20,41
10,10	-19,23
11,07	-19,31

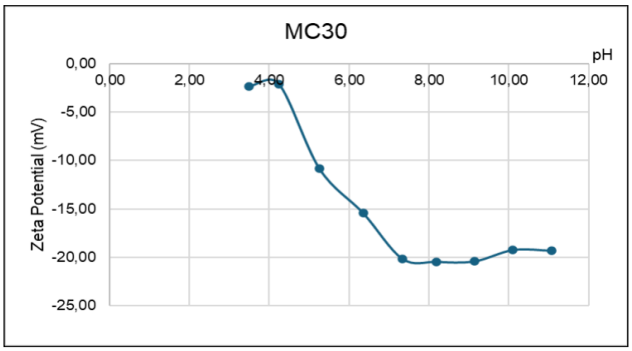


Figure 10: Relationship between the Zeta potential and pH of the MC30 Prime

Furthermore, the relationship between the Zeta potential and pH of the Bituminous products are shown in Figures 9 and 10 respectively. From the graphs, it is observed that the aggregates showed decreasing trend in Zeta potential with increasing pH from -4.73 to -14.00. Consequently, the Bituminous products showed differential behaviours in pH levels. The MC30 showed negatively charged surface with increasing pH levels from -2.34 to -19.31. The C55 Emulsion Prime showed a positively charged surface levels from +4.55 to –16.75. This relationship indicates that since the C55 Emulsion has more positively charges it has increased potential to adhere and bond with the aggregates compared to the MC30 Prime without stripping. This thus indicated that the formulation in the C55 Emulsion Prime has anti stripping agents that ensures improved adhesion of the bituminous Prime to aggregates. The Hefer (2004) dissertation stated that bitumen conventionally has a net acidic character, where function groups such as carboxylic acids will act as proton donors. These proton donors tend to form more durable bonds with strong protons accepting aggregate surfaces. Thus, there is a higher susceptibility for proton accepticity between the C55 Emulsion prime and the aggregate material which has the potential to form stronger bonds compared with the MC30 prime.

3.5 Bituminous Penetration Power Test (EN12849):

3.5.1 Determination of penetration power of Bituminous emulsions

Two samples of silica sand type (Silica No2 and Amber White Silica) were obtained from Greens Sands material supply quarry in Pretoria, Gauteng Province of South Africa. The samples were then placed in separate pans and put in the oven to be dried at a temperature of 110^0C. This was then allowed to cool. After cooling, the two samples were placed in a flat pan and allowed to roll for 3 hours (refer to Figure 2). The two samples of (50,0 ± 0,1) g of the sand was mixed intimately until a homogenous mix was attained. The homogenous mixture was then transferred via a funnel to the upper part of the test apparatus. Figure 11 A and B.

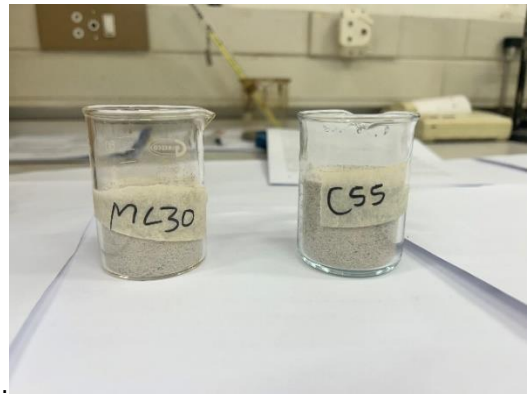
A.  B. 

Figure 11: A-. Sample of Silica Sand placed in pan to cool. B-. Sample of Silica Sand placed in test apparatus

.

The mixture was levelled at the surface by knocking the bottom of the apparatus three times on to a wooden board until a uniform surface level was reached.

Two samples each of (10,0 ± 0,1) g of C55 emulsion and MC30 were each poured at standard room temperature into the weighted masses of silica sand.

During the poring process the timer was used to record as per EN12849 procedure penetration time from the time of initial pouring of the C55 emulsion and MC30 onto glass apparatus until the emulsion samples weights were attained.

The two bituminous samples containing of C55 emulsion and MC30 with both silica sand was left on a levelled table to penetrate through and the times for penetration recorded. The figure 12 shows the placement of the samples into the test apparatus.

