# PERFORMANCE EVALUATION OF SELF HEALING ASPHALT CONCRETE USING RECYCLED REJUVENATING AGENT

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(2022)

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A thesis submitted to the National University of Sciences and Technology, Islamabad,

in partial fulfillment of the requirements for the degree of

Masters of Science in

Transportation Engineering

Thesis Supervisor: Dr. Arshad Hussain


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# DEDICATION

[To My Family and Teachers]

## ACKNOWLEDGEMENTS

All praise and appreciation to ALMIGHTY ALLAH for providing me with the patience and tenacity necessary to accomplish my master's degree. I am immensely appreciative of our parents' earnest prayers and our family's support during the duration of my course and research work. My deepest appreciation and gratitude to Dr. Arshad Hussain for his invaluable counsel and direction during my study effort. My gratitude to the transportation lab team, who worked tirelessly and contributed considerably to the successful completion of my research's experiment.

# ABSTRACT

Asphalt is the most preferred binding material for roadway construction due to the advantages of cost efficiency, noise reduction, and comfortable driving experience. Meanwhile, several factors including UV radiation, oxidation, temperature, moisture, and traffic loads damage asphalt and becomes brittle. Deterioration begins at the micro-level and gradually transforms into macro-cracks that eventually lead to pavement failure. Researchers have proven that asphalt has intrinsic self-healing ability, but it is limited. Thus, damages accumulate beyond asphalt natural healing capacity. This research uses the in-situ crack healing method of encapsulated rejuvenator technology to improve the limited self-healing ability of roads and enhance its service life so that extrinsically induced self-healing ability of asphalt can help it recover the damage incurred during service life. When a crack is induced in the pavement, capsules rupture and releases a rejuvenator which diffuses into the crack and heals it. Ca-Alginate capsules with rejuvenator were prepared. The surface texture and morphology of capsules was assessed using optical microscopy. The thermogravimetric analysis was done to analyze the thermal stability of capsules in asphalt mixture. A moisture susceptibility test was conducted to investigate the water sensitivity of capsules in the asphalt mix. It was found that capsules will improve the moisture susceptibility of rejuvenator encapsulated asphalt mixture. To illustrate the benefits of encapsulated rejuvenator on the pavement service life, the self-healing behavior of capsule-induced asphalt was investigated using the semi-circular bending test and three-point bending beam test. Results of these tests were used to explain the relationship between healing time, temperature, and healing level. Overall, Ca-Alginate containing recycled rejuvenator was found to be suitable for healing for asphalt mix with increased healing temperature and healing time better for healing index.



**Keywords:** Self-healing asphalt, Rejuvenator, 3 Point Bending, Healing Index

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# LIST OF SYMBOLS, ABBREVIATIONS AND ACRONYMS

NHA – National Highway Authority

OBC – Optimum Bitumen Content

RTFO – Rolling Thin Film Oven

HI – Healing Index

VFA – Voids Filled with Asphalt

VMA – Voids in Mineral Aggregates

Gmb – Bulk Specific Gravity

Gmm – Maximum Specific Gravity

ITS – indirect Tensile Strength

TSR – Tensile Strength ratio

SCB – Semi Circular Bending test

3PB – 3 Point bending test

UTM – Universal Testing Machine

ASTM – American Society for Testing and Materials

HMA – Hot Mix Asphalt

RAP – Reclaimed Asphalt Pavement

UV – Ultravoilet

# CHAPTER 1: INTRODUCTION

## 1.1 Background

During service, asphalt pavement deteriorates owing to environmental causes such as oxidation, ultraviolet radiation (UV), moisture damage, and traffic stresses, increasing the stiffness of the pavement by altering the binders rheological characteristics. Thus, as pavement ages, it becomes more susceptible to deterioration such as cracking and fretting. While ageing degrades the quality and performance of flexible pavement, many ways have been developed to mitigate this impact. Asphalt rejuvenation is a promising technology that utilizes industrial rejuvenators and waste oils to replenish the bitumen's lost qualities and extend the life of the pavement. Asphalt rejuvenators have been used to obtain a 100 percent reclaimed asphalt pavement (RAP) in pavement recycling. While rejuvenators employed in surface course rejuvenation provide good results, they do have a drawback. It barely penetrates the top 2 cm of the pavement; hence, internal fractures and degradation are unaffected by this technique. This approach necessitates on-site labor, resulting in traffic congestion and adverse environmental consequences.

Over the past several years, self-healing materials have attracted a great deal of attention, with the majority of the research focusing on their potential uses in engineering materials such as polymers, concrete, and asphalt (Bergman and Wudl, 2008). The majority of microcracks observed in asphalt pavements are self-healing, provided that the surrounding environment is suitable. This results in self-healing asphalt pavements that are capable of fixing cracks on their own (Bazin and Saunier, 1967). A self-healing characteristic of asphalt was discovered in laboratory studies, as well.

Due to the viscoelastic characteristics of asphalt, this occurs (Kim et al., 2003). A rest interval between two loading cycles aids in the healing of fractures in the road surface, but it takes time for the pavement to restore its stiffness and strength once the rest period has been applied (Anderson et al., 1994). With the ability of asphalt mixes to self-heal, the possibility of active repair of asphalt pavements is increased. This means that rather than allowing the cracks to expand, it is preferable to repair them as soon as they appear (Li et al., 1998).

Road conditions have deteriorated because of a lack of maintenance as a result of high volumes of traffic and the time restrictions that come with fixing roads while also maintaining delays tolerable for motorists. If enclosed rejuvenator systems are available, they may be able to assist reduce the harmful effects of rejuvenators on the environment.

## 1.2 Problem Statement

The intrinsic self-healing capacity of bitumen is insufficient to keep up with the rate at which asphalt pavement degrades, as seen in Figure 1. The employment of external aid in the form of a rejuvenator or to speed the process is necessitated because of this. Because it makes use of a waste resource to generate a self-healing surface, using a low-cost rejuvenator in an enclosed form is a viable alternative to traditional pavement construction.

Food preparation requires cooking oil, which is why Pakistan imports a massive 2.6 million tonnes of cooking oil each year, ranking it as the world's third largest importer of cooking oil behind the United States and China. In Pakistan, hotels use and sell cooking oil to local merchants, which helps to alleviate poverty. Local restaurant proprietors then utilize this oil, and it eventually makes its way down the food chain to street food sellers. When they are finished with it, they dump it on the ground or into bodies of water, contaminating both groundwater and bodies of water. This waste cooking oil may be collected from vendors and centrifuged to produce a particle- and solid-free cooking oil that can be used as a rejuvenator to build long-lasting, self-healing pavements. The cooking oil can be used as a rejuvenator to create long-lasting, self-healing pavements.

Cracks appearance is inevitable and after occurrence of such macro cracks the options to revitalize pavement to original condition are limited. They include fog seal, crack seal, micro surfacing etc. but it not only time and resource consuming but also ineffective in longer run. 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 overcome by use of a promising technique of encapsulated rejuvenator.

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.

This research study develops on previous work to use multi-compartmented sodium alginate capsules containing waste cooking oil as rejuvenator to produce asphalt pavements that can produce localized internal healing of cracks thus mitigating development of macro-cracks that lead to early need deterioration of pavement. The mechanism is that due to crack propagation, capsule will rupture causing rejuvenator to leak out of capsule rise through capillary action to recover cracks. The encapsulated rejuvenator is an excellent choice owing to the following four reasons:

1. Reduced Maintenance Cost
2. Extended Service Life
3. Less Waste Oil Disposal
4. Reduced Green House Gas Emissions

The application of encapsulated rejuvenator to improve the self-healing efficiency of asphalt mixtures has remarkable advantages. Therefore, it was selected as the external heating method in this study

## 1.3 Research Objectives

1 Determine surface morphology and thermal stability of Ca-Alginate capsule Induced rejuvenator
2 Determine moisture susceptibility of capsules in asphalt mixture
3 Evaluating the effect of capsules on healing efficiency of Ca-Alginate capsule induced rejuvenator under different temperature and healing interval
4 Cost Comparison of Control Section with Encapsulated Section

### 1.4 Scope of Study

Literature review and collection of chemicals for capsules manufacturing, capsule production and characterization using thermo gravimetric analysis.

Moisture Susceptibility test of Marshall Samples with and without capsules.

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

**Table 1 : Test Matrix**

| Sr. No | Material | | | | Performance Tests | | | |
|---|---|---|---|---|---|---|---|---|
| | | **%age of capsules in Mix** | **OM** | **TGA** | **SCB** | **Moisture Susceptibility Test** | **TPB @ °C (20,25,30,40)** | **Total** |
| **1.** | HMA | 0% | 12 | 3 | 3 | 6 | 12 | 36 |
| **2.** | HMA + Capsules | 0.5% | | | 3 | 18 | 12 | 54 |
| | | | | | | | 12 | |
| | | | | | | | 12 | |
| | | | | | | | 12 | |
| | Total | | 12 | 3 | 6 | 24 | 60 | 105 |

### 1.5 Thesis Outline

This thesis is comprised of 5 chapters which are briefly described below:

Chapter 1 gives information about background, problem statement, research objectives, scope of study and provides thesis outline. It briefly all these heading and layouts case for need for self-healing asphalt pavement.

Chapter 2 chapter provides information about binder, asphalt mixture, waste cooking oil and chemicals needed for making self-healing mixture. It discusses literature review about moisture susceptibility, semi-circular bending test and three point bending test. It explains why certain healing methods are superior to other healing methods.

Chapter 3 explains bitumen testing, aggregate testing and preparation of capsules. It explains marshal sample preparation and calculating Optimum bitumen content. Further it explain optical microscopy, thermogravimetric analysis, semi-circular bending test and 3 point bending test procedure.

Chapter 4 discusses the results of using encapsulated rejuvenator in asphalt mixture. The results of OM, TGA, SCB and three point bending are explained in relation to self-healing pavements.

Chapter 5 The most important findings and suggestions for further research are presented in this chapter.

# 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 Asphalt Binder

Asphalt binder is mostly utilized in the construction sector, such as on roads and airport runways, among other things. A solid at room temperature, asphalt binder changes state from solid to liquid as the temperature rises, reaching a liquid state at temperatures over 80°c. The asphalt binder is mixed with the aggregate in an asphalt factory at temperatures ranging from 140 to 160 degrees Celsius for 30-40 seconds before being put on site at temperatures ranging from 140 to 150 degrees Celsius. It consists of various molecular compositions with various chemical structures; especially carbon and hydrogen, termed hydrocarbon. Bitumen is a viscoelastic substance. Other structure consists of components such as oxygen, sulphur and nitrogen, which is called a heteroatom structure. The characteristics of asphalt split into two portions, namely solid asphalt and liquid maltenes, are therefore well balanced. Solid asphalt contributes to viscosity and rigidity while saturated, aromatic and resins fragmented maltene gives a bitumen flexibility. These characteristics can be evaluated by various rheology assays. The characteristics of bitumen should be understood, because asphalt pavement's behavior varies widely with changes in bitumen's properties. The presence of organic compounds effect the behavior of binder that in turn causes things like ageing, early deterioration and resistance to healing. The ratio of malentene to asphaltene is important matric that is currently under study. After cooling, the asphalt binder returns to a semi-solid condition, and driving is permitted on the newly paved roadway after 24 or 48 hours, depending on the temperature. Asphalt binder is created by the fractional distillation of crude oil. During the fractional distillation process, various byproducts are generated, including gasoline, diesel, and kerosene, among others (Speight, 2015).

## 2.3 Asphalt Self-healing

Because to its inherent characteristics, asphalt has the ability to repair itself naturally. Qiu (2012b) characterizes self-healing of bitumen as a viscosity-driven mechanism that works by inhibiting crack formation in bitumen to restore the bitumen's original characteristics. The fracture healing mechanism in bitumen is seen in Figure 2.1. A time and temperature dependent process, self-healing of asphalt is also controlled by other physical variables, such as moisture content.

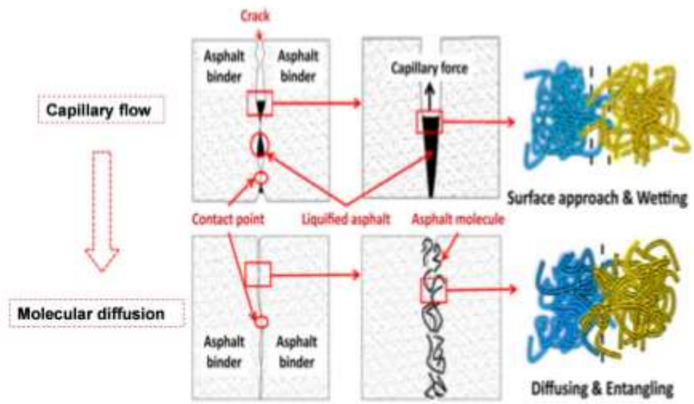


**Figure 2. 1: Crack healing process in bitumen (Sun et al., 2017).**

Asphalt pavements degrade with time as a result of factors such as oxidative stress, UV radiation, moisture damage, and traffic loads. As a result, the stiffness of the pavement increases as the rheological characteristics of the binder used in the pavement's production change. Consequently, the stiffness of the pavement is enhanced. In the course of the natural ageing process, the degradation of pavements, including cracking, becomes more severe. Since Bazin and Saunier first proposed the concept of self-healing asphalt concrete in 1967, a great deal of study has been conducted to better understand the behaviour of self-healing asphalt concrete in a variety of settings. A number of technologies are currently available on the market that may help to accelerate the healing process in asphalt mixes. These technologies include: Technology such as microwave heating, electromagnetic induction (also known as pulsed electromagnetic induction), and microcapsule technology are examples of this kind of technology (also known as infrared heating). Internal stresses reach a certain level and the capsule ruptures, allowing the release of regenerative chemicals that assist in the mending of fractures and the restoration of the pavement's functioning to its original state of operation. It is also commonly recognized that raising temperatures in the surrounding environment may speed the self-healing process of asphalt mixtures, which is an extra benefit.

The ability of asphalt mixes to self-heal has seen substantial advancement in recent years, thanks to the efforts of researchers. There is a practical and effective approach for enhancing the self-healing capabilities of asphalt concrete that have been demonstrated. This approach involves embedding healing chemicals within asphalt concrete rather than relying on external induction or microwave heat to achieve the desired results. As asphalt pavements age, they fracture and lose their qualities as a result of the impacts of fatigue loading and the environment on their structural integrity. As a result of capillary force, the healing chemical will seep through the inner layer and into the asphalt, softening the surrounding area and enabling the crack to repair on its own. Several researches have been carried out on the characteristics of rejuvenators, with the purpose of understanding their self-healing abilities, which may be employed to return the original properties of old asphalt to their original state.

When it comes to microcapsule-based healing, microcapsules with a core–shell structure on the order of a micron are used as a carrier. The microcapsule is formed mostly of a core and a shell, with the shell consisting primarily of polymer material and the core consisting primarily of a rejuvenating repair agent; the core is composed primarily of polymer material. Achievable results may be achieved with it in asphalt binder and mortar applications because to its micron size. Numerous investigations have shown that the oil contained within the microcapsules cannot be released in sufficient quantities to repair the fractures that have been formed. If the microcapsule is broken by fracture or compression, these healing chemicals may be released; however, they are only effective once and cannot be released again. However, there was a drawback to using this technique: once the capsules were fractured, they could no longer be utilised again.

