INVESTIGATING SELF-HEALING BEHAVIOUR OF MICRO-CAPSULE INDUCED ASPHALT CONCRETE

FINAL YEAR PROJECT UG 2015

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2019

This is to certify that the

Final Year Project Titled

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has been accepted towards the requirements for the undergraduate degree

in

CIVIL ENGINEERING

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DEDICATION

To our Parents and Teachers who kept us motivating.

ACKNOWLEDGEMENTS

We would like to thank our supervisor Dr. Arshad Hussain for providing us the opportunity to work under his supervision and utilize the state-of-the-art testing facilities and equipment present in National Institute of Transportation in National University of Science and Technology. Special thanks to Mr. Hassan Farooq Afridi who constantly coordinated with us and helped us out of our problems. Special thanks to Mr. Hassan Farooq Afridi who constantly coordinated with us and helped us out of our problems.

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LIST OF ACRONYMS

NHA – National Highway Authority

OBC – Optimum Bitumen Content

RTFO – Rolling Thin Film Oven

SCB – Semi Circular Bending test

3PB – 3 Point bending test

UTM – Universal Testing Machine

VA – Air Voids

VFA – Voids Filled with Asphalt

VMA – Voids in Mineral Aggregate

RAP – Reclaimed Asphalt Pavement

TGA – Thermogravimetric Analyis

AC – Asphalt Concrete

ASTM – American Society for Testing and Materials

HMA – Hot Mix Asphalt

ITS – indirect Tensile Strength

TSR – Tensile Strength ratio

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ABSTRACT

Binders rheological properties are affected due to several factors including UV radiation, oxidation, temperature, moisture damage and traffic loads. This leads to pavements more prone to damage. Encapsulated rejuvenator technique is used to enhance the self-healing ability of asphalt mix. When crack appears, capsules ruptures and releases rejuvenator, which diffuses into crack and heals it. In this research, Ca-Alginate capsules containing waste cooking oil with changing oil/water ratios were prepared. The thermogravimetric analysis was done to analyze thermal stability of capsules in asphalt mix . Moisture susceptibility test was conducted to confirm the water sensitivity of capsules. To illustrate the benefits of encapsulated rejuvenator on the pavement engineering, the self-healing behavior of capsule-induced self-healing asphalt was investigated by Semi Circular Bending Test (SCB) and 3 Point Bending Test. Cracked asphalt mixture recovered 40 % of intial peak load at 25°C versus 14% of asphalt mixture without capsules. 3 Point Bending test was performed at four different temperature. Samples with capsules recovered 53% of initial strength in comparison to 11% of samples without capsules.

CHAPTER 1

INTRODUCTION

1.1 Background

Asphalt pavement deteriorate during service due to external factors like oxidation, ultraviolet radiations, moisture damage and traffic loads which increases the stiffness of pavement by affecting the binders rheological properties. Ageing, thus makes pavement more prone to damages such as cracking and fretting (Hagos, 2008). Ageing of binder has serious financial implications for county's economy. National highway is facing budgetary constraints for maintenance and rehabilitation of pavements amidst deteriorating quality of roads due to excessive traffic loads. As a result, only 7% roads are in good condition, and 26% are in poor or very poor condition.

Although the ageing reduces the quality and performance of flexible pavement, several techniques have been used to counter the effect of ageing. Asphalt rejuvenation is a promising technique using industrial rejuvenator and waste oils to restore the lost properties of bitumen and increase the life of pavement. 100 % RAP in pavement recycling has been achieved by using asphalt rejuvenators **(**Zaumanies et al., 2014).

Rejuvenators used in surface course rejuvenation have favorable outcomes but it has a disadvantage. It only reaches top 2 cm pavement depth; this means that internal cracks and deterioration is not impacted by this method. These technique calls on for use of onsite work, which brings about traffic congestions and unfavorable environmental impacts.

Snowballing traffic loads have worsened road conditions by decreasing pavement performance and restricting the opportunity for road maintenance and rehabilitation due to limited time available for maintenance while keeping traffic delay at a tolerable minimum level. Drawbacks of using rejuvenators can be overcomed by use of a promising technique of encapsulated rejuvenator.

1.2 Problem Statement

The natural self-healing rate of bitumen is not sufficient to deal with the deterioration rate of asphalt pavement. This requires use of external aid in form of rejuvenator or to speed up the process. Using a low-cost rejuvenator in encapsulated form is an alternate to traditional pavement which uses a waste resource to produce a self-healing pavement.

Cooking oil is one the essential item of every household and restaurant, that is why, imports of cooking oil in Pakistan amount to staggering 2.6 million ton per annum making Pakistan 3rd largest importer of cooking oil in world. In Pakistan, cooking oil is being used and sold by hotels to local vendors.

This oil is then used by local restaurants owner and further travels down the hierarchy and goes to street food makers. They use it and then dump it on ground or in water, which causes land and water pollution. This waste cooking oil can be collected from vendors and processed in centrifuge to obtain a particle and solid waste free cooking oil that can be used as a rejuvenator to produce sustainable, life long, self-healing pavements.

1.3 Research Objectives

1. To Produce and Characterize capsules based on their temperature susceptibility.
2. Evaluate thermal stability of capsules
3. Evaluate the self-healing property of asphalt mixtures using Semi Circular Bending test and 3 Point Bending test.

1.4 Research Methodology

Phase 1: Literature review and collection of chemicals for capsules manufacturing, capsule production and characterization using thermogravimetric analysis.

Phase 2: Moisture Susceptibility test of Marshall samples with and without capsules.

Phase 3: Self-healing investigation of asphalt samples using SCB test and 3 Point Bending test.

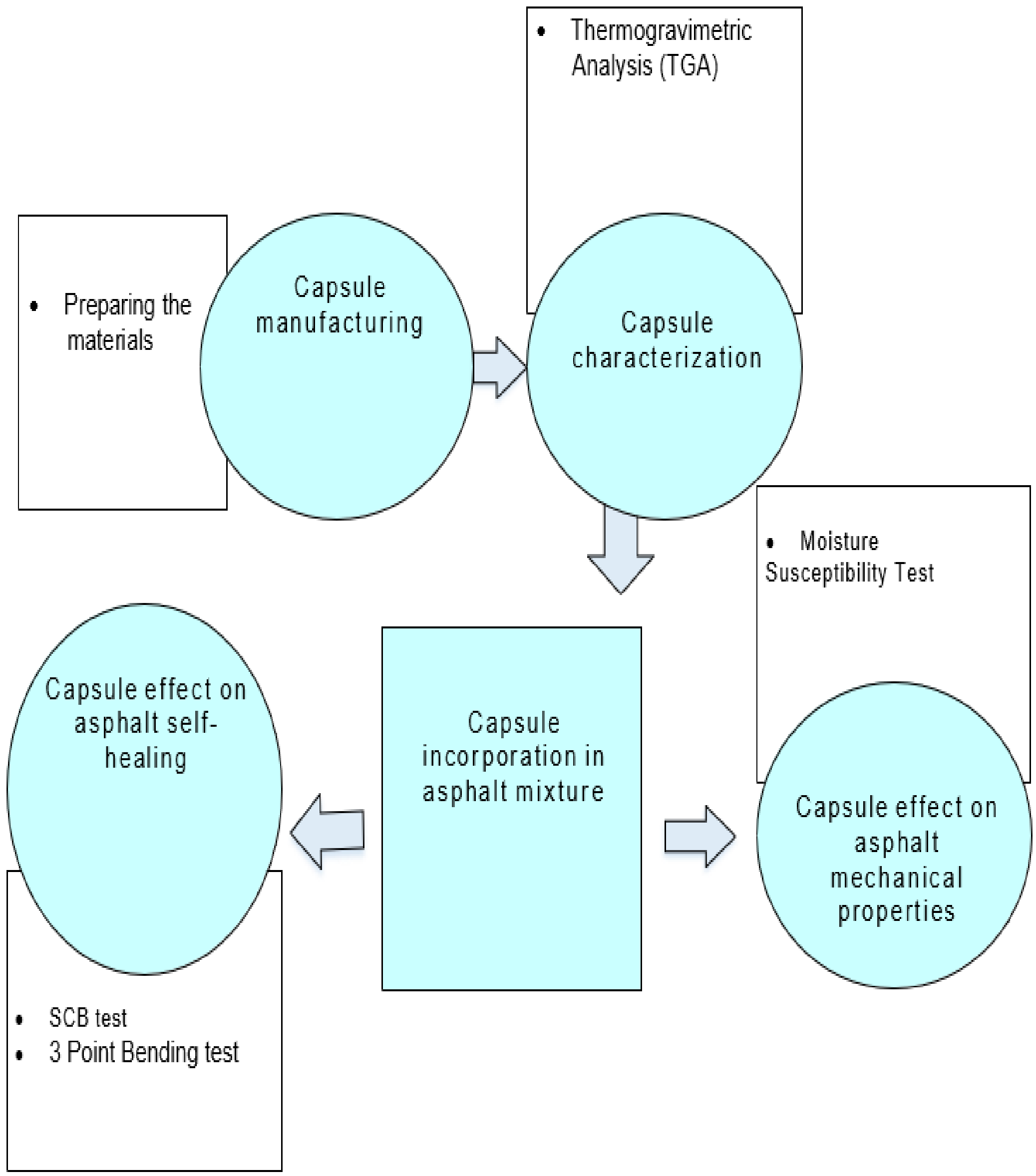

Figure 1.1: Elements of the Research Methodology

1.5 Report Outline

Chapter	Description
Chapter 1	An overview and general background information about asphalt pavement maintenance, bitumen ageing and asphalt self-healing. This chapter also includes the problem statement, research objectives as well as the research methodology.
Chapter 2	This chapter provides an overview, self-healing materials, the general concept of encapsulated rejuvenators, diffusion of rejuvenators inside an asphalt pavement, asphalt self-healing by induction heating and asphalt self-healing by encapsulated rejuvenators.
Chapter 3	This chapter presents the materials and steps involved in capsule production and asphalt mixture preparation, testing of capsules and asphalt mixture testing.
Chapter 4	In this chapter, results of capsules and asphalt mixture testing are presented. Effect of capsules on mechanical and self-healing properties of asphalt mix capsule properties are presented.
Chapter 5	In this chapter, the main conclusions and recommendations for future research are presented.

CHAPTER 2

Literature Review

2.1 Overview

This chapter aims to provide suitable background into topic of self-healing asphalt by giving literature review of the topic. It describes the behavior of asphalt mixture in correspondence to chemical structure and ageing. It details the intrinsic self-healing and engineering self-healing efforts by previous researchers.

2.2 Bitumen

Bitumen is a viscoelastic material composed of different molecular composition with varied chemical structure; mainly carbon and hydrogen, called as hydrocarbon. Other structure is called as heteroatom structure composed of elements such as oxygen, Sulphur and nitrogen. (Wang, 2011). This gives a fine balance to asphalt's properties divided into two fractions; namely solid asphaltenes and liquid maltenes. Solid asphaltenes contribute to viscosity and stiffness while maltene subdivided into saturated, aromatics and resins give flexibility of bitumen. These properties can be tested through wide range of rheological tests. It is imperative to understand the properties of bitumen because behavior of asphalt pavement highly varies with change in properties of bitumen.

2.3 Asphalt Self-healing

Asphalt has natural self-healing abilty due to its intrinsic properties. Qiu (2012b) describes self-healing of bitumen as viscosity driven process, that functions on reinstating the bitumen properties by impeding crack growth in bitumen. Bhasin et al. (2009) named the tip of crack as fracture/healing zone. Figure 2.1 shows fracture zone. Self-healing of asphalt is time and temperature dependent activity and is also influenced by other physical factors. (Qiu et al., 2012b)

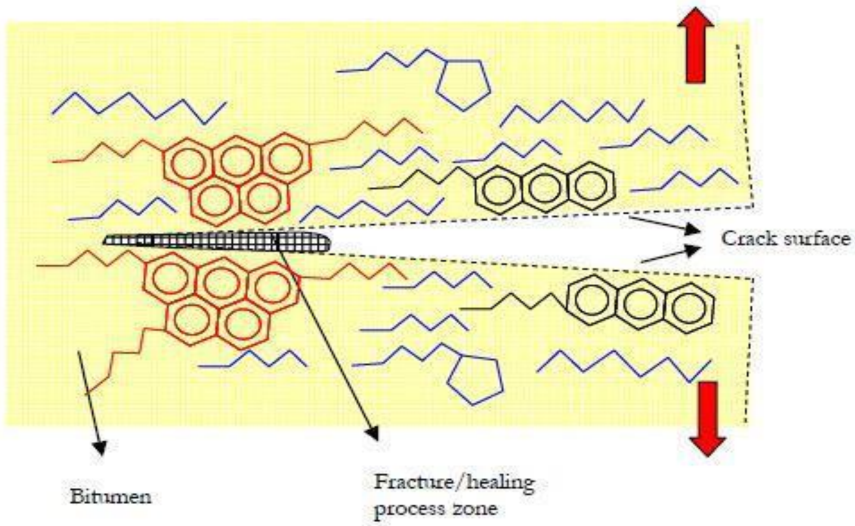

Figure 2. 1: Fracture/healing process of bitumen (Bhasin et al., 2009).

2.4 Cooking oil as a rejuvenating agent

Zargar et al. (2012) found that cooking oil can effectively rejuvenate aged binder due to its highly aromatic properties. Self-healing of bitumen binder is greatly influenced by aromatic content of rejuvenator. Ji et al. (2016) found that 6-8 % of cooking oil is optimum content to reduce aged binder stiffness and improved its rheological and physical properties.

2.5 Engineered self-healing techniques

Nature gives substantial evidence of self-healing phenomena. White et al. (2001) stated that encapsulated polymeric substance showed crack healing and upheld 75 % of its strength.

Following on, self-healing technology has been employed in metals, polymers, concrete and bitumen.