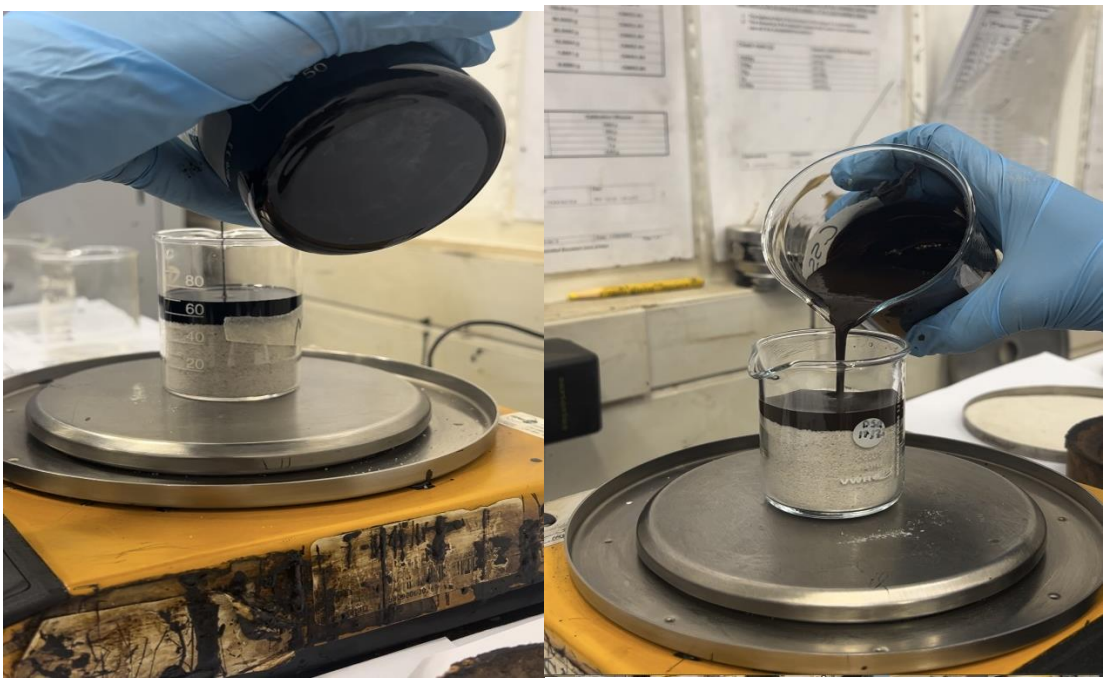


Figure 12: Sample of Silica Sand placed in pan to cool. B. Sample of Silica Sand placed in test apparatus

After placing to a levelled platform and allowed to penetrate through the homogenous sand mixture, the figure 13 - 14 indicates the visual representation of the effective penetration times for each sample. Three samples each was carried out using this procedure and the mean result is presented in the Table 2 and table 3.

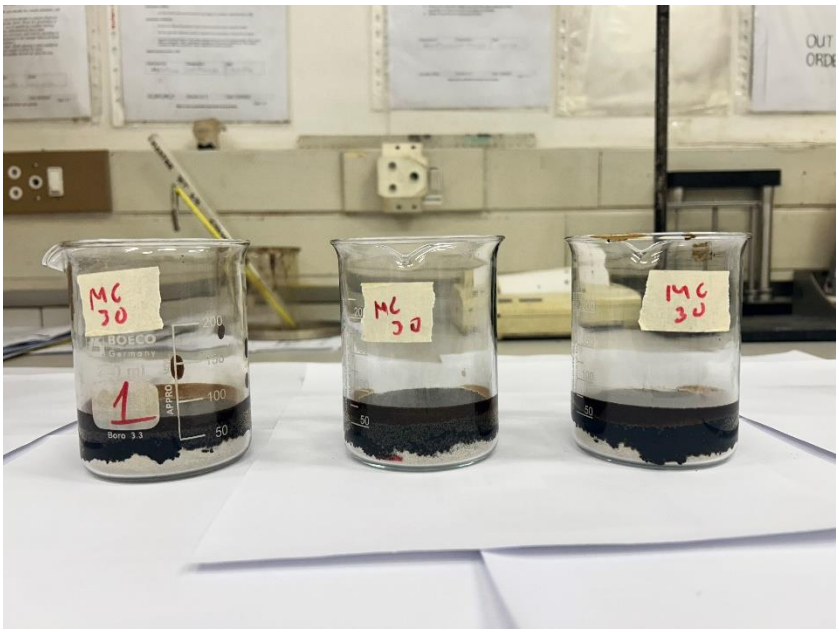


Figure 13: MC30 Sample after test

.