The capsule behavior is advantageous for self-healing asphalt because it allows for the regeneration of old binder, which is excellent for the environment. The reuse of rejuvenating agents, as well as the creation of capsules with a multi-compartmented structure that may be used multiple times for fracture healing, as opposed to traditional shell-core capsules that rupture only once, are the primary focus of our research. In order to build a mathematical model defining the link between various healing variables, multi-compartmented capsules will be tested for their influence on the self-healing capacities of asphalt mixes at various healing intervals and temperatures in all of the aforementioned studies.

## 2.4 Waste Cooking Oil

Yan et al. 2022 studied the effect of Waste Cooking Oil to find out the effect of WCO on mechanical properties of asphalt mixtures. It was found out that WCO positively impacts the low-temperature properties of mixture and has unfavorable results for high temperature performance. They used European rock bitumen (ERB) in addition to waste cooking oil to counter the unfavorable impacts of WCO on asphalt mixture.

Oldham et al. (2021) investigated the benefits of esterifying waste cooking oil to reduce its acidity for use as a rejuvenator for old asphalt. Apart from their role in bitumen, the effect of acidic chemicals on the moisture susceptibility of bituminous composites made of siliceous stones and bitumen was also investigated. Bio-modifiers such as used cooking (vegetable) oil have raised the amount of acidic compounds in bio-modified bitumen significantly. Acids form fast bonds with siliceous stones but are promptly replaced by water molecules, resulting in stone degradation. By reducing or removing acidic components, transesterification may improve the resistance of waste cooking oil to moisture deterioration. Not only is waste cooking oil a suitable rejuvenator for old bitumen, but it is also resistant to moisture damage, according to the research.

An investigation into how adding WCO enhanced the low-temperature performance of a neat modified warm-mix asphalt binder (Li et al., 2020) was conducted (WEAB). The addition of WCO to the clean WEAB increased the damping and thermostability of the material. When the waste oil content was less than 4 percent wt, the addition of WCO enhanced the elongation at break of the neat WEAB by a significant amount.

Researcher Li et al. (2019) studied how WCO treatment affects the SARA fractions of varying asphalt binders and concluded that WCO can revitalise weathered bitumen by radically affecting the macroscale attributes of the aged. Although it is still a challenging challenge, predicting molecular interactions between the binder and rejuvenator chemical components, the colloidal index only predicts a general rearrangement of discrete fractions, which is of little utility.

Cooking and frying produce large amounts of waste cooking oil (WCO), which has the potential to be a great rejuvenator because of the lighter oil components included in WCO that are analogous to those found in asphalt (Sun, Yi, Huang, Feng, and Guo, 2016). Due to WCO's lesser oil components, it has the potential to be an effective rejuvenator. Asphalt, on the other hand, contains heavier oil components.

When modified WCO was used, the researchers (Azahar, Jaya, Hainin, Bujang, and Ngadi, 2016) detected a change in viscosity. The viscosity and internal resistance of processed WCO are greater than those of untreated WCO. This may explain why chemical modification of WCO improves adhesion to asphalt and makes the binder firmer than unmodified WCO, compared to unmodified WCO. Also associated with better adhesive properties of the combinations is a greater viscosity of the mixtures. As a result, the performance of treated WCO suggests that it may be better appropriate for use as a rejuvenator.

## 2.5 Encapsulation Method

(Norambuena-Contreras et al., 2020) created Ca-alginate capsules with bio-oil as the primary component utilising a Syringe pump and a falling funnel lately. Instead of dropping funnel-made capsules, the syringe pump created capsules that had better size distribution, form regularity, and density.

Shu et al. (2019) used a microfluidic system to generate fibres with three distinct shells. The Ca-alginate/grapheme oxide fibre demonstrated a variety of responses to cracking and heating, suggesting that it has the potential to considerably improve the self-healing capability of asphalt mixes.

(Shu et al., 2019) employed varying flow rates of core and shell material to adjust the internal structure of Ca-alginate fibres, and they were successful in changing the internal structure. Additionally, ca-alginate fibres in a variety of configurations, such as the compartmented fibre containing rejuvenator droplets and the hollow fibre containing a rejuvenator jet, were produced. It is possible to observe that the core material is not kept in an egg-box structure, but rather that it is just wrapped in the shell material if you use a microfluidic device.

Al-Mansoori et al. (2018) developed manufactured capsules which were found to increase the healing level of asphalt mixtures when temperatures were lower than 40 degrees Celsius. Even though Ca-alginate capsules accounted for small portion of the asphalt binder weight in dense-graded asphalt mixtures, their rheological properties were not impaired.

Sun et al. (2018) shoed important point to be taken care for the success of microcapsule manufacturing and has been demonstrated. Microcapsules of acceptable quality must be produced under strict control of the production parameters.

The performance of microcapsules can be improved by integrating inorganic elements inside the shell, such as carbon nanotubes, nanoscale calcium carbonate (Wang et al., 2016), nanoscale aluminium oxide (Jiang et al., 2015). When compared to organic substances, inorganic substances have superior adhesion to asphalt concrete and hence increase interaction between microcapsules and asphalt concrete. The formaldehyde component in the shell of the microcapsule, on the other hand, is a source of worry for both the environment and human health.

It was reported in 2016 by Micaelo et al. that CaCl2 coagulation solution was used to coagulate a saline alginate solution containing a rejuvenator, which was then washed and dried to get Ca-alginate capsules/fibers after the procedure was complete. The addition of sodium alginate/rejuvenator solution to a CaCl2 coagulation bath and the pouring of the solution via an aperture results in Ca-alginate capsules with particle sizes greater than 1 mm. In order to get Ca-alginate fibres with diameters on the order of micrometres, the sodium alginate/rejuvenator solution must be continuously injected into the calcium chloride coagulation solution using a needle. Before the fibre can be combined with the asphalt, it must first be cut into short fibres of a specific length and then chopped again.

To develop the first porous sand capsules, Garcia et al. (2016) employed conventional rejuvenator/vegetable oil as the core material, which was then encased in an epoxy resin and cement shell to form the first porous sand capsules.

## 2.6 Moisture Susceptibility

The sensitivity of HMA pavements to moisture is the most prevalent cause of pavement distress. High-performance asphalt mixes may be termed sensitive to moisture if it weakens in the presence of water. A substantial level of weakness could result in stripping (Figure 2.2).

**Figure 2.2: Pavement with moisture damage**

Using a hydrophobic material on the aggregate surface, (Hamedi, Shabani, & Safargar, 2020) investigated the effects of coating the aggregate surface with a hydrophobic substance on the polar features and hydrophilic properties of the aggregate mixture. It was discovered that the hydrophobic coating on aggregate surfaces increased the acidic component of the surface free energy while reduced the alkaline component for both types of aggregates. These adjustments will result in improved bitumen-aggregate adhesion as well as a better bitumen coating on the aggregate surface as a consequence of the modifications. Thermal analysis revealed that the aggregate surface coating reduced the separation energy of the system and reduced the desire to peel the aggregate surface coating.

(Ameli et al. 2020) found out major of the issues with asphalt mixes' durability is moisture damage, which is sometimes referred to as stripping. This is often caused by water in conjunction with repetitive traffic loads, resulting in a scouring effect as the water is forced into and drawn out of the pavement spaces. Stripping occurs when water enters between the aggregate and binder film, rupturing and weakening the adhesive connection between the aggregate and binder, resulting in the binder film's separation and removal from the aggregate's surface.

(Omar et al. 2020) reviewed the elements affecting vary according to the materials' characteristics, the mixture's qualities, the construction circumstances, and field factors after construction. Bitumen and aggregate have distinct chemical, physical, thermodynamic, and electrostatic characteristics, and their behavior vary in dry and wet environments, creating misunderstanding over how to prevent moisture damage.

According to the findings of Ahmed and Hossain (2020) the hot mix asphalt has properties owing to binder and aggregate that makes it susceptible to deterioration via moisture. As moisture infiltrates the binder and aggregate bond gets negatively affected leading to stripping and moisture related damages. One of examples of prolonged exposure to moisture damage is potholes that really impair traffic flow on road and damage traffic safety.

According to the findings of (Majidifard, Tabatabaee & Buttlar, 2019), used reclaimed asphalt and waste cooking oil and found out that increasing RAP and WCO percentage negatively affected the moisture resistivity of asphalt pavement.

According to the findings of (Zaumanis, Mallick, Poulikakos & Frank, 2014), 100 percent RAP regenerated asphalt with 12 percent waste cooking oil led to anti-synergistic effect on to moisture deterioration and failed to fulfil the minimum moisture degradation criteria.

## 2.7 Semi Circular Bending

A large number of researchers are interested in the SCB test because of its ease of manufacture and conformity to the genuine mechanical condition of asphalt pavement, among other factors.

To produce a notch in the bottom of the SCB , Xu et al. (2019) prepared asphalt mixture and placed the specimen in the SCB test chamber. They conducted three SCB experiments at 0°C on the same material, followed by a 20-hour healing treatment at 23°C. In testing, it was discovered that a porous asphalt mixture had high healing efficacy; however, this benefit was lessened when the injury exceeded the maximum healing capacity.

Using the SCB technique to measure the quantity of crack healing in an asphalt mixture containing calcium alginate fibres, Tabakovi et al. (2017) were able to detect how much crack healing occurred. His innovative idea of using self healing fiber that has rejuvenator in it has limitation of size and rejuvenator space.

## 2.8 Three Point Bending

The materials were heated at 150°C for around 2 hours before being mixed for approximately 3.5 minutes. The following raw materials were added to the mixing bowl in the following sequence: bitumen and fibres first, coarse aggregate second, fine aggregate third, and filler fourth, all in the same order. The flexural strength and strain capacity of asphalt concrete were determined by the use of a three-point bending test method.

Researchers (Norambuena-contreras et al., 2019) evaluated the self-healing potency of a bituminous mixtures containing calcium alginate capsules using a modified 3PB analysis to test the healing performance, and determined that the adequate capsule content is 0.5 percent by weight of asphalt mixture, bearing in mind the healing performance and rheological properties of the asphalt mixture.

Despite several hours of conditioning at -20°C, the 3PB test was able to break apart an asphalt mixture beam with a transverse notch at the mid-point of the bottom surface, resulting in two pieces of fragmented asphalt mixture. Second, the capsules are activated. It took several hours for the broken beam to be inserted in a steel mould at a temperature of 20 degrees Celsius with a thin plastic barrier between the fractured surfaces. Afterwards, a compressive pressure was applied evenly to its top surface, causing the capsules to burst and the rejuvenator to be released into the environment. (3) The process of recuperation (recuperation). After being freed from the plastic barrier, the broken beam was re-encased in the steel mould for a few hours in order to enable the fractures to repair.

Al-Mansoori et al. (2018) performed a three-point bending test on an asphalt mixed beam containing calcium alginate capsules in order to determine its strength. Capsules of calcium alginate significantly enhanced the healing levels of asphalt beams when the temperature was less than 40°C, but they were useless when the temperature was more than 40°C. Therefore, it was recommended that the crack healing test be performed at temperatures below 40 degrees Celsius.

With the help of Qiu's research, Su et al. (2015b) investigated the self-healing behaviour of asphalt materials containing microcapsules by utilising a modified BOEF model. Microcapsules having a core–shell structure of a micron or less are utilised as carriers in microcapsule-based healing. The microcapsule is made up of a core and a shell, with the shell being predominantly polymer and the core being mostly rejuvenating repair agent. Due to its micron size, it may be used in asphalt binder and mortar applications. Numerous studies have revealed that the microcapsules' oil cannot be released in sufficient amounts to mend the cracks. These therapeutic chemicals may be released from the microcapsule if it is fractured or compressed, but they are only effective once. However, once the capsules were shattered, they could no longer be used.

According to Qiu (2012a), a unique test procedure known as BOEF was created in which the beam was linked to a rubber base, allowing the fractured surface to close automatically when the beam was broken.

## 2.9 Chapter Summary

Asphalt's self-healing qualities have been known for some time, but the method by which they may be expedited and used in the most suitable manner is still being researched. It is reported in the literature that two novel methods of self-healing asphalt pavements have been developed:

encapsulated rejuvenators and induction heating. Both methods are designed to help asphalt pavements begin and accelerate their natural self-healing processes. Due to the fact that it creates heat inside the asphalt binder, induction heating has been demonstrated to be an effective self-healing technique. This is because it reduces the viscosity of the asphalt binder near cracks. For the time being, a key restriction with regard to this technology is that it is only effective on surfaces that are close to the pavement's surface and is unable to penetrate all the way down to the asphalt layer's depth. Aside from that, the induction heating equipment used on roadways is large and slow to move, resulting in delays during maintenance and repair operations. A different kind of self-healing mechanism is addressed in this section. Encased rejuvenators are used in the asphalt mixture, and they are encapsulated in the asphalt mixture. When used in conjunction with other techniques, this technology has the ability to restore the asphaltene/maltene ratio in asphalt pavements. The stress created by the traffic load reaches a point when the capsules burst, releasing their rejuvenator to mend the cracks in the asphalt. The results of the first investigation into this strategy have been promising.

# CHAPTER 3: RESEARCH AND TESTING METHODOLOGY

## 3.1 Introduction

This chapter outlines the technique used to accomplish the study's goals, which include material collecting, material testing, specimen preparation, and numerous specimen tests. The investigation included a control sample as well as a capsule-containing sample. This chapter discusses determining OBC using Marshal Mix Design. On the basis of the OBC, performance samples will be created with and without capsules. Moisture Susceptibility and Healing Evaluation are included in the performance testing. This chapter will cover the equipment used, the technique for preparing samples, and the input parameters utilised during testing on the specimen obtained.

## 3.2 Methodology

The investigation was conducted on asphalt concrete specimens constructed using aggregate from the Margalla quarry 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 and compaction. The optimal binder content was determined by experimenting with various binder percentages with a 0.5 percent increase. Marshall samples comprising waste materials were made with the optimal binder content in order to detect moisture damage. The tests were conducted under controlled settings and at the temperatures stated for each test. Marshall samples were produced at OBC for TSR testing (unconditioned and conditioned), and then tested using a Universal Testing Machine (UTM). ALDOT-361 was used to condition samples for TSR. Additionally, this chapter explains the exact processes for the tests stated above, as well as the testing equipment and input parameters utilized during these tests. To examine the healing process, two different tests were performed: the semi-circular bending test and the three-point bending beam test. By comparing the findings of these samples to those of control samples, the effectiveness encapsulated rejuvenator was determined.

