AMERICAN UNIVERSITY OF BEIRUT

EVALUATION OF THE MOISTURE SUSCEPTIBILITY OF ASPHALT MIXTURES USING THE DIRECT TENSION TEST AT THE BINDER AND MASTIC SCALES

By
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ABSTRACT
OF THE THESIS OF

Mohammad Nour Faysal Fakhreddine for Master of Engineering
Major: Materials and Pavement Engineering

Title: Evaluation of the Moisture Susceptibility of Asphalt Mixtures Using the Direct Tension Test at the Binder and Mastic Scales

The objective of this research is to study the interaction between different binders, aggregates, and mineral fillers in terms of their ability to resist moisture damage using an innovative direct tension test which is conducted at the binder and mastic levels. The direct tension test is used to study the adhesion between the binder or mastic and the aggregate surface as well as the cohesive strength of the binder or mastic. This is done by testing at various extension rates that cause adhesive and cohesive failures. The study uses non-modified and SBS-modified binders, locally used aggregates and fillers such as limestone and hydrated lime respectively, in addition to recycled concrete aggregates (RCA) and recycled asphalt pavement (RAP). The results show that the test method can evaluate the effect of moisture conditioning on the fabricated samples and differentiate between binders, aggregates, and fillers in the context of moisture damage susceptibility. The advantages of the developed test are that it is easy to conduct, repeatable, uses affordable equipment, and allows to measure strength as well as strain data. It is concluded that the non-modified binder performs better than the SBS-modified binder in resisting moisture damage when paired with limestone and RCA. Replacing limestone coarse aggregate with RCA worsens the moisture susceptibility, whereas incorporating RCA filler limits the loss of the adhesive bond strength after moisture conditioning. The positive effect of incorporating hydrated lime, which is a common anti-stripping agent used to improve adhesion, is dependent on the type of aggregate and binder it is used with.

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ABBREVIATIONS

AUB	American University of Beirut
AASHTO	American Association of State Highway and Transportation Officials
ASTM	American Society for Testing and Materials
BBS	Bitumen Bond Strength
RCA	Recycled Concrete Aggregate
RAP	Recycled Asphalt Pavement
MDR	Moisture Damage Ratio
APA	Asphalt Pavement Analyzer
HWTD	Hamburg Wheel Tracking Device
MMLS	Model Mobile Load Simulator
ECS	Environmental Conditioning System
UTM	Universal Testing Machine
DSR	Dynamic Shear Rheometer
SBS	Styrene-Butadiene-Styrene
PG	Performance Grade
RTFO	Rolling Thin Film Oven
MSCR	Multiple Stress and Creep Recovery
NCHRP	National Cooperative Highway Research Program

CHAPTER 1

INTRODUCTION

1.1. Motivation and Problem Statement

Roads and highways are considered a significant part of the global transportation infrastructure network as they provide a means for transporting goods and people and are a pillar of a thriving economy. They provide a hard surface for vehicles to travel on, known as a pavement, which on more than 90% of roadways is flexible due to several favorable factors such as faster construction time, lower cost, easier maintenance, and more comfortable ride quality (Gong et al., 2019). Flexible pavements consist of a mix of aggregates bound together by a bituminous substance referred to as the asphalt binder or bitumen. Although asphalt pavements are typically made to accommodate traffic over a period of 20 years, they require maintenance to rectify the damage caused by traffic loads and climatic conditions.

The damage inflicted to asphalt pavements manifests itself in the form of various distress types that prompt routine maintenance and rehabilitation intervention. One such example of a pavement distress is asphalt rutting, which is a depressed channel that mostly runs along the wheel path and is accompanied by upheavals of the displaced asphalt on its sides (Mirzahosseini et al., 2011). Rutting compromises traffic safety as it restricts the free movement of a vehicle in and between lanes. Rutting also creates a space for water to pond, which increases the risk of traffic accidents (Javilla et al., 2017; Sousa et al., 1991), particularly in areas with sub-freezing temperatures where the available water inside the rut freezes and drastically decreases a vehicle's ability to accelerate and decelerate. Rutting is one of several pavement distresses which include, among others, cracking and raveling, the latter defined as the dislodgement of aggregate particles from the pavement surface. The presence of water, though not only caused by rutting, in the asphalt pavement leads to the accelerated appearance of pavement distresses by negatively affecting the materials' adhesive and cohesive durability in a phenomenon known as moisture damage (Cho & Kim, 2010). Moisture damage has been a topic of research since the 1900s (Kakar et al., 2015), yet it remains a critical problem in asphalt pavements.

The evaluation of moisture susceptibility in asphalt pavements is challenging since several factors such as traffic loads, mixture properties, climate conditions, construction practices, and material characteristics are involved (Taylor & Khosla, 1983). Widely used methods such as the AASHTO T283 and Hamburg Wheel Tracking Test under AASHTO T324 have several drawbacks such as their empirical procedures, inaccurate simulation of field conditions, and results that are dependent on the moisture conditioning process (Abuawad et al., 2015). Consequently, the results of these tests correlate poorly with field performance. Since it is very difficult to predict moisture damage using a single test, additional testing is required, particularly at the level of the asphalt-aggregate interface.

1.2. Objectives and Scope of Work

The objective of this study is to introduce an innovative procedure to study the interaction between mixture components, which are the asphalt binder, aggregates, and mineral filler (particles whose dimensions are smaller than 0.075 mm) in the context of resisting moisture damage. The procedure employs the direct tension test and is conducted at the binder and mastic levels to isolate the impact of mixture components on moisture damage. The test is based on the pull-off approach and provides improvements over other tests that use the same approach such as the Bitumen Bond Strength (BBS) test (AASHTO T361-16). The test is conducted by pulling apart two aggregate disks that are bound together by a film of binder or mastic.

The setup and sample geometry of the direct tension test allow for easy manipulation of parameters such as the binder or mastic film thickness and the rate at which the aggregates are pulled apart, which have a significant effect on the type of failure that can occur. Moisture damage affects the adhesive bond, which keeps the binder or mastic adhered to the aggregate surface, and the cohesive bond which, keeps the binder or mastic film from tearing apart. As such, another objective of this research is to evaluate the effect of moisture conditioning on the adhesive and cohesive bonds by testing at different extension rates.

The final objective of this research is to evaluate the effect of recycled concrete aggregate (RCA) and recycled asphalt pavement (RAP) on moisture damage by incorporating them in the testing plan as aggregates and mineral fillers.

1.3. Thesis Structure

The next chapter contains a review of the moisture damage phenomenon and the tests which are used to predict moisture susceptibility. Chapters 3 and 4 outline the testing methodology and experimental plan, respectively. The analysis and results are discussed in Chapter 5, which is followed by the research conclusions and recommendations in Chapter 6 and future work in Chapter 7.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

Moisture damage is a problem that has plagued AC pavements ever since their introduction into the transportation network and is one of the main factors that affect their durability (Airey & Choi, 2002). It has been recognized since the 1930s, its quantification dates back to the 1960s (Caro et al., 2008b). It attracted the attention of highway agencies as well as the pavement industry in the 1980s (Taylor & Khosla, 1983). Moisture damage causes the premature deterioration of asphalt pavements by accelerating the appearance of distresses such as rutting, cracking, and raveling (Kim et al., 2004), and thus significantly increases pavement maintenance costs, vehicle operation costs (Copeland, 2005), and puts the safety of travelers at risk if not attended to promptly. Researchers have defined moisture damage in several ways, but the most comprehensive definition was given by Kiggundu & Roberts, 1988 as " the progressive functional deterioration of a pavement mixture by loss of adhesive bond between the asphalt cement and the aggregate surface and/or loss of the cohesive resistance within the asphalt cement principally from the action of water". According to Caro et al., 2008a, the moisture damage mechanism is composed of moisture transport and the response of the system. Moisture transport is the phase in which water infiltrates into the asphalt mixture, reaching the asphalt-aggregate interface as well as the binder or mastic. The presence of moisture inside the asphalt mixture prompts a response by the system whereby the outcome is the degradation of the adhesive bond between the binder and aggregate as well as the cohesive bond between the particles of the binder itself. Moisture can be transported into the asphalt mixture mainly through the infiltration of surface water, capillary rise of subsurface water, and diffusion of water vapor. The mode of transport in controlled by material and system attributes such as the air voids percentage in the system, aggregate shape, texture, and angularity (Caro et al., 2008a). The response of the system may include one or more of the following processes:

- Detachment, which is the microscopic separation of the binder film from the aggregate surface without an obvious break in the binder film (Little, 2003)
- Displacement, which results from the penetration of the water to the aggregate surface through a break in the bitumen film leading to the loss of material from the aggregate surface and the possible separation of the aggregate or mastic (Little, 2003)
- Dispersion of mastic which is the weakening of the cohesive bond of the asphalt binder or mastic (Kringos & Scarpas, 2005)
- Rupture of mastic or aggregates
- Desorption of mastic, where the outer layer is washed due to the flow of water (Kringos & Scarpas, 2005)
- Spontaneous emulsification, which is the inverted emulsion of water in the bitumen (Little, 2003). It leads to the separation of bitumen particles from each other and consequently causes adhesive failure when water infiltrates through the bitumen film to the aggregate surface (Omar et al., 2020)

The loss in adhesion and cohesion are the final steps in the moisture damage process, which results in the acceleration of distresses such as rutting, cracking, and raveling. Stripping, which is perhaps the term most commonly associated with moisture damage in literature, is the separation of the binder or mastic from the aggregate surface due to the loss of adhesion (Taylor & Khosla, 1983). The loss of adhesion is a complicated process that is explained by several theories which are believed to interact together and are outlined as follows:

- Weak boundary layers, which states that adhesive failure may occur due to the presence of an interface region that has low cohesive strength (Packham, 2003) such as a film of dust covering the aggregate surface
- Electrostatic forces, which attributes the adhesive strength between two materials to the coulombic forces of attraction at their surfaces (Allan, 1992)
- Chemical bonding, which suggests that the adhesive bond between the asphalt binder and aggregate surface results from a chemical reaction that leads to the formation of a new material (Caro et al., 2008a)
- Mechanical bonding, which assumes that the adhesion happens due to mechanical interlock when the binder is forced into the irregularities of the aggregate surface
- Surface free energy, which states that adhesion between two materials is a function of their surface energies and that unbalanced intermolecular surface forces are responsible for the surface tension of liquids and surface energy of solids (Al Basiouni Al Masri et al., 2019), and is currently the most popular approach in adhesion science (Omar et al., 2020)

For additional information about the mechanism of moisture damage and the theories that explain adhesion, please refer to the studies by Kakar et al., 2015; Little, 2003; and Omar et al., 2020.

2.2. Experimental Methods

Although the problem of moisture damage has been widely studied, its complexity has made it very difficult to propose a test to accurately measure moisture susceptibility and predict field performance (Solaimanian et al., 2003). This is because there are several variables that interact in the moisture damage phenomenon such as traffic, construction practices, environmental conditions, and material properties. Numerous tests have been developed to assess the moisture susceptibility of asphalt mixes dating back to the 1930s, yet there is still no universally acceptable procedure (Terrel & Shute, 1989). According to Caro et al., 2008b, the available characterization methods can be classified as follows:

- Subjective qualification
- Quantification of a performance parameter
- Quantification of a moisture damage ratio (MDR)
- Mathematical Modeling (which is beyond the scope of this study)

The subjective qualification relies on visual inspection to determine the percentage of aggregate surface that lost the binder. An example of such a test would be the boiling water test (ASTM D 3625).

The visual inspection method was improved using a quantifiable performance parameter which is obtained by conducting a mechanical or chemical test related to moisture damage on loose or compacted samples and with or without loading, such as the methylene blue test.

Using a moisture damage ratio has become a very popular approach to measure the moisture susceptibility of asphalt mixtures. The MDR is the ratio of a certain parameter such as the strength or number of cycles to failure after moisture conditioning to that before moisture conditioning. Moisture conditioning is meant to accelerate moisture damage and simulate field conditions, and the MDR represents the retained value of the parameter in use. The tests which rely on a MDR are generally easy to conduct, and they can be based on a single or multiple parameter ratio. The single parameter tests are able to capture the deterioration of an asphalt mixture property after moisture conditioning, but they don't provide any indication to the cause of the deterioration. They also do not take into account the interaction between different chemical and physical properties that can be affected by moisture damage, which makes it very hard to propose a solution for the mixtures that perform poorly. Multiple parameter tests consider more than one parameter and allow testing the effect of moisture conditioning on the chemical, physical, and mechanical properties of the asphalt mixture. This provides a more comprehensive evaluation of its moisture susceptibility.

Several other studies have documented and classified the methods which are used to determine the moisture susceptibility of asphalt mixes (Airey & Choi, 2002; Kakar et al., 2015; Kiggundu & Roberts, 1988; Omar et al., 2020; Solaimanian et al., 2003; Taylor & Khosla, 1983; Terrel & Shute, 1989).

2.2.1. Tests on Asphalt Mixes

The AASHTO T283 has the widest acceptance in the pavement industry for testing the moisture susceptibility of asphalt mixes (Chen & Huang, 2008), yet it has been reported that the test does not correlate well with field performance due to the empirical nature of the test procedure (Caro et al., 2008b; Kim et al., 2008). Also, the test does not give any insight regarding the reason behind sub-par performance. Kringos et al., (2009) conducted a study to understand the reasons behind the discrepancies of the AASHTO T283, reported as follows:

- Poor correlation with field results such that mixtures which performed well in the lab could at times perform poorly in the field and vice versa.
- The test results are dependent on the size of the specimens which could be 100 mm Marshsall compacted or 150 mm gyratory compacted.
- The conditioning regime does not always promote stripping and leads to levels of saturations which are inconsistent and less than those in the field.
- The type of loading does not simulate the pumping action of traffic loads.