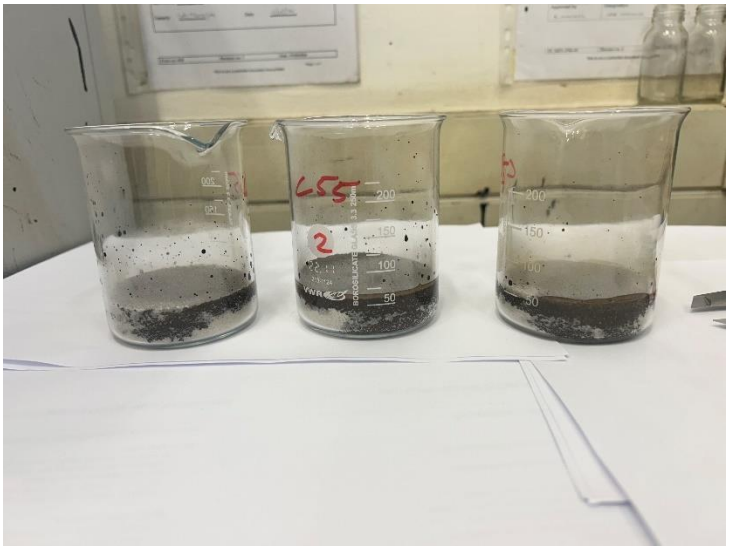


Figure 14: C55 Sample after test.

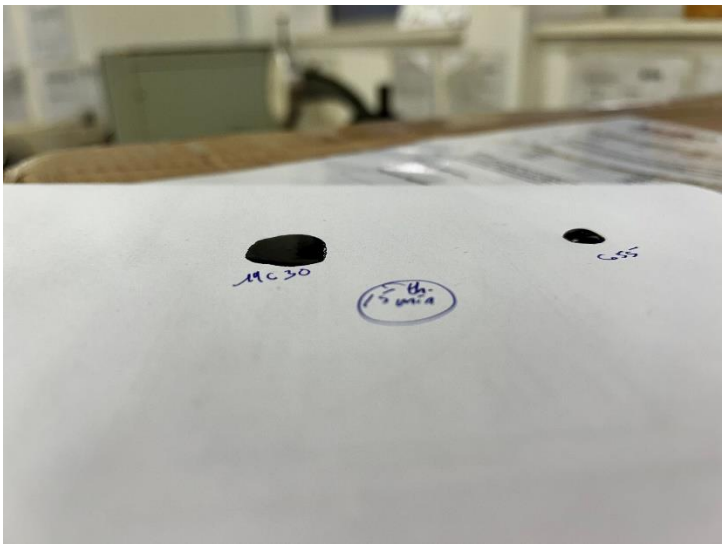


Figure 15: Binder Sample drop test for Prime curing rate.

Figure 15 displays a drop test regarding the spread rate and curing rate for a drop of each prime sample. Although, this method does not give a detailed procedure or with any test method, a thin spatula was used to collect a drop of each of the samples (C55 and MC30) and allowed to drop on a clean white paper. The essence of this test was to observe the rate of stickiness and spreading rate of the sample while its being cured under standard ambient temperature. It was observed that the MC30 sample being an oil-based prime had a wider spreading rate over the paper compared to the C55 sample at the 15th Minute. This C55 sample on the other hand maintained its drop form and began to cure immediately. After a set time of 15min, the C55 was non-sticky while the MC30 remained sticky beyond 24hours.

Table 2: Detailed test report and sample description for MC 30 penetration power

Test Description	MC30	MC30	MC30
Mass of Silica Sand mix (g)	100.035	100.016	100.027
Mass of Bituminous Material (g)	10.016	10.006	10.020
Maximum penetration depth (mm)	9.95mm	9.86mm	13.47mm
Average Penetration depth (mm)	11.09mm		
Penetration time (t)	8 min 12sec	9 min 52 sec	10 min 5sec
Difference Penetration time (s)	0sec	1min 40 sec	2 min 38 sec
Average Penetration time (s)	9 min 38 sec		

Table 3: Detailed test report and sample description for C55 penetration power

Test Description	C55	C55	C55
Mass of Silica Sand mix (g)	100.019	100.002	100.026

Mass of Bituminous Material (g)	10.010	10.016	10.040
Maximum penetration depth (mm)	12.47mm	14.17mm	13.33mm
Average Penetration depth (mm)	13.32mm		
Penetration time (t)	9 min 05sec	10 min 57 sec	13 min 10sec
Difference Penetration time (s)	0sec	1min 52 sec	2 min 53 sec
Average Penetration time (s)	11min		

Furthermore, when mixing the bituminous binder with distilled water using a ratio 1:1 on the C55. Thus (5,0 ± 0,1) g distilled water was mixed with (5,0 ± 0,1) g C55 emulsion.