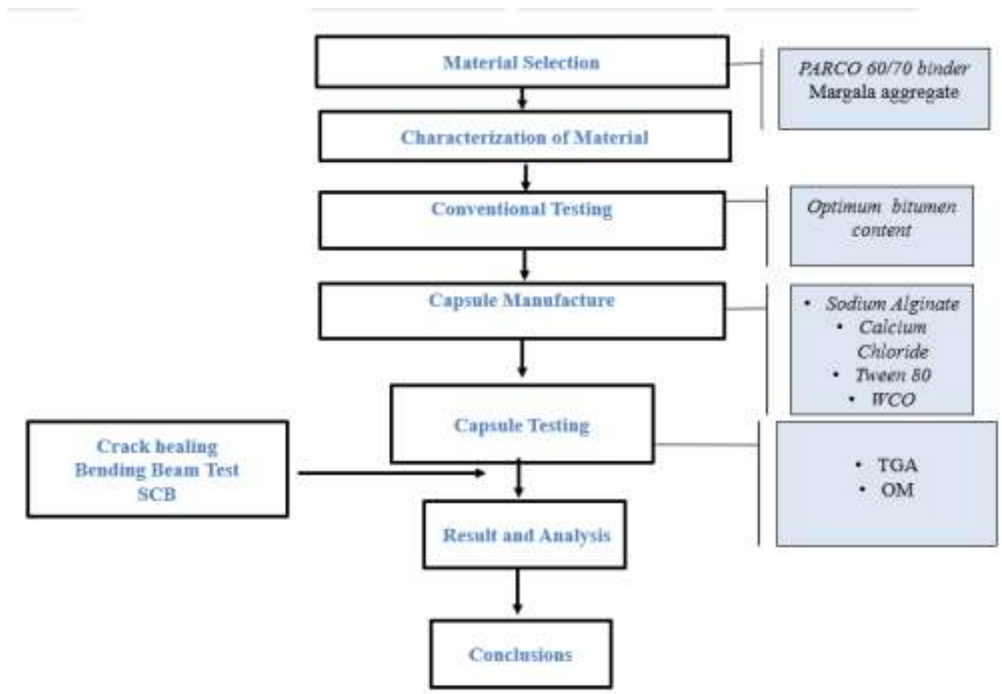


**Figure 1.1: Research Methodology**

### 3.3 Material Selection

For this study, fine and coarse aggregate were obtained from the Margalla quarry, and a binder with a penetration grade of 60/70 was obtained from Parco. Binder 60/70 grade was chosen since it is widely utilised in Pakistan's asphalt mixtures. Additionally, it is suitable for usage in places with mild to cold temperatures. Asphalt is a mixture of aggregates, bitumen, and air. Typically, the aggregate is 95 percent by weight since it offers the majority of resistance to permanent deformation, while the bitumen makes up the remaining 5%. Due to the fact that air is weightless, it plays no part in the equation. Asphaltic mix is made of 85% aggregate, 10% bitumen, and 5% air. To ensure that asphaltic mixes fulfil needed criteria, extensive laboratory testing of chosen components, aggregate, and bitumen is required.

#### 3.3.1 Aggregate

Margalla hills crushed stone was gathered. Aggregate is critical for the strength and longevity of HMA pavements. It bears the utmost weight that the pavement can bear. The texture and form of aggregate have a significant effect on these strength-related parameters. In general, aggregates with a greater degree of angularity and rough texture are better able to withstand stresses in pavement caused by repetitive loads. Numerous tests are conducted in accordance with ASTM and BS standards to determine the aggregate qualities that effect the pavement.

**Figure 3. 1 (a) : Margalla Hills Crush Plant**

### 3.3.2 Bitumen

Bitumen is used as a binder in asphalt mixtures. It is blackish or dark brownish and is produced from residues distillation of petroleum. Bitumen's consistency, safety, and purity are required for engineering and building needs. In Pakistan, mostly bitumen of grade 60/70 is utilized per weather conditions. So bitumen of grade 60/70 was collected from PARCO.


**Figure 3. 1 (b) : Bitumen**

### 3.3.3 Preparation of Capsules

Sodium Alginate has been used as emulsifying agent with oil while calcium chloride supplied as granular pellets acquired from Daejung Chemicals.

**Figure 3. 2 (c): Sodium Alginate & Calcium Chloride**

Sodium alginate is the major ingredient that forms the capsule shell. It was discovered in 1881 for its exceptional capacity to concentrate liquids and fabricate membranes. After 50 years, large-scale commercial manufacturing of sodium alginate began because to its cheap cost and environmental friendliness. It offers promising usage in capsule making due to its polymer nature.

Capsules were manufactured using a calcium-alginate polymer to contain waste cooking oil. The pills included filtered waste cooking oil bought at a very low price from a local merchant. Sodium alginate was powdered, and calcium chloride was pelletized. To produce capsules, we use 2 percent of sodium alginate and mix it in water along with oil and water. Separately, a 2.5 percent calcium chloride solution in water is made. Emulsion of sodium alginate is poured into a dropping funnel. Tween 80 is used with calcium chloride and sodium alginate to help decrease the size of capsules. The emulsion is put into a solution of calcium chloride. Capsules are created by an ionotropic gelation process. Throughout the procedure, a calcium chloride solution is agitated. Capsules are decanted and placed in a 40°C oven for 24 hours. Following that, these capsules may be employed in self-healing asphalt.

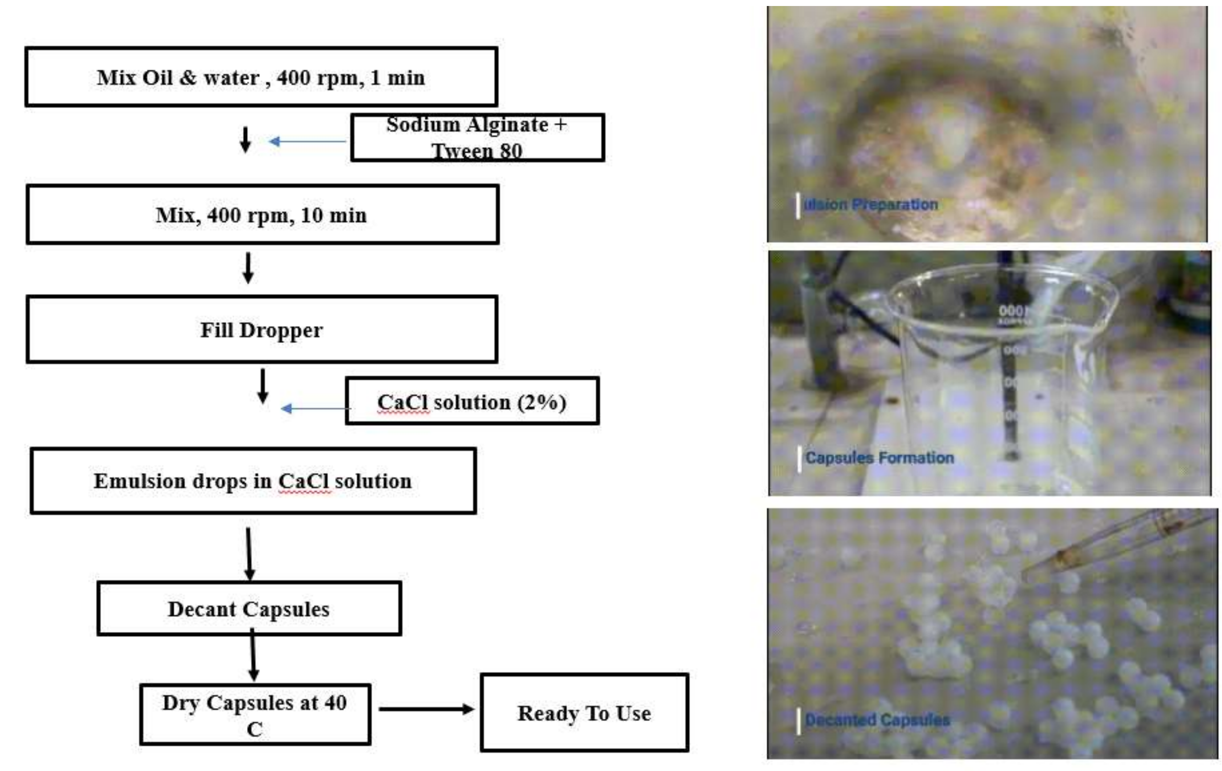


**Figure 3. 2 (b): Preparation of Ca-Alginate Capsules**

### 3.3.4 Waste Cooking Oil

Waste cooking oil was obtained from local vendor and used in our research. Waste cooking oil has been proved to be rejuvenating agent that an revitalize asphalt lost properties. Waste cooking oil is produced in excessive quantities in Pakistan.

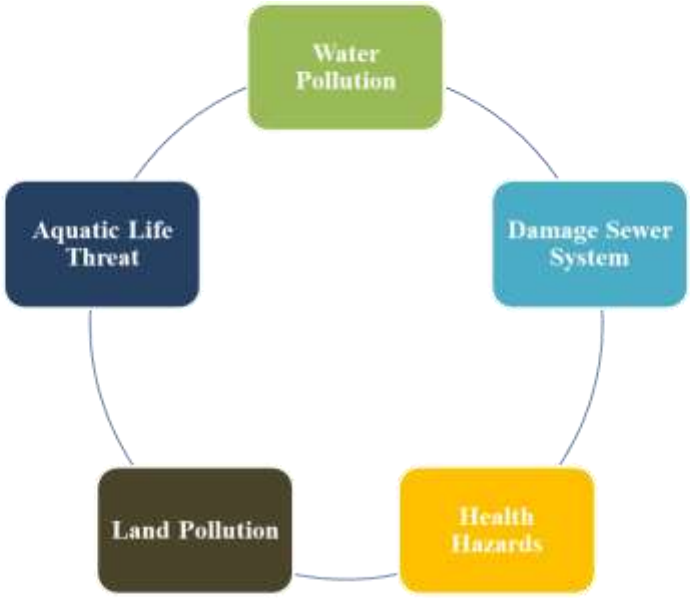


**Figure 3. 2 (c): Disadvantages of Waste Cooking Oil**

### 3.4 Material Testing

In most cases, the term "aggregate" refers to a variety of different types of mineral minerals. They can either be natural or man-made. There are many types of natural aggregates that can be extracted from a quarry. After that, the rocks are broken down into smaller pieces that can be used. Paving materials are made up of aggregate, which serves as a skeleton and is responsible for their structural integrity and load-bearing capacity. Asphaltic mixtures' basic strength attributes are determined by the surface's size, shape, texture, and aggregate gradation, all of which must be strong, durable, tough, hard, appropriately graded, and low in porosity. It must be free of dirt, roughness, and water. The chosen aggregate has undergone a slew of tests in the lab.

#### 3.4.1 Aggregate Tests

The fundamental strength attributes of asphaltic mixes are determined by the size, shape, texture, and gradation of the aggregate, which must be strong, durable, tough, hard, appropriately graded, and have a low porosity. It must be free of impurities, abrasive, and have hydrophobic surfaces. Table 3.1 shows all the tests carried out on both fine and coarse aggregate to find their properties and suitability. The standards are also mentioned in the table below.

**Table 3. 1: Test performed on aggregates**

| Test Description | Specification Reference | |
|---|---|---|
| Elongation Index (EI) | ASTM D 4791 | |
| Flakiness Index (FI) | ASTM D 4791 | |
| Aggregate Absorption | Fine Agg: | ASTM C 127 |
| | Coarse Agg: | |
| Impact Value | BS 812 | |
| Los Angles Abrasion | ASTM C131 | |
| Specific Gravity | Fine Agg: | ASTM C 128 |
| | Coarse Agg: | ASTM C127 |

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

In accordance with ASTM D 4791, flaky particles are classified as such if their lesser dimension is less than 0.6 of their mean sieve size, while elongated particles are classified in the same manner as flaky particles if their length exceeds 1.8 of their mean sieve size. During the practise of compaction, these flat and elongated particles might lock together more quickly, making the procedure more difficult to complete. The aggregate particles reorient during the process of compaction, and these particles tend to shatter during compaction, causing the aggregate gradation to become finer and so aiding in the reduction of Voids in Mineral Aggregates (VMA). According to the ASTM requirements, the percentage of elongated and flat particles must be less than or equal to 15% of the total number of particles. The results of the tests carried out on chosen aggregates fall within an acceptable range of values. The results of the flat and elongated particles tests are shown in the table below. This test is used to assess the proportion of flaky and elongated particles in an aggregate mixture. An angular shape is preferred in order to ease the interlocking of aggregate particles. The flakiness index must be less than 15 percent, and the elongation index must be less than 15 percent, to be considered acceptable.

**Figure 3. 3: Shape Test Apparatus**

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

Three weights were established for the purpose of calculating Specific gravity is defined as the weight of oven dried aggregate, the weight of aggregate entirely submerged in water, and the weight of aggregate at the saturated surface. The specific gravity and water absorption of fine aggregate were evaluated using ASTM C 128.

The specific gravity of fine and coarse aggregates is critical in the manufacture of asphalt paving mixes. Engineers typically employ it in the design of pavements and buildings as well. Bulk specific gravity is used to determine the amount of binder absorbed and the VMA. The phrase "relative density" is also used to refer to the specific gravity of aggregate material, which represents the weight volume properties of the material. It refers to a material's mass to volume ratio at a constant temperature. The coarse aggregates are those that remain on sieve No. 4 after passing through sieve No. 4. The fine aggregates are those that pass through sieve No. 4. Separately, the specific gravities of coarse and fine aggregate were determined.

The specific gravity and water absorption of coarse aggregate particles were determined using ASTM standard technique C 127. The aggregates were first passed through sieve #4, and those that remained were dried in an oven and then soaked in water for 24 hours. The aggregates were then rolled on a cloth and their saturated weight determined. Following that, the aggregates' submerged weight was evaluated, as well as their specific gravity and water absorption. The oven-dried sample contains no water, but water fills the aggregate pores in the saturated surface-dry state.

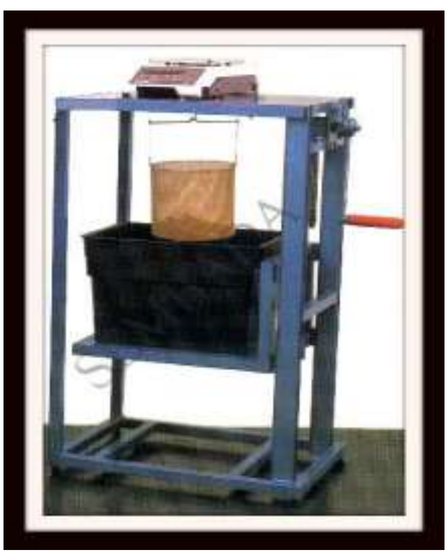

**Figure 3. 4: Specific Gravity Test**

#### 3.4.1.3 Impact Value of Aggregate (BS 812)

An aggregate's impact value demonstrates its resistance to a sudden shock. This procedure is recognised by British Standard BS 812. The impact testing equipment consists of an impact testing machine, a cylindrical mould with a diameter of 75 mm and a depth of 50 mm, a tamping rod with a circular section of 10 mm and a length of 230 mm, and sieves in the sizes 1/2", 3/8", and #8. (2.36mm.) Around 350g of aggregate was obtained by passing it through a 1/2" sieve and keeping it on a 3/8" sieve and layering it in three (3) layers in the mould of the Impact Testing Machine, with each layer tamped 25 times. The sample was transferred to the larger mould on the machine and 15 blows were given from a 38cm height with a 13.5 to 14kg hammer. The resulting aggregate was detachable and went through filter #8. The impact value was calculated by the proportion of aggregate that passed through a 2.36mm diameter sieve. The findings are summarised in the table below.