In their study, the authors showed that although the samples were grouped with similar air voids to achieve similar moisture conditions, the outside porosities were not taken into consideration in the calculations to obtain the air voids which causes different degrees of saturation and in turn, different severities of moisture conditioning. The authors also showed that during testing, samples experienced at all locations, and at all times, different strain levels and strain rates through a simulation of the indirect tensile strength test using a finite element software as shown in Figure 1. They recommended that the testing protocol should be based on various strain rates.

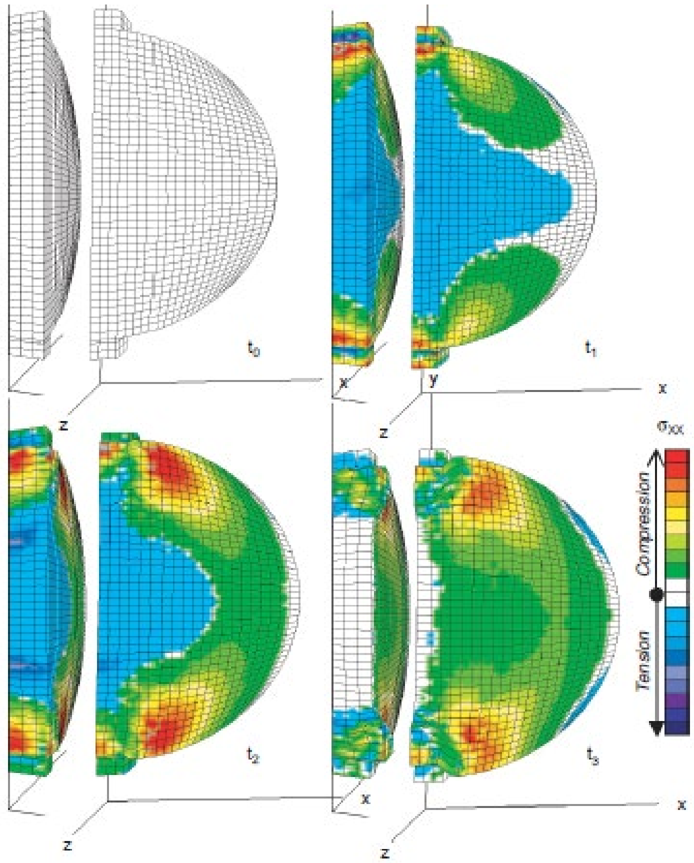


Figure 1 Finite Element Analysis of Dry Indirect Tensile Strength (Kringos et al., 2009)

Water immersion wheel tracking tests such as the Environmental Conditioning System (ECS) (AASHTO TP34), Asphalt Pavement Analyzer (APA), Hamburg Wheel Tracking Device (HWTD) and Model Mobile Load Simulator (MMLS3) are testing equipment used to evaluate moisture damage and high temperature stability under the combined effect of water, temperature, and vehicle loading through measuring the effect on rutting (Wang et al., 2019). These methods differ in the way they simulate the hydraulic scouring effect. The APA uses a pressurized hose between the wheel and the specimen whereas the MMLS applies the load directly from pneumatic tires. Other methods use rubber or stainless-steel wheel. These methods also differ in the way they measure deformation. The APA and HWTD measure deformation in real time using displacement sensors fixed on the testing wheel. In the case of the MMLS, the deformation is measured by the operator using a profilometer at set intervals of wheel passes (Smit et al., 2003). The tests can also be used to collect stress, strain, and pore water pressure data by loading sensors inside the sample. However, these tests are limited in their ability to simulate dynamic hydraulic scouring due to the large difference between the slow speed of the testing wheel and the actual high speed of a moving vehicle and further research on the accuracy and effectiveness of dynamic pore water pressure produced under the low speed of a testing wheel is required (Wang et al., 2019). Other issues such as the need for expensive equipment limit the use of these tests (Taib et al., 2019). Similar to the AASHTO T283, these tests do not accurately predict the degree of moisture damage of different asphalt mixes in the field because they are exposed to different boundary conditions, time frames, and moisture infiltration modes in addition to applying a fixed load at a fixed temperature (Airey et al., 2008; Kim et al., 2008).

2.2.2. Pull-off Tests

Researchers also proposed studying the moisture susceptibility of asphalt mixtures at the micro-scale using smaller samples rather than compacted ones to understand the stripping phenomenon at the level of the asphalt-aggregate interfacial bond where it takes place. The smaller samples generally consist of an asphalt film sandwiched between an aggregate substrate and a metal stub, or 2 cylindrical aggregates which are attached to the testing machine from each side. The use of such samples allows to eliminate mixture-related factors which cause a variability in the results such as the air void distribution and surface porosity.

Cho & Bahia, 2010 used the dynamic shear rheometer (DSR) to measure the rheological response of the asphalt film adhered to the top and bottom aggregates after moisture conditioning. The use of the DSR enabled the authors to have accurate control over the asphalt film thickness and the loading rate so that field conditions can be emulated. The authors established the wet to dry yield stress ratio (W/D YSS) as a parameter to evaluate the moisture conditioning effect on the asphalt-aggregate combinations and concluded that while the W/D YSS was sensitive enough to do that, other issues such as the complexity and practicality of the procedure prevent the use of the DSR to characterize moisture damage.

The pull-off method has been very popular recently in measuring the moisture susceptibility and studying the adhesive and cohesive properties of asphalt-aggregate systems (Kakar et al., 2015). It uses the simplified format of a binder or mastic film sandwiched between two aggregate cylinders, or an aggregate surface and a metal stub. The aggregates are typically 25 mm in diameter by 5 mm in thickness, and there have been different approaches to selecting an appropriate film thickness. The widest accepted pull-off test is the Bitumen Bond Strength Test (BBS) which uses the Pneumatic Adhesion Tensile Testing Instrument (PATTI), shown in Figure 2, and is standardized under AASHTO T361-16. The BBS was used by Canestrari et al., 2010 to study the effect of moisture conditioning on the adhesive and cohesive properties of several asphalt-aggregate combinations and concluded that the test was repeatable and could discriminate between different materials and moisture states. A similar conclusion was reached by (Moraes et al., 2012) who measured the effects of moisture conditioning time and asphalt modification on the strength of the asphalt-aggregate bond. The authors also concluded that moisture conditioning for 96 hours is sufficient to induce significant damage. Both studies noted that longer conditioning times caused the mode of failure to go from cohesive to adhesive as a general trend, although materials like limestone aggregate showed strong binder affinity and did no fail in adhesion even after long conditioning times. Another study was done by (Chaturabong & Bahia, 2018) to investigate the effect of mineral fillers on the bond strength between asphalt mastics and the aggregate surface using the BBS test. The study found that while the mineral filler does significantly affect the bond strength, the effect is highly dependent on what type of filler was used. The surface area of the mineral filler was found to be a significant factor to resist moisture damage and fillers with higher surface areas in the mastic resulted in a lower loss of the pull-off tensile strength.

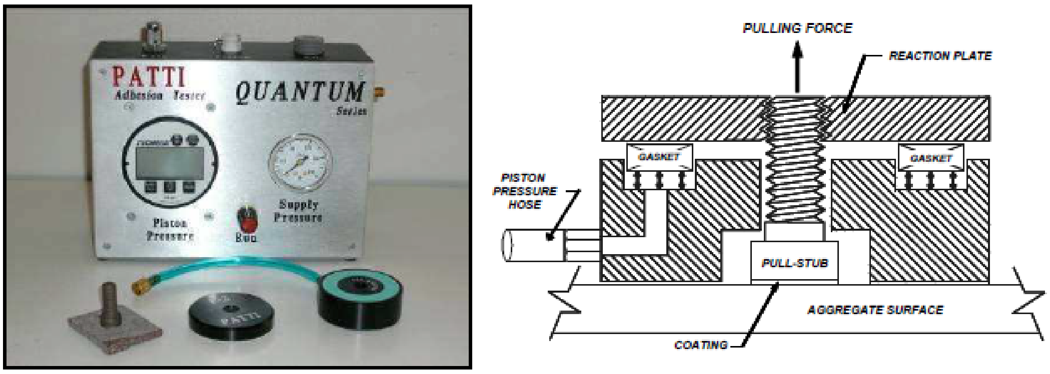


Figure 2 Bitumen Bond Strength Test Device (Canestrari et al., 2010)

The published research which used the BBS has shown that it has been successful in studying the moisture susceptibility of asphalt mixtures in terms of the change in pull-off strength and mode of failure for different combinations of binders, aggregates, and fillers. The test, however, does have some areas where it could be improved. The BBS is limited in its output such that only the maximum pull-off tensile strength can be extracted. In addition to that, the binder film only adheres to an aggregate substrate on one side while the other side adheres to a metal stub, which does not accurately represent field conditions. The test also uses a fixed loading rate and a film thickness which does not properly simulate field conditions. (Huang & Lv, 2016) investigated the influence of the asphalt film thickness and loading rate using the BBS and found that both factors highly affect the result such that the pull off tensile strength decreased, and the mode of failure transitioned from adhesive to cohesive as the asphalt film thickness increased and the loading rate decreased.

Rahim et al., 2019 and Zhang et al., 2015 modified the Universal Testing Machine (UTM) using custom made parts so that it can fit an asphalt film sandwiched between two aggregate cylinders which measured 25 mm in diameter and 5 mm in thickness rather than an aggregate substrate and a metal stub. The samples were fabricated using a modified DSR and a custom-made gap assembly respectively to accurately control the film thickness. The test was run as a pull-off test at a rate of 10 mm/min in tension and was sensitive enough to distinguish between different moisture conditions and different aggregate-bitumen combinations. While the setup did allow for the extraction of strain and elongation data and manipulating the asphalt film thickness and the loading rate, the setup required modifications to already very expensive machinery and thus rendered it not practical. Cala et al., 2019 also attempted to overcome the challenges faced when using the BBS test by fabricating specimens using a modified micrometer setup which allows to control the asphalt film thickness with an accuracy of 1 micron. The specimens constituted a binder film with a thickness of 20 microns that is sandwiched between an aggregate disk and a metal stub. Despite having a film thickness that is very close to the reported 10 micron optimum film thickness in in-service pavements (Al-Khateeb, 2018), achieving such a low and uniform film thickness requires a complicated and precise procedure to ensure that the surface of the aggregate is completely parallel to the surface of the metal stub, which also makes the sample preparation procedure not practical.

2.3. Research Needs

The literature review has shown that moisture damage is a very serious problem that leads to the accelerated deterioration of asphalt pavements, which in-turn leads to high maintenance fees, high vehicle operation costs, and possible traveler safety risks. Although moisture damage has been recognized since the early 20th century and extensive efforts have been undertaken by researchers and departments of transportation to limit the susceptibility of asphalt mixes to moisture damage, the issue remains prominent till this day due to the complexity of its mechanisms and the abundance of factors which impact it. Efforts to predict the moisture susceptibility of asphalt mixes using a single test such as the AASHTO T283 have shown to be insufficient due to the empirical nature of the test and the fact that it is not possible to account for all the factors which impact moisture damage in one test. As such, the use of different testing procedures at multiple scales is necessary to provide an accurate prediction of the moisture susceptibility of asphalt mixes.

CHAPTER 3

TESTING METHODOLOGY

This chapter outlines the testing methodology followed in this study with a focus on materials used, description of laboratory tests, equipment used, materials preparation, sample fabrication, and test execution.

3.1. Materials

3.1.1. Binders

The study incorporates two kinds of asphalt binders, a locally available neat binder that does not contain any additives and polymers and is commonly used in the Lebanese pavement industry, and a Styrene-Butadiene-Styrene (SBS) modified binder that was obtained from Total. Superpave Performance Grading (PG) testing and viscosity testing were conducted to characterize the binders as detailed in the following sections.

3.1.1.1. Neat Binder

According to the specifications of the manufacturer, the performance grade of the binder based on the Superpave criteria is PG 64-16. To verify the PG specification, high temperature performance grading was done in accordance with AASHTO M320. Testing was done for unaged samples as well as RTFO aged samples in accordance with ASTM D2872. The obtained high temperature PG grade for the neat binder was found to be 70˚C and the results shown in Figure 3 are for the unaged and RTFO aged samples respectively. Verification of the low temperature grading was not possible due to the lack of the required equipment. As such, the low temperature grade specified by the manufacturer was adopted and the neat binder is considered as PG 70-16.