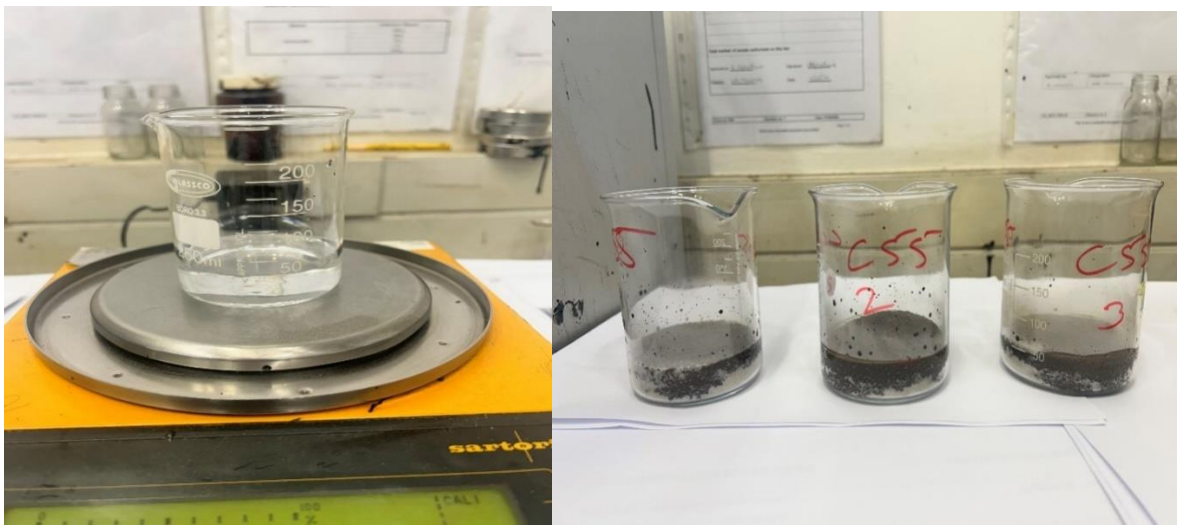


Figure 16:C55 and Distilled water (1:1): Binder penetration Sample after test.

It was observed that the penetration power improved tremendously within seconds of its application to the Silica Sand sample. The results are as displayed in the Table 4.

Table 4: Detailed test report and sample description for C55 penetration power

Test Description	C55	C55	C55
Mass of Silica Sand mix (g)	100.10	100.046	100.061
Mass of Bituminous Material (g)	10.061	10.012	10.048
Maximum penetration depth (mm)	14.41mm	15.64mm	15.21mm
Average Penetration depth (mm)	15.09mm		
Penetration time (t)	39sec	48 sec	31sec
Difference Penetration time (s)	0sec	+9 sec	- 8 sec
Average Penetration time (s)	39.33 sec		

From the Bituminous power penetration test procedure, it was observed that although the MC30 prime had a faster penetration time compared to the C55 emulsion prime, the penetration depth of the C55 emulsion prime was more than the penetration depth of the MC30 into the Silica Sand sample by 2.23mm. Furthermore, the dropping test visual experiment performed was used to represent the curing rate of the sample. The C55 sample drop was non-sticky when touched under 20mins while the MC30 sample remained sticky beyond 24 hours. Thus, the C55 emulsion prime has a higher propensity for penetrating into Silica samples faster and remain non-sticky when compared to the MC30 in accordance with the EN12849 procedure.
In addition, using a mixing ratio of the C55 with distilled water (1:1), the penetration time decreased which resulted to improved penetration depth. The material was also able to set and break within 60 mins of its application to the Silica Sand mix. This thus gives a good representation of its effectiveness for a fast-track construction process with reduction in construction delay times for curing as well as its applicability to wet surfaces or during raining seasons.