Lately, two new techniques have been advanced in literature that have promising future in pavement industry. They are: encapsulated rejuvenator and Induction heating mechanism.

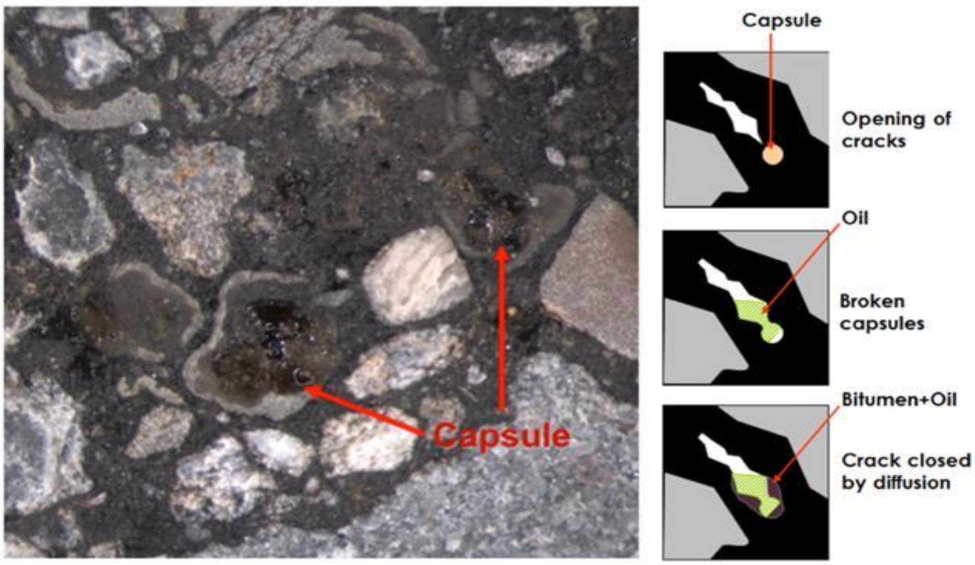

Figure 2. 2: The mechanism of induction heating(García et al., 2011c).

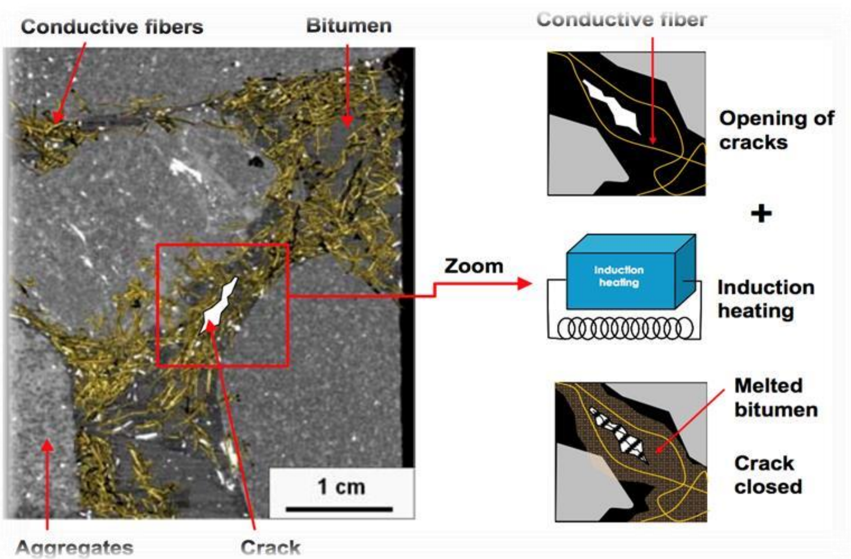

Figure 2. 3: Encapsulated rejuvenator healing mechanism (García et al., 2010b).

2.6 Encapsulated Rejuvenator

Garcia et al. (2010) used porous sand capsules containing rejuvenator to study healing in asphalt mixture. Sun et al (2013) developed methanol modified melamine resin as shell and rejuvenator in core to evaluate properties of these microcapsules. Tabakovic et al. (2016) used alginate fibers to study self-healing effect of encapsulated rejuvenator. Ji et al. (2016) studied the optimum amount of vegetable cooking oil that can be used as asphalt rejuvenator to recover aged bitumen without affecting its desired physical and mechanical properties. The test was performed with five different cooking oil contents to recover aged asphalt binder. Shirzad et al. (2017) studied effect of encapsulated sunflower oil on performance of asphalt mixture. Sun et al. (2018) found out that microcapsules can improve healing of bitumen binder and asphalt mixture.

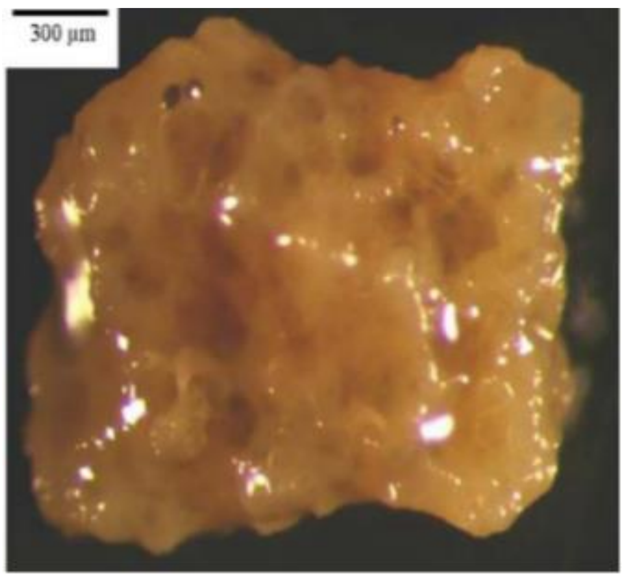

Figure 2. 4: Encapsulated rejuvenator in porous stone (García et al., 2010b).

CHAPTER 3

RESEARCH AND TESTING METHODOLOGY

3.1 Introduction

This chapter discusses the methodology adopted to achieve the objectives of the study which includes acquisition of material, testing of material, specimen preparation and various tests on specimen. The study was carried out on control sample as well as sample containing capsules. In this chapter, determination of OBC using Marshal Mix Design is discussed. Based on the OBC samples for performance testing will be prepared with and without addition of capsules. Performance testing includes Moisture Susceptibility and Healing Evaluation. The equipment used, the procedure used for the preparation of samples, along with the input parameters used during testing on the specimen prepared will be discussed in this chapter.

3.2 Research Methodology

Virgin aggregate was collected from Margalla hills crush plant site, and bitumen(60/70) was procured from PARCO. Chemicals including Sodium Alginate, Calcium Chloride and Deionized Water were obtained from DAEJUNG. Waste Cooking oil was acquired from local vendor. These materials were brought to laboratory and several tests were conducted on aggregate and bitumen. Capsules were prepared in laboratory. Thermogravimetric test was performed on capsules to ensure thermal stability of capsules at high temperature of mixing. After that Marshall specimen were prepared for finding OBC of samples. These OBC's were then used to prepare samples to perform Performance test on samples. Healing Evaluation was done using Semi-Circular bending test and 3 Point bending beam test.

3.3 Aggregate Testing

3.3.1 Aggregate

Aggregate was collected from Margalla hills crush site. Aggregate plays a vital role in defining the strength, durability of HMA pavements. It takes maximum load of pavement. These strength related properties of aggregate are greatly influenced by the texture and shape. Generally, more angular and rough textured the aggregate is more capable to resist stresses in pavement due to application of repeated loads. Several tests are performed per ASTM and BS to check properties of aggregate affecting pavement.

Figure 3. 1: Margalla Hills Crush Plant

3.3.2 Bitumen

In Pakistan, mostly bitumen of grade 60/70 is utilized per weather conditions. So bitumen of grade 60/70 was collected from PARCO.

3.3.3 Sodium Alginate & Calcium Chloride

Sodium Alginate & Calcium Chloride were bought from DEJUNG.

Figure 3. 2: Sodium alginate & calcium chloride

3.3.4 Waste Cooking Oil

Waste cooking oil was obtained from local vendor.

3.4 Aggregate Testing

3.4.1 Aggregate Tests

Aggregate resists deformation in pavements so it should have sufficient strength, texture so that it can withstand its purpose in pavement. Following tests were performed on aggregate.

- Shape Test.
- Specific Gravity and Water absorption test.
- Impact value of Aggregate.
- Los Angles Abrasion Test.

Three samples were prepared for each test and their result were compiled in the table below:

3.4.1.1 Shape test of Aggregate (ASTM D 4791-99)

This test determines the percentage of flaky and elongated particles in aggregate. For better interlocking of aggregate particles angular shape is preferred. The flakiness index should be less than 15% while elongation index less than 15%.

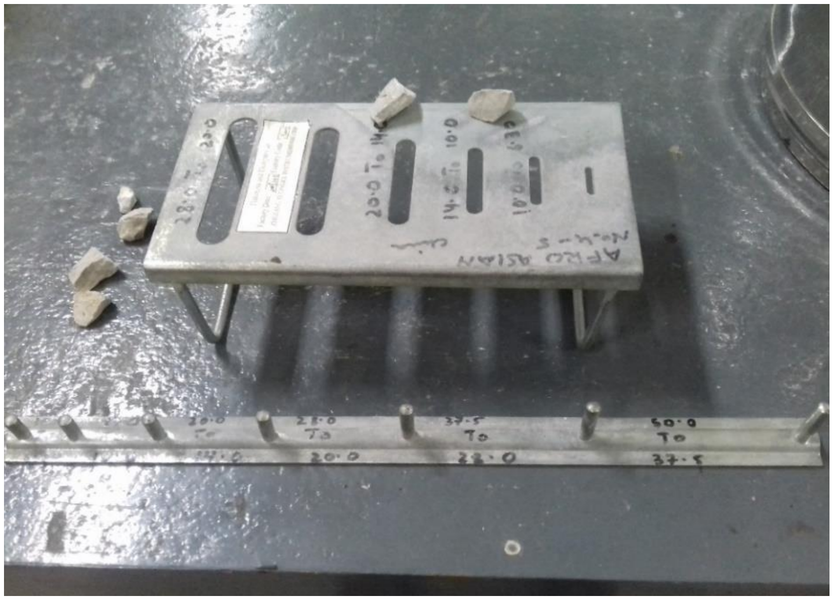

Figure 3. 3: Shape Test Apparatus

3.4.1.2 Specific Gravity Test (ASTM C 127 & ASTM C 128)

This test was performed only on coarse aggregate per ASTM C 127-88. Three weights were determined for calculating Specific gravity i.e. weight of oven dried aggregate, weight of aggregate completely submerged in water, and Saturated surface dry weight of aggregate. Specific Gravity of Fine aggregate and water absorption was determined using ASTM C 128.

Figure 3. 4: Specific Gravity Test

3.4.1.3 Impact Value of Aggregate (BS 812)

The impact value of aggregate gives its relative strength against impact loading. The equipment required was impact testing machine, temping rod, sieves of sizes 1/2”, 3/8” and #8 (2.36mm). About 350 grams of sample passing sieve ½” and retained on 3/8” was transferred impact testing machine cup in three layer while each layer was temped 25 times with the help of temping rod. After that it was subjected to 25 blows from rammer of impact machine weighing 14 kg and free fall of 38 cm. After that material was taken out from cup and passed through sieve #8. The percent passing through sieve #8 gives impact value of aggregate. The result are summarized in table below.

Figure 3. 5: Impact value Test Apparatus

3.4.1.4 Los Angles Abrasion Test (ASTM C 535)

This test is used to check resistance of aggregate to wear and tear due to heavy traffic load. More the abrasion value of aggregate more the performance of pavement is adversely affected. The equipment used was LOS angles machines, sieve set, balance steel balls. NHA Gradation B was selected for this test. About 5000g of sample, containing 2500g retained on sieve ½" and 2500g retained on 3/8", was placed in Los Angles Machine along with 11 steel balls. Rotation speed of drum was 30 rpm for 500 revolutions. After that material from machine was passed through 1.7mm sieve and weight (W2) of sample passing it was noted. The abrasion value is defined to be = W2/W1 *100

Figure 3. 6: Los Angles Abrasion Machine

Test	Specification		Result	Limits
Flakiness Index	ASTM D 4791		11.5%	≤15%
Elongation Index	ASTM D 4791		3.24%	≤15%
Aggregate Absorption	Fine	ASTM C127	2.13%	≤3%
	Coarse		0.69	≤3%
Impact Value	BS 812		18.5%	≤30%
Specific Gravity	Fine Agg	ASTM C128	2.617	-
	Coarse Agg	ASTM C127	2.633	-
LOS Angles Abrasion	ASTM C131		24.5%	≤45%

Table 3. 1: Test results of Aggregate

3.5 Test on Bitumen

3.5.1 Bitumen

Bitumen act as binder material in pavement and keeps aggregate in place. Bitumen is a semi-solid type material, behavior of which vary under different temperature and weather conditions. Performed tests are explained below.

3.5.1.1 Penetration Test (AASHTO T 49-03)

The penetration test is used to determine the penetration grade of bitumen by measuring the depth in tenths of a millimeter up to which a standard loaded needle will vertically penetrates the bitumen specimen under given conditions of loading, time and temperature. Softer binder gives greater values of penetration.

According to AASHTO T 49-03 temperature used was 25°C, load of 100 grams, while time for the test equal 5 seconds, until unless the situations are not explicitly stated. Using two ARL 60/70 specimens, five values from each specimen were taken after performing penetration tests. All values obtained fulfilled the required criteria of penetration test as per specifications. Penetration test result is presented in Table 3.3

Figure 3. 7: Penetration Test Apparatus

3.5.1.2 Softening Point (AASHTO-T-53)

For softening point determination of asphalt as per AASHTO-T-53 specifications ring and ball apparatus was used.

3.5.1.4 Ductility (AASHTO T 51-00)

Different samples of bitumen were tested and all gave the ductility value greater than the least limit of 100 cm.