**Figure 3. 5: Impact value Test Apparatus**

#### 3.4.1.4 Los Angles Abrasion Test (ASTM C 535)

Los Angeles (LA) Abrasion Testing is widely performed to test the resilience of an aggregate against deterioration. This experiment exhibits the aggregate's toughness and abrasion resistance, or its ability to withstand wear caused by high traffic loads. Abrasion resistance is crucial to evaluate because the aggregate in the mix is repeatedly stressed, leading in fragmentation, degradation, and crushing. This test is accomplished with the use of a LA Abrasion machine, a weight balance, a set of sieves, and steel balls known as charge. This procedure was based on testing or grading B. 2500 g of aggregate retained on 12" and 3/8" sieves, totaling 5000 g (W1) of material, were loaded into the Los Angeles abrasion instrument, together with 11 steel balls or charges. Following that, it was spun 500 times at a speed of 30–33 revolutions per minute. After that, the material was sieved using a 1.7mm sieve. The weight of the sample going through it was observed (W2). Calculate the abrasion value using the formula = W2/W1 100. Coarse aggregates having an abrasion value of 30% or less are permissible under NHA standards. This test followed the ASTM C 131 standard. The following table highlights the LA Abrasion test results.

**Figure 3. 6: Los Angles Abrasion Machine**

### 3.4.2 Bitumen

Bitumen acts as a binder in pavements, securing aggregate in place. Bitumen is a semi-solid substance whose behaviour varies according on temperature and meteorological conditions. Bitumen is a binder that is used in asphalt mixes. It is blackish or dark brownish in colour and is derived from petroleum distillation leftovers. It occurs naturally in asphalt lakes or is synthesised in petroleum refineries from crude oil leftovers. Bitumen's consistency, safety, and purity are required for engineering and building needs (MS-4 Manual 2003). These qualities have a significant impact on the performance of asphalt mixtures. Due of the substantial fluctuations in these characteristics over time, the age of the binder is crucial for understanding its behaviour. The SHRP has developed a unique technique for forecasting the physical characteristics of bitumen in the field. The Performance Grading (PG) system is the name given to this system. It emphasises the need of viscosity control at low temperatures, although this is not an issue in our nation due to the ambient temperature. Numerous experiments were conducted to determine the appropriateness of binders for HMA. Typically, tests on binders are conducted at a temperature of 25 oC to compare the consistencies of asphalt binders, as consistency varies with temperature. The table below details the tests and their associated standards that were conducted on these binders. The experiments that were conducted are detailed below.

#### 3.4.2.1 Penetration Test (AASHTO T 49-03)

AASHTO T 49-03 specified a temperature of 25°C, a weight of 100 grammes, and a duration of 5 seconds for the test. This was one of the first experiments used to assess the consistency of asphalt binders. It quantifies the binder's softness and hardness in order to grade it. A higher penetration value indicates a flexible and thin binder. Binders with a low penetration value are desirable in hot regions; binders with a high penetration value are advised in cold climates. To begin, the binder is heated to a suitable temperature to ensure that it flows freely and does not trap air; nevertheless, the temperature should not be increased excessively, since this will compromise the binder's properties. The binder is then placed into a test container and maintained in a temperature-controlled water bath at a constant temperature of 25 C. After the container reaches the necessary temperature, it is removed and tested in a penetrometer with a 100g load passed via a needle for 5 seconds. Five values were gathered from each of two ARL 60/70 specimens during penetration testing. All findings obtained met the penetration test's requirements. The outcome of the penetration test is shown in Table 3.3

**Figure 3. 7 (a) : Penetration Test Apparatus**

### 3.4.2.2 Softening Point (AASHTO-T-53)

ASTM D 36 is the standard that specifies how this test with the ring and ball equipment should be conducted. To begin, the binder was heated to a temperature that permitted free flow without compromising its characteristics. Then, using a mould, it was flattened into horizontal discs. Once the discs were placed in the device, the balls were positioned on them. The temperature was raised until the binder allowed the balls to fall through the previously indicated distances.


**Figure 3. 7 (b) : Softening Test Apparatus**

#### 3.4.2.3 Ductility (AASHTO T 51-00)

It is conducted in accordance with ASTM D 113, which evaluates the binder's elasticity and adhesion properties. The physical property of ductility of bitumen is critical. It illustrates bitumen's behaviour as a result of temperature fluctuations. This test is conducted at a standard temperature of 25 C. Ductility is defined as the distance that a standard-sized specimen of binder (in a 1 in2 cross-sectional area briquette) lengthens without breaking when its two ends are dragged apart at a rate of 5 cm/minute at a specified temperature of 25 oC. The specimen must be a minimum of 100cm in length to pass the ductility test. Asphalt mixtures generated from less ductile bitumen fractured when subjected to repeated heavy traffic loads. The ductility test results for both binders are summarised in Table 3.

**Figure 3. 8: Ductility Test of bitumen**

#### 3.4.2.4 Flash and Fire point (D3143/D3143M-13)

The flash and fire point tests were performed in accordance with D3143/D3143M-13 standards. The flashpoint of a binder is the temperature at which bitumen fumes in COC cause a flash in the presence of flame. The binder's surface catches fire and emits flames for at least five seconds; this is the fire point. Bitumen was poured into a metal cup at a specific volume. It was then continually heated, and at specified intervals, a test flare 30 was passed over it. The temperature at which flash and fire erupted was determined when the parameters described above were satisfied. These temperatures were determined in three different experiments for each binder.

**Figure 3. 8 (b) : Flash and Fire point Test of bitumen**

#### 3.4.2.5 Viscosity test (ASTM D 4402 – 06)

A rotational viscometer uses the concept of torque. Using a rotating viscometer, AASHTO technique T-316 is utilised to evaluate the viscosity of the binder at increased temperatures. Viscosity is crucial at increased temperatures because it governs pumping, workability, mixing ability, and compaction. It may be performed at a range of temperatures, but for Performance Grade bitumen, it is performed between 135°C and 160°C due to the consistent production temperature independent of the climatic circumstances.

The Brookfield RV equipment was used for this purpose, as defined in ASTM D 4402 and AASHTO T 316. To begin, we heated the sample chamber, spindle, and environmental chamber to 135 and 160 degrees Celsius, respectively. After stirring to eliminate any trapped air bubbles, the bitumen sample was heated to aid flow and appropriately put into the sample chamber. After placing the sample into a temperature controller device, the sample was gently dropped into a spindle No. 27. After thirty minutes, it was heated to the appropriate temperature (135 o C or 160 o C) and left to equilibrate for ten minutes at that temperature. The spindle should be turned at a pace of 20 revolutions per minute to create between 2 and 98 percent torque. After the sample has reached room temperature and equilibrated, take three measurements, waiting one minute between each. Averaging the three values yields the viscosity.

**Figure 3. 9: Viscosity Test Apparatus**

**Table 3. 2: Test results on Bitumen**

| Test Description | Specification Reference | | Result(%) | Limits(%) |
|---|---|---|---|---|
| **Elongation Index (EI)** | ASTM D 4791 | | **2.9** | ≤ 15 |
| **Flakiness Index (FI)** | ASTM D 4791 | | **11.6** | ≤ 15 |
| **Aggregate Absorption** | Fine Agg: | ASTM C 127 | **2.15** | ≤ 3 |
| | Coarse Agg: | | **0.71** | ≤ 3 |
| **Impact Value** | BS 812 | | **17.2** | ≤ 30 |
| **Los Angles Abrasion** | ASTM C131 | | **23.9** | ≤ 45 |
| **Specific Gravity** | Fine Agg: | ASTM C 128 | **2.612** | - |
| | Coarse Agg: | ASTM C127 | **2.638** | - |

## 3.5 Capsule Testing

Calcium alginate capsules incorporating waste cooking oil were prepared using ionotropic gelation technique and were tested for surface morphology and thermal stability. These properties show us whether capsules are suitable for addition in asphalt pavement and can bear harsh environment of asphalt mixing and compaction.

#### 3.5.1.1 Morphological Testing of Capsules-

The morphology of capsules were tested to find out the surface morphology of capsules. This helps in understanding of bitumen interaction with capsules. The surface of capsules can impact the bond in asphalt matrix. Moreover, this shows us whether capsules are multicavity or egg-shell based. Multi-compartmented capsules can do healing multiple times and has longer life time.

#### 3.5.1.2 Thermal Stability of Capsules

Because the temperature at which asphalt is mixed is 160 °C, capsules must be able to withstand this temperature. TGA was used to determine the thermal stability of capsules when mixed at a high temperature of 160 C. The testing environment contains nitrogen. The TGA findings were plotted across a temperature range of 0-1000°C with a 10°C/min increment. The mixing temperature of asphalt can go upto 180 C and this means that capsules should be able to survive in such high temperatues. Thermogravimetric analysis tells us the percentage loss of mass of capsules upon heating at range of 0-1000°C.

**Figure 3. 11: TGA Test Apparatus**

## 3.6 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 3. 3: Test Matrix of Marshal Testing**

| Performance Testing Matrix | |
| --- | --- |
| Marshall Testing | |
| Bitumen Content % | No. of Samples |
| 3.5 | 3 |
| 4 | 3 |
| 4.5 | 3 |
| 5 | 3 |
| 5.5 | 3 |
| Total | 15 |

### 3.6.1 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: Sieving**

**Figure 3. 12 Mixing Machine**

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**

### 3.6.2 Mixing of Materials

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

**Figure 3. 15: Mixing of Material**

### 3.6.3 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. 17: Marshal Sample Preparation**

### 3.7 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**

Following the determination of Gmb, the specimen was conditioned and then established utilising Marshall Equipment for Marshall Stability and Flow. The sample is placed into the Marshall Apparatus at a continuous deformation rate of 5mm/minute until the specimen fails. The Stability value indicates the maximum load that the specimen can withstand, and the flow number in millimetres indicates the strain that occurs when the specimen is subjected to the maximum load. The Marshal Mix Design Criteria MS-2 suggests that the Stability value be at least 8.006 KN and the flow rate be between 2 and 3 millimetres per second for a surface intended to sustain heavy traffic loads.

To calculate the Gmm, weigh the loose mix first, then determine the calibration weight of the apparatus, transfer the mix to the apparatus, and apply vacuum. After removing the air contained in the mix, re-weigh the equipment containing the mix. Equipment.

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

**Table 3. 4: Volumetric Properties**

| Sr No | Bitumen % | Gmb | Gmm | VA % | VMA % | VFA % | Stability, KN |
|---|---|---|---|---|---|---|---|
| 1 | 3.5 | 2.33 | 2.48 | 6.13 | 14.35 | 57.32 | 10.37 |
| 2 | 4 | 2.36 | 2.47 | 4.27 | 13.57 | 68.54 | 12.23 |
| 3 | 4.5 | 2.39 | 2.46 | 3.62 | 13.22 | 77.66 | 12.53 |
| 4 | 5 | 2.39 | 2.45 | 2.15 | 13.38 | 83.92 | 11.38 |
| 5 | 5.5 | 2.4 | 2.44 | 1.73 | 13.68 | 86.04 | 9.77 |

At 4% air voids, the asphalt content is consistent with OBC. The mixture contains 4.3 percent OBC. Additionally, the volumetric qualities of the OBC obtained are verified. If all characteristics are within the MS -2 tolerances, everything is okay; otherwise, OBC must be changed. According to MS-2 Criteria, the VMA number should be larger than 13%, however it is 13.45 percent in this situation, which is within the range. The VFA number should be between 65 and 75 percent, however the estimated result is 69.22 percent. Stability should be at least 8.006 KN, but was judged to be 12.6 KN in this instance, and the flow rate of 2.7 mm is within the range of 2-3.5 mm.

**Table 3. 5: Specification Check**

| Parameters | Value Measured | Limits |
|---|---|---|
| **OBC** | 4.3 | – |
| **VMA (%)** | 13.45 | ≥13 |
| **VFA (%)** | 72.5 | 65−75 |
| **Stability (KN)** | 12.6 | ≥8.006 |
| **Flow (mm)** | 2.7 | 2−3.5 |

The graph between Asphalt content and different volumetric properties were plotted to find out OBC as shown in figure 3.19

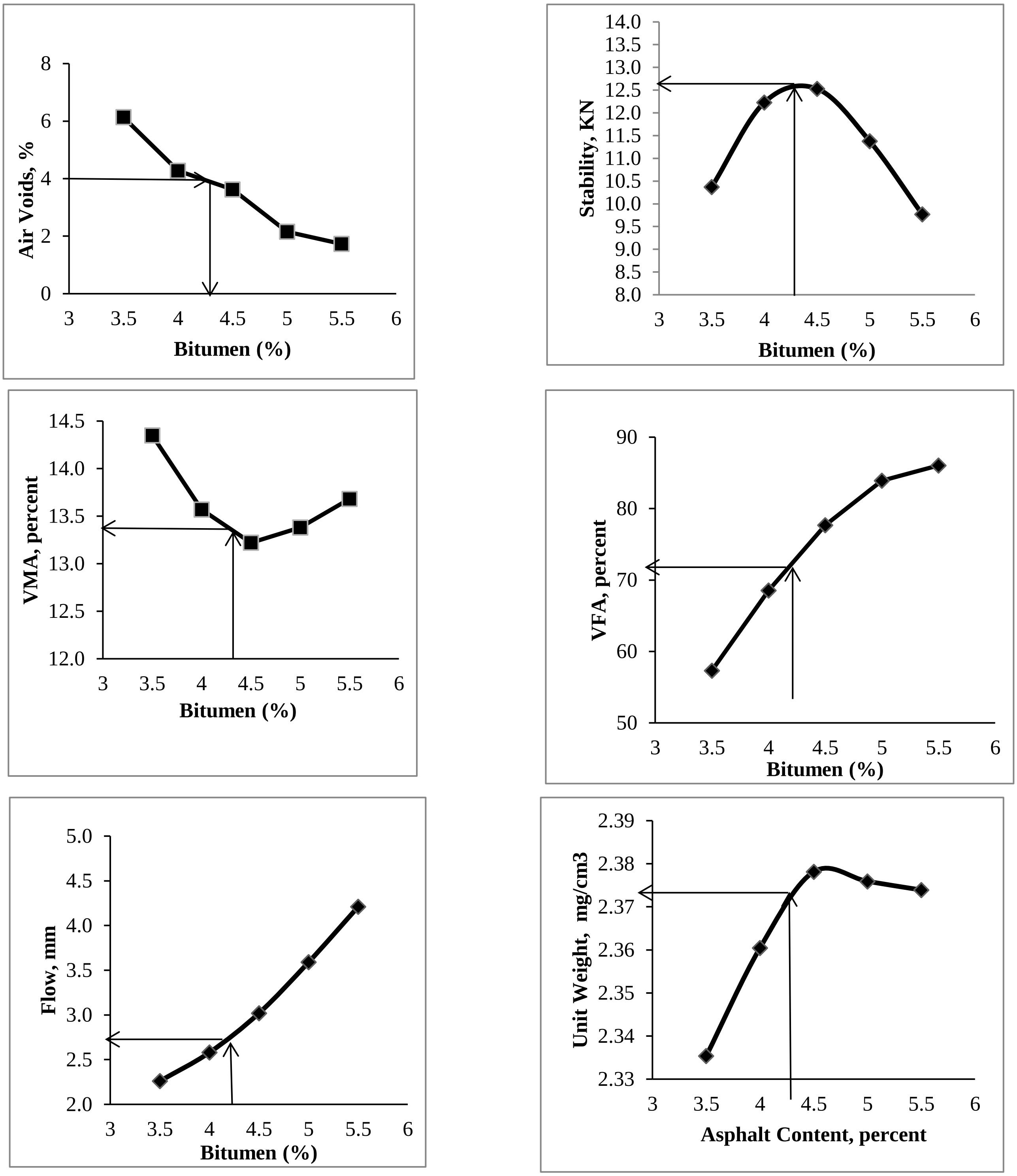


**Figure 3. 19: Volumetric Properties**

## 3.8 Preparation of Sample for Performance Tests

After finding out OBC, sample for performance tests were prepared i.e. for moisture susceptibility test, semi circular bending test and three point bending beam test.