4. Conclusion

Based on the test results, C55 Emulsion Prime demonstrates superior interlayer bond strength compared to MC30 Cutback Bitumen Prime in wet regions or when dry aggregate condition is not feasible. The C55 Emulsion prime has a rapid curing rate within 60mins of application to a wet surface making it an efficient protective layer over granular surfaces to adhere and form a good bond strength property with the asphalt layer. This superiority is evident in both the direct shear test, rolling bottle tests, penetration power test and Zeta Potential test making C55 Emulsion Prime a more reliable choice for enhancing pavement performance in accordance with environmental friendliness and climate change sequences. Furthermore, the adhesive strength of the emulsions can be determined by identifying the isoelectric point (IEP). IEP is the pH condition where the surface charge = 0 mV. The IEP of C55 is approximately at pH 4.1. The IEP of the MC30 has been extrapolated to be approximately at pH 2.1. Material where the IEP is at higher pH form the strongest bond because they have a high proton acceptability. It can be predicted that the C55 forms a stronger bond with the aggregate when compared to MC30.This consolidated technical report provides a comprehensive comparison of the interlayer bond strength of C55 Emulsion Prime and MC30 Cutback Bitumen Prime, offering valuable insights for pavement construction and maintenance. Previous study has indicated that more aggregate bonding can be achieved with other aggregate types (Nomlala, et al, 2022) using the C55 Emulsion Prime with Limestone, Dolomite, Andesite or Quartzite. The penetration power test conducted in accordance to EN12849 demonstrate that the C55 emulsion prime has improved effective penetration depth compared to the MC30 prime.

5. Recommendations

- **For fast-track Improved Pavement Performance interlayer bond on wet/moist granular surface without stripping**: Use C55 Emulsion Prime for applications requiring adequately strong interlayer bonding.

References

- ASTM|D2397_D2397M-20|EN-US Standard Specification for Cationic Emulsified Asphalt.
- American Society for Testing and Materials (ASTM) D907-2015: Standard Terminology of Adhesives. ASTM International. West Conshohocken. PA. USA
- EN 12697-1. Rolling Bottle Test method. **Test** methods for hot **bituminous** conglomerates—Part 11.
- EN 12849:2009 Bitumen and bituminous binders - Determination of penetration power of bituminous emulsions
- EN 13808, 2013 Bitumen and bituminous binders - Framework for specifying cationic bituminous emulsions.
- Hefer, A.W., 2004. Adhesion in bitumen-aggregate systems and quantification of the effects of water on the adhesive bond. Texas A&M University.
- Labib, ME, Hefer, AW & Little, DN, 2007. Surface Chemistry at the Bitumen-Aggregate Interface. Presented and published. Proceedings of the International Conference on Advanced Characterisation of Pavement and Soil Engineering Materials, Athens, Greece.
- Luxbacher, T, 2014. The ZETA guide: Principles of the streaming potential technique. Anton Paar GmbH, Austria p. 9-16 (2014).
- Nomlala, B., Mthembu, Z., Mturi, G. and Hefer, A., 2022. Bitumen-aggregate adhesion: A predictive study based on zeta potential analysis using the streaming potential technique. Southern African Transport Conference.
- Rolling Stones Test Method/ Rolling Bottle Test Method/ Stripping Test Method: BE-TMBINDER-7-2011. Council for Scientific and Industrial Research. Pretoria. South Africa.
- SANS 4001-BT4: 2016. Specifications for bituminous road materials. Part BT4: Cationic Road Emulsion.
- SurPASSTM3 Reference Guide. 2019 . Anton Paar. Anton Paar GmbH. Austria.

Appendix A

Material:	**Soil**		
Relative Dry density:	81%	Cohesion (kPa):	69,4
Saturation:	68%	Friction angle (deg):	50,7