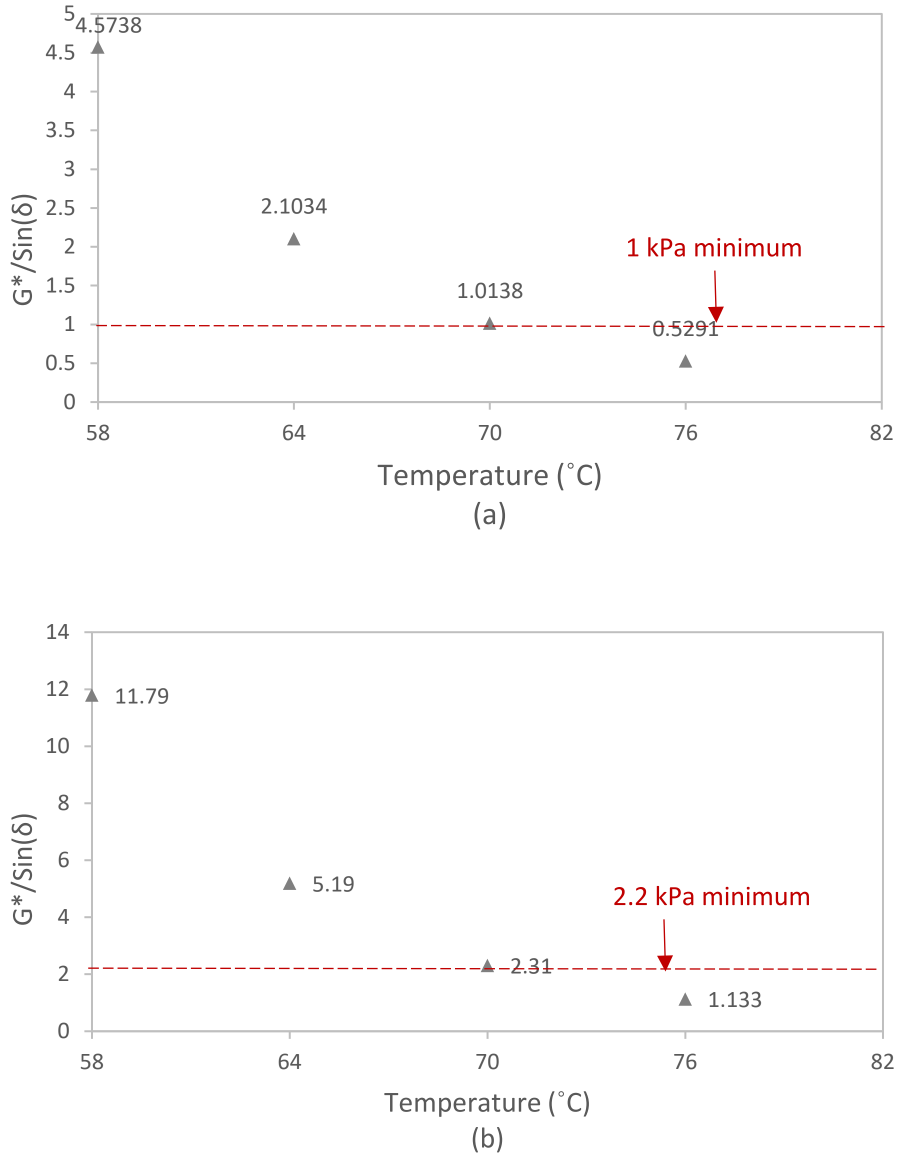


Figure 3 High Temperature PG Grading for a) Unaged and b) RTFO Aged Sample of Neat Binder

	G*/SIN(Δ) (kPa)			
TEMPERATURE	Unaged		Aged	
58˚C	4.4499	4.5738	11.7946	12.1555
64˚C	2.0950	2.1034	5.1973	5.3647
70˚C	1.0055	1.0138	2.3142	2.3597
76˚C	0.5128	0.5291	1.1337	1.1746

Viscosity testing was done in accordance with ASTM D4402 to determine the mixing temperature which was found to be 150˚C. The results of the viscosity tests are shown in Figure 4.

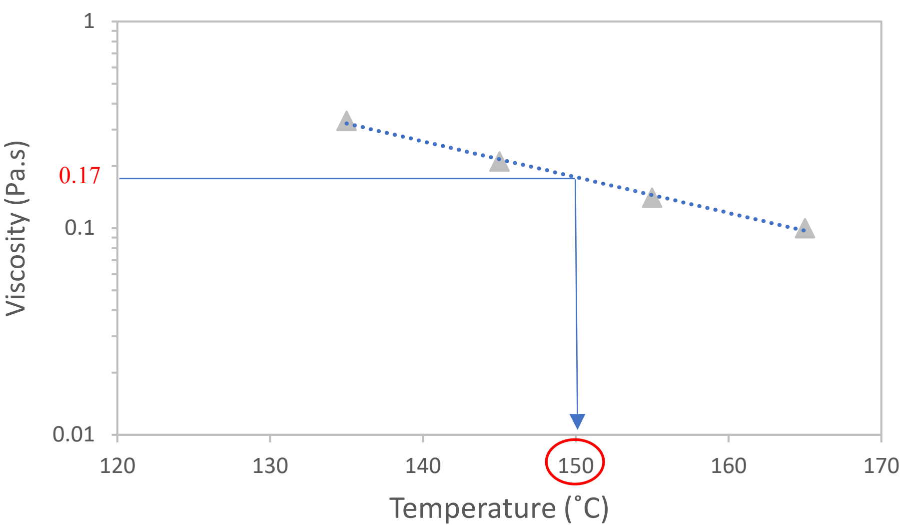


Figure 4 Viscosity as a Function of Temperature for the Neat Binder

3.1.1.2. SBS-Modified Binder

The Styrene-butadiene-styrene (SBS) modified binder obtained from Total was classified as a PG 76-28 by the manufacturer. To verify the performance grade, high temperature grading was done in accordance with AASHTO M320 for unaged and RTFO aged samples. Figure 5 shows the results of the RTFO-aged samples, which complies with a high temperature grade of 88˚C.

The multiple stress and creep recovery (MSCR) test, AASHTO T350, is typically done for modified binders and provides a high temperature grading that more accurately represents rutting performance. The test uses two parameters to characterize the binder, which are the non-recoverable creep compliance at 3.2 kPa ($J_{nr3.2}$) and the percent difference in non-recoverable creep compliance between 0.1 kPa and 3.2 kPa (J_{nrdiff}). Testing was done at 76˚C, which is the high temperature grade provided by the manufacturer, and the results showed $J_{nr3.2}$ values between 0.5 and 1, which corresponds to a very heavy traffic "V" grade. The binder, however, failed to meet the J_{nrdiff} criteria which should not exceed 75% for the binder to be resistant to rutting as values ranging between 250% and 800% were measured.

Based on the high temperature grading results, the PG 76-28 "V" specification was adopted for reporting purposes throughout the study.

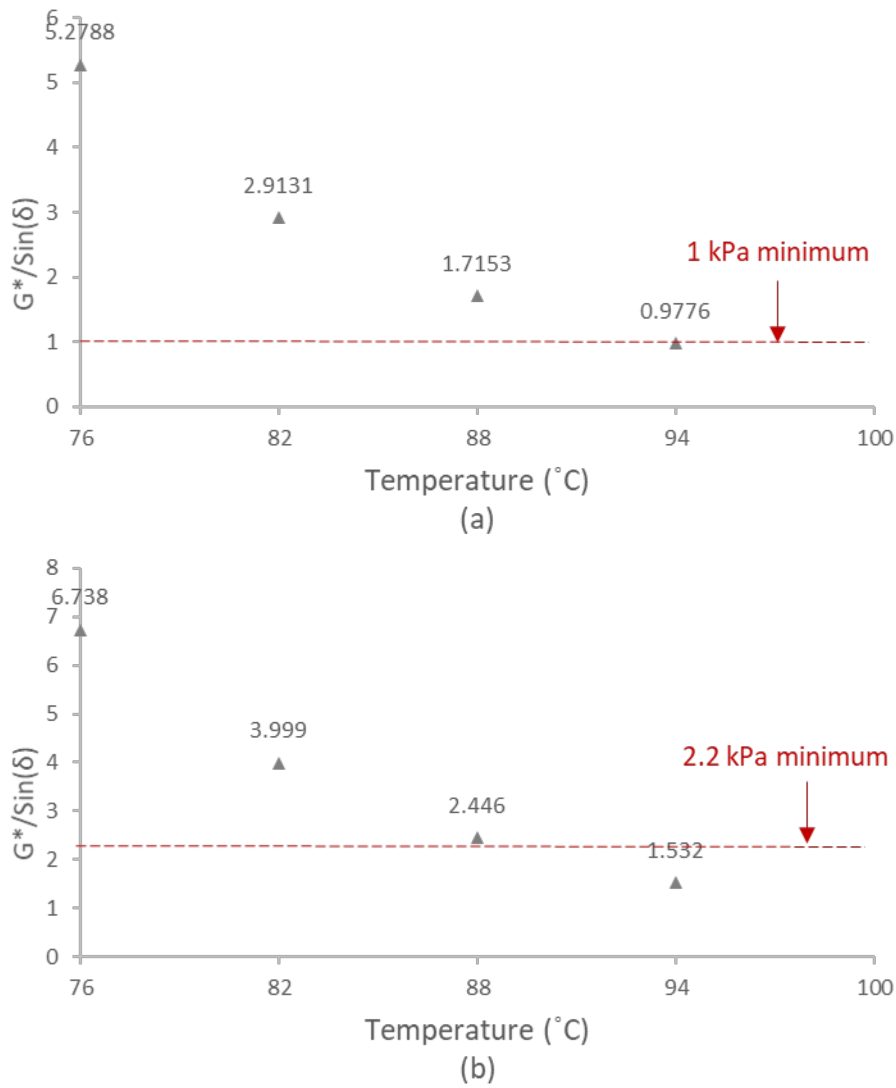


Figure 5 High Temperature PG Grading for a) Unaged and b) RTFO Aged Samples of SBS-Modified Binder

Adopting the mixing temperature specified by Superpave would result in unreasonably high temperatures for modified binders and thus damage them. To achieve proper coating of the aggregate at a temperature which would not damage the binder, the NCHRP report 459 states that targeting temperatures which correspond to a viscosity in the range of 0.75±0.05 Pa.s would be sufficient. While preparing the samples, it was observed that the obtained mixing temperature, 145˚C, was not sufficient since the binder cools down before the sample preparation process is completed. Therefore, a temperature between 165˚C and 170˚C was adopted, which translates to a viscosity in the range of 0.3 to 0.4 Pa.S recommended for some polymer-modified binders in the NCHRP Report 459.

3.1.2. Aggregates

Three different types of aggregates are used in this study: limestone, recycled concrete aggregate (RCA), and recycled asphalt pavement (RAP). The limestone, widely used in the Lebanese paving industry, was sourced locally. The RCA was obtained from the structural engineering laboratory at the American University of Beirut (AUB) in the form of a cylinder which was cast for the purpose of testing the compressive strength of the concrete. It was not possible to obtain the RCA from local construction and demolition waste because the geometry of the local waste does not comply with the requirements of sample preparation procedure for the direct tension test, which will be further discussed in the coming sections. As for the RAP, it was obtained from a local milled asphalt pavement. The use of recycled material is an important aspect of this study as more and more attention is directed towards sustainable infrastructure development for the purpose of lowering construction costs, conserving natural resources, and lowering the costs and pollution associated with moving the waste to landfills and dumping sites. Studying the moisture susceptibility of recycled materials at the level of the asphalt-aggregate interface may uncover insights that are not apparent when testing at the mixture level, thus enabling to better understand their behavior and predict their performance. The use of recycled material in this study aims to compliment the research on their effect on moisture susceptibility by evaluating the behavior of the asphalt with recycled materials at the binder and mastic levels.

3.1.3. Fillers

Three types of mineral fillers were used throughout this study: limestone, RCA, and hydrated lime. The limestone filler is of the same source as the aggregate and was obtained by crushing the aggregates and collecting the material passing the #200 sieve (0.075 mm size). Similarly, RCA filler was obtained by crushing the concrete cylinder and collecting the material passing the #200 sieve. As for the hydrated lime, it was purchased from Holcim, a local construction materials manufacturer.

3.2. Experimental Testing

This section provides a description of the direct tension test, the equipment used, as well as the materials preparation and sample fabrication processes.

3.2.1. Test Method

The direct tension test is based on the pull-off approach adopted in the Bitumen Bond Strength (BBS) test which is standardized under AASHTO T 361-16. The sample geometry of the direct tension test consists of a binder or mastic film confined between two aggregate disks as shown in Figure 6. The test is conducted by pulling the upper plate at a specified extension rate while recording load and displacement data until the sample cannot carry any load and completely fails.

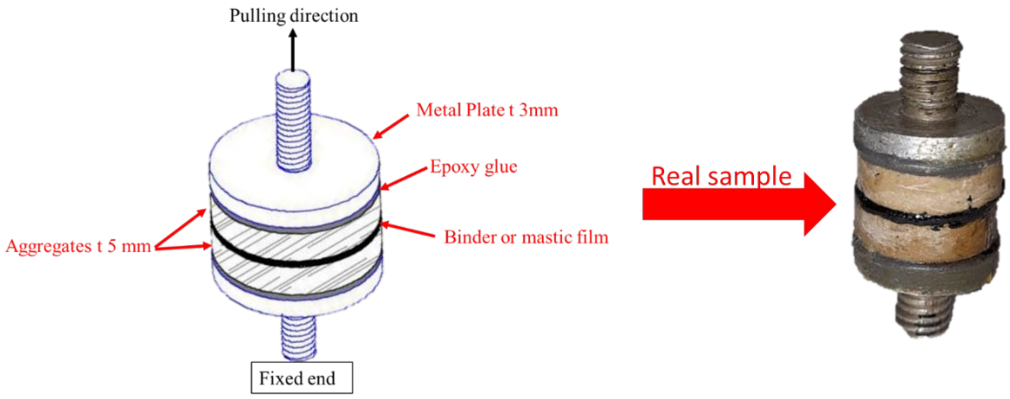


Figure 6 Direct Tension Test Sample Geometry

3.2.2. Equipment and Instrumentation

<u>3.2.2.1. Lloyd LS-1 UTM</u>

To perform the direct tension test, the Lloyd LS-1kN universal testing machine, shown in Figure 7, was used. The LS-1 belongs to the LS series UTMs offered by Lloyd Instruments and has a maximum pulling capacity of 1 kN. The UTM is capable of recording load and displacement data and conducting tension tests under stress or strain control mode. The extension rate can be selected between 0.01 to 2032 mm/min and the binder film thickness can be manually controlled to the nearest 0.1 mm. To accommodate the sample geometry, custom grips were designed and manufactured at the AUB engineering shops. The grips are shown in Figure 8 and serve the purpose of providing anchor points for the upper and lower plates of the testing sample.