### 3.8.1 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 0.5% percentages of capsules.



**Figure 3. 20: Dried & Decanted Capsules containing Oil**

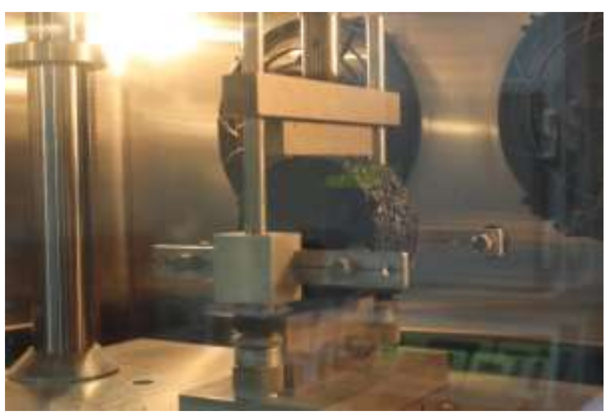

**Figure 3. 21: Moisture Susceptibility Test**

**Figure 3. 21: Unconditioned and Conditioned Samples**

## 3.9 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. Healing index is relationship between first load and subsequent peak loads and it tells us whether sample has recovered form crack or not. Healing index for control as well as encapsulated samples is calculated and shown in graphs.

$$K_{Ic} = Y_{Ic(0.8)}\sigma_o\sqrt{\pi a} \quad \text{------------------------------------------------eq(1)}$$

where:

$$K_{lc} = \text{ Fracture Toughness } \left(\text{Pa} \cdot \text{m}^{1/2}\right)$$
$$\sigma_{\text{o}} = \text{ Stress } (\text{N/m}^2),$$

These results can be used to find out a lot of properties related to cracks. The energy dispersed in producing these cracks so that a comparison can be built to find out the relationship between control specimen and encapsulated specimen. Figure 3.22 shows the setup in a UTM machine where sample are placed after conditioning. The samples are conditioned so that the crack produced is brittle not ductile. Three back-to-back loads are placed with repetitive conditioning. The results are explained in next chapter.

**Figure 3. 22: Schematic of SCB test**

## 3.10 3 Point bending

3 Point bending test was used to find out healing efficiency of asphalt mortar. 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. 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

**Figure 3. 23: Schematic of 3 point bending test**

### 3.11 Chapter 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. Capsules have been manufactured in the laboratory. The properties of capsules are discussed below.

### 4.2.1 Optical Microscope

Fig. 4.1(b) shows OM images of capsule cross sections at a 20x equivalent magnification. As can be seen, the capsules' interior structure is multi-cavity rather than core–shell. The little drops of oil were encased and encircled by a thick layer of cross-linked Ca-alginate shells. The cross-linked multi-cavity construction has a number of benefits over the conventional shell-core configuration. Additionally, the random distribution of the cross-linked structure inside the capsule may result in compartmentalized oil encapsulations. Thus, when a fracture is reached, the capsules will not release all of the oil, suggesting that capsules may allow multiple crack repair and long-term healing.

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**Figure 4. 1 (a): Capsules**

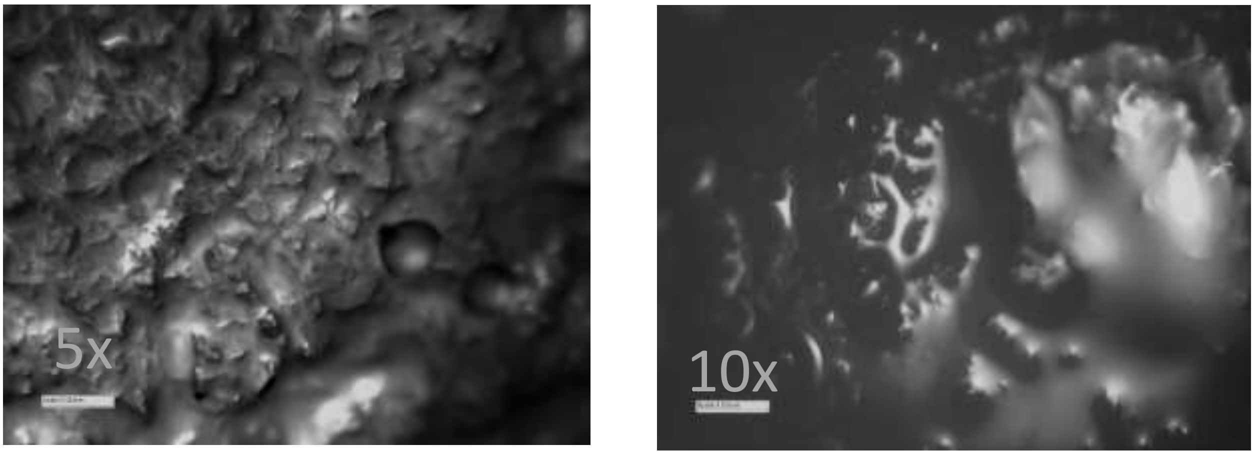


**Figure 4. 1(b): OM**

### 4.2.2 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 %.

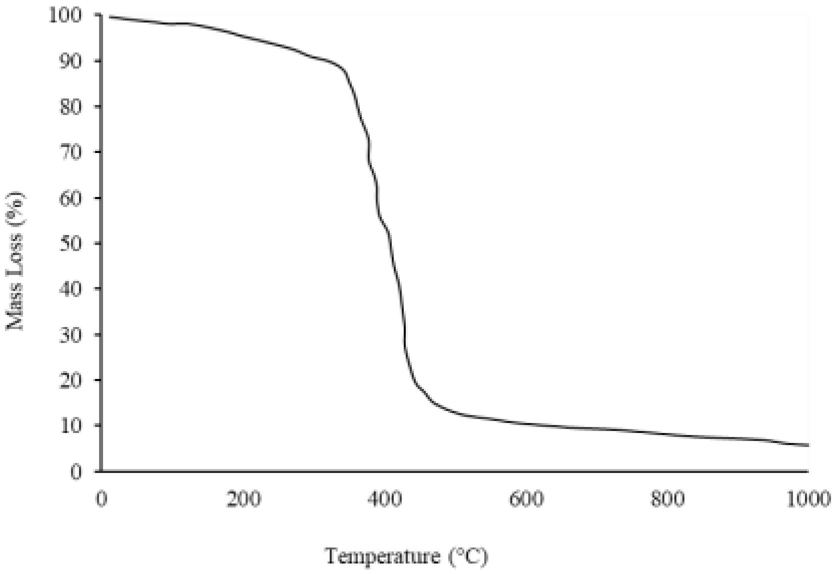


**Figure 4. 2: TGA Graph**

### 4.3 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.

$$S_t = \frac{2P}{\Pi t D}$$

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

### 4.3.1 Moisture susceptibility test result [ALDOT-361-88]

Figure shows that sample with 0.5% capsules has highest value of TSR. Thus, the incorporation of capsules decreases moisture susceptibility of asphalt sample. The reason behind increase in moisture susceptibility with increase in capsule content till 0.5 % is the increase in packing of asphalt matrix. It is proposed that if capsules increase beyond 0.5 % the quantity of capsules has increased beyond optimal dose and the moisture starts negatively affecting asphalt mixture. We take 0.5 % as optimal dose for our healing tests because higher doses might compromise other mechanical properties as well.

Asphalt undergoes high temperature during mixing and compaction phase and it increases beyond 170 C. This high temperature is important factor to consider during self healing evaluation using encapsulated rejuvenators. Our test results conclude that the tensile strength for unconditioned samples is 794.1 and 802.4 while tensile strength for conditioned samples is 734.2 and 779.3 for 0 % and 0.5 % capsules respectively. This indicates that conditioning has decreased tensile strength of samples. TSR for 0 % capsules is 92.46 while for 0.5 % capsules it has increased to 97.12 %.

**Table 4. 1: Moisture Susceptibility test**

| Fiber content (%) | Tensile Strength (Uncond.) (kpa) | Tensile Strength (Cond.) (kpa) | TSR(%) | Remarks |
|---|---|---|---|---|
| 0% | 794.1 | 734.2 | 92.46 | Pass |
| 0.5% | 802.4 | 779.3 | 97.12 | Pass |

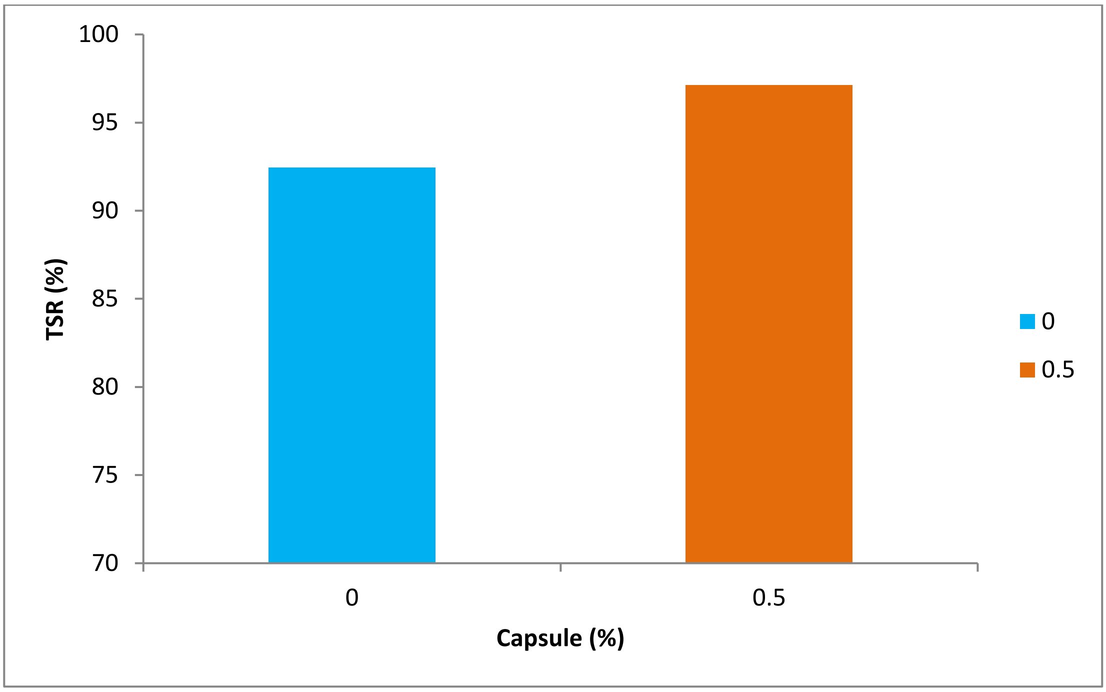


**Figure 4. 3: TSR vs Capsule %**

## 4.4 Semi Circular bending test

The SCB test was used to determine the healing capacity of the asphalt mixture. Marshall samples are sliced in half and a 3 mm notch is inserted in the centre. The sample is loaded on top to determine the peak load at which the sample fractures. This information is then utilized to calculate the Healing Index.

### 4.4.1 Semi Circular Bending Test Results

The results of peak load tests on SCB samples are shown in Figure 4.4. Although the peak loads of the second and third SCBs are lower than the first peak load, the findings are much better than those from samples without capsules. This indicates that a 20-hour healing interval considerably increased SCB sample healing. The healing index is 38% for the first healing and lowers to 16% for the second healing owing to capsule fracture caused by cracks. The findings indicate that SCB specimens equipped with capsules were able to regain 16% of their original peak force, but control samples were only able to restore 9%.

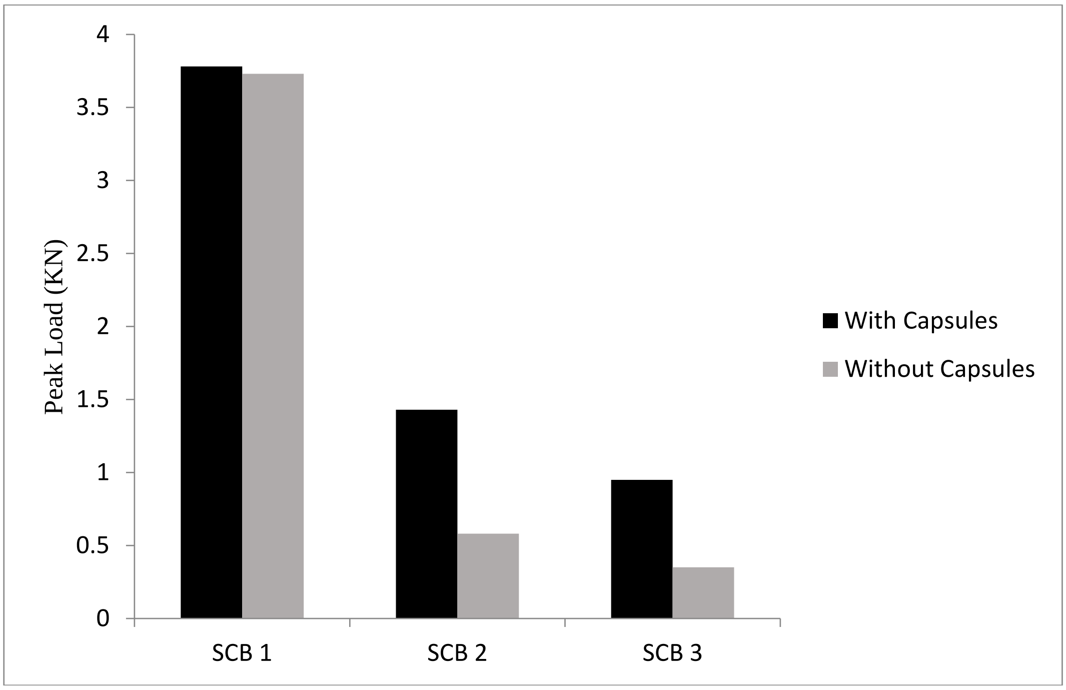


**Figure 4. 4: Peak Loads of SCB**

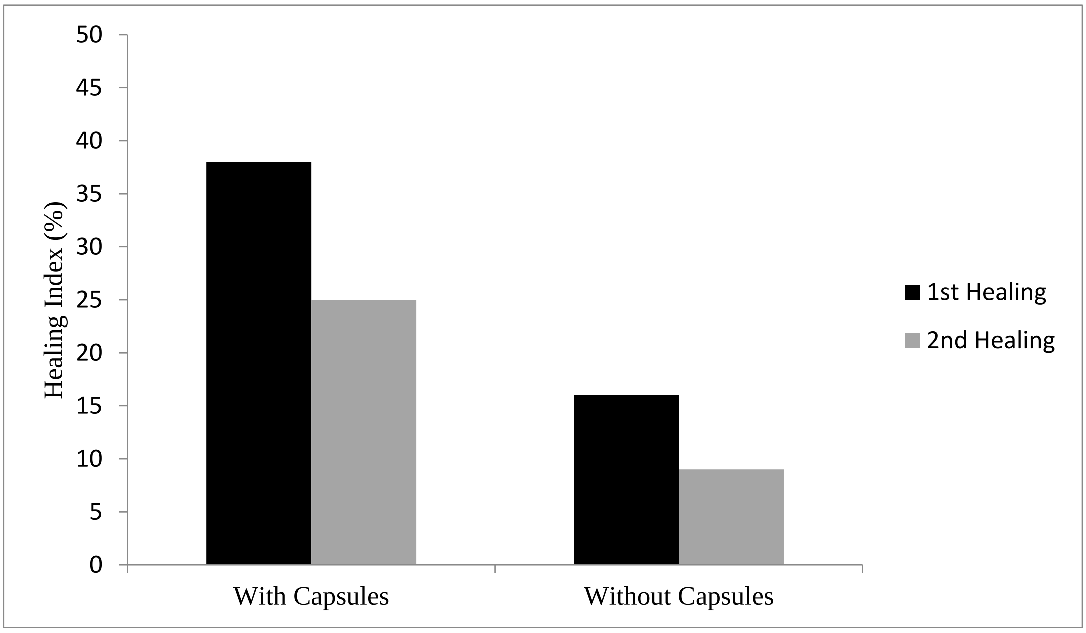


**Figure 4. 5: Healing Index SCB**

## 4.5 3 Point Bending test and Healing Efficiency of Asphalt Mortar

The three-point bending test was done to determine the asphalt mortar's healing capacity. To demonstrate a link between healing time and healing temperature, samples were generated and rigorous testing was performed. The strength of beams is evaluated before and after healing to determine the healing index.