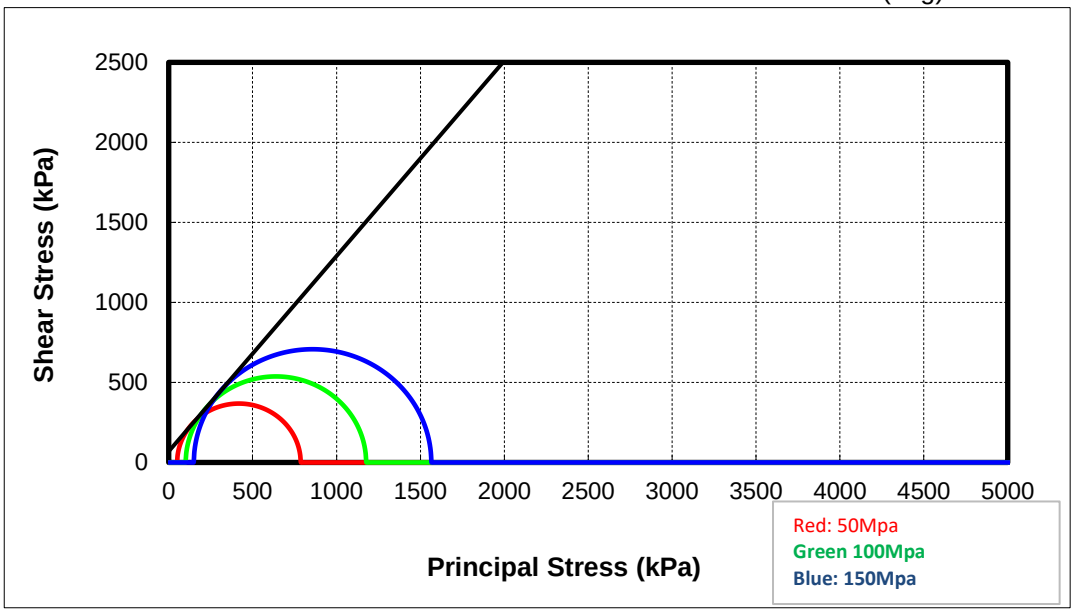


Figure 17: Direct Shear Results

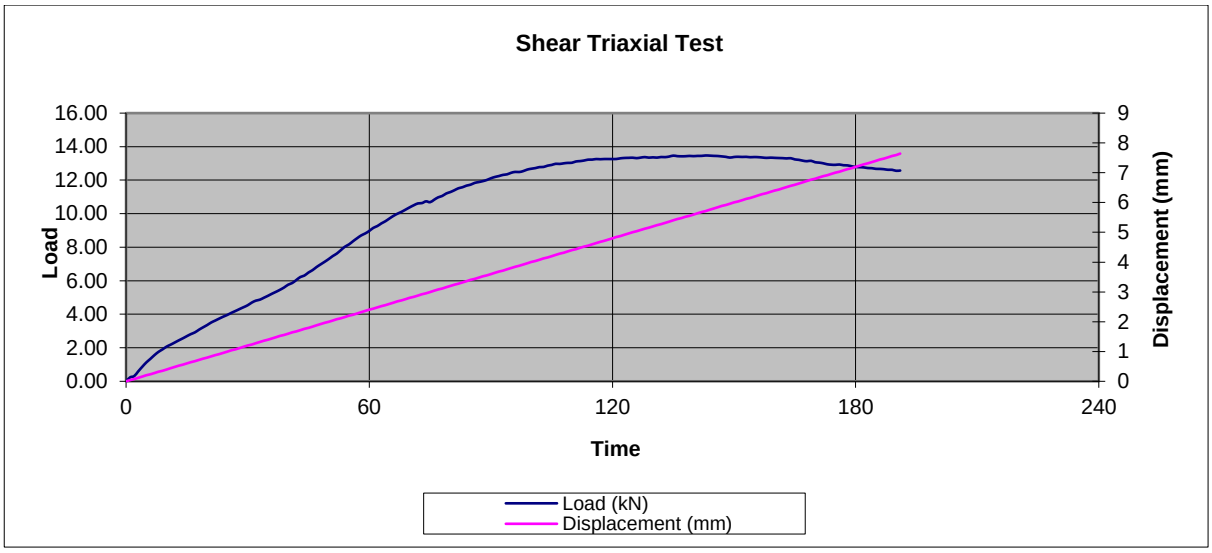


Figure 18: C55: Sample 1(50Mpa)