Figure 7 Lloyd LS-1 UTM (from the LS-1 User Manual)

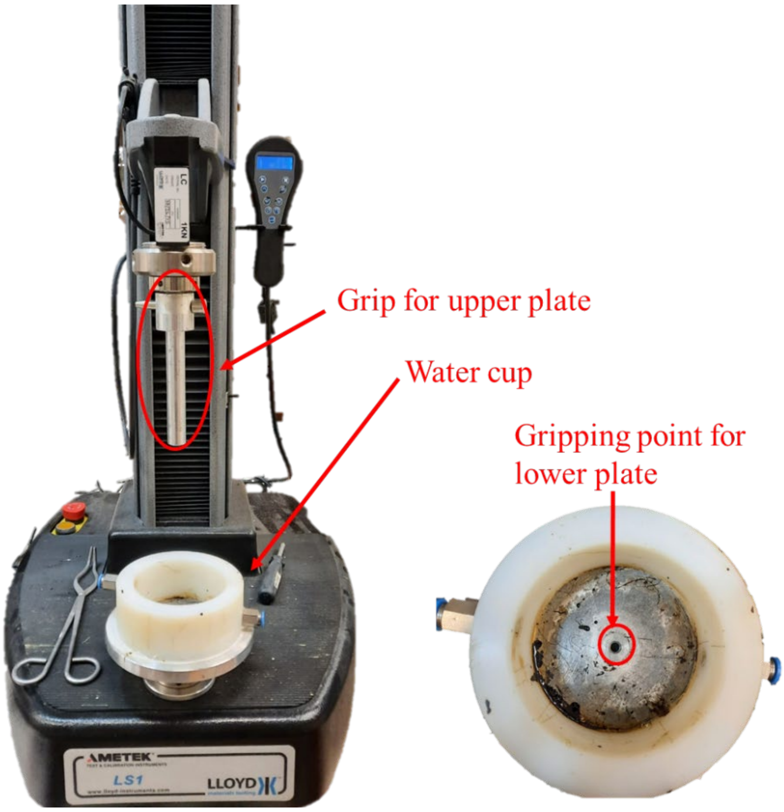


Figure 8 Lloyd LS-1 with Custom Grips

3.2.2.2. Thermal Imager

To verify the temperature of the binder before applying it on the surface of the aggregate, the Milwaukee M12 thermal imager is used. Other applications of the thermal imager include verifying the temperature of the soaking tub in addition to the temperature of the samples before testing. Figure 9 shows the thermal imager and a sample of the output displayed on the screen.

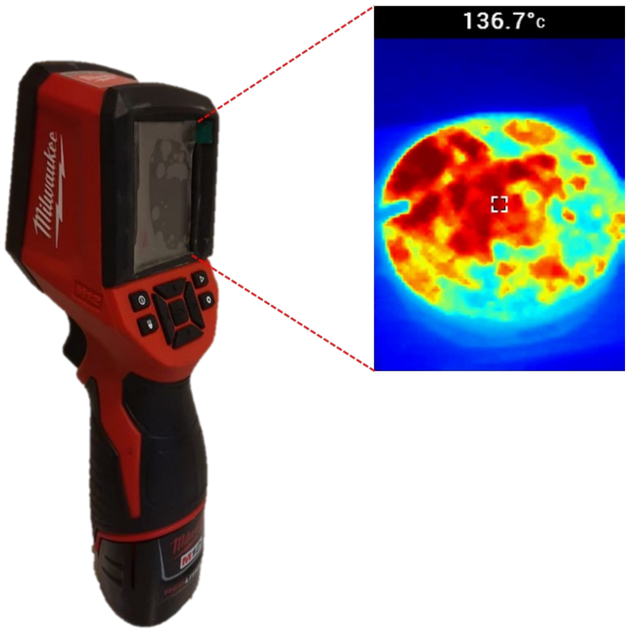


Figure 9 Thermal Imager and Sample Output

3.2.3. Sample Preparation

<u>3.2.3.1. Coring</u>

The sample preparation process starts with coring the aggregate blocks to obtain cylinders which have a diameter of 14 mm. Figure 10 shows the Hilti DD120 coring machine as well as the limestone, RCA, and RAP samples. In the case of the RAP, directly extracting cores from the milled asphalt sample was not possible due to the presence of excessive air voids, which caused the core to break inside the drill bit. To circumvent this issue, the RAP was heated in the oven and re-compacted using the Superpave gyratory compactor, and the cores were extracted from the compacted specimen.

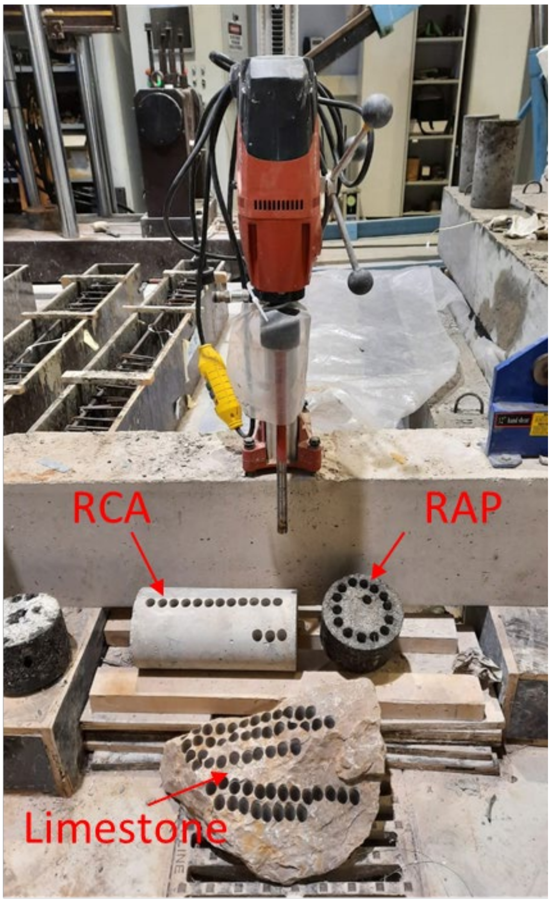


Figure 10 Cored Aggregates and Coring Machine

3.2.3.2. Sawing

After extracting the aggregate cores, they were sawed into small disks having a 5 mm thickness using a table saw. The process is illustrated in Figure 11.

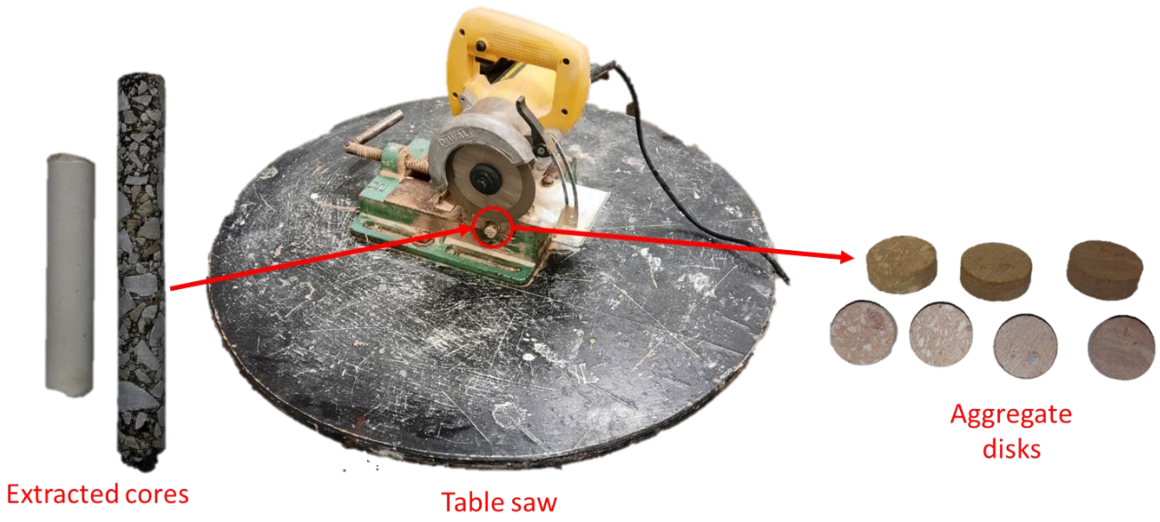


Figure 11 Sawing Process

3.2.3.3. Applying Epoxy between Aggregate and Metal Plate

The aggregate disks obtained after sawing the aggregate cores were cleaned to remove residues and then dried. The disks were then glued to the metal plates. In this study, Devcon 10110 plastic steel putty was used. After the disk was glued to the plate, it was left to cure for 24 hours to reach maximum strength.

3.2.3.4. Preparing Asphalt Mastic

The steps explained above are required for preparing samples that are meant to be tested at the binder level. Testing at the mastic level requires the additional step of preparing the asphalt mastic. The mastic is prepared by mixing the mineral filler with the asphalt binder at a ratio of 60% by mass of filler to binder, which is the minimum set by the Superpave mixture design guide. The mineral filler is the portion of fine aggregates that passes the #200 sieve, and the dimensions are smaller than 0.075 mm.

The mastic preparation was done by heating the binder to its mixing temperature and adding the mineral filler. The binder and filler were then mixed thoroughly to ensure that the filler is uniformly dispersed.

3.2.4. Sample Fabrication

Fabricating the samples required for the direct tension test started with cleaning the aggregate surface with acetone as any dust or contaminants could affect the quality of adhesion between the aggregate and the binder or mastic. The plates were then screwed to the top and bottom grips of the UTM. After that, the top plate was lowered until a load of 2N was recorded, which indicates that the aggregate surfaces were in contact (zero gap). The zero gap is essential for reaching the desired film thickness. The binder was then heated to the appropriate temperature and directly poured onto the aggregate surface. Immediately after that, the top plate was lowered until the desired film thickness was reached. It is very important to lower the top plate as soon as the binder is poured onto the lower aggregate to avoid losing temperature and consequently achieving poor adhesion. It is also important that little to no load is recorded while pressing the aggregates against each other as it would mean that the binder has cooled down. After the desired film thickness was achieved, the sample was left to cool down and then the excess binder was trimmed. The sample was then removed from the UTM and left to rest for 24 hours before it was tested in the dry condition as specified in the BBS procedure under AASHTO T361-16. During the resting period the sample must be supported in a way that prevents the binder film from creeping from the sides due to the weight of the upper plate and aggregate. Small metal bars were glued to the top and bottom plates as shown in Figure 12, which allows the self-weight of the top plate and aggregate to travel through the bars rather than the binder or mastic film.

Figure 12 Fabricated Sample with Supports

3.2.5. Sample Testing

<u>3.2.5.1. Dry Testing</u>

24 hours after the samples was fabricated, the supports were removed, the samples were re-installed in the UTM, and the tests were run at the desired extension rate. After the tests were completed, load and displacement data were extracted for analysis and images of the failure surface were taken to determine the mode of failure.

<u>3.2.5.2. Testing after Moisture Conditioning</u>

Samples which were subjected to moisture conditioning were placed in a water bath after the 24-hour rest period at a temperature of 40˚C and for 96 hours. This procedure was adopted by Chaturabong & Bahia, 2018 and proved to be effective in inducing moisture damage. 20 minutes before the 96-hour soaking period ends, the samples were conditioned in another water bath at the testing temperature and then re-installed in the UTM for testing. The subsequent steps were identical to those followed when testing in the dry condition.

3.2.6. Cleaning and Re-using Metal Plate

After a test is completed, the samples were removed from the UTM and placed in an oven at 170˚C to soften the epoxy holding the metal plate and aggregate together. A cutter blade was used to separate the plate and the aggregate. While the plate was still hot, the epoxy that was left over on the metal plate was scraped off as it was very hard to remove at low temperatures. The plates were then soaked in acetone to break down any residues left.

3.2.7. Challenges Faced and Proposed Solutions

Developing a protocol for sample preparation and testing required overcoming several challenges:

1. The binder or mastic film thickness has a major effect on the strength measured in the direct tension test. As such, the sawing process of the aggregate cores was one that requires great accuracy to achieve aggregate disks whose surfaces were as parallel as possible, thus ensuring a uniform binder or mastic film thickness. It is important that the sawing angle be parallel to the cross-sectional plane of the aggregate core. Since 100% parallelism between both planes cannot be achieved with a standard table saw, avoiding the rotation of the core in the plane of its cross-section,

illustrated in Figure 13, can highly decrease the risk of obtaining a non-uniform film thickness.

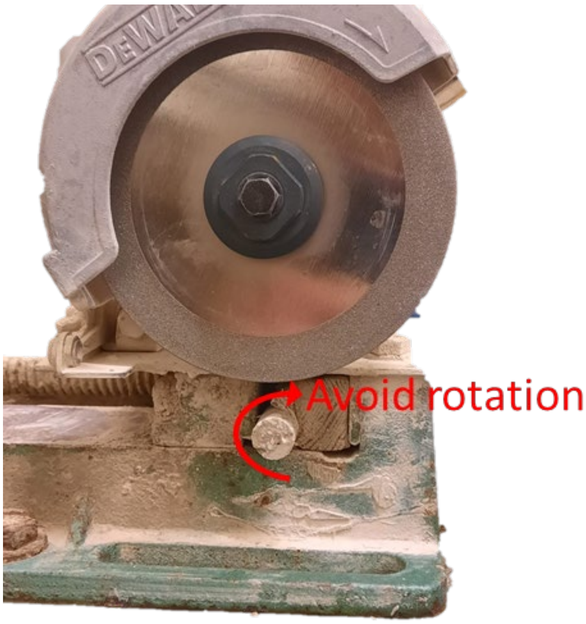


Figure 13 In-Plane Rotation of the Aggregate Core

2. Using epoxy that is resistant to water is essential as it must be able to withstand soaking for 96 hours at 40˚C. Although the Devcon 10110 is resistant to water as stated in the technical sheet, the epoxy failed when testing the samples after moisture conditioning. Covering the area where water can access the epoxy film with a thin film of sealant can prevent the deterioration of the epoxy. Den Braven Clear Fix was used to seal the area around the epoxy film and proved to be an effective solution.