Figure 3. 8: Ductility Test of bitumen

3.5.1.5 Flash and Fire point (D3143/D3143M-13)

Flash and Fire point test was conducted as per D3143/D3143M-13 standards.

3.5.1.6 Viscosity test (ASTM D 4402 – 06)

A rotational viscometer uses the concept of torque. It measures the torque required to rotate an object submerged in fluid. (Asphalt in this case) and relates it to the viscosity of the fluid. This test is performed per the test standard ASTM D 4402 – 06.

Figure 3. 9: Viscosity Test Apparatus

S No.	Test Description	Specification	Result
1	Penetration test@25°C	ASTM D: 5-06	68
2	Flash Point(°C)	ASTM:D-92	269
3	Fire Point(°C)	ASTM:D-92	297
4	Softening point(°C)	ASTM D 36 – 95	49
5	Ductility Test(cm)	ASTM-113-99	117
6	Viscosity Test(Pa.sec)	ASTM D 88 – 94	0.2533
7	Specific Gravity	ASTM D 70	1.02

Table 3. 2: Test results on Bitumen

3.6 Capsule Preparation and Testing

3.6.1 Introduction

Calcium alginate capsules containing waste cooking oil were prepared by ionotropic gelation of sodium alginate to increase the healing of asphalt mixtures. Thermal stability of these capsules is also tested.

3.6.2 Capsule Preparation

Capsules were made of a calcium-alginate polymer that encapsulated the waste cooking oil . The healing agent used in the capsules was a centrifuged waste cooking oil obtained from a local vendor for a very cheap price. Sodium alginate was in powdered form while calcium chloride was in form of pellets. To prepare capsules 50 g of oil and 550 g of water are mixed at 400 rpm for 1 min. Calcium chloride solution of 2.5% with water is prepared separately. Sodium Alginate emulsion is put into dropping funnel. Emulsion is dropped into calcium chloride solution. Capsules are formed by process known as ionotropic gelation. Calcium chloride solution is stirred during whole process. Capsules are decanted and put into oven at 40 °C for 24 hrs. Afterwards these capsules can be used in asphalt self-healing.

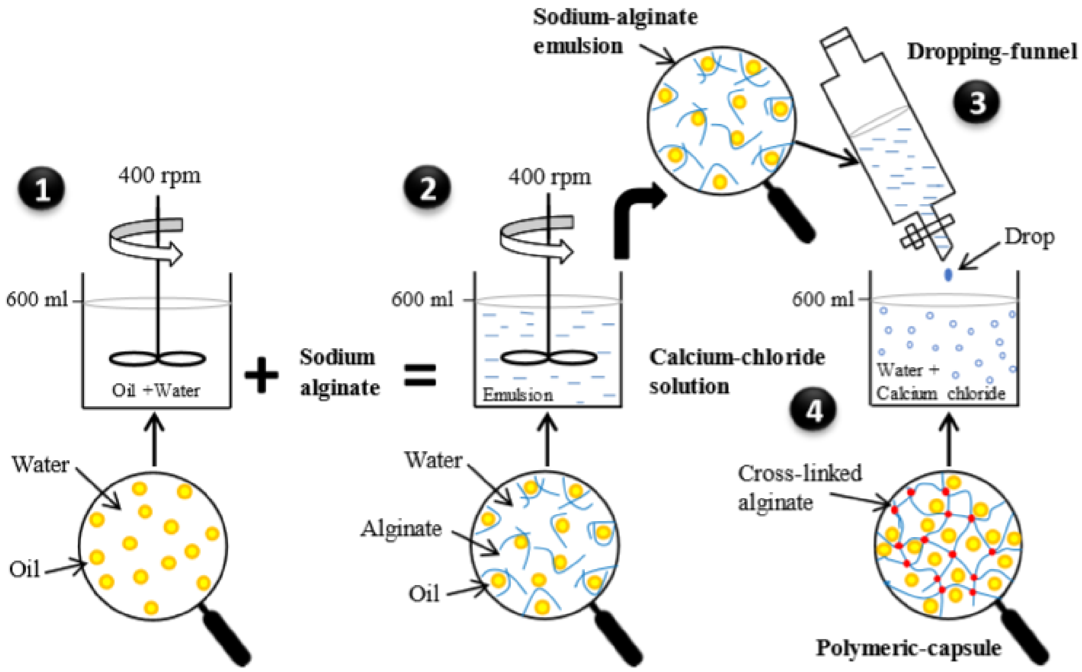

Figure 3. 10: Capsule Preparation

3.6.3 Capsule Testing

TGA test were performed on the capsule which is as follows:

3.6.3.1 Thermal Stability of Capsules

Asphalt mixing temperature is 160 °C, therefore capsules must be able to beat this temperature. TGA has been used to evaluate the thermal stability of capsules at high mixing temperature i.e 160 C. TGA results were graphed over a temperature range of 0-1000°C with increment of 10 °C/ min..

Figure 3. 11: TGA Test Apparatus

3.7 Asphalt Mixture Preparation

These samples were prepared as per Marshall Mix Design method in order to determine the optimum bitumen content (OBC). The Test matrix for determining OBC is shown in Table 3.

<table>
<tr><th colspan="3">Performance Testing Matrix</th></tr>
<tr><th colspan="3">Marshall Testing</th></tr>
<tr><th>Bitumen Content %</th><th colspan="2">No. of Samples</th></tr>
<tr><td>3</td><td colspan="2">4</td></tr>
<tr><td>3.5</td><td colspan="2">4</td></tr>
<tr><td>4</td><td colspan="2">4</td></tr>
<tr><td>4.5</td><td colspan="2">4</td></tr>
<tr><td>5</td><td colspan="2">4</td></tr>
<tr><td>5.5</td><td colspan="2">4</td></tr>
<tr><td></td><td>Total</td><td>24</td></tr>
</table>

Table 3. 3: Test results on Bitumen

3.7.1 Preparation of Materials for Mixing/Pre-Heating

Aggregate was sieved for different sizes and oven dried. Total weight of a sample of Marshall Mix is 1200g and weight of aggregate and bitumen varied according to percentage of binder in each sample. NHA gradation B is followed for aggregate specs.

Total Sample Weight = Weight of Aggregate + Weight of Bitumen

Figure 3. 12: Mixture Preparation

Figure 3. 13: Mixture Preparation

Prior to mixing of material, Bitumen and Aggregate were pre-heated in a temperature range of 110-120 ℃ in an Electric Oven.

Figure 3. 14: Pre-Heating of Aggregate and Bitumen

3.7.2 Mixing of Materials

Mixing was done at 120-150°C. Aggregates and bitumen/PMB were preheated and mixed thoroughly. Mixing was continued until all particles were well coated.

Figure 3. 15: Mixing of Material

3.7.3 Compaction of Sample

Number of blows are determined by type of traffic surface is being prepared for. For heavy traffic, 75 blows are given on each side for compaction.

Mold was oiled and filter paper was placed in it. Samples were then transferred to mold and given 75 blows on each side to achieve compaction using Marshall Compactor.

Figure 3. 16: :Compaction of Sample

Figure 3. 17: Marshall Samples

3.8 Determination of OBC

After the cooling of Specimen to room temperature the volumetric of specimen are calculated by determining G_{mb} and G_{mm} values. The tests for G_{mb} and G_{mm} are performed in accordance with ASTM D2726 AND ASTM D2041 respectively. For determination of G_{mb} firstly weight in air of specimen is determined, after which its weight in water and SSD weight are determined.

Figure 3. 18: Gmb Calculation for Marshall Samples

After the determination of G_{mb}, the specimen conditioned and then tested for Marshall Stability and flow using Marshall Equipment. After placing the sample in Marshall Apparatus, it is loaded at constant deformation rate of 5mm/minute until the specimen fails. The maximum load that the specimen takes is its Stability value and the strain that occurs at maximum load is recorded as flow number in mm. According to Marshal mix design Criteria MS-2, for surface designed for heavy traffic load should have Stability value not less than 8.006 KN and Flow should be between 2 to 3.5 mm.

For G_{mm} calculation weight the loose mix, then find the calibration weight of apparatus, after that transfer the mix to apparatus and apply vacuum. After the removal of air entrapped ari in mix weigh again the apparatus containing mix also.

3.8.1 Volumetric properties of mix

The flow and stability, volumetric properties of controlled mix are shown in table.

Asphalt%	G_{mb}	G_{mm}	Va (%)	VMA (%)	VFA (%)	Stability (KN)	Flow (mm)
3.5	2.35	2.508	6.09	13.419	54.6	11.973	2.25
4	2.368	2.489	4.86	13.4004	63.8	13.276	2.55
4.5	2.389	2.482	3.76	13.0951	71.3	11.879	2.74
5	2.394	2.467	2.97	13.36	77.8	8.954	3.548
5.5	2.401	2.448	1.92	13.56	85.8	8.315	4.08

Table 3. 4: Volumetric Properties

The graph between Asphalt content and different volumetric properties were plotted to find out OBC as shown in figure 3.27

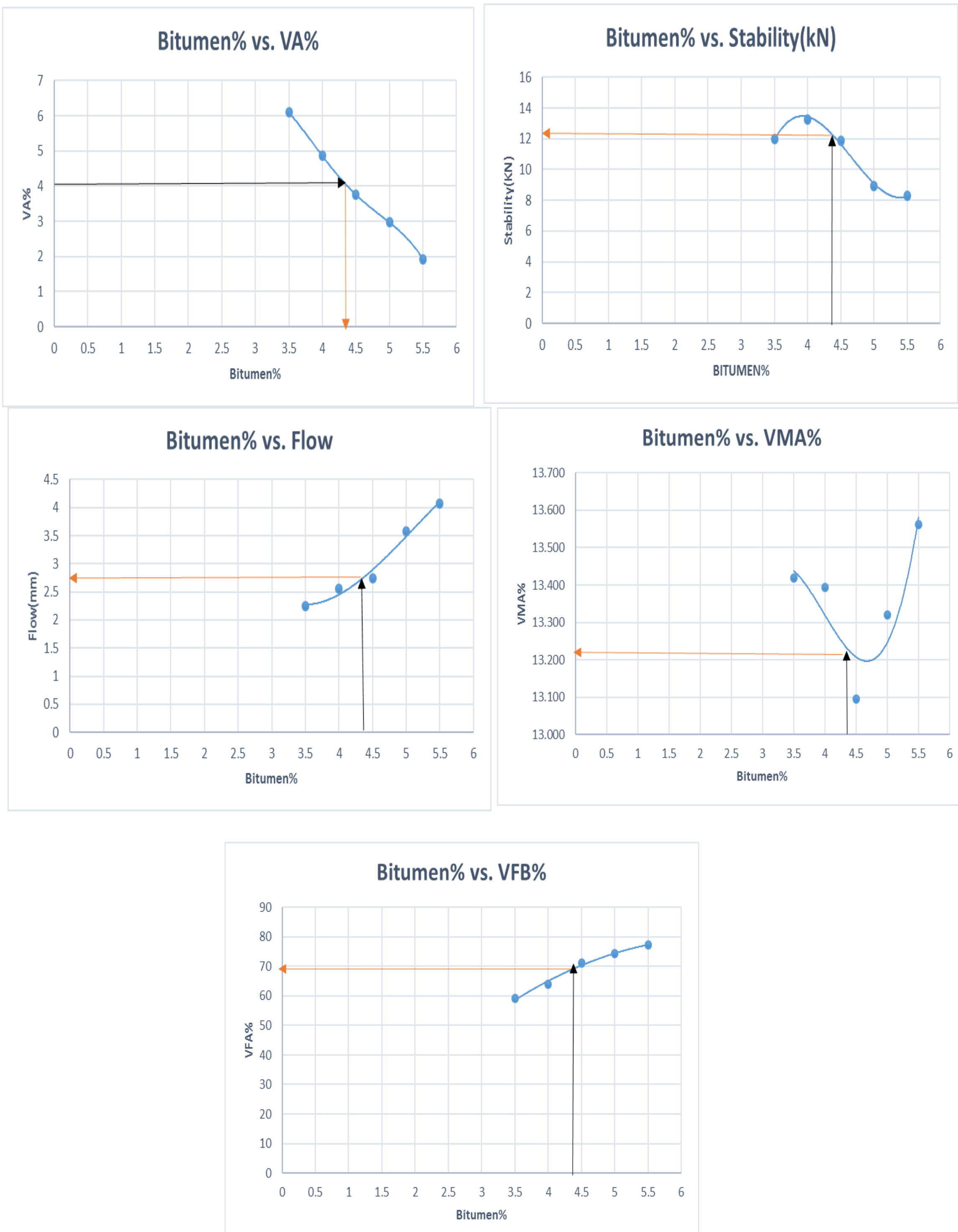

Figure 3. 19: Volumetric Properties of 0% CAPSULES

The asphalt content at 4% air voids corresponds to OBC. The mix has an OBC of 4.37%. Other volumetric properties are checked at the OBC obtained. If all the properties are within the limits specified by MS -2 then well and good otherwise OBC will have to be adjusted. According to MS-2 Criteria, the VMA value should be greater than 13% while in this case it is 13.24% i.e. within the range. VFA should be between 65-75, while the calculated value is 69.22%. Stability should be minimum of 8.006 KN while it is determined to be 12.4 KN in this case while flow is 2.69 mm lies within the range of 2-3.5 mm.

Parameters	Value Measured	Limits	Remarks
OBC	4.37	-	-
VMA (%)	13.24	≥13	Pass
VFA (%)	69.22	65-75	Pass
Stability (KN)	12.4	≥8.006	Pass
Flow (mm)	2.69	2-3.5	Pass

Table 3. 5: Job Mix Formula

3.9 Preparation of Sample for Performance Tests

After finding out OBC, sample for performance tests were prepared i.e. for Moisture Susceptibility and Fatigue Tests.