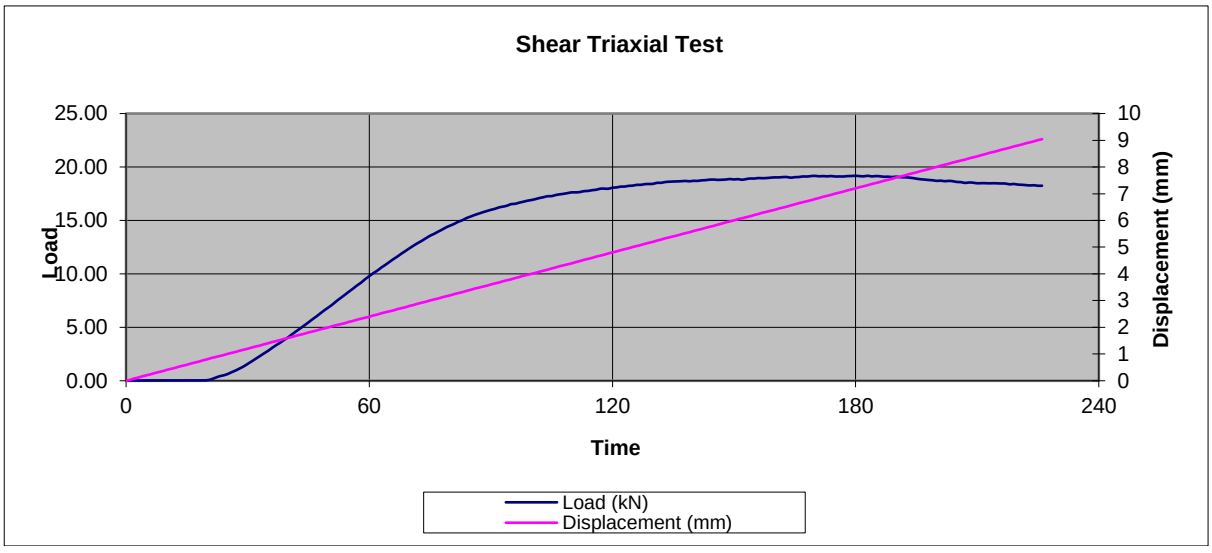


Figure 19: C55: Sample 2 (100Mpa)

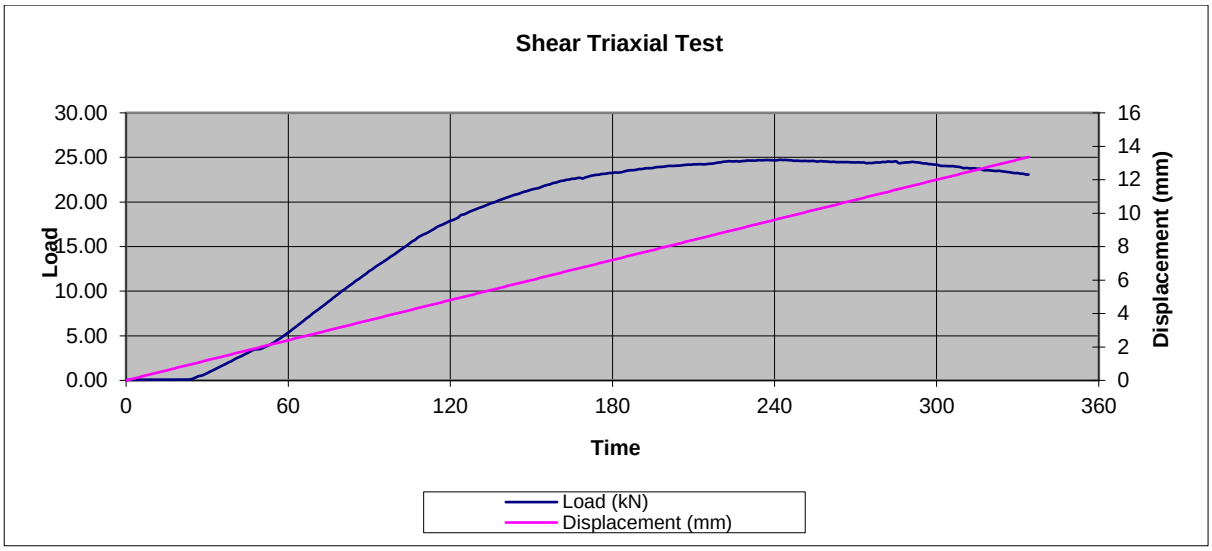


Figure 20: C55: Sample 3 (150Mpa)

Sample Details

Project description	C55 Bitumen Emulsion Prime	
Sample no (CSIR)	17497	
Sample description (Client)	XY530211	
Date sample received	10 April 2024	
Date sample tested	24 April 2024	
Sample condition	Sample is smooth without lumps or sedimentation	
Tests carried out	**Test**	**Test method**
	Saybolt Furol Viscosity @ 50°C	ASTM D88
	Binder Content, %	ASTM D95
	Fluxing Agent, %	ASTM 6997
	Residue on Sieving	SANS4001:BT4
	Particle Charge	ASTM D244-BT4
	Sedimentation	SANS4001:BT4
	Coagulation with Silica Flour	SANS4001:BT4
Sample preparation	Stirred by hand for 2 minutes and sieved through a 710µm sieve	

SIGNED: RConrad Name: 6 May 2024

SIGNED: Keneilwe Mphathiwa Mphathiwa Date: 06 May 2024
Laboratory Manager (Name / Signature)