CHAPTER 4

EXPERIMENTAL PLAN

The asphalt binder is the bituminous material that bonds the aggregates together, but it does not exist alone in the asphalt mixture. The binder exists with the filler material and is known as the asphalt mastic. In this study, testing was done for the asphalt binder and its mastic with different fillers to isolate the effect of the binder and filler types on moisture susceptibility. The sections below describe the test conditions as well as the experimental plan for each testing level.

4.1. Phase One: Binder Level

To assess the resistance of a binder-aggregate combination to moisture damage, the direct tension test was conducted before and after moisture conditioning and the changes to performance parameters such as the maximum attained strength and the strain at maximum strength were measured. A critical factor which affects the performance parameters as well as the mode of failure is the extension rate. It is well documented in literature that testing at high loading rates increases strength and promotes adhesive failure, while the opposite is true at low loading rates (Omar et al., 2020). The asphalt binder is a viscoelastic material which exhibits time-dependent behavior, meaning that the duration for which a load is applied affects properties such as its stiffness, viscosity, strength, and failure mode. Knowing that traffic moves at different speeds when crossing over a pavement, the evaluation of moisture susceptibility must be done at different extension rates to evaluate the effect of moisture conditioning on the adhesive and cohesive bonds. Based on trial tests, 5 mm/min and 80 mm/min were chosen as the extension rates to target cohesive and adhesive failure respectively, and 20 mm/min was chosen to target a mix of adhesive and cohesive failure. The remaining testing parameters are detailed in Table 1.

Table 1 Phase 1 Testing Parameters

Testing temperature	22.5 ˚C
Soaking temperature	40 ˚C
Soaking solution	Distilled water
Soaking time	96 hours
Conditioning time before testing	20 minutes at 22.5˚C

The testing plan for Phase 1 is detailed in Table 2. It was not possible to test all binder-aggregate combinations at all 3 extension rates due to time and logistics limitations.

Table 2 Phase 1 Testing Plan of Binder Samples

Combinations			Replicates	
Binder	Aggregate	Extension Rate (mm/min)	Dry	Wet
PG 70-16	Limestone	5, 20, 80	3	3
	RCA	20	3	3
	RAP	20	3	3
PG 76-28	Limestone	5, 20, 80	3	3
	RCA	20	3	3
	RAP	20	3	3

4.2. Phase Two: Mastic Level

In Phase 2 of the experimental plan, the effect of incorporating different mineral fillers on moisture damage was evaluated. RAP was not tested as a mineral filler since it is mostly incorporated in paving projects as a replacement for coarse and fine aggregates rather than filler material. The testing plan is detailed in Table 3.

Table 3 Phase 2 Testing Plan of Mastic Samples

Combinations				Replicates	
Binder	Aggregate	Filler	Extension Rates (mm/min)	Dry	Wet
PG 70-16	Limestone	Limestone	5, 20, 80	3	3
	Limestone	Lime	20	3	3
	RCA	Lime	20	3	3
	Limestone	RCA	20	3	3
PG 76-28	Limestone	Lime	20	3	3

CHAPTER 5

ANALYSIS AND RESULTS

5.1. Phase One

To analyze the results of the tests conducted in Phase 1, the data was split into two groups as show in Table 4.

Table 4 Grouping of Experiments for Phase 1

	GROUP 1	GROUP 2
AGGREGATES	Limestone	Limestone – RCA - RAP
BINDERS	PG70 – PG76	PG70 – PG76
EXTENSION RATES (MM/MIN)	5 – 20 – 80	20
MOISTURE STATES	Dry – Wet	Dry – Wet

Since the limestone aggregate is tested with both binders, at all extension rates, and in both moisture states, the analysis of group 1 allowed us to compare the effect of different combinations of binders, extension rates, and moisture states for the limestone aggregate. The analysis of group 2, on the other hand, allows to draw comparisons between different combinations of aggregates, binders, and moisture states at a 20 mm/min extension rate. The considered performance parameters are strength, strain at maximum strength, and mode of failure. Strength is a measure of the load that can be resisted by the binder (cohesive strength) or the interface bond between the binder and the aggregate (adhesive strength). The strain at maximum strength is a measure of ductility as it indicates how much the binder can deform before it cannot resist loading anymore.

The analysis for both groups was done using load-displacement curves, boxplots, and the failure surfaces of the samples. Furthermore, statistical analysis was done by constructing a three-way analysis of variance model using R Studio to validate the trends and observations highlighted in the boxplots and load-displacement curves. The model diagnostic plots were examined to verify that the ANOVA assumptions regarding normality and equality of variance. The significance level (α) was chosen to be 10% as using 5% was considered conservative given the variability between the replicates.

5.1.1. Group One: Effect of Rate of Loading and Binder Type

5.1.1.1. Strength

The results of the strength data for group 1 are summarized in Figure 14. For the samples tested using the PG70 binder in the dry state, the strength increased when the extension rate increased from 5 mm/min to 20 mm/min and then decreased when reaching 80 mm/min. After moisture conditioning, the strength also increased between 5 mm/min and 20 mm/min but did not change upon reaching 80 mm/min.

For the samples tested using the PG76 binder, the strength increased with the increase in extension rate from 5 mm/min to 80 mm/min in the dry state as well as after moisture conditioning.

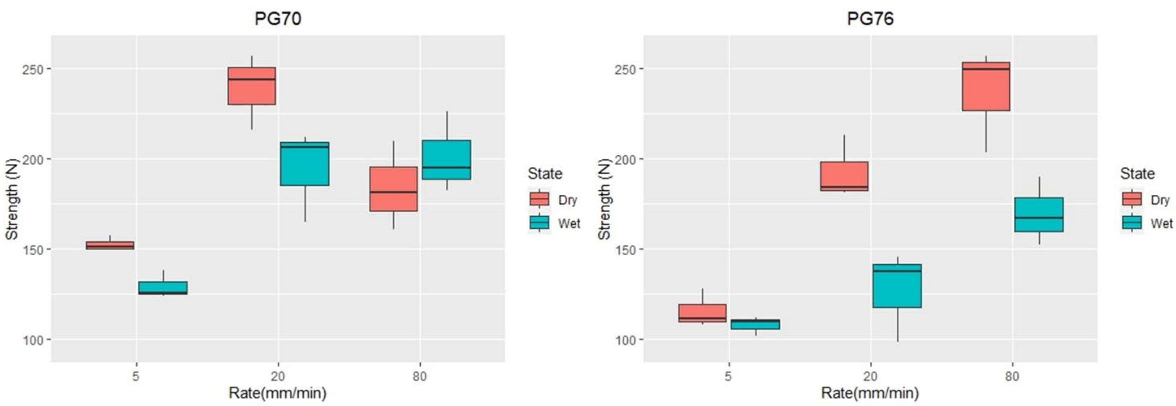


Figure 14 Boxplot Summary of Strength Data for Group 1 Split by Binder Type

An ANOVA model was constructed to evaluate the effect of varying the extension rate on the measured strength of the samples. The results for the PG70 and PG76 binders are shown in Table 5 and Table 6 respectively and confirm the observed trends.

Table 5 Results of ANOVA Analysis for the Samples Made with PG70 Binder

	EXTENSION RATE	P-VALUE	SIGNIFICANT?
DRY	5 mm/min - 20 mm/min	0.0007196	Yes
	20 mm/min - 80 mm/min	0.0689436	Yes
MOISTURE CONDITIONED	5 mm/min - 20 mm/min	0.0174315	Yes
	20 mm/min - 80 mm/min	0.9999999	No

Table 6 Results of ANOVA Analysis for the Samples Made with PG76 Binder

	EXTENSION RATE	P-VALUE	SIGNIFICANT?
DRY	5 mm/min - 20 mm/min	0.003	Yes
	20 mm/min - 80 mm/min	0.2584	No
MOISTURE CONDITIONED	5 mm/min - 20 mm/min	0.9831	No
	20 mm/min - 80 mm/min	0.2932	No
	5 mm/min – 80 mm/min	0.0269	Yes

An important parameter to consider when evaluating the resistance to moisture damage is the percentage of lost strength after moisture conditioning. Figure 15 shows the lost strength after moisture conditioning for both binder types which was obtained according to the following equation:

$$Lost\ Strength(\%) = 1 - \frac{Strength_{wet}}{Strength_{dry}}$$

The strength used in the above equation was obtained by averaging the 3 replicates at each condition.

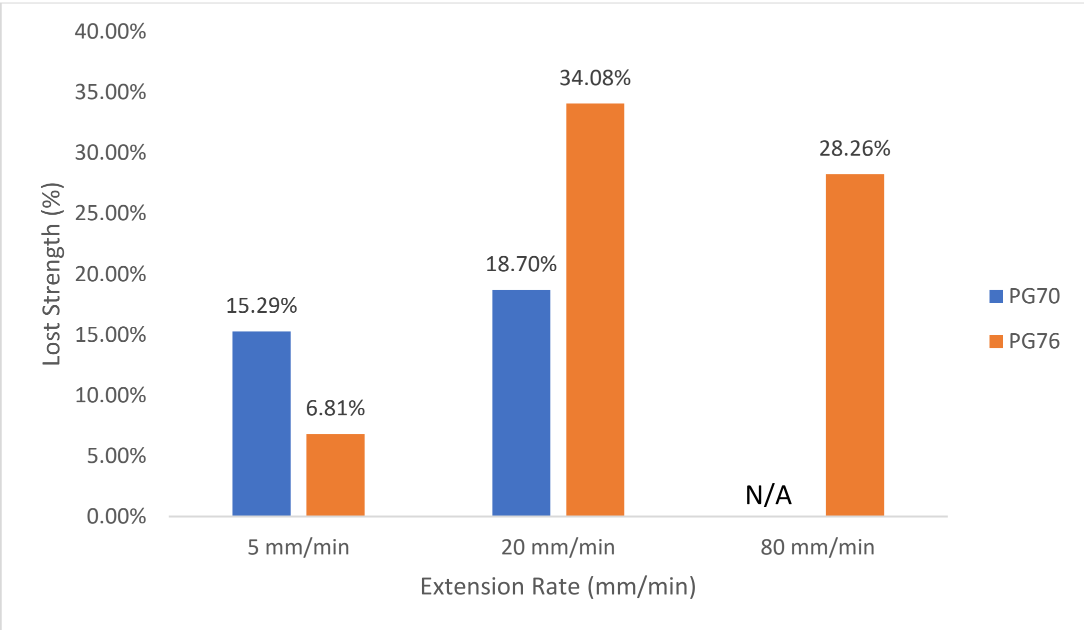


Figure 15 Lost Strength after Moisture Conditioning

The polymer-modified binder loses only 6.8% of its strength at 5 mm/min as opposed to the non-modified binder which loses 15.2%. At 5 mm/min, the binder stretches and expands as the two aggregates are pulled apart and remains adhered to the top and bottom aggregate surfaces. Therefore, failure occurs within the asphalt binder and the measured strength is indicative of its cohesive bond. The polymer-modified binder contains polymers which improve its cohesive strength, and this may explain why the lost strength when testing at 5 mm/min is lower when using the polymer-modified binder than it is with the non-modified binder. At 20 mm/min, the samples fabricated with PG70 binder lose only 18.7% of their strength as compared to 34% for the samples fabricated with PG76, which is a sharp increase from 6.8% at 5 mm/min. At 80 mm/min, samples fabricated with PG76 binder lose 28.3% of their strength, a 6% decrease over that at 20 mm/min. On the other hand, samples fabricated with PG70 binder do not exhibit a decrease in strength. Table 7 presents the results of statistical analysis (ANOVA) done to test the effect of moisture conditioning on strength for each extension rate. Although the samples fabricated with the PG70 binder, on average, lose a percentage of their strength after moisture conditioning, the ANOVA results show that there is no statistical evidence to suggest that moisture conditioning has a significant effect on strength. In the case of the PG76 binder, the analysis shows that there is a significant effect of moisture conditioning at 20 mm/min and 80 mm/min but not at 5 mm/min.

Table 7 Results of Statistical Analysis to evaluate the effect of Moisture Conditioning on Strength

Effect of Moisture Conditioning	Extension Rate	P-Value	Significant?
PG70	5 mm/min	0.938	No
	20 mm/min	0.242	No
	80 mm/min	0.992	No
PG76	5 mm/min	0.999	No
	20 mm/min	0.016	Yes
	80 mm/min	0.013	Yes

When testing at 5 mm/min, the samples fabricated with PG76 binder lose a smaller percentage of their strength after moisture conditioning compared to those fabricated with the PG70 binder. At the same time, the samples fabricated with PG70 binder achieve a higher strength in the dry and moisture conditioned states. This observation may indicate that the polymer modified binder displays superior performance in resisting moisture damage to its cohesive bond, but poorly adheres to the aggregate surface.

Another observation that requires investigation is the fact that the samples fabricated with the PG70 binder achieved their maximum strength at an extension rate of 20 mm/min and then decreased in strength when testing at 80 mm/min. As mentioned before, it is well known in literature that increasing the rate of loading leads to an increase in strength, which was not the case with the PG70 binder.

An explanation for these observations is provided in the following sections as additional input are acquired and used.

5.1.1.2. Strain at Maximum Strength

The strain at maximum strength data for group 1 are shown in Figure 16. As can be seen for the PG70 binder, the trend of strain at maximum strength seems to follow that of strength. In dry and moisture conditioned states, values increased when the extension rate increased from 5 mm/min to 20 mm/min, but they decreased at 80 mm/min in the dry state and remained unchanged in the moisture conditioned state. Samples tested at 20 mm/min rate achieved higher strain at maximum strength as compared to those tested at 5 mm/min, although it is at 5 mm/min that the binder is less stiff and consequently able to strain more. The load vs. displacement curves were plotted for the samples fabricated with PG70 binder and tested after moisture conditioning at 5 mm/min and 20 mmm/min (Figure 17). These curves help us to distinguish between the total strain and the strain at maximum strength. At the 5 mm/min rate the samples achieved higher total strain than that at 20 mm/min since they failed in a ductile manner. At 20 mm/min, the samples achieved higher strain at maximum strength but exhibited brittle behavior which caused a lower total strain as compared to samples tested at 5 mm/min.