### 4.5.1 3 Point Bending Test Results

Figure 4.8 illustrates the healing levels obtained by the asphalt samples at four different heating temperature and eight different healing temperature with, and without capsules. Each bar graph represents set of beams tested for healing evaluation. The sheer quantity of tests confirms the huge data available for analysis of healing index of beams.

Encapsulated beams showed a superior performance in terms of healing than non-capsulated beams as already confirmed in SCB. This test further tests the effect of increasing temperature on healing. As healing temperature increases the flowability of bitumen naturally increases and crack closure becomes easier. This results in improved healing performance with increasing temperature.

**Figure 4. 6: 3 Point Bending Test**

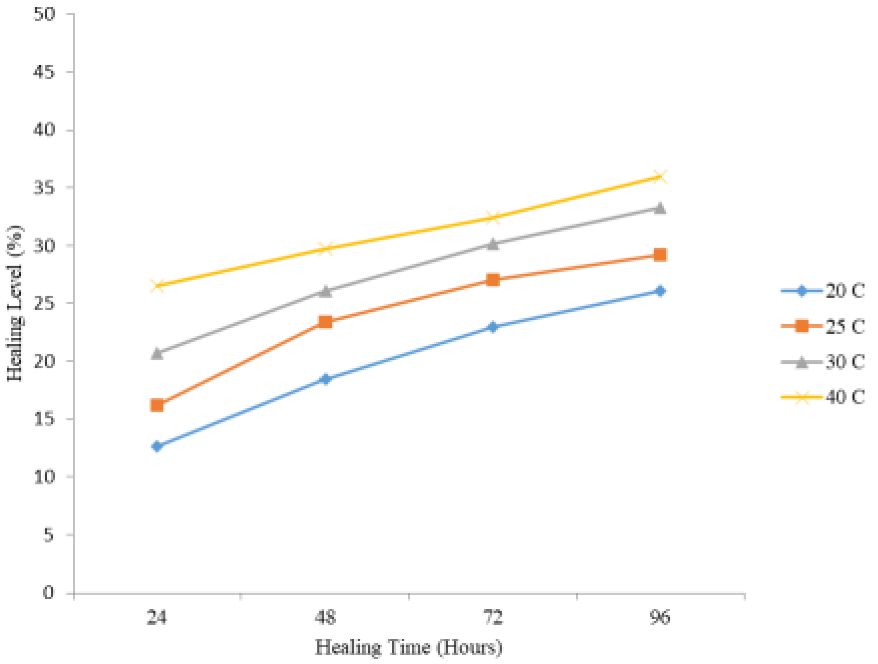


**Figure 4. 7: 3 Point bending Results Without Capsules**

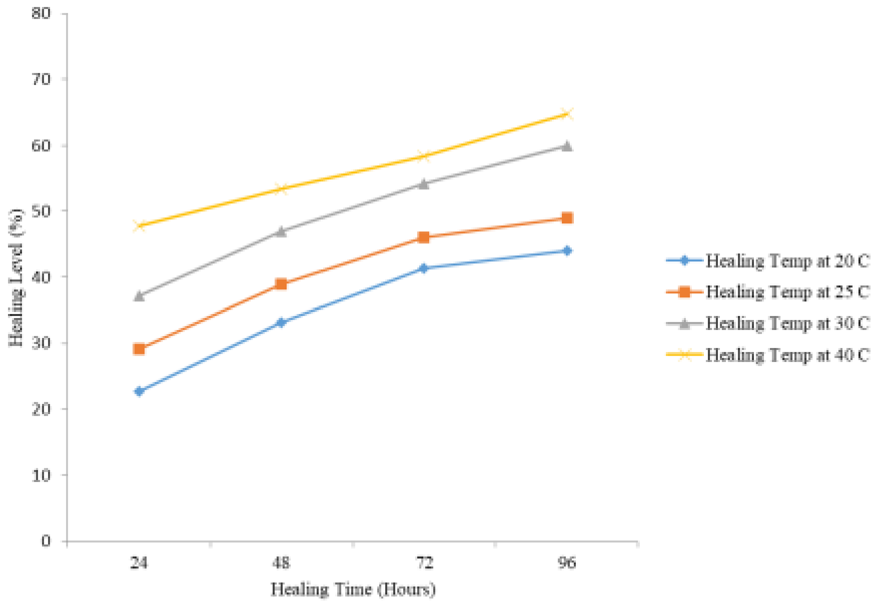


**Figure 4. 8: 3 Point bending Results With Capsules**

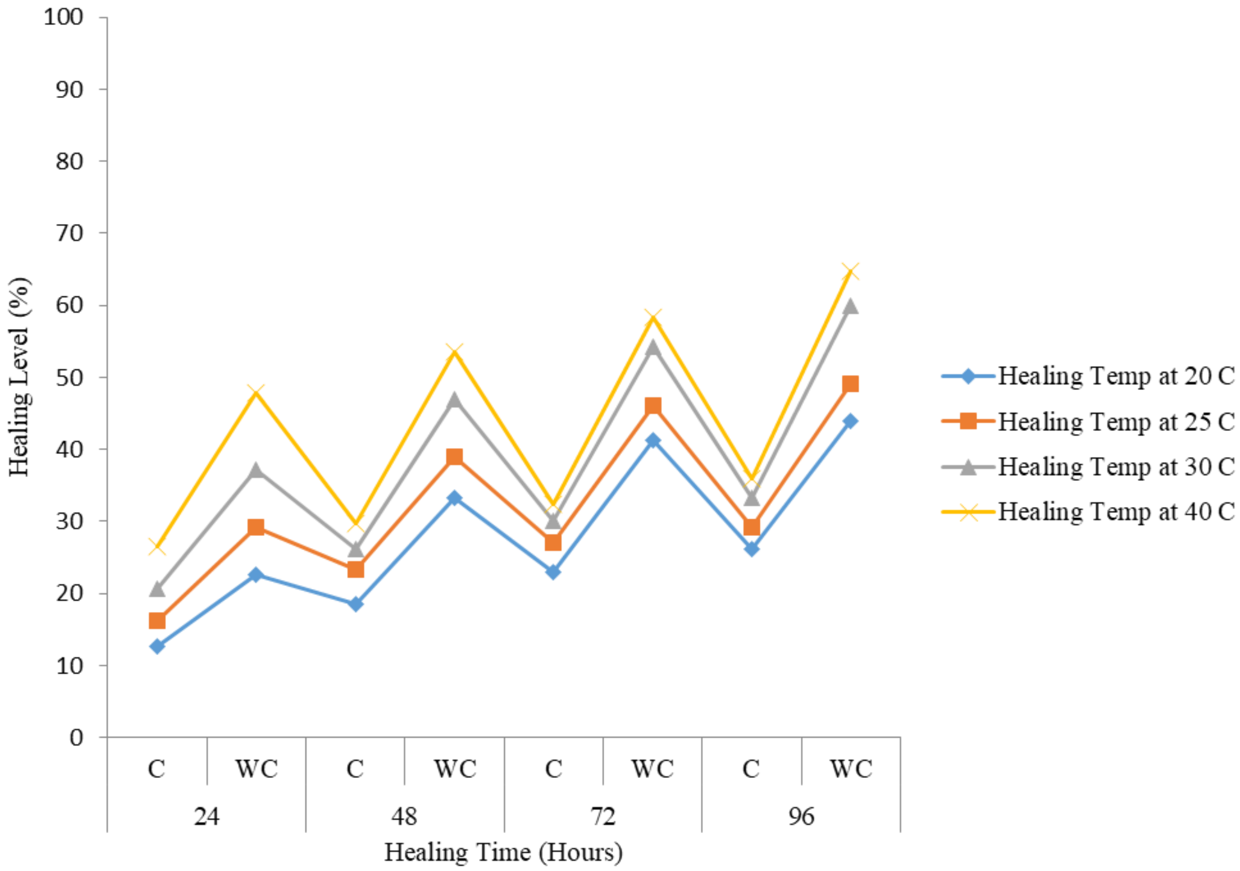


**Figure 4. 9: Healing Comparison**

## 4.6 Cost Estimation

A one-kilometer length of road is intended to be used to compare the costs of virgin mix with modified mix containing capsules. A standard lane width of 3.6 metres with a thickness of 63.5 mm was assumed. The cost of the lower layers' base and sub-base, as well as the cost of subgrade preparation, were totally neglected since they were assumed to have similar qualities for both mixtures. The cost of the HMA surface course is the only factor considered in this comparison. Cost estimates are made using NHA Composite Schedule Rates (CSR). The cost of virgin HMA and capsules modified HMA per kilometre per lane of a road stretch was determined and the results are presented below.

**Table 4.2: Cost Comparison (Without Capsules)**

| Quantity and Cost Estimation | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Single Lane Highway | | | | | | | | | |
| Description | Length | Lane Width | Layer Thickness | Volume of Material | Density of Asphalt | Asphalt Mix | Bitumen (4.3%) | Crush | Furnace Oil |
| | (m) | (m) | (m) | (m3) | | (ton) | (ton) | (ton) | (per ton HMA) |
| Wearing Course | 1000 | 3.6 | 0.0635 | 228.6 | 2.36 | 539.436 | 23.195748 | 516.2403 | |
| | | | Cost per Ton | | | | 110,037 | 1000 | 1339.23 |
| | | | Cost per Total HMA | | | | 2552390.523 | 516240.3 | 722428.874 |
| | | Total Cost (Pakistani Rupees) | | | | 3791059.649 | | | |

**Table 4.3 : Cost Comparison (With Capsules)**

| Quantity and Cost Estimation | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Description: Single Lane Highway | | | | | | | | | | |
| Description | Length | Lane Width | Layer Thickness | Volume of Material | Density of Asphalt | Asphalt Mix | Bitumen (4.3%) | | Crush | Furnance Oil |
| | (m) | (m) | (m) | (m3) | | (ton) | (ton) | | (ton) | (per ton HMA) |
| | | | | | | | Virgin | 0.5 % Cap | | |
| Wearing Course | 1000 | 3.6 | 0.0635 | 228.6 | 2.36 | 539.436 | 22.73183304 | 0.463915 | 516.240252 | |
| | | | Cost per Ton | | | | 110,037 | 100000 | 1000 | 1339.23 |
| | | | Cost per Total HMA | | | | 2501342.712 | 46391.5 | 516240.252 | 722428.874 |
| | | Total Cost (Pakistani Rupees) | | | | 3786403.335 | | | | |

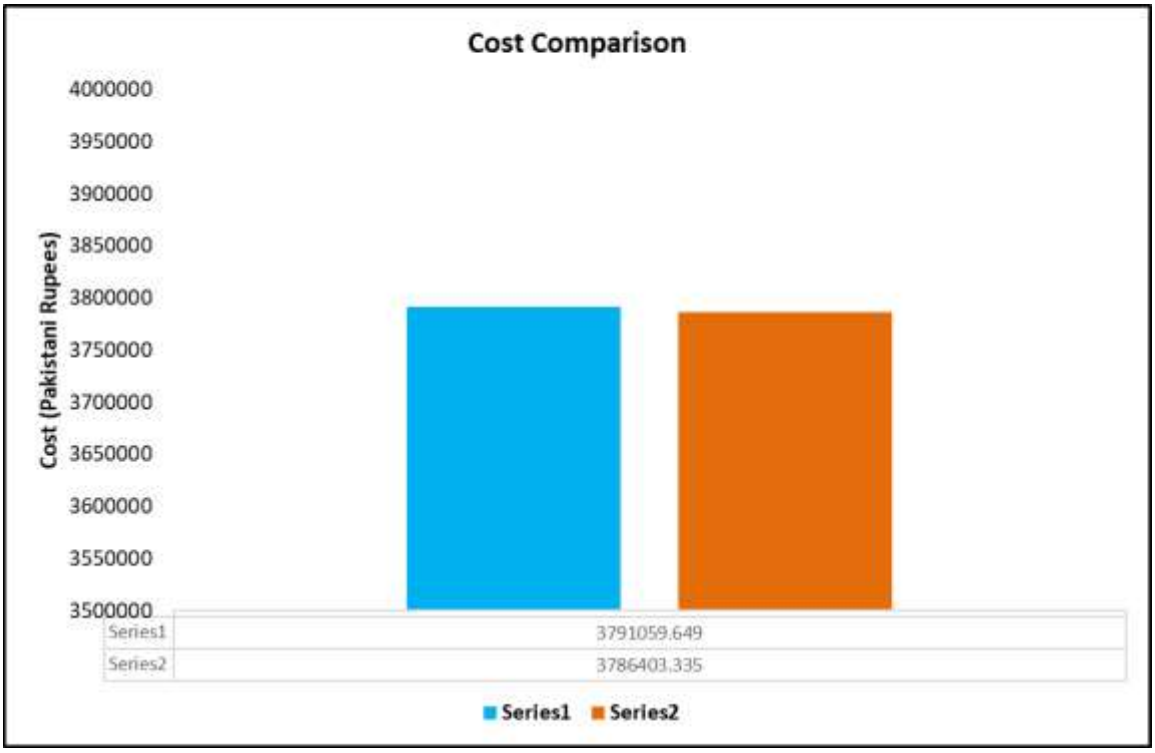


**Figure 4. 10: Cost Comparison**

## 4.7 Chapter Summary

This chapter examined the extensive study of performance testing results. The UTM results are examined in relation to the growth in TSR levels. Tables and graphs were used to assess the performance testing results.

The results of the Semi Circular Bending Test and the Three Point Bending Test are presented in detail using graphics. The rise in Healing Index is given as a function of healing time and temperature. From graphs illustrating the link between healing level, healing temperature, and healing time demonstrates optimal conditions for self-healing roads.