3.9.1 Moisture Susceptibility

The Samples for Moisture Susceptibility were prepared according to ALDOT 361, for which Marshall sample having 2.5" height and 4" diameter were prepared. The samples for moisture susceptibility falls into two category normal Marshall Samples and other having capsules.

Sample having capsules and normal samples were prepared at OBC with the different percentages of capsules i.e. 0.2%, 0.3% and 0.5

Figure 3. 20: Dried Capsules containing Oil

CAPSULES%	Unconditioned Samples	Conditioned Samples	Sub-Total
0	2	2	4
	2	2	4
0.2	2	2	4
	2	2	4
0.3	2	2	4
	2	2	4
0.5	2	2	4
	2	2	4
Total	40		

Table 3. 6: Test Matrix for Moisture Susceptibility Test

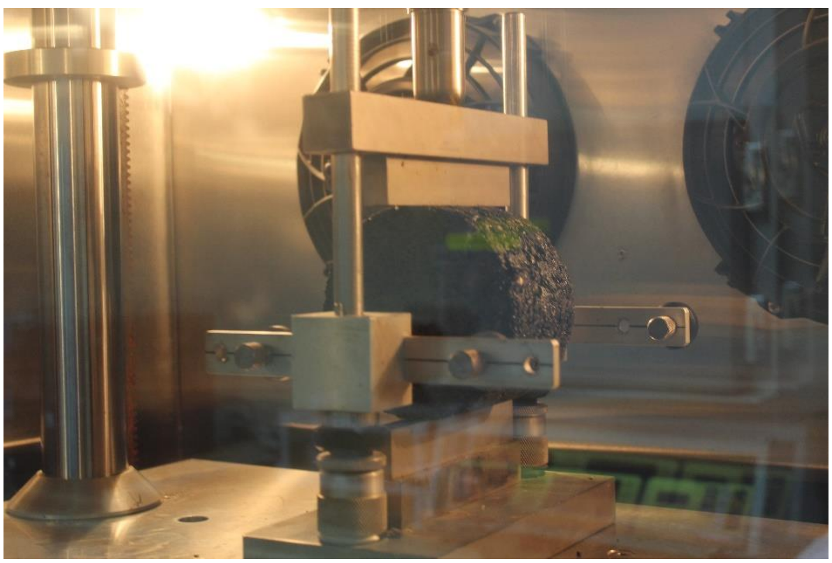

Figure 3. 21: Sample for Moisture Susceptibility Test

3.10 Semi Circular Bending Test

SCB test was performed to find out the healing level of asphalt mixture. Marshall samples are cut into halves and a notch of 3 mm is placed in middle of sample. Load is applied on top of sample to find out peak load at which sample cracks. This is then used to find out Healing Index.

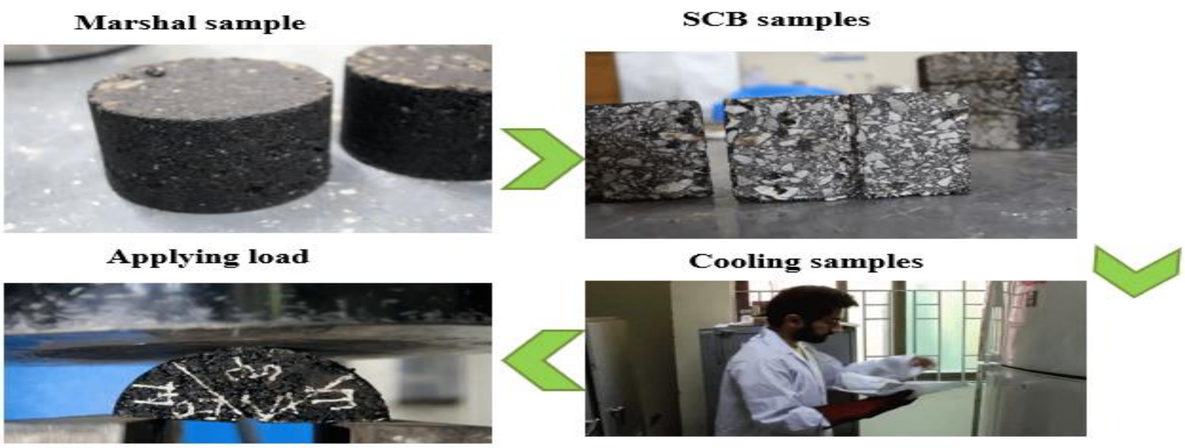

Figure 3. 22: Schematic of SCB test

Capsules%	**Samples**	
0	10	
	10	
0.5	10	
	10	
	Total	**40**

Table 3. 7: Test Matrix for SCB test

3.11 3 Point bending

3 Point bending test was used to find out healing efficiency of asphalt mortar. 96 samples were prepared and extensive testing was done to establish relationship between healing time and healing temperature. Samples were healed at 20 C, 25 C, 30 C & 40 C. Healing efficiency was evaluated at different rest times i.e 24 Hrs, 48 Hrs, 72 Hrs, 96 Hrs, 120 Hrs, 144 Hrs, 168 Hrs, 192 Hrs. Formula for Healing index:

$$HI = Cb/ Ca * 100\%$$

where HI is the healing index , Ca is before healing bending strength (MPa) and Cb is the bending strength (MPa) after b cycles of healing.

Capsules%	Samples	Temp. 20C	Temp. 25C	Temp. 30C	Temp. 40C
0.5	96	24	24	24	24

Table 3. 8: Test Matrix for 3 Point Bending

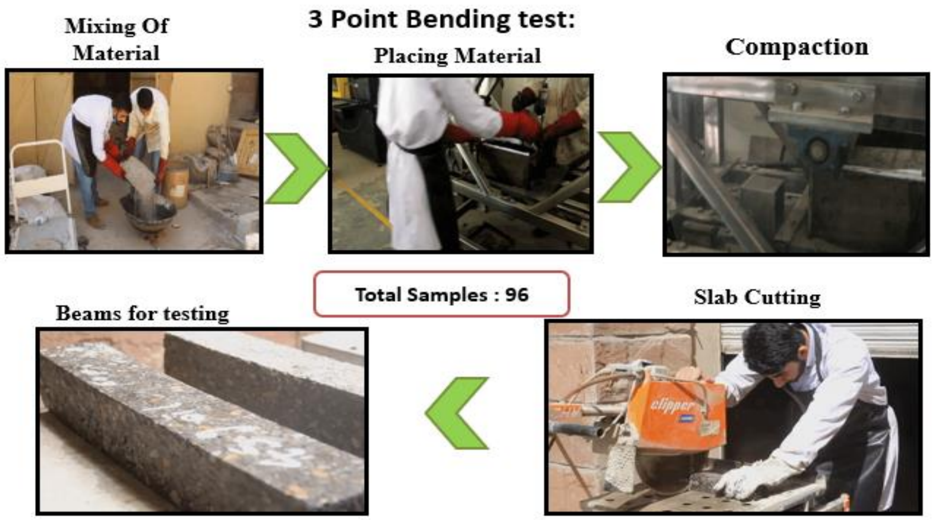

Figure 3. 23: Schematic of 3 point bending test

3.12 Summary

This chapter explains the testing of Aggregate, Bitumen and capsules. The material satisfying the criteria was then used to prepare Bituminous Mix samples. The volumetric properties of mix were calculated and OBC was determined. The OBC determined was then used to prepare samples containing encapsulated rejuvenator for performance testing i.e. Moisture Susceptibility and Self-healing test. In the end of Chapter Moisture Susceptibility, Semi Circular test and 3 point Bending were elaborated.

Chapter 4

RESULTS AND ANALYSIS

4.1 Introduction

After capsules preparation, Thermogravimetric analysis is performed to find out thermal stability of capsules. Then, marshall samples are prepared and OBC is determined. Further on, Moisture Susceptibility Test, Semi Circular Bending Test and 3 Point Bending Test are conducted.

4.2 Capsule Testing

Calcium alginate capsules containing waste cooking oil were prepared by ionotropic gelation of alginate. Five different sizes of capsules have been manufactured in the laboratory. This chapter investigates experimentally temperature susceptibility of capsules.

Figure 4. 1: Capsules

4.3 Thermogravimetric analysis (TGA)

TGA result shows the mass change of capsules with increasing temperature. Capsules with lower oil content loses more mass while capsules with greater oil content loses less mass. C4 capsule only loses 2 % of mass even at 200 ºC. With further increment in temperature, between 300 ºC to 500 ºC mass loss considerably increases. After 1000 ºC, remaining mass for C4 is just 3 %.

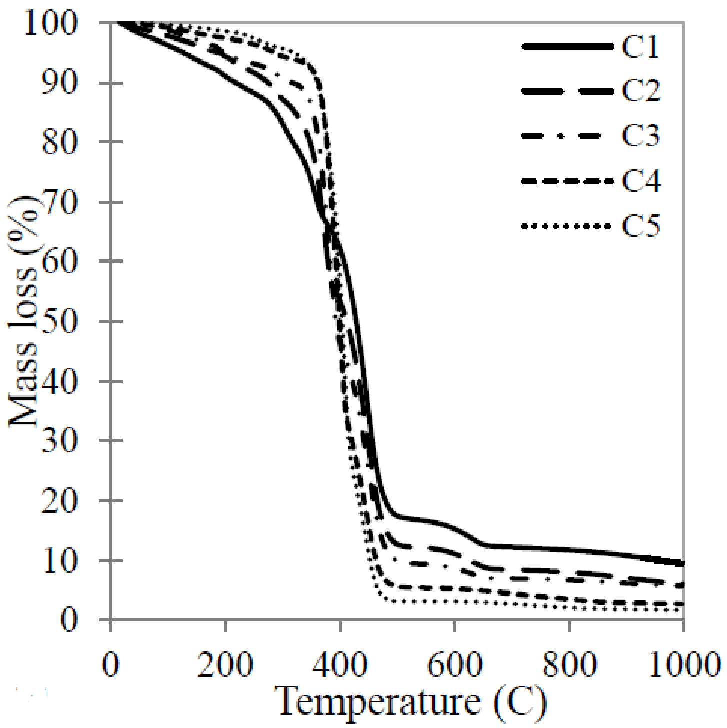

Figure 4. 2: TGA Graph

4.4 Moisture Susceptibility Test [ALDOT-361-88]

The Moisture Susceptibility test was performed according to ALDOT 361-88. One set for conditioned the other one for unconditioned samples was prepared. Un-Conditioned sample just required 1 hr. of conditioning by keeping the sample at 25 °C (i.e. the Testing Temperature) in temperature controlled Chamber prior to testing. However, for Conditioned Sample, the sample was kept in water bath for 24 hrs. at a temperature of 60°C, later it was kept for 1 hr. at 25 °C (i.e. the Testing Temperature) in water bath. Both sets of samples were then placed in UTM machine where the load was applied at 50mm/min. The maximum load at failure was noted in kN. Tensile Strength was determined by the given Formula. The ratio of average Tensile Strength of conditioned to that of un-conditioned sample was determined as per the formula given.

$$S_t = \frac{2000\,P}{\pi * D * t}$$

Where, St = Tensile Strength (kpa)

P = Maximum Load (N)

D = Diameter of Sample (mm)

t = Thickness of Sample (mm)

The Tensile Strength ratio for each set of sample was determined by the following equation

$$TSR = \frac{S2}{S1}$$

Where,

S_2 = Average Tensile Strength of Conditioned sample

S_1 = Average Tensile Strength of Un-Conditioned sample

4.4.1 Moisture susceptibility test result [ALDOT-361-88]

Figure shows that sample with 0.5% capsules has highest value of TSR. Thus, incorporation of capsules decreases moisture susceptibility of Asphalt sample.

Performance testing							
test	#2	Moisture susceptibility test					
B.C 4.4							
sample	unconditioned sample (S1)			Conditioned sample (S2)			TSR (S2/S1)*100
	1 (kpa)	2 (kpa)	average	1 (kpa)	2 (kpa)	average	percentage %
virgin	1120.4	1124.7	1122.5	1052.6	1054.1	1053.35	93.83
0.2 %	1110.1	1115.1	1112.6	1050.1	1053.1	1051.6	94.51
0.3 %	1100.3	1103.9	1102.1	1048.0	1051.9	1049.95	95.26
0.5 % capsules	1090.5	1078.6	1084.5	1045.5	1049.3	1047.4	96.57

Table 4. 1: Moisture Susceptibility test

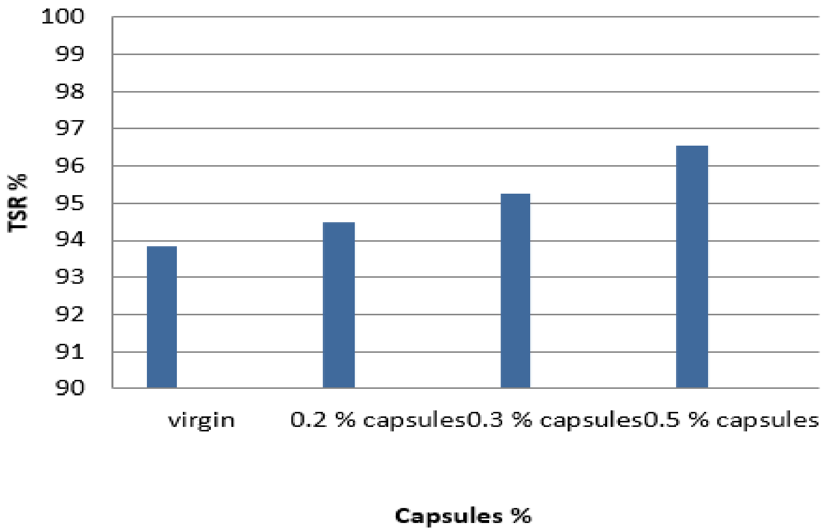

Figure 4. 3: TSR vs Capsule %

4.5 Semi Circular bending test

SCB test was performed to find out the healing level of asphalt mixture. Marshall samples are cut into halves and a notch of 3 mm is placed in middle of sample. Load is applied on top of sample to find out peak load at which sample cracks. This is then used to find out Healing Index..