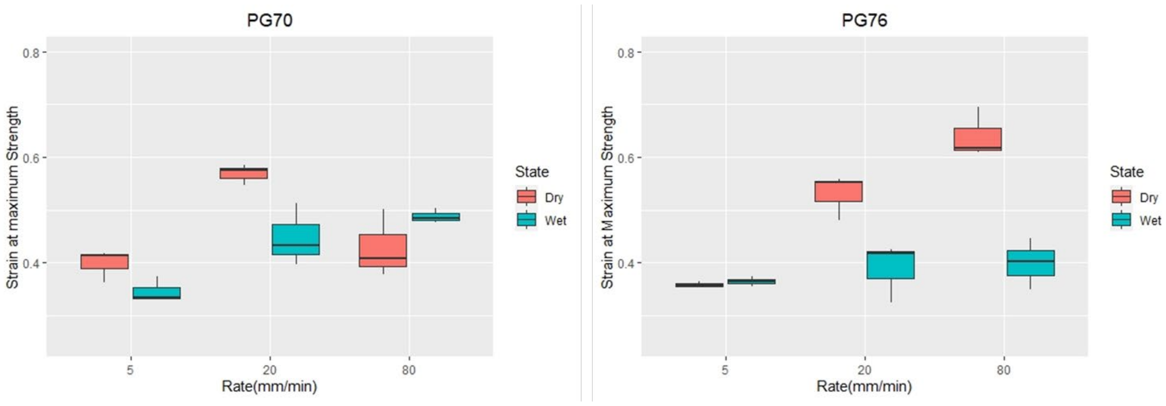


Figure 16 Boxplot Summary of Strain at Maximum Strength for Group 1 Split by Binder Type

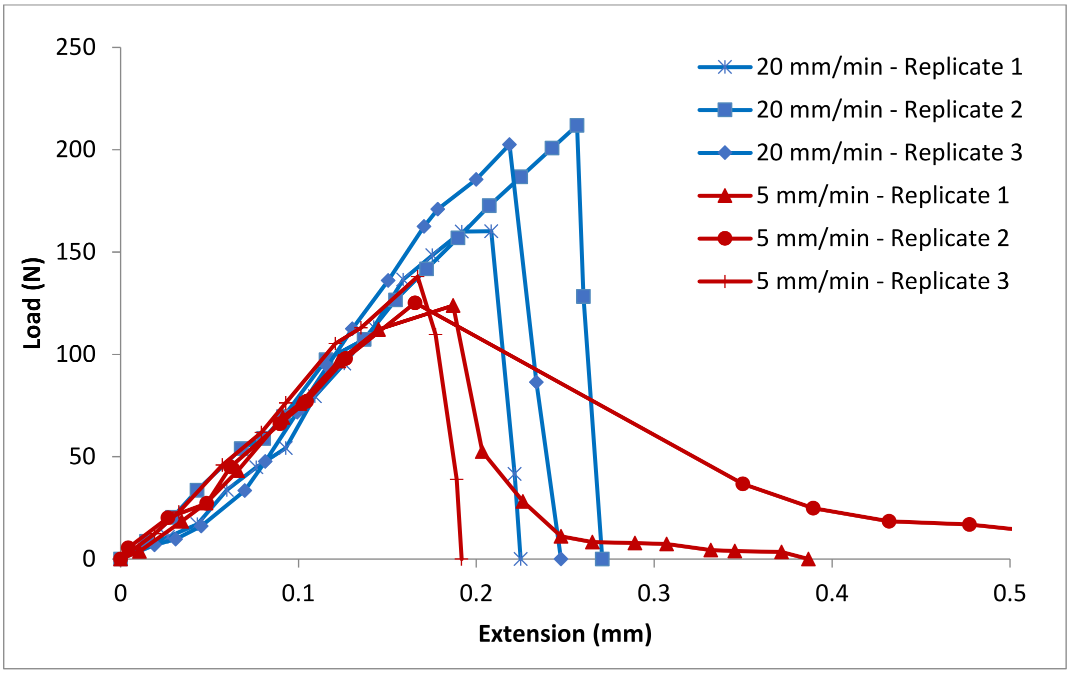


Figure 17 Load vs Displacement for Samples Fabricated with PG70 Binder and tested after Moisture Conditioning

For samples fabricated with the PG76 binder, the strain at maximum strength increased with the increase in the extension rate in the dry condition, but remained at around 0.4 when the samples were subjected to moisture conditioning, regardless of extension rate.

5.1.1.3. Mode of Failure

For the samples fabricated with the PG70 binder and tested in the dry and moisture conditioned states, the strength increased when the extension rate increased from 5 mm/min to 20 mm/min. At 80 mm/min, strength decreased in the dry state and remained statistically the same after moisture conditioning. The decrease in strength at 80 mm/min was not expected since it is consistent in literature that increasing the loading rate leads to an increase in strength. To understand the reason behind this behavior, it is necessary to examine the modes of failure before and after moisture conditioning which are presented in Figure 18.

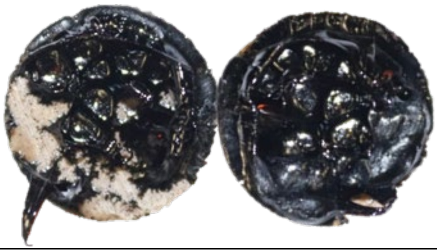
MOISTURE STATE / EXTENSION RATE	DRY	MOISTURE CONDITIONED
5 MM/MIN		
20 MM/MIN		
80 MM/MIN		

Figure 18 Failure Surfaces for Samples Fabricated with PG70 Binder

In dry conditions, the mode of failure was cohesive when testing at 5 mm/min and adhesive when testing at 20 mm/min, both of which were expected since increasing the loading rate also leads to a shift from cohesive to adhesive failure. When the extension rate is increased to 80 mm/min, the mode of failure reverted to cohesive although it was expected that the sample would continue to gain strength and fail in adhesion. A possible explanation to this behavior may be that the binder is not able to withstand the high stresses resulting from the high loading rate and is thus prematurely failing in cohesion. The fact that the PG76 binder, which is modified with a polymer that increases its cohesive strength, did not fail in cohesion at 80 mm/min may serve as support for the aforementioned justification.

After the samples were subjected to moisture conditioning, the mode of failure at 5 mm/min, shown in Figure 19, became inconsistent and dependent on the degree of damage inflicted by the water to the adhesive bond. Figure 20 compliments the observations presented in Figure 19. It can be seen from replicates 1 and 3, which failed in adhesion, brittle behavior was dominant after reaching the maximum strength. On the other hand, replicate 2, which has a large cohesive failure area on its surface, exhibited ductile behavior after reaching its maximum strength. At 20 mm/min the mode of failure was a mix between adhesive and cohesive. The effect of moisture conditioning can be clearly seen in the areas where adhesive failure occurred as there are no remains of the binder on the aggregate surface. At 80 mm/min, similar to testing in the dry state, the mode of failure was cohesive, and it is accompanied by a brown-ish film that is left adhered to the aggregate surface on 2 of the 3 tested replicates. The brown film could be a concentration of oils that had separated from the binder because of moisture conditioning.

Replicate 1	
Replicate 2	
Replicate 3	

Figure 19 Failure Surfaces of Samples Fabricated with PG70 Binder and Tested at 5 mm/min after Moisture Conditioning

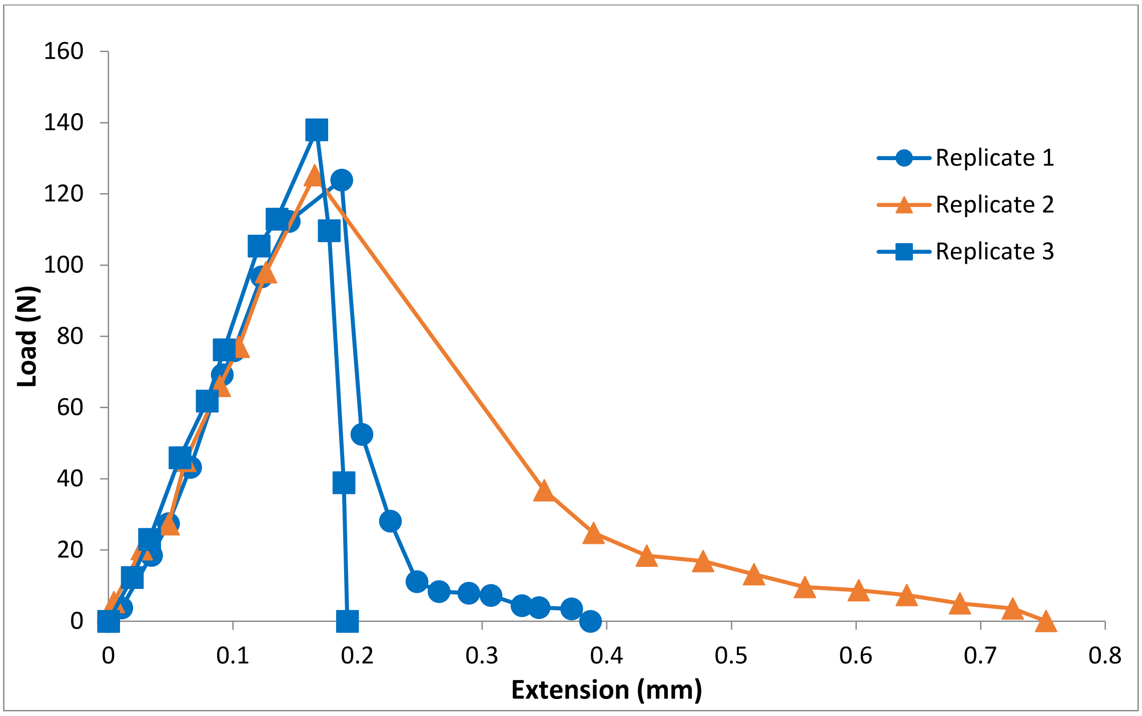


Figure 20 Load vs Displacement for Samples Fabricated using PG70 Binder and Tested in the Moisture Conditioned State at 5 mm/min

The samples fabricated with the PG76 binder and tested at 5 mm/min only lost 6.8% of their strength following moisture conditioning, yet achieved significantly lower strength figures compared to the samples fabricated with the PG70 binder. This observation may imply that the polymer-modified binder improves moisture resistance but does not improve the cohesive strength of the binder. Since it is well established that polymer modification increases the cohesive strength of the binder, the observation was further investigated by plotting the load-displacement curves for the samples fabricated with the PG76 binder in both dry and moisture conditioned states. The curves representing each state are plotted by averaging all 3 replicates and are shown in Figure 21.

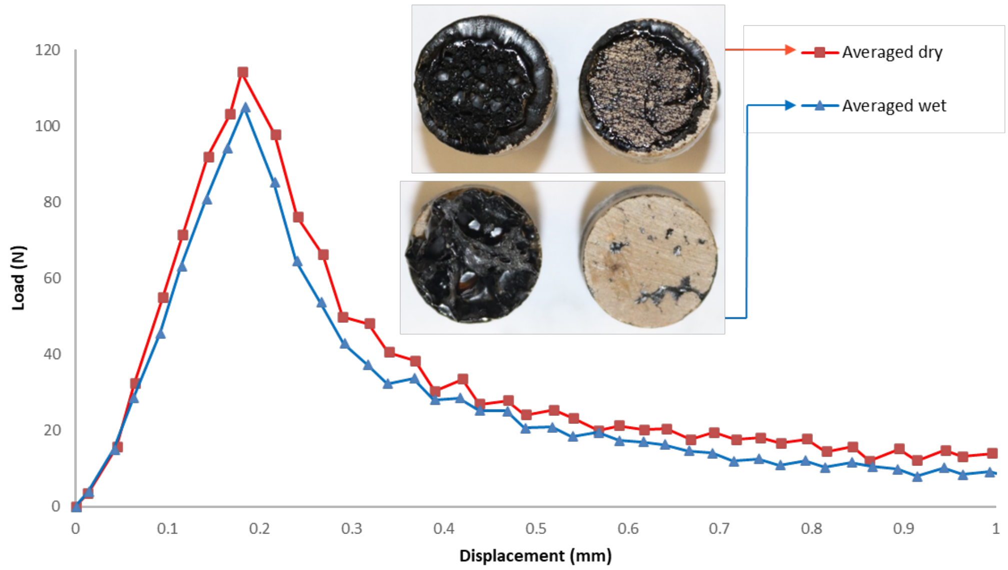


Figure 21 Averaged Load vs Displacement Curves and Failure Surfaces for Samples with PG76 Binder in Dry and Moisture Conditioned States at 5 mm/min

The samples in both conditions gained load in a similar way and unloaded after reaching the maximum strength in a ductile manner that indicates cohesive failure. A close look at the failure surface in Figure 22 shows that despite the ductile behaviour after reaching the maximum strength, the mode of failure was adhesive rather than cohesive. Another close examination of the samples during testing in Figure 23 shows a thin film of binder that remains attached to the aggregate surfaces and is the likely explanation behind the ductile behaviour as this thin film fails in cohesion. Based on the available information, the likely explanation as to why the PG76 binder achieves significantly lower strength at 5 mm/min when compared to the PG70 binder is poor adhesion with the limestone aggregate, which possibly makes it prone to stripping even without moisture conditioning. Recalling that the samples fabricated with the PG76 binder had similar strain at maximum strength after moisture conditioning regardless of the extension rate at which they were tested reinforces the previous claim regarding the moisture susceptibility of the PG76 binder with the limestone aggregate.