# CHAPTER 5: CONCLUSION & RECOMMENDATIONS

## 5.1 Introduction

It is concluded that used cooking oil may effectively reverse the ageing of bitumen used in asphalt pavement. The rejuvenation of bitumen using waste cooking oil improved the aromatic content and decreased the viscosity of the binder. When the capsule is ruptured by crack fracture energy, the oil escapes and fills the fissures, healing them. The self-healing ability of an asphalt mixture containing capsules is evaluated at various temperatures and healing periods.

## 5.2 Capsule production and characterization

The encapsulated rejuvenator (waste cooking oil) is made by ionotropic gelation of sodium alginate with calcium chloride.

- Capsules containing up to 80% of the healing agent are manufactured.
- Thermogravimetric analysis was performed on capsules to confirm their thermal stability. The results demonstrated that all capsules could withstand the mixing temperature of asphalt and that oil loss was never more than 2%.

### 5.2.1 Capsules effect on Asphalt performance

Capsules were engineered to rupture in the case of a trigger, such as a fracture. To begin, a moisture susceptibility test of asphalt shown that capsules increased the asphalt sample's water sensitivity.

- Asphalt specimens subjected to the Semi Circular Bending Test revealed that samples containing 0.5 percent capsules healed at a rate of 40% during the first SCB. Regaining peak load suggests that capsule healing had a beneficial effect.
- Three-point bending tests conducted at various temperatures and healing durations demonstrated that as temperature and healing time rise, the healing level increases. Maximum healing levels were 44.25 percent, 49.25 percent, 58.87 percent, and 64.75 percent, respectively, for asphalt mixes at 20 C, 25 C, 30 C, and 40 C of capsules. These findings account for the rejuvenator release, healing time, and temperature-healing index link. This is because a greater temperature and a longer healing period improve the likelihood of released oil and probable repair.

## 5.3 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, and rubber, may be used to modify the composition of capsules in various ways. A capsule's mechanical strength may be measured by the oil to water ratio present in the capsule.

- The usage of FTIR may be utilised to determine the amount of oil created during mixing and compacting operations. Environmental effects may also be investigated in terms of their impact on the quality of the capsules. In the case of capsules, fungi and bacteria may have an impact.

- If capsules are investigated in combination with the induction heating technique, it is possible that they will reveal whether or not there is a synergy between the two.

- In order to evaluate the life cycle cost of a product, an economic feasibility study may be performed.

- CT scans may be utilised to determine the effect of the quantity of capsule oil present on the amount of healing that occurs in relation to the fracture width.

- It is possible to model the dispersion of rejuvenators as well as the propagation of cracks in order to have a better knowledge of capsule rupture and bitumen healing.

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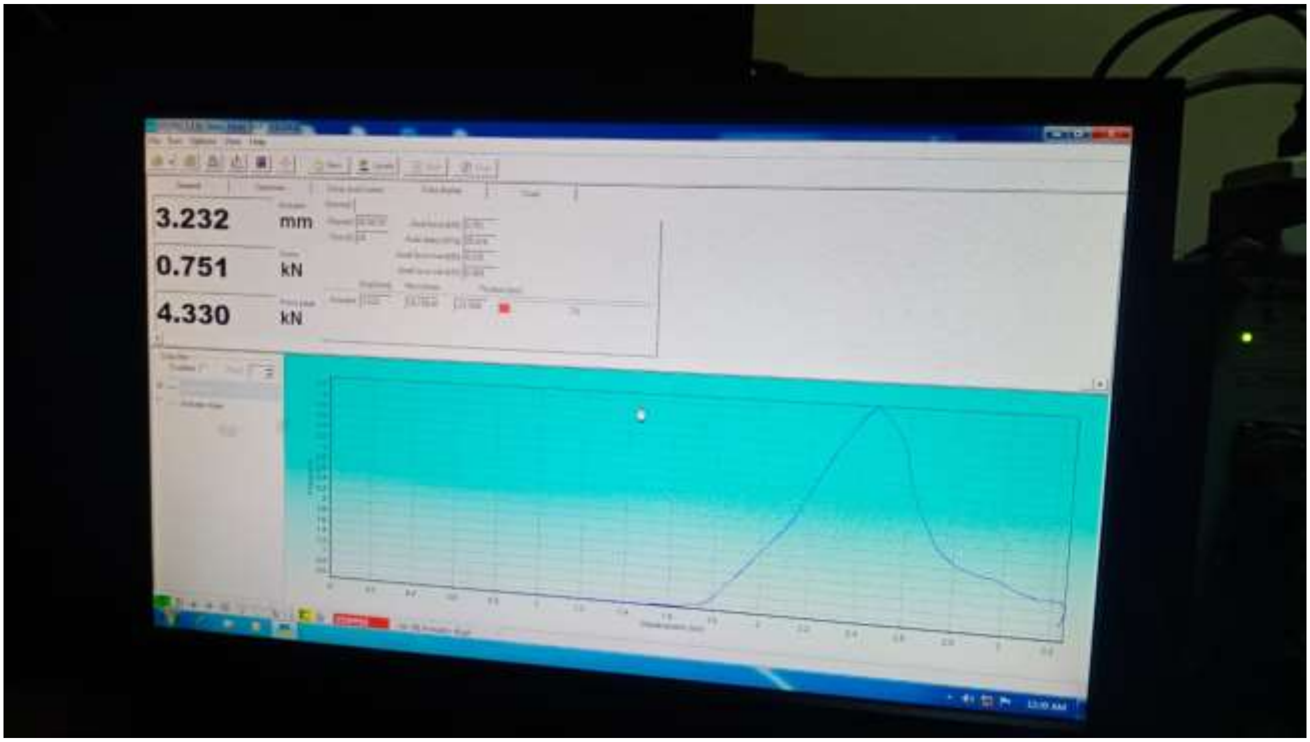
# APPENDIX 1: MARSHAL MIX DESIGN REPORTS

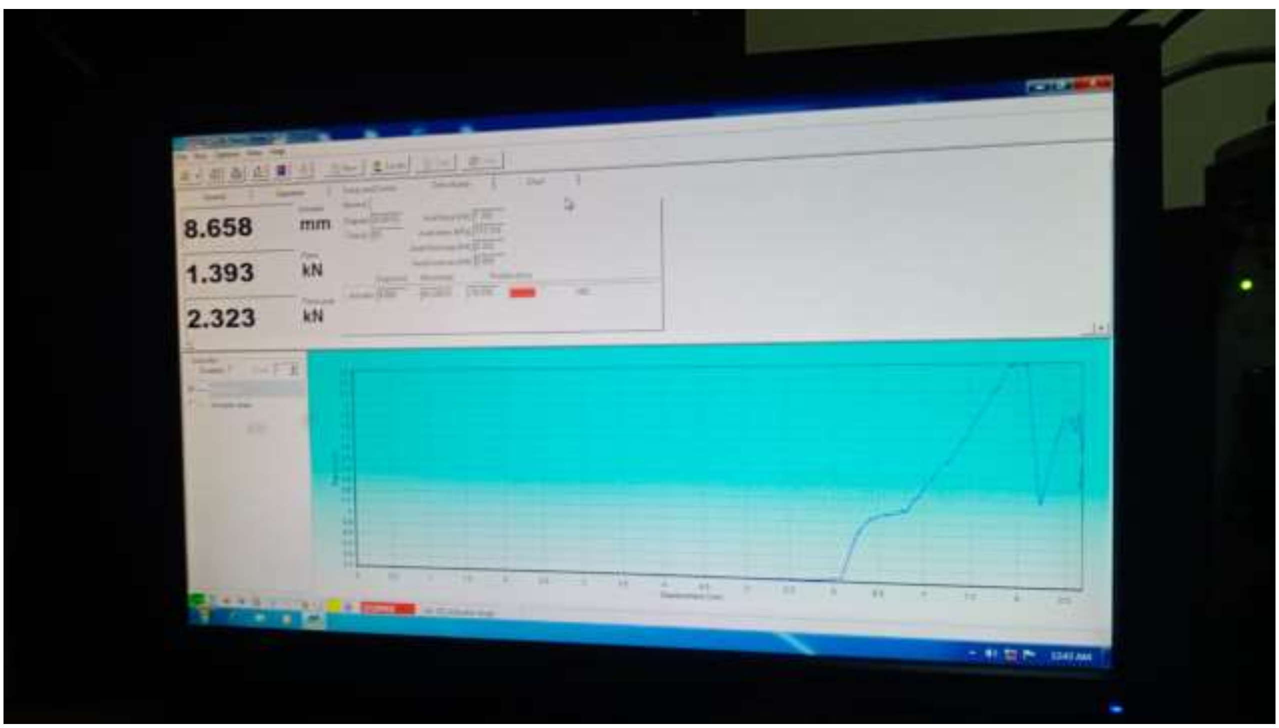
# APPENDIX 1: MARSHAL MIX DESIGN REPORTS

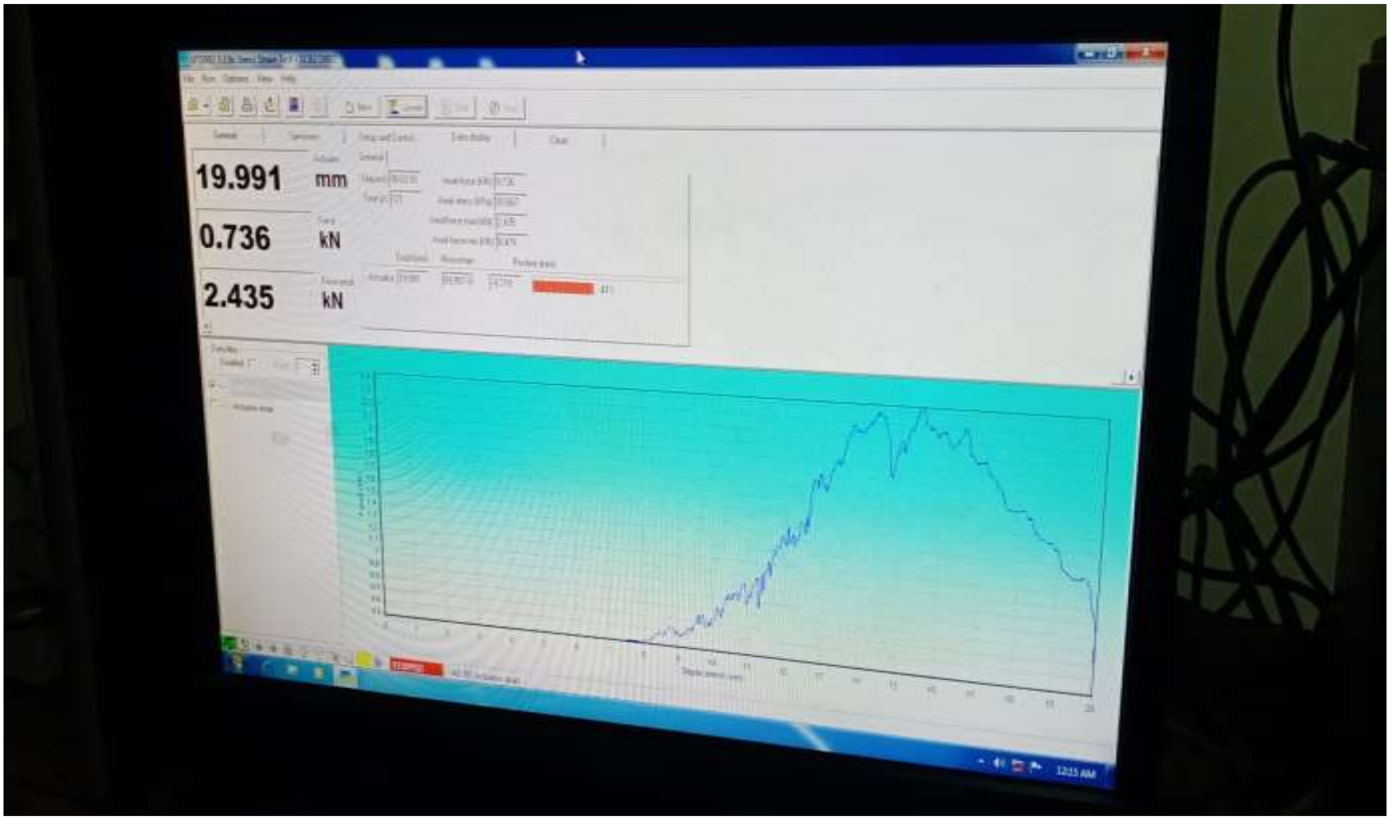
| **Marshall Test Report** | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Project: | MS Thesis | | Marshal Samples | | | | | | | | | | | | | |
| Aggregate Source:Margalla | | | | | | | | | | | | | | | | |
| Bitumen 60/70 | | Ali Zain Ul Abadeen | | | MS- 19 | | | | | | | | | | | |
| | | Mass, grams of compacted Specimen | | | | | Mass, grams of loose Mix | | | | | | | | | |
| % AC by wt. of mix, Spec. No. | Spec. Height in. (mm) | In Air (A) | In Water (C) | Sat. Surface Dry (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 | Stability, (KN) Measured | Adjusted | Flow, mm |
| 3.5-A | 64.5 | 1188.65 | 692.21 | 1200.82 | 508.61 | 2.337056 | 1187 | 6765 | 7473.6 | 2.481187 | 5.808969 | 14.08537 | 58.75886 | 10.533 | 10.11168 | 2.063 |
| 3.5-B | 62 | 1109.35 | 651.71 | 1128.52 | 476.81 | 2.326608 | 1106 | 6765 | 7425.22 | 2.481044 | 6.224652 | 14.46946 | 56.98075 | 10.133 | 11.04497 | 2.388 |
| 3.5-C | 65.25 | 1171.75 | 689.12 | 1192.92 | 503.8 | 2.325824 | 1164 | 6765 | 7460.27 | 2.483306 | 6.341636 | 14.49829 | 56.25942 | 10.361 | 9.94656 | 2.322 |
| Average | 63.91667 | 1156.583 | 677.68 | 1174.087 | 496.4067 | 2.329829 | 1146.5 | 6765 | 7453.03 | 2.481846 | 6.125085 | 14.35104 | 57.31957 | 10.34233 | 10.36774 | 2.257667 |
| 4.0-A | 65 | 1197.35 | 697.31 | 1202.82 | 505.51 | 2.368598 | 1194 | 6765 | 7476.02 | 2.472152 | 4.18882 | 13.37699 | 68.68637 | 12.7704 | 12.25958 | 2.31 |
| 4.0-B | 62.5 | 1181.55 | 696.31 | 1196.22 | 499.91 | 2.363525 | 1148 | 6765 | 7447.952 | 2.468562 | 4.254985 | 13.5625 | 68.62684 | 12.13 | 12.6152 | 2.567 |
| 4.0-C | 64.5 | 1180.75 | 695.31 | 1196.12 | 500.81 | 2.357681 | 1180 | 6765 | 7466.35 | 2.465267 | 4.364085 | 13.77625 | 68.32169 | 12.323 | 11.83008 | 2.869 |
| Average | 64 | 1186.55 | 696.31 | 1198.387 | 502.0767 | 2.363268 | 1174 | 6765 | 7463.441 | 2.46866 | 4.269297 | 13.57191 | 68.54496 | 12.4078 | 12.23495 | 2.582 |
| 4.5-A | 63.75 | 1174.85 | 691.21 | 1181.92 | 490.71 | 2.394184 | 1171 | 6765 | 7461.46 | 2.467653 | 2.97728 | 12.89731 | 76.91549 | 13.085 | 13.085 | 2.967 |
| 4.5-B | 63.25 | 1180.85 | 691.11 | 1183.12 | 492.01 | 2.400053 | 1178 | 6765 | 7466.07 | 2.469964 | 2.830458 | 12.68379 | 77.68445 | 12.171 | 12.171 | 3.022 |
| 4.5-C | 63.75 | 1192.25 | 700.31 | 1205.22 | 504.91 | 2.361312 | 1174 | 6765 | 7466.99 | 2.487235 | 5.062791 | 14.09322 | 64.07642 | 12.833 | 12.31968 | 3.073 |
| Average | 63.58333 | 1182.65 | 694.21 | 1190.087 | 495.8767 | 2.385183 | 1174.333 | 6765 | 7464.84 | 2.474951 | 3.62351 | 13.22477 | 72.89212 | 12.69633 | 12.52523 | 3.020667 |
| 5.0-A | 63.54 | 1191.75 | 701.01 | 1197.22 | 496.21 | 2.401705 | 1187 | 6765 | 7468.59 | 2.455473 | 2.189707 | 13.08116 | 83.2606 | 11.135 | 11.135 | 3.772 |
| 5.0-B | 62.5 | 1175.25 | 687.71 | 1180.52 | 492.81 | 2.384793 | 1166 | 6765 | 7453.12 | 2.439943 | 2.260289 | 13.69319 | 83.49334 | 11.532 | 11.99328 | 3.342 |
| 5.0-C | 64 | 1183.75 | 696.51 | 1191.02 | 494.51 | 2.393784 | 1182 | 6765 | 7463.12 | 2.442754 | 2.004731 | 13.36783 | 85.00331 | 11.019 | 11.019 | 3.641 |
| Average | 63.34667 | 1183.583 | 695.0767 | 1189.587 | 494.51 | 2.393427 | 1178.333 | 6765 | 7461.61 | 2.446057 | 2.151576 | 13.38073 | 83.92034 | 11.22867 | 11.38243 | 3.585 |
| 5.5-A | 62.75 | 1166.15 | 683.51 | 1169.32 | 485.81 | 2.400424 | 1161 | 6765 | 7450.62 | 2.442257 | 1.71287 | 13.58473 | 87.39121 | 9.29 | 10.1261 | 4.253 |
| 5.5-B | 63.25 | 1164.15 | 681.91 | 1168.22 | 486.31 | 2.393843 | 1161 | 6765 | 7448.57 | 2.43177 | 1.559632 | 13.82164 | 88.71601 | 8.395 | 9.15055 | 4.032 |
| 5.5-C | 64 | 1152.45 | 677.71 | 1158.12 | 480.41 | 2.398888 | 1142 | 6765 | 7440.12 | 2.446025 | 1.927054 | 13.64002 | 85.87205 | 9.636 | 10.02144 | 4.333 |
| Average | 63.33333 | 1160.917 | 681.0433 | 1165.22 | 484.1767 | 2.397719 | 1154.667 | 6765 | 7446.437 | 2.440017 | 1.733186 | 13.68213 | 87.33249 | 9.107 | 9.76603 | 4.206 |