4.5.1 Semi Circular Bending Test Results

Figure 4.4 shows the results of peak loads results performed on SCB samples. Although, the peak loads of 2nd SCB and 3rd SCB shows peak values lower than intial peak load, yet the results are substantially better than samples without capsules. This means that 20 hrs of healing period has significantly improved healing of SCB sample. Healing Index in 1st healing is 52 % and decreases to 46 % for 2nd healing because of fracturing of capsules due to cracks. The results demonstrate that SCB specimen with capsules were able to restore 52 % of initial peak force while control samples could only restore 14 %.

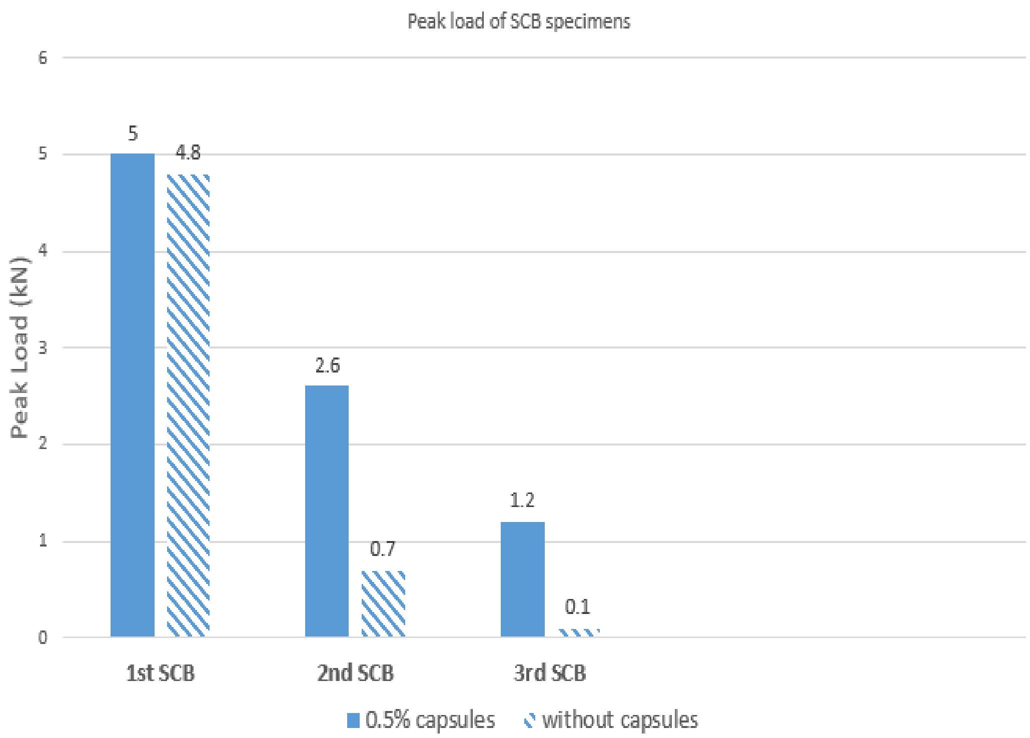

Figure 4. 4: Peak Loads of SCB

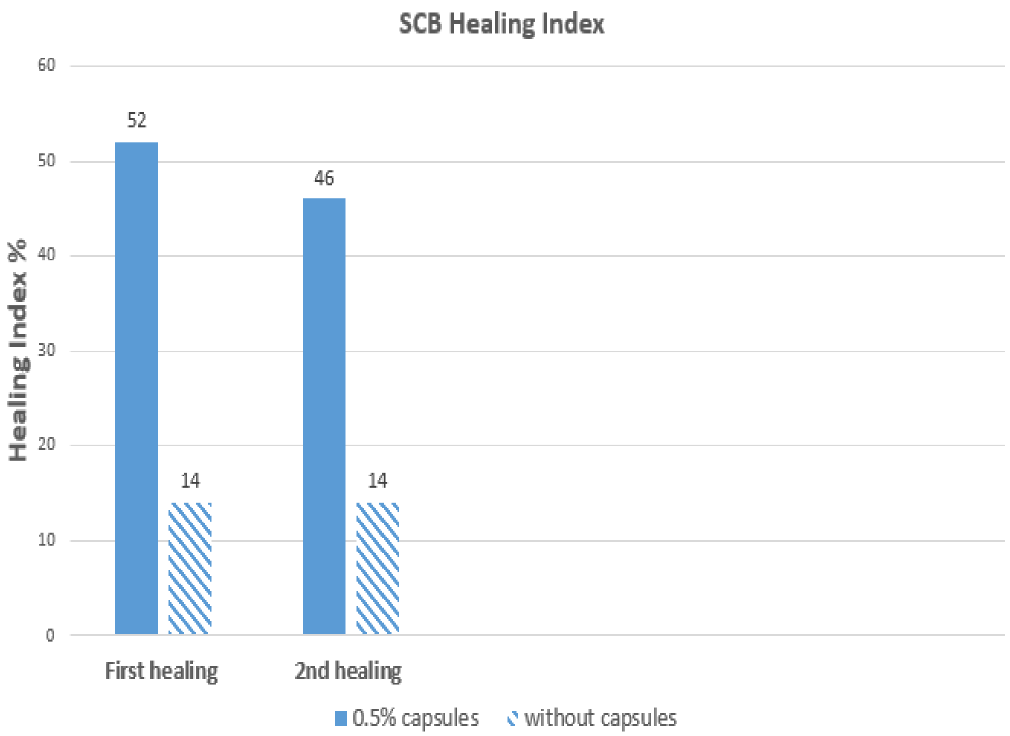

Figure 4. 5: Healing Index SCB

4.6 3 Point Bending test and Healing Efficiency of Asphalt Mortar

3 Point bending test was used to find out healing efficieny of asphalt mortar. 96 samples were prepared and extensive testing was done to establish relationship between healing time and healing temperature. Strength of beams before healing and after healing are compared to find out healing index.

4.6.1 3 Point Bending Test Results

For aged samples with capsules, the initial bending strength was recorded as 4.7 MPa, that reduced to 2.5 MPa after first healing. In comparison to 11 % healing index of samples without capsules, the healing index of capsulated beams was 53 %. .

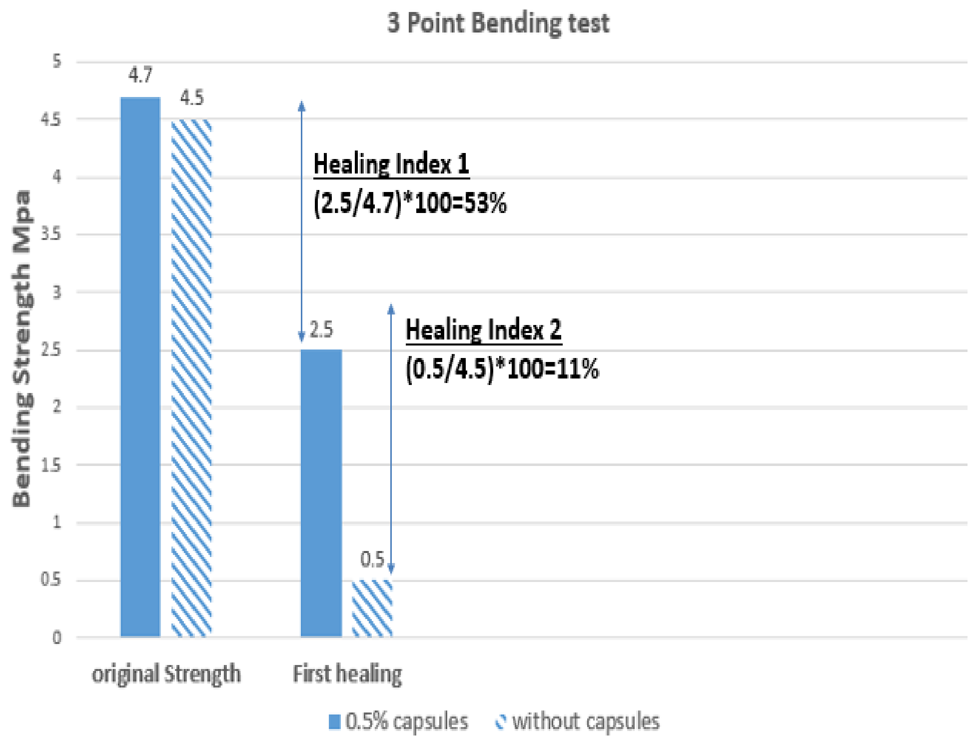

Figure 4. 6: 3 Point Bending Test

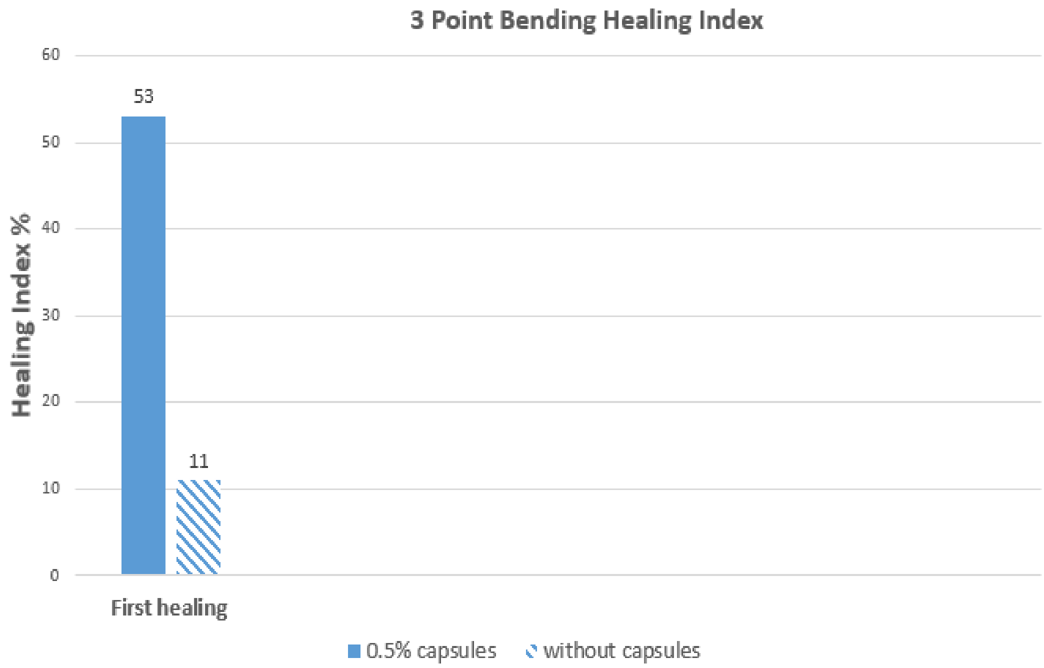

Figure 4. 7: 3 Point bending Healing Index

4.6.2 Mathematical Model for Time & Temperature Dependent Asphalt Healing

After finding out healing level at different healing time and temperature, a mathematical model is developed with R^2 greater than 0.96. Figure shows logarithmic equations to find out healing level at different healing time and temperature.