Figure 22 Failure Surface for Sample Fabricated with PG76 Binder and Tested at 5 mm/min in the Dry State

Figure 23 Thin Layer of binder around adhesive failure area

Despite it being well documented in literature that SBS modification increases moisture resistance (Hu et al., 2020; Iskender et al., 2012; Kok & Yilmaz, 2009; Sengul et al., 2013; Vamegh et al., 2020), a possible explanation for the poor moisture resistance of the PG76 binder may come from the surface energy concept. Little & Bhasin, 2006 stated that one material can wet the surface of another material if the cohesive bond energy of the former is less than the work of adhesion of the latter. Based on this information, the cohesive bond energy of the polymer-modified binder could be exceeding the work of adhesion of the limestone aggregate thus leading to an adhesive bond which could be easily displaced by the action of water. Further investigation using the surface energy method is required to verify the accuracy of this justification.

Figure 24 presents the failure surfaces for the samples fabricated with the PG76 binder. Similar to the samples tested at 5 mm/min, those tested at 20 mm/min and 80 mm/min failed in adhesion in both the dry and moisture conditioned states as it can be seen that there is complete stripping of the binder film. The effect of moisture conditioning can also be seen in

Figure 25 such that it caused a shift from ductile to brittle behavior after reaching the maximum strength.

MOISTURE STATE / EXTENSION RATE	DRY	MOISTURE CONDITIONED
20 MM/MIN		
80 MM/MIN		

Figure 24 Failure Surfaces for Sample Fabricated with the PG76 Binder

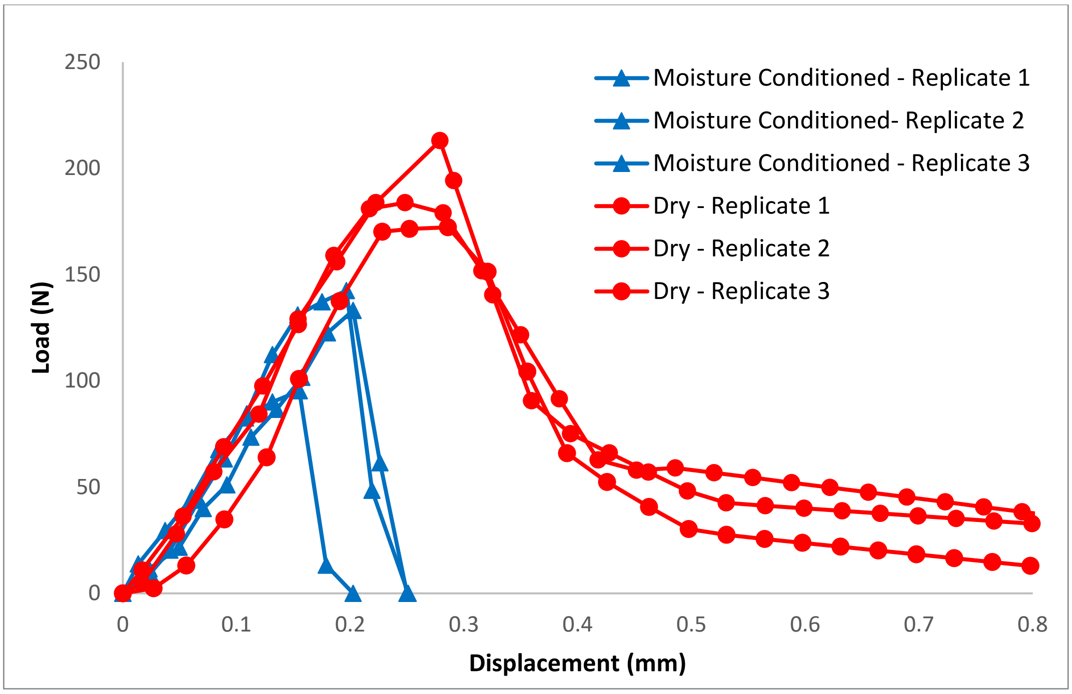


Figure 25 Load vs Displacement for Samples Made with PG76 Binder at 20 mm/min

5.1.1.4. ANOVA Models Results

A three-way ANOVA model was constructed to determine the factors and interaction terms which affect strength as well as the strain at maximum strength for the data in group 1. The explored variables are the binder type, extension rate, as well as the moisture state. The significance level (α) is chosen to be 10% instead of the traditional 5% to account for the variability which may be caused by the nature of the experimental procedure as well as materials, particularly the RAP and RCA. The results for the strength and strain at maximum models are presented in Table 8. For the strength model, all variables and their interaction terms are significant, meaning that the strength of a tested sample cannot be accurately predicted without considering the binder type, extension rate, and moisture state. The interaction terms mean that considering the variables as standalone factors is not enough to predict the response. The variables interact to produce a combined effect which influences the response. The results are mostly similar with the strain at maximum strength model with the exception that the binder as a standalone factor is not significant, but its interaction with the remaining variables is. Figure 26 shows the diagnostic plots for the strength model which did not indicate any problems with the ANOVA assumptions such as the normal distribution of the population and the variance being common for the populations.

Table 8 ANOVA Results for Strength and Strain at Maximum Strength Models

		P-values	
		Strength Model	Strain at Maximum Strength Model
Predictor Variables	Rate	$4.65*10^{-9}$	$6.86*10^{-8}$
	State	$5.64*10^{-5}$	$2.55\text{-}10^{-6}$
	Binder	0.00078	0.97
Interaction terms	Rate:State	0.0516	0.01
	Rate:Binder	0.0011	0.00920
	State:Binder	0.0308	0.00317
	Rate:State:Binder	0.0154	$3.05*10^{-5}$

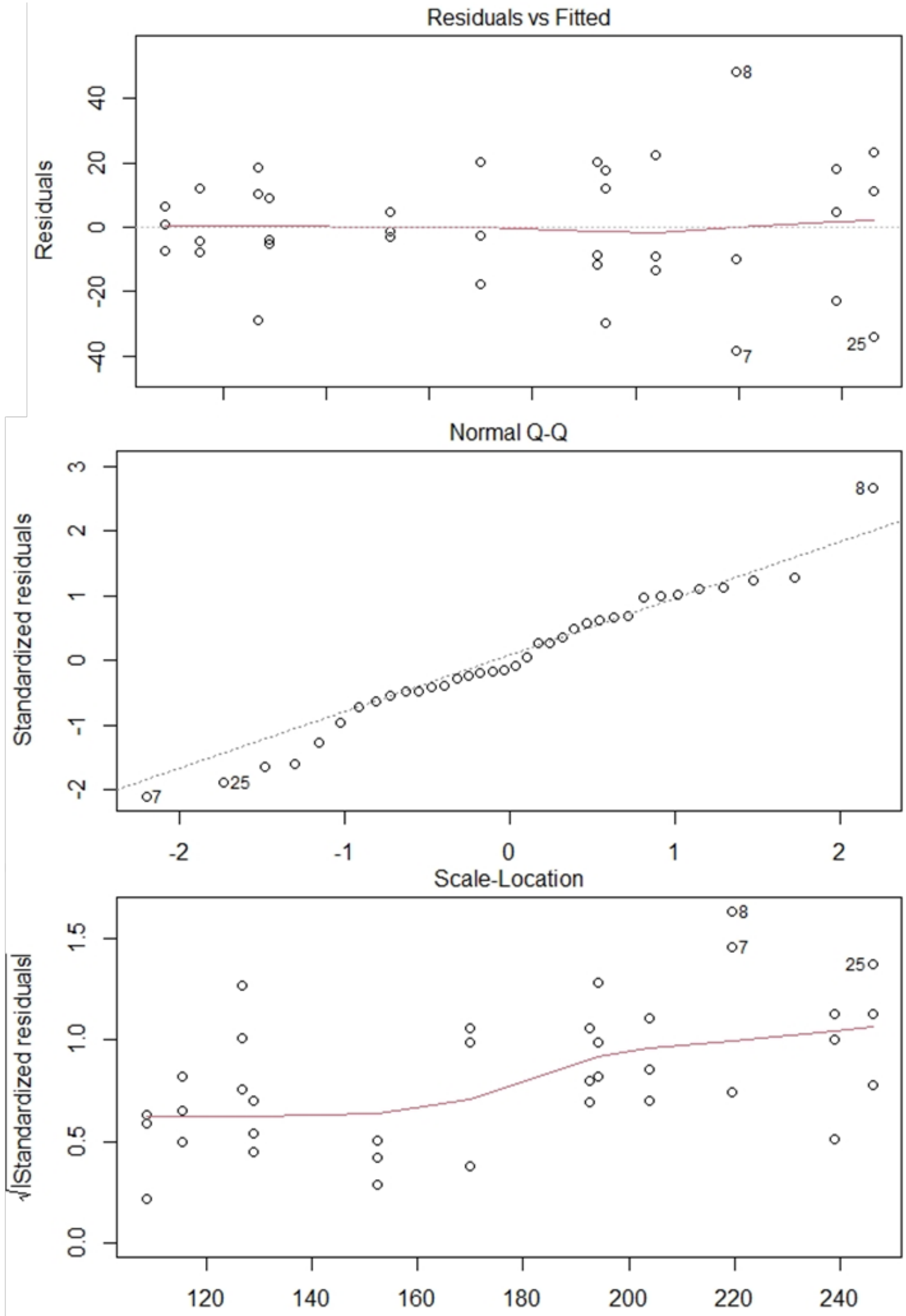


Figure 26 Strength Model Diagnostic Plots

5.1.2. Group Two: Effect of Aggregate Type

Compared to group 1, group 2 fixes the extension rate at 20 mm/min and adds RCA and RAP as aggregates, thus allowing us to evaluate the performance of different aggregates with the 2 different binder types in moisture damage resistance.

5.1.2.1. Strength

Figure 27 presents a summary of the strength data for group 2. It is important to recall that the RAP samples are obtained from a milled asphalt pavement that is re-compacted, cored, and sliced into 5 mm thick disks. The obtained disks constitute aggregates which are held together by the aged binder as shown in Figure 28. The PG76 and PG70 binders are then sandwiched between 2 of the disks, thus forming the samples which are tested in direct tension.

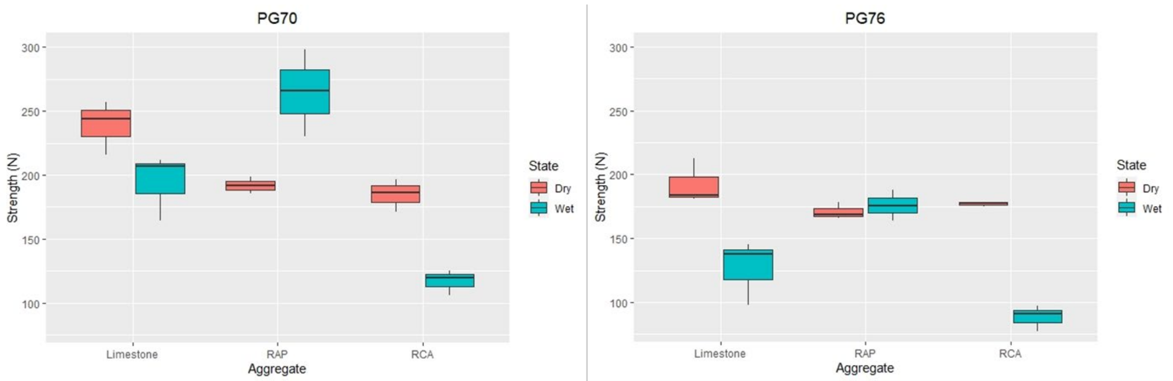


Figure 27 Boxplot Summary of the Strength Data for Group 2 Split by Binder Type

For the samples fabricated with RAP and PG70 binder, failure occured at the epoxy level and the strength after moisture conditioning exceeded that in the dry state. Since the samples failed at the epoxy level, it was not possible to examine the failure surface to understand the reason behind the gain in strength after moisture conditioning. One sample, however, failed at the level of the asphalt aggregate interface and is shown in Figure 29. It can be seen that the large, highlighted aggregate is the likely reason behind the low strength as it causes poor adhesion. It is also apparent that the areas with cohesive failure are the same areas where the aged binder is located on the RAP surface. A possible explanation as to why the samples exhibited an increased strength after moisture conditioning is that the 96-hour soaking period at 40˚C may have caused the virgin binder to soften and adhere better to the aged binder in the RAP surface, thus leading to an increase in strength.

In a study by Carpenter & Wolosick, 1980, the authors showed that when adding virgin binder to RAP, the diffusion and rejuvenation process can take an extended period of time and is also dependent on the composition of the new and aged asphalt. Therefore, it is necessary to account for the time and conditions required for the unaged binder to blend with the aged binder in the RAP when testing in dry conditions. In addition, the sample geometry may not be suitable for RAP is that aggregates in the RAP surface do not comply with the 4:1 representative volume element (RVE) criteria set by ASTM D3497, which necessitates that the size of the largest aggregate cannot exceed one-fourth the smallest dimension of the sample. To comply with the RVE criteria, a sample with a larger diameter may be required. Alternatively, the RAP could be obtained from a mix with a smaller nominal maximum aggregate size.

Figure 28 RAP Disk

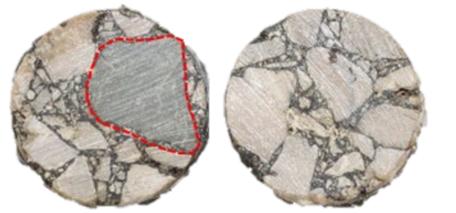
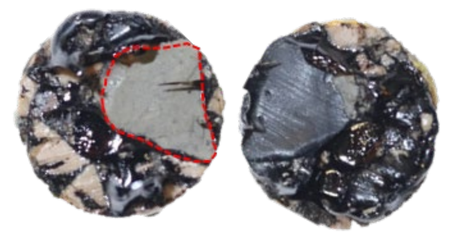
Aggregate Surface	
Failure Surface	

Figure 29 RAP Surface Before and After Testing in the Moisture Conditioned State

The strength results show that the combination of limestone aggregate and PG70 binder performs the best in terms of the maximum achieved strength. Changing the aggregate type to RCA leads to a significant drop in strength. Furthermore, coupling the RCA aggregate with PG76 binder leads to a further reduction in strength specifically after moisture conditioning. The strength when testing in the dry condition remained the same when the binder type was changed from PG70 to PG76. This may mean that the RCA is not sensitive to a change in the binder type.