Form no: R10b	Revision no: 10	Date: 14/03/2024

Figure 21: BT4 Test Results: C55 Sample

Sample Details

Project description	C55 Bitumen Emulsion Prime	
Sample no (CSIR)	17497	
Sample description (Client)	XY530211	
Date sample received	10 April 2024	
Date sample tested	24 April 2024	
Sample condition	Sample is smooth without lumps or sedimentation	
Tests carried out	**Test**	**Test method**
	Saybolt Furol Viscosity @ 50°C	ASTM D88
	Binder Content, %	ASTM D95
	Fluxing Agent, %	ASTM 6997
	Residue on Sieving	SANS4001:BT4
	Particle Charge	ASTM D244-BT4
	Sedimentation	SANS4001:BT4
	Coagulation with Silica Flour	SANS4001:BT4
Sample preparation	Stirred by hand for 2 minutes and sieved through a 710µm sieve	

SIGNED: BConrad Name: 6 May 2024

SIGNED: Keneilwe Mphathiwa Mphathiwa Date: 06 May 2024

Laboratory Manager (Name / Signature)

Form no: R10b	Revision no: 10	Date: 14/03/2024

The laboratory received a 5L C55 bituminous emulsion sample. The test results contained in this report are for the sample as provided by the client. The test results are shown in Table 1. BT4 Table is appended to show the SANS 4001- BT4:2014 for correlation of results.

Table 5: Testing as per SANS 4001-BT4: 2014 specification requirements

Table 1: Testing as per SANS 4001-BT4: 2014 specification requirements.

Property	17497 (XY530211)	Test Method or sub clause
Saybolt Furol Viscosity at 50°C, (seconds)	19	ASTM D244
Binder content (%)	56.17	ASTM D244
Fluxing agent content (%)	2.89	ASTM D6997
Residue on sieving (g/100ml) 710 µm 150 µm	 0.004 0.262	Clause 5.2
Particle charge a) Std procedure (10 mA) b) Modified procedure (50mA)	 Positive -	 ASTM D244 Clause 5.4
Binder deposit after 30min (g, min)	0.173	Clause 5.4
Sedimentation after 60 rotations	Passed at 55 Rotations	Clause 5.2
Coagulation with silica flour (% mass fraction of binder)	0	Clause 5.6

SANS 4001-BT4:2014
Edition 1

Table 1 — Type and grade requirements

1	2	3	4	5	6	7	8
Property	Type and grade requirement						Test method or subclause
	Spray type (CRS)			Pre-mix type (CMS)		Stable-mix type (CSS)	
	Grade						
	60	65	70	60	65	60	
Viscosity at 50 °C, Saybolt Furol seconds	15 to 50	51 to 200	51 to 400	20 to 50	51 to 200	50 max.	ASTM D244
Binder content[a], % (mass fraction of emulsion)	60 to 63	65 to 68	70 to 73	60 to 63	65 to 68	60 to 63	
Fluxing agent content[b], % (mass fraction of binder)	5 max.	5 max.	5 max.	5 to 10	5 to 10	Nil	
Residue on sieving, g/100 mL							5.2
710 µm	0,10 max	0,10 max.	0,10 max.	0,10 max.	0,10 max.	0,10 max.	
150 µm	0,25 max.	0,25 max.	0,25 max.	0,25 max.	0,25 max.	0,25 max.	
Particle charge							5.4 ASTM D244
a) Standard procedure (10 mA)	Positive	Positive	Positive	Positive	Positive	–	
b) Modified procedure (50 mA)	–	–	–	–	–	Positive	
Binder deposit on the cathode after 30 min, g, min.	1,0	1,0	1,0	–	–	–	5.4
Sedimentation after 60 complete rotations	Nil	Nil	Nil	Nil	Nil	Nil	5.2
Aggregate coating water resistance test	–	–	–	pass	pass	–	5.5
Coagulation value when mixed with standard silica flour, % (mass fraction of binder)	–	–	–	–	–	2,0 max.	5.6

[a] By difference from water content determined in accordance with method ASTM D244.

[b] In ASTM D244 "fluxing agent" is referred to as "oil distillate". ASTM D244 gives an approximate estimate of light fractions added to bitumen, thus enabling the emulsion binder to be characterised giving an indication of the permanent characteristics of the residual binder. If the CRS emulsion contains no fluxing agent, the type of emulsion should be succeeded by the letter "t" in brackets, for example, CRS 65(t).