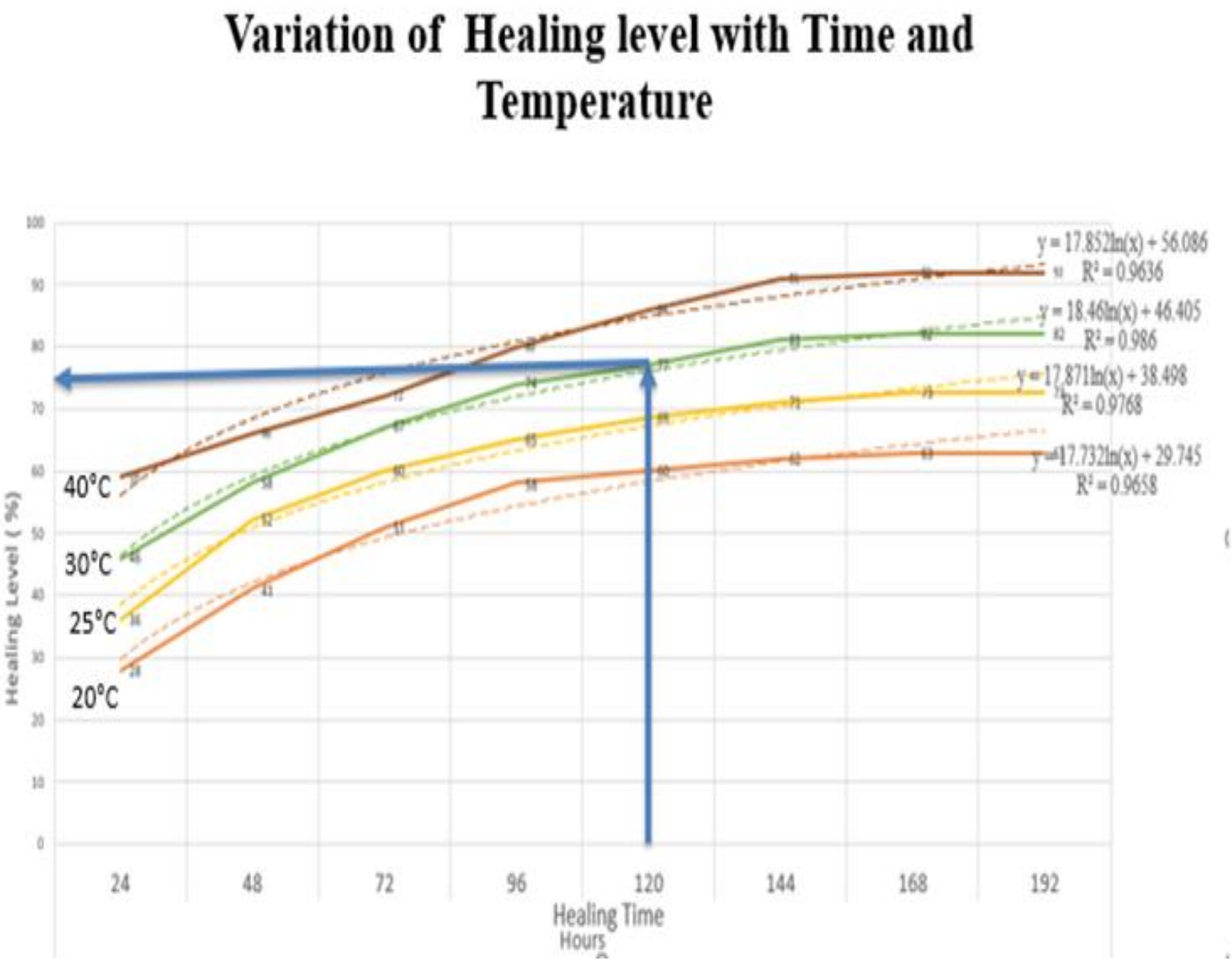

Figure 4. 8: Healing Level vs Time

Equations to Find Time & Temp Dependent Asphalt Healing

- At 20°c

  $$y = 17.732\ln(x) + 29.745$$
  $$R^2 = 0.9658$$

- At 25°C

  $$y = 17.871\ln(x) + 38.498$$
  $$R^2 = 0.9768$$

- At 30°C

  $$y = 18.46\ln(x) + 46.405$$
  $$R^2 = 0.986$$

- At 40°C

  $$y = 17.852\ln(x) + 56.086$$
  $$R^2 = 0.9636$$

Figure 4. 9: 3 Point bending Mathematical Model

4.7 Summary

In this chapter, detailed analysis of the result obtained from performance testing were discussed. The result obtained from UTM are discussed in reference of increase in TSR values. Tables and Graphes were presented to analyze the data obtained from performance testing. Results obtained from Semi Circular Bending Test and 3 Point Bending Test are discussed along with graphs. Increase in Healing Index with increase in healing time and temperature is presented. Logarithmic Mathematical model is extracted from graphs showing relationship between healing level, healing temperature and healing time.

Chapter 5

Conclusion & Recommendation

5.1 Conclusions

Waste Cooking oil has been found to be successfully reverse ageing of bitumen used in asphalt pavement. Bitumen rejuvenation via waste cooking oil increased the aromatic content in bitumen and reduced the viscosity of binder. Oil comes out of capsule when it is broken by crack fracture energy and fills the cracks to heal it. Self-healing of asphalt mixture incorporating capsules is assessed at different temperature and healing times.

5.1.1 Capsule production and characterisation

Encapsulated rejuvenator (waste cooking oil) is produced using ionotropic gelation of sodium alginate in the company of calcium chloride.

- Capsules encapsulating healing agent up to 80% are fabricated.

.

- Capsules were subjected to Thermogravimetric analysis to ensure thermal stability of capsules. Results proved that all capsules could resist the mixing temperature of asphalt and oil loss never exceeded 2%.

5.1.2 Capsules effect on Asphalt performance

Capsules were designed to break in event of trigger such as crack. First, moisture susceptibility test of asphalt proved that capsules improved the water sensitivity of asphalt sample.

- Asphalt specimens tested on Semi Circular Bending Test proved that sample with 0.5% of capsules showed 40% healing during 1st SCB. Peak load regain demonstrates that capsules healing had positive impact .

- 3 Point Bending test performed at different temperature and healing times showed that with increasing temperature and healing time, healing level increases. Healing level increases from 52% to 69% at 25 °C for 48 hrs. and 120 hrs. healing time.

5.2 Recommendations

This part presents recommendations that were not included this particular study due to shortage of time and funds:

- Different materials such as silica/ limes/ rubber can be used to modify capsule composition. Capsules mechanical strength can be evaluated with reference to oil / water ratio.
- Oil amount released during mixing and compaction can be quantified using FTIR. The effect of environmental factors on capsule quality can also be evaluated. For example, effect of fungi and bacteria on capsules.
- Capsules can be studied in combination with induction heating technique to develop a synergy between these techniques.
- Economic feasibility analysis can be done to study life cycle cost assessment.
- Healing can be studied using CT scans to evaluate effect of capsules oil content on healing level with respect to crack width.
- The process of rejuvenator diffusion and crack propagation can be modelled to better understand the process of capsule rupture and bitumen healing

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APPENDIX 1: MARSHAL MIX DESIGN REPORTS

Marshall Test Report

Project: FYP UG 2k15 Trial Mix: control (1) Grade AC: ARL 60/70

		Mass, grams of compacted Specimen					Mass, grams of loose Mix							Stability, (KN)		
% AC by wt. of mix, Spec. No.	Spec. Height in. (mm)	In Air (A)	In Water (C)	Sat. Surface Dry in Air (B)	B-C	Bulk S.G. Specimen (Gmb) Gmb =A/(B-C)	Dry Weight (a)	Calibration Weight = wt: of Pycnometer + Glass Lid + Water (b)	wt: of Sample + Water + Pycnometer and Lid (c)	Max. S.G. (loose Mix) (Gmm) =a/b-(c-a)	% Air Voids	% VMA	% VFA	Measured	Adjusted	Flow mm
3.5-A	70.485	1182	706.1	1208	501.9	2.355	1174	6774	7479.5	2.506	6.0186	13.424	55.165	12.443	10.328	2.355
3.5-B	66.675	1176	695.4	1193	497.6	2.363	1180	6774	7484.5	2.513	5.9669	13.118971	54.517	14.335	13.332	2.291
3.5-C	68.58	1169.1	692.5	1190.6	498.1	2.347	1162	6774	7472.1	2.505	6.297	13.715433	54.088	13.775	12.25975	2.1
Average	68.58	1175.7	698	1197.2	499.2	2.355	1172	6774	7478.700	2.508	6.0947	13.419	54.583	13.517667	11.972997	2.248666667
4.0-A	66.04	1158	683	1172	489	2.368	1158	6774	7465.5	2.482	4.6012	13.395267	65.65	13.886	12.91398	2.452
4.0-B	69.85	1172.5	695.6	1192	496.4	2.362	1172	6774	7475	2.488	5.0764	13.61805	62.723	15.887	13.18621	2.542
4.0-C	65	1178.1	698.4	1194.7	496.3	2.374	1178	6774	7480	2.496	4.8882	13.187991	62.935	14.3	13.728	2.655
Average	66.96333	1169.533333	692.333333	1186.23333	493.9	2.368	1169.333333	6774	7473.500	2.489	4.8555	13.400436	63.769	14.691	13.276063	2.549666667
4.5-A	66.99	1178	696.5	1190.2	493.7	2.386	1183	6774	7479.5	2.477	3.6901	13.192704	72.029	12.443	11.07427	2.766
4.5-B	66.04	1184	699.8	1196	496.2	2.386	1186	6774	7483	2.486	4.0315	13.19015	69.435	13.885	12.91305	2.699
4.5-C	64.5	1189.6	702.7	1199.6	496.9	2.394	1189	6774	7484	2.482	3.5537	12.902433	72.457	12.113	11.62848	2.763
Average	65.84333	1183.867	699.667	1195.267	495.600	2.389	1186.000	6774	7482.167	2.482	3.7586	13.095096	71.307	12.813667	11.871933	2.742666667
5.0-A	70.167	1186	714.5	1210	495.5	2.394	1187	6774	7481	2.473	3.2098	13.37658	76.005	11.497	9.54251	3.3445
5.0-B	64.8	1185	703.1	1199	495.9	2.390	1180	6774	7475	2.463	2.9987	13.519431	77.82	9.124	8.75904	3.654
5.0-C	64	1181.1	696.5	1188.7	492.2	2.400	1180	6774	7475.5	2.466	2.6928	13.156092	79.532	8.9	8.544	3.755
Average	66.32233	1184.033	704.700	1199.233	494.533	2.394	1182.333	6774	7477.167	2.467	2.9673	13.350701	77.774	9.8403333	8.9485167	3.5845
5.5-A	66.36	1178	696.4	1187	490.6	2.401	1178	6774	7471	2.449	1.9568	13.558907	85.568	9.45	8.7885	4.24
5.5-B	63.5	1180	703.5	1193	489.5	2.411	1178	6774	7472	2.454	1.7743	13.217569	86.576	8.98	8.98	4.133
5.5-C	62.75	1149.8	677	1157.8	480.8	2.391	1140	6774	7447	2.441	2.0352	13.908486	85.367	7.163	7.163	3.866
Average	64.203	1169.267	692.300	1179.267	486.967	2.401	1165.333	6774.000	7463.333	2.448	1.9219	13.561654	85.829	8.531	8.3105	4.079666667

Worksheet for Volumetric Analysis of Compacted Paving Mixture

(Analysis by Weight of Total Mixture)