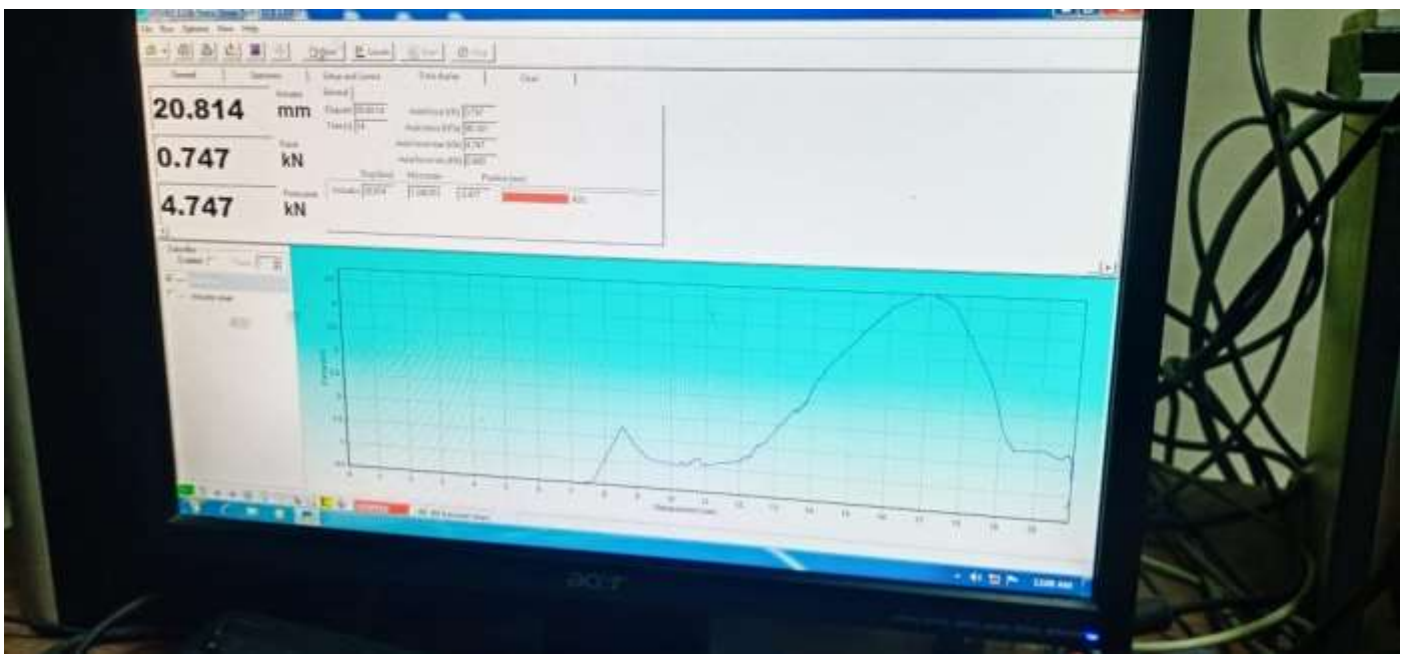
# APPENDIX 2: PERFORMANCE TESTING

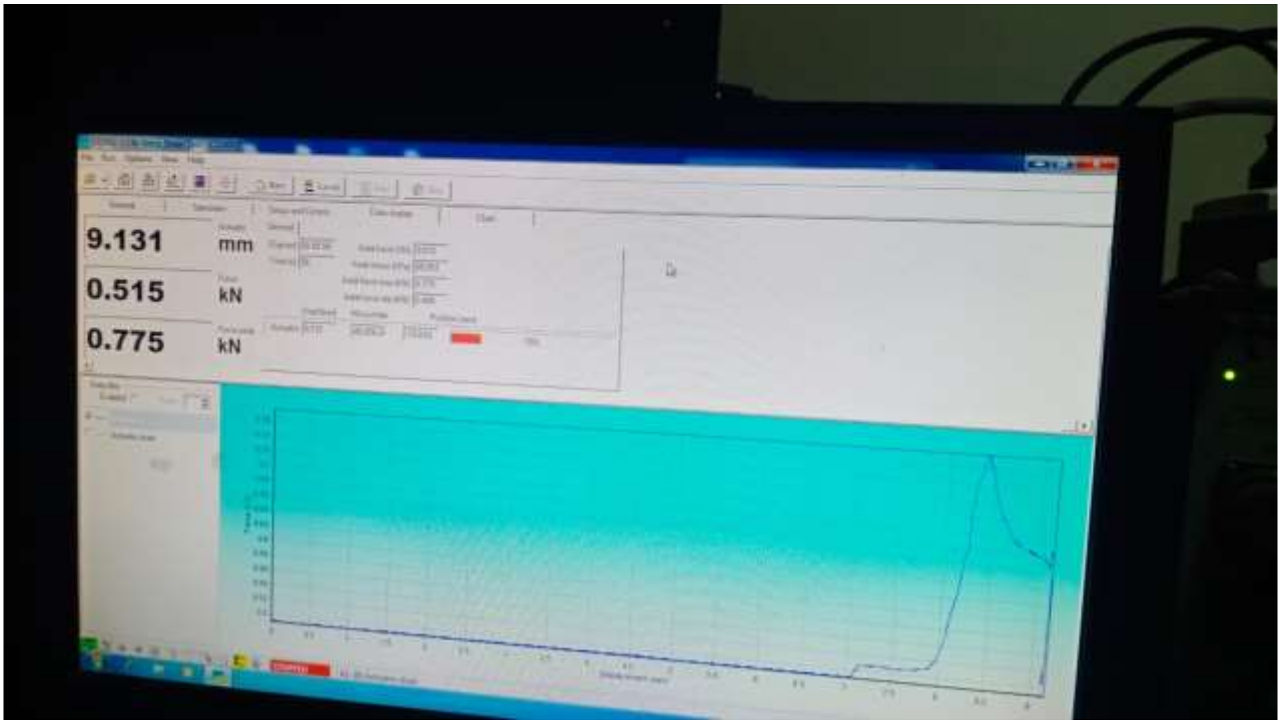
## APPENDIX 2: PERFORMANCE TESTING

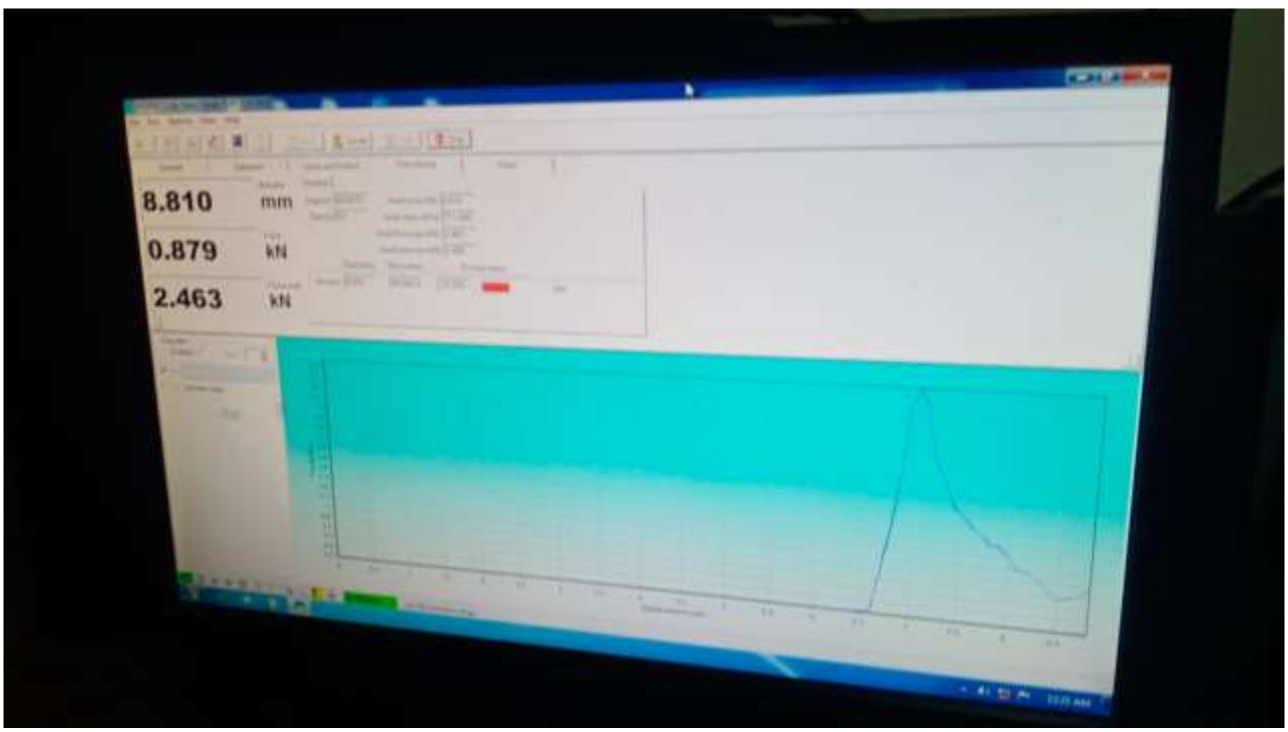


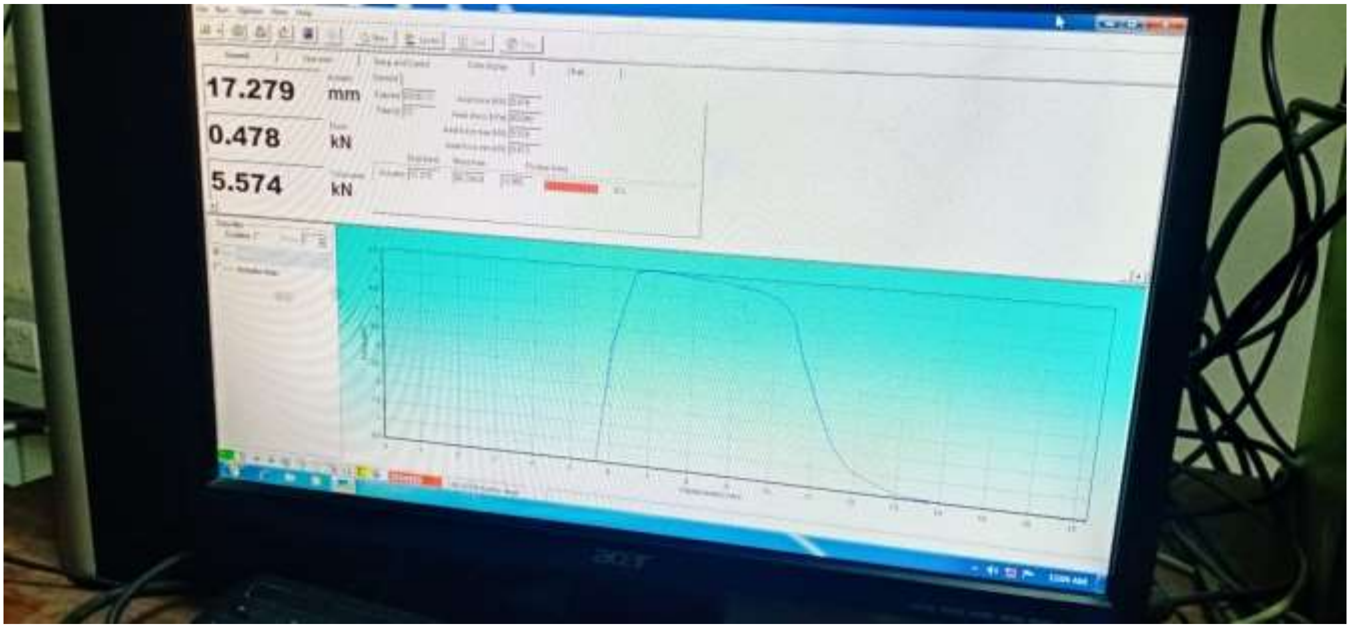




19.991
mm
0.736
kN
2.435
kN


20.814
mm
0.747
kN
4.747
kN


9.131
mm
0.515
kN
0.775
kN


8.810
mm
0.879
kN
2.463
kN


17.279
mm
0.478
kN
5.574
kN