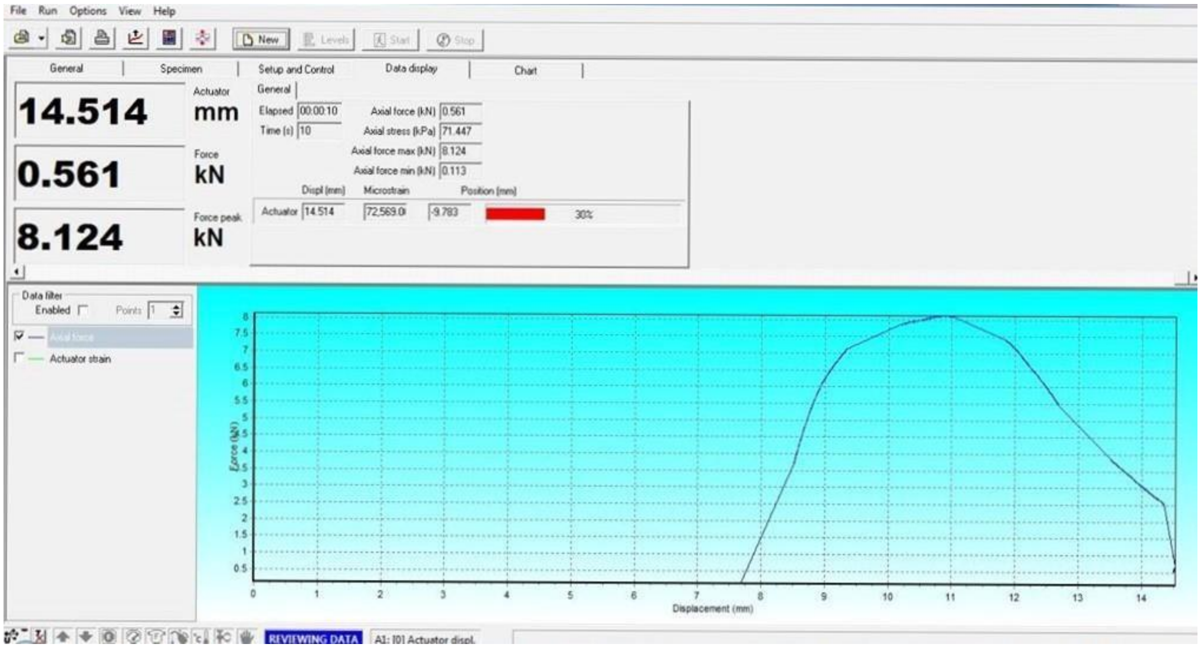
Sample: Controlled sample

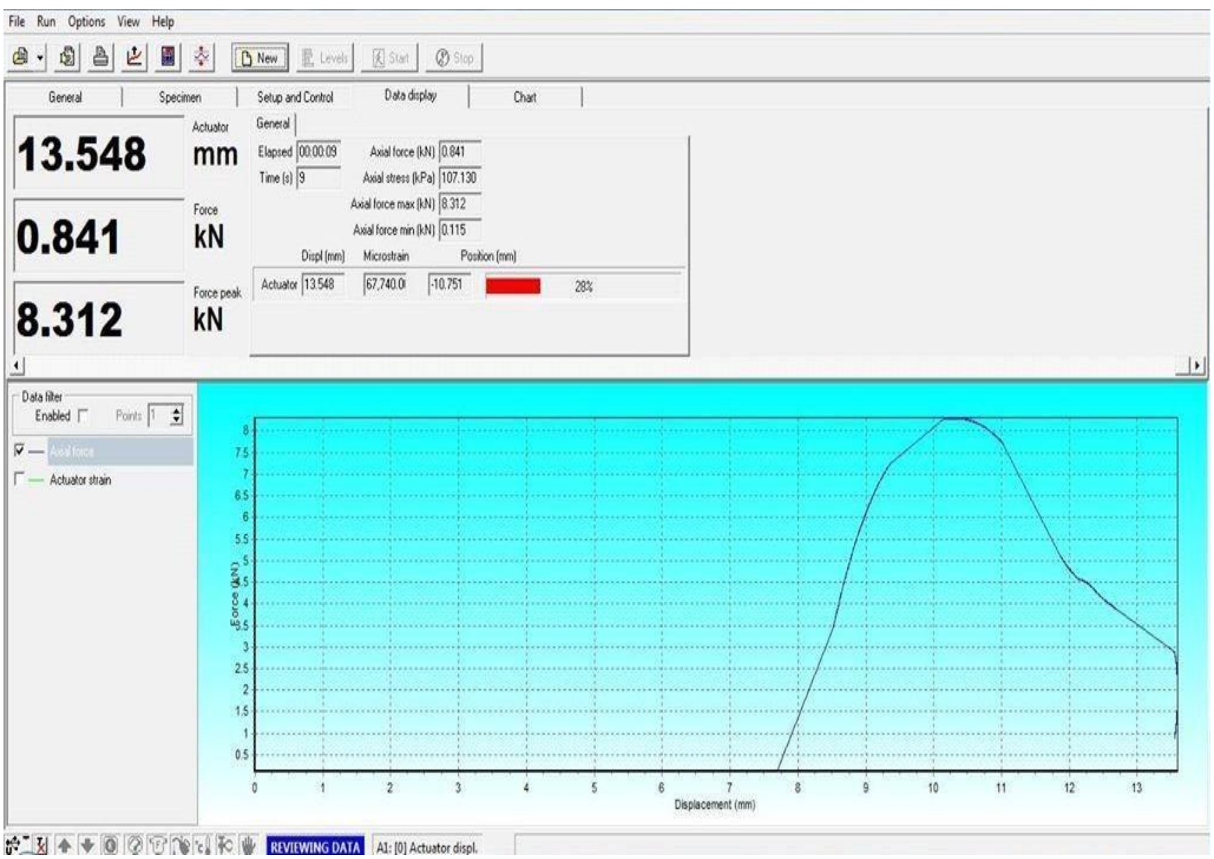
Identification: Margalla

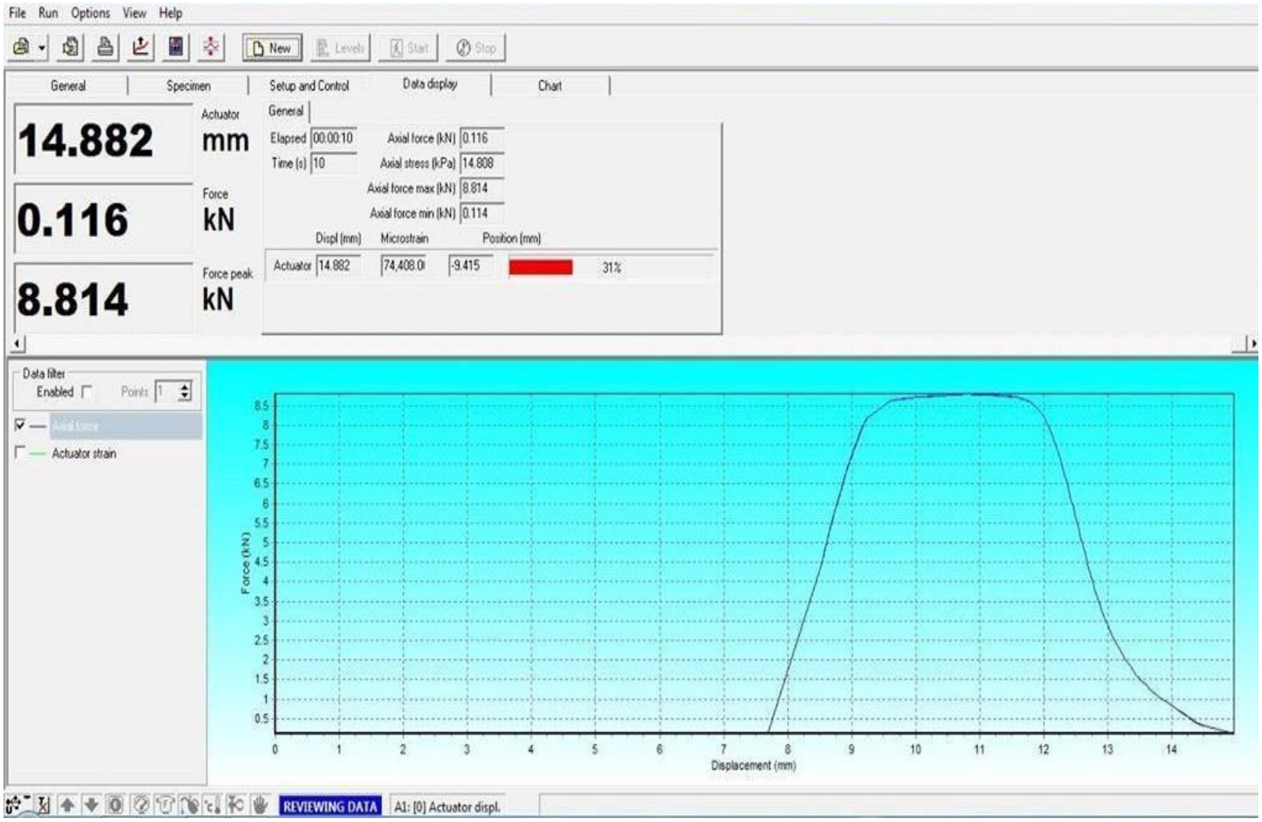
Composition of Paving Mixture

	Specific Gravity, G			Mix Composition, % by Wt. of Total Mix, P	Mix or Trial Number				
			Bulk		1	2	3	4	5
1. Coarse Aggregate	G1		2.632	P1	48.250	48.000	47.750	47.500	47.250
2. Fine Aggregate	G2		2.618	P2	48.250	48.000	47.750	47.500	47.250
4. Total Aggregate	G4	---	2.625	Ps	96.50	96.00	95.50	95.00	94.50
5. Asphalt Cement	G5	1.03	-----	Pb	3.50	4.00	4.50	5.00	5.50
6. Bulk Sp. Gr. (Gsb), total aggregate					2.625	2.625	2.625	2.625	2.625
7. Max. Sp. Gr. (Gmm), paving mix					2.508	2.489	2.482	2.467	2.448
8. Bulk Sp. Gr. (Gmb), compacted mix					2.355	2.369	2.389	2.395	2.401
9. Effective Sp. Gr. (Gse), total aggregate					2.646	2.645	2.659	2.663	2.661
10. Absorbed Asphalt (Pba), % by wt. total agg.					0.307	0.299	0.496	0.553	0.534
CALCULATIONS									
11. Effective Asphalt content (Pbe)					3.204	3.713	4.026	4.475	4.995
12. Voids in Mineral Aggregate, VMA (percent of bulk vol.					13.425	13.362	13.085	13.323	13.563
13. Air Voids (Va)					6.100	4.821	3.747	2.919	1.920
14. Voids Filled with Aggregate, VFA					54.559	63.918	71.365	78.094	85.845

APPENDIX 2: ITS TEST RESULTS



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New Levels Start Stop
General Specimen Setup and Control Data display Chart
14.882
Actuator
mm
0.116
Force
kN
8.814
Force peak
kN
Elapsed 00:00:10
Time (s) 10
Axial force (kN) 0.116
Axial stress (kPa) 14.808
Axial force max (kN) 8.814
Axial force min (kN) 0.114
Displ (mm) Microstrain Position (mm)
Actuator 14.882 74,408.0 -9.415 31%
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Axial force
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Force (kN)
Displacement (mm)
REVIEWING DATA
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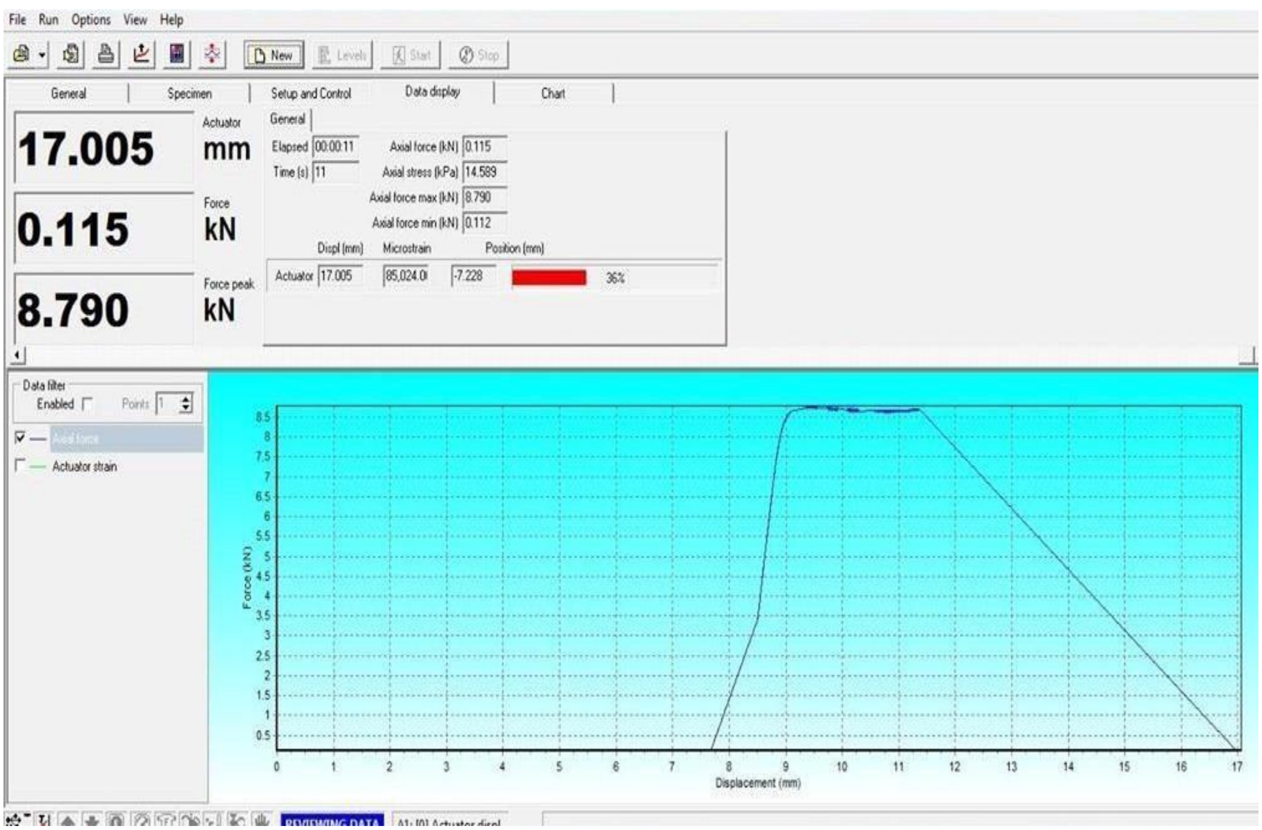
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Force
kN
8.790
Force peak
kN
Elapsed 00:00:11
Time (s) 11
Axial force (kN) 0.115
Axial stress (kPa) 14.589
Axial force max (kN) 8.790
Axial force min (kN) 0.112
Displ (mm) Microstrain Position (mm)
Actuator 17.005 85,024.0 -7.228 36%
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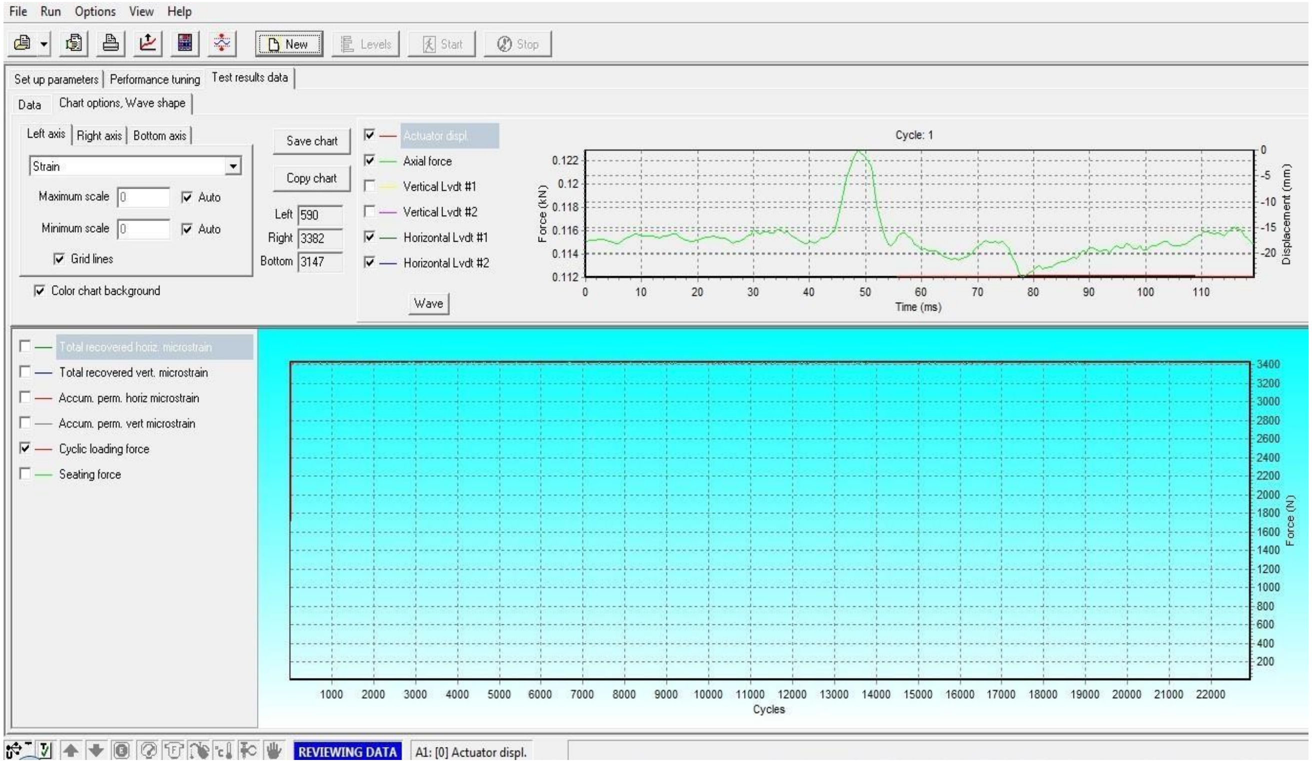
Points 1
Axial force
Actuator strain
Force (kN)
Displacement (mm)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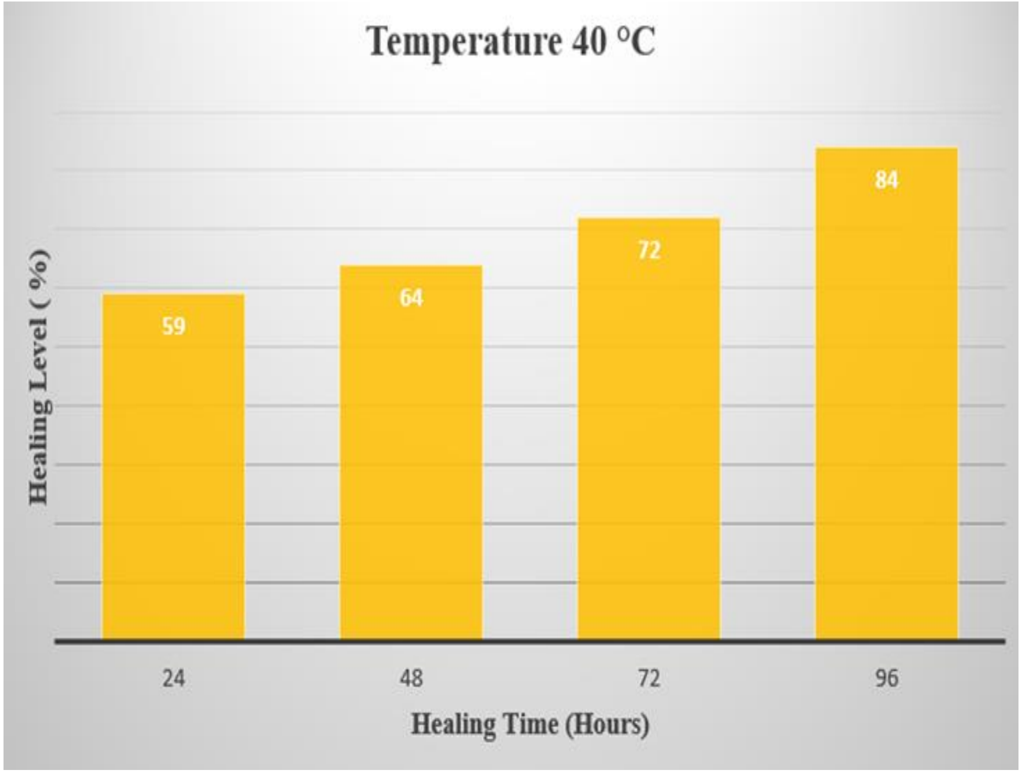
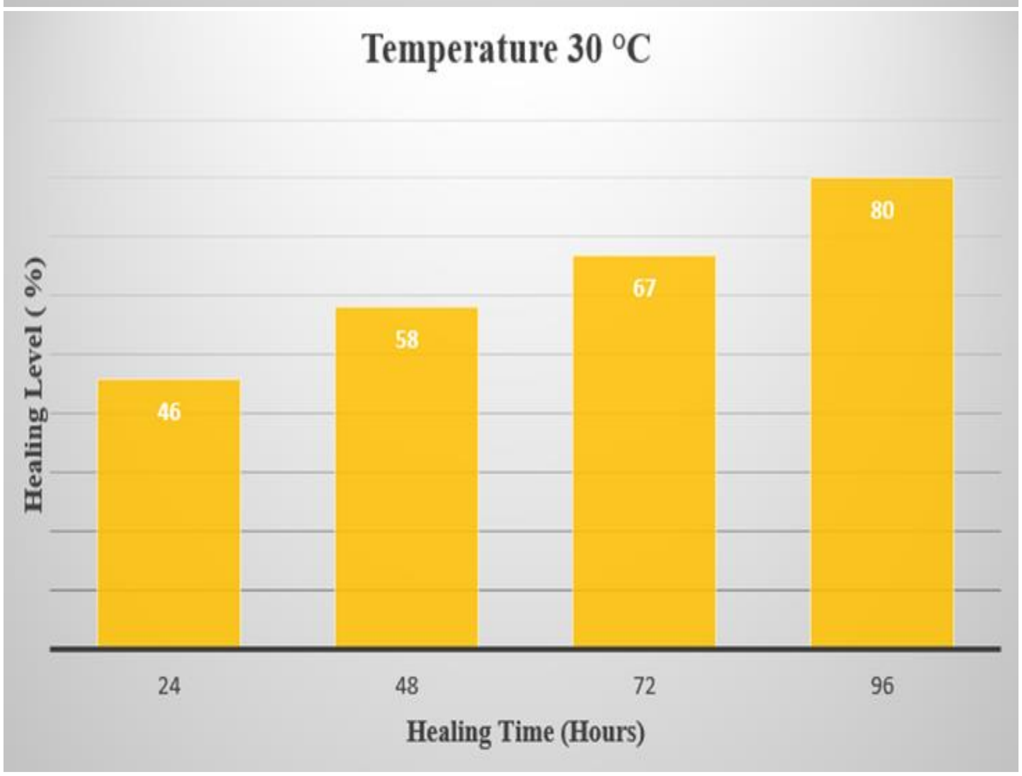
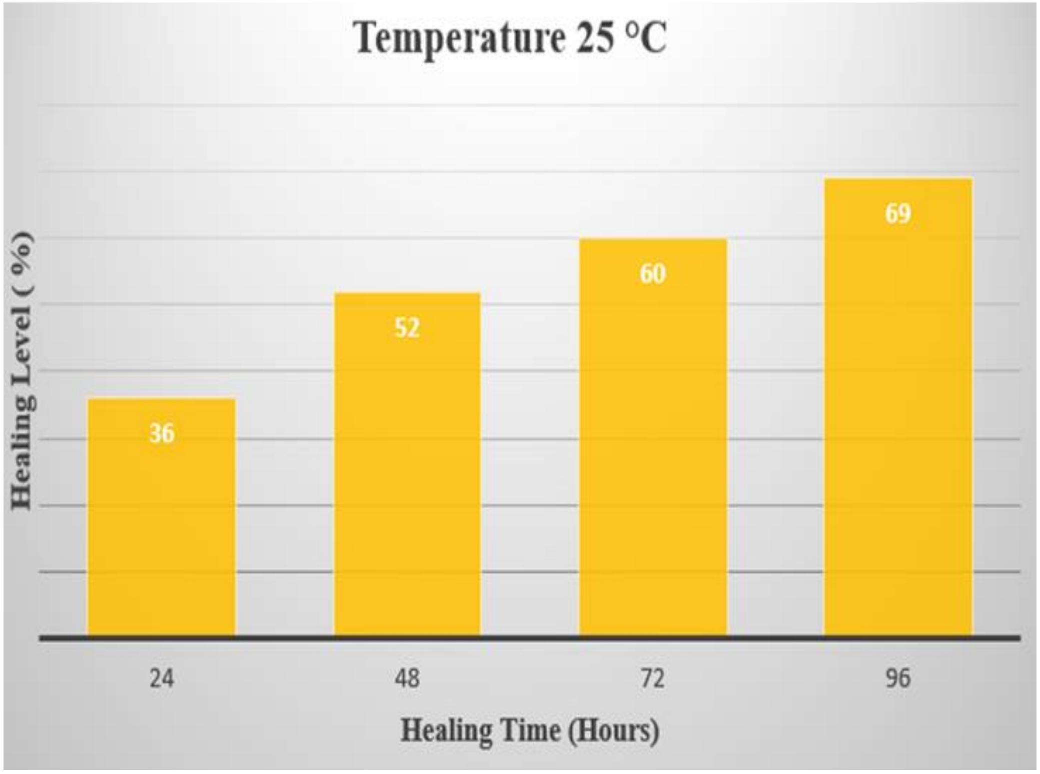
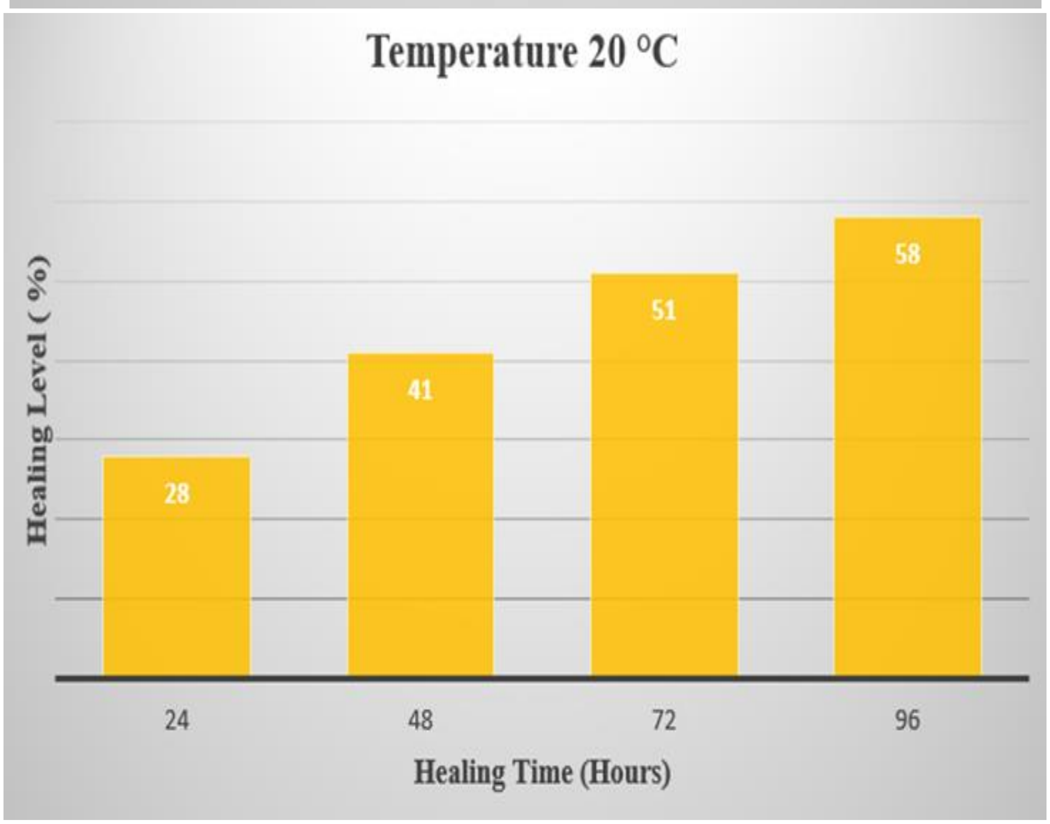
New
Levels
Start
Stop
Set up parameters
Performance tuning
Test results data
Data
Chart options, Wave shape
Left axis
Right axis
Bottom axis
Strain
Maximum scale
Minimum scale
Auto
Grid lines
Color chart background
Save chart
Copy chart
Left 590
Right 3382
Bottom 3147
Actuator displ.
Axial force
Vertical Lvdt #1
Vertical Lvdt #2
Horizontal Lvdt #1
Horizontal Lvdt #2
Wave
Cycle: 1
Force (kN)
Displacement (mm)
Time (ms)
Total recovered horiz. microstrain
Total recovered vert. microstrain
Accum. perm. horiz microstrain
Accum. perm. vert microstrain
Cyclic loading force
Seating force
Force (N)
Cycles
REVIEWING DATA
A1: [0] Actuator displ.

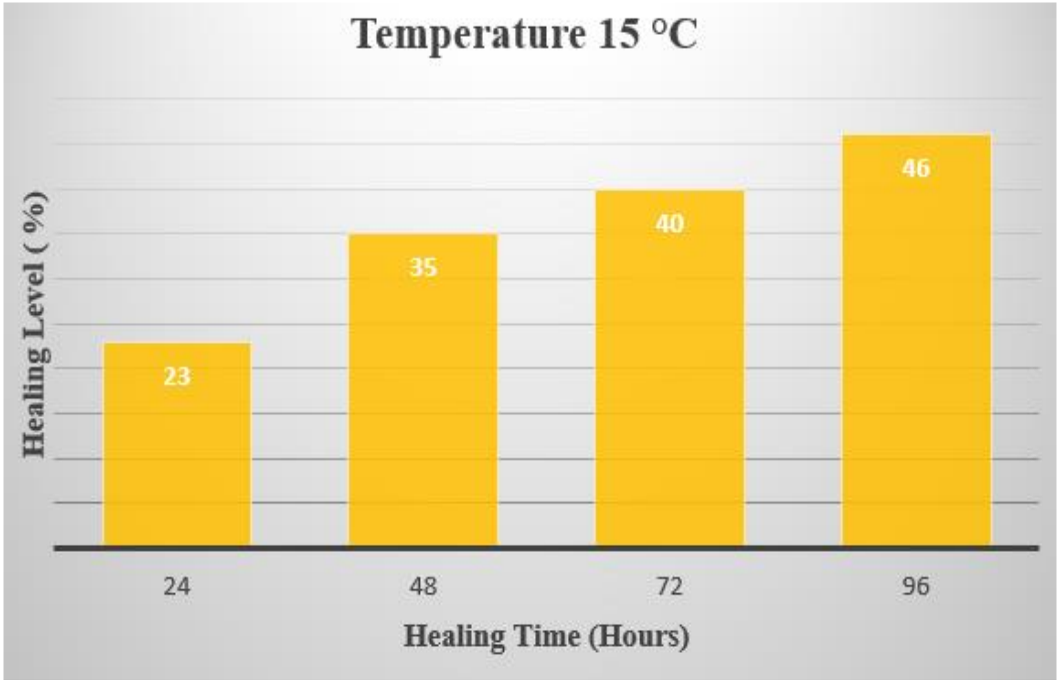
APPENDIX 3: 3 Point Bending Graphs






Temperature 25 °C
Healing Level (%)
36
52
60
69
24
48
72
96
Healing Time (Hours)


Temperature 20 °C
Healing Level (%)
28
41
51
58
24
48
72
96
Healing Time (Hours)


Temperature 15 °C
Healing Level (%)
23
35
40
46
24
48
72
96
Healing Time (Hours)