Figure 30 shows that switching from limestone aggregate to RCA leads, on average, to a 17% increase in the lost strength after moisture conditioning. The results shown in Figure 30 support the work of Gopalam et al., 2020; Ossa et al., 2016; Paranavithana & Mohajerani, 2006; Zhu et al., 2012 regarding the increased moisture susceptibility of asphalt mixes due to the incorporation of RCA. The authors attribute the increased moisture susceptibility to the high porosity and high water absorption of the RCA. The authors also state that the mortar which keeps the RCA intact is weak and easily breaks under load, which causes poor adhesion.

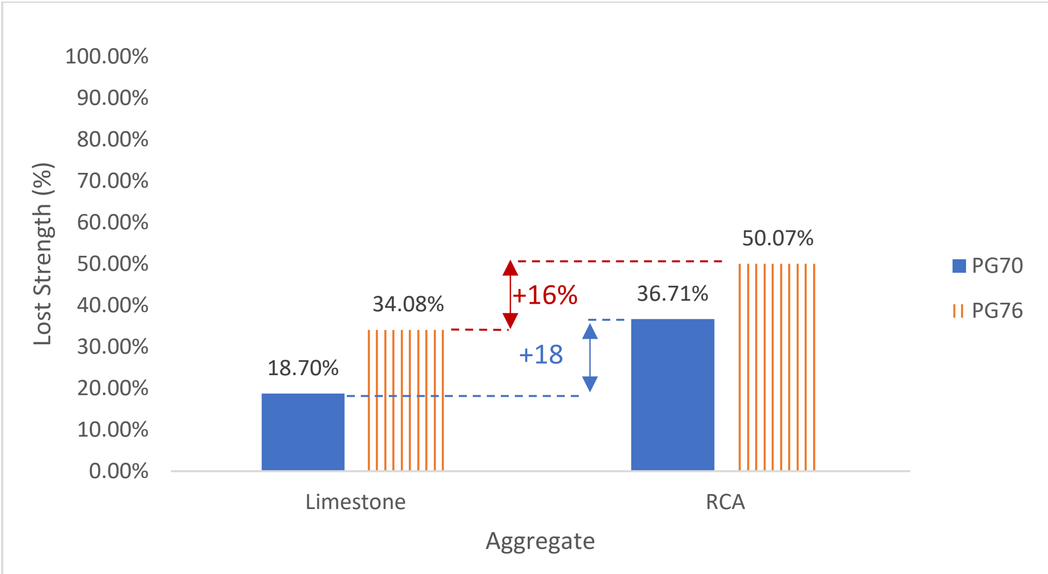


Figure 30 Lost Strength after Moisture Conditioning

5.1.2.2. Strain at Maximum Strength

Figure 31 shows that for the samples fabricated with RCA, the strain at maximum strength is not sensitive to moisture conditioning, which is the opposite to what was observed with the strength values. The reason behind the insensitivity could be due to the poor adhesion between RCA and both binder types, even in the dry condition. On the other hand, the drop in strength could be explained by the binder softening and consequently losing a percentage of its load-carrying ability. The data on RCA shows that for the same strain at maximum strength before and after moisture conditioning, there is a reduction in strength, which could mean that the impact of moisture conditioning manifests itself in the loss of cohesive strength of the binder.

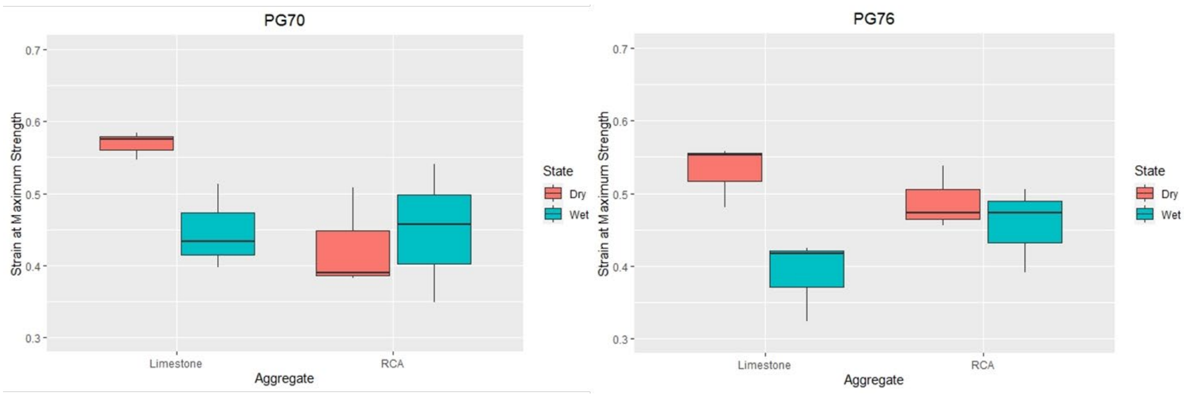


Figure 31 Boxplot Summary for Strain at Maximum Strength Results

5.1.2.3. Mode of Failure

Figure 32 shows the mode of failure for the samples fabricated with RCA and limestone, respectively.

When the samples are fabricated with the PG70 binder, the mode of failure before moisture conditioning is a mix of adhesive and cohesive. After moisture conditioning, the mode of failure becomes mostly adhesive with a small area of cohesive failure and visible binder softening. The areas of cohesive failure are represented in the load-displacement curve such that ductile behavior is exhibited after reaching the maximum strength as shown in Figure 34. When the samples are fabricated with the PG76 binder, the mode of failure in the dry and moisture conditioned states is adhesive, which is expected as the same mode of failure occurred with the limestone samples shown in Figure 32. RCA seems to be highly susceptible to stripping as evident from the mode of failure as well as the strength values.

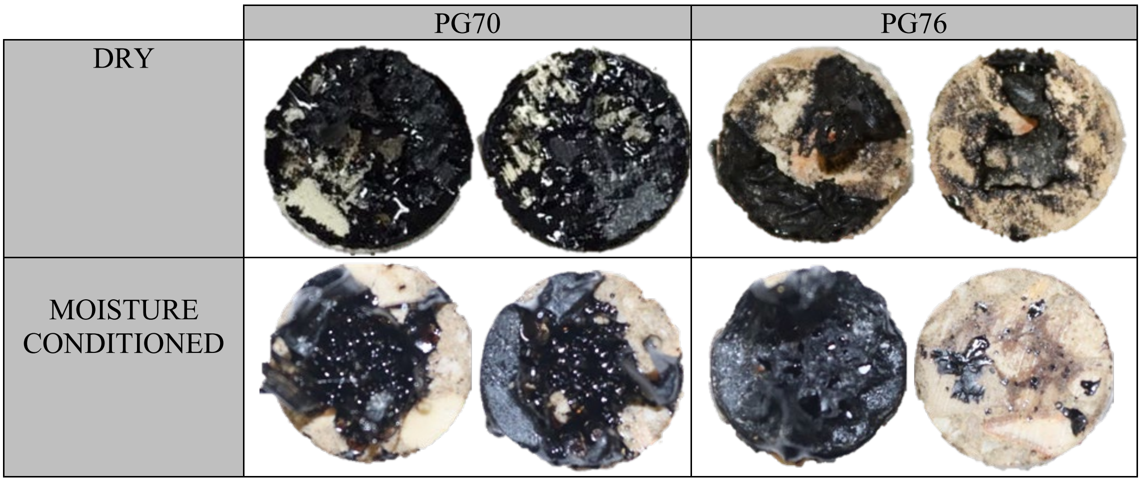


Figure 32 Mode of Failure for Samples Fabricated with RCA

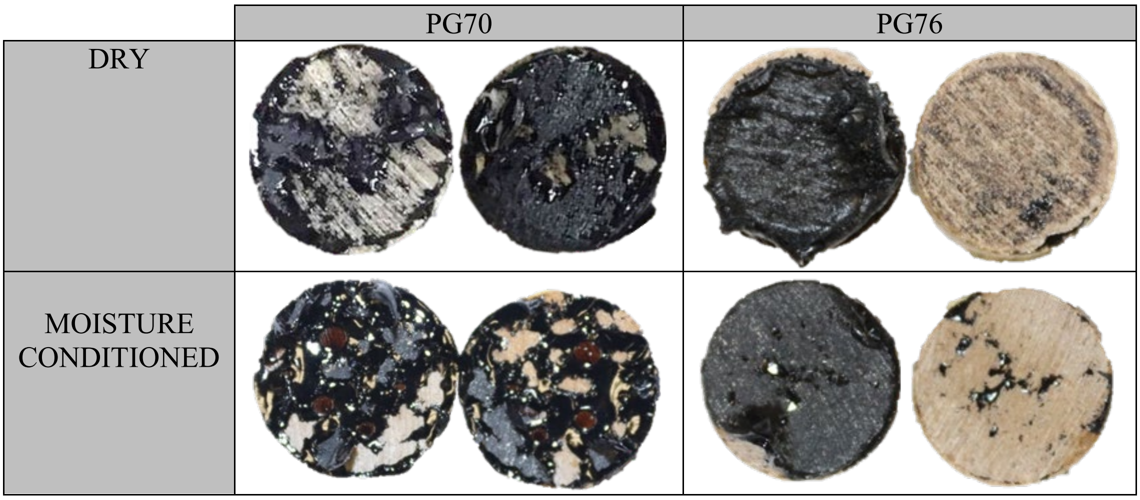


Figure 33 Mode of Failure for Samples Fabricated with Limestone

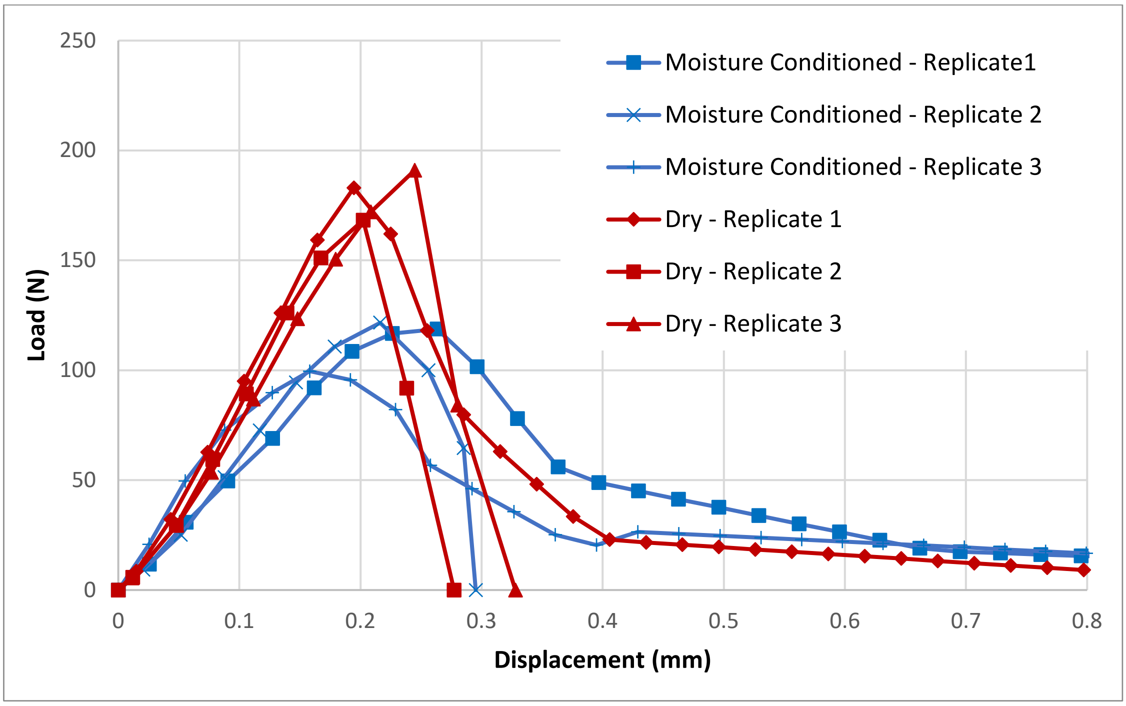


Figure 34 Load vs Displacement for RCA with PG70 Binder

<u>5.1.2.4. ANOVA Models Results</u>

A three-way ANOVA model was constructed for the strength as well as strain at maximum strength data. The results are summarized in Table 9. The model for strength showed that the binder type, aggregate, and moisture state, are significant factors. The model also showed that there is a significant interaction between the aggregate and binder. The interaction is further explored by conducting Tukey's honest significance test and the results confirmed that the strength for the samples fabricated with RCA is not sensitive to a change in binder type.

The model for the strain at maximum strength showed that the binder type is not a significant predictor variable. The most important predictor of strain at maximum strength was the moisture state, which interacted with the aggregate type.

Table 9 ANOVA Results for Strength and Strain at Maximum Strength Models

		P-values	
		Strength Model	Strain at Maximum Strength Model
Predictor Variables	Aggregate	$7.33*10^{-6}$	0.25
	State	$7.16*10^{-6}$	0.0129
	Binder	$7.16*10^{-6}$	0.7862
Interaction terms	Aggregate:State	0.1266	0.02
	Aggregate:Binder	0.0157	0.1051
	State:Binder	0.1638	0.45
	Rate:State:Binder	0.99	0.73

5.2. Phase Two

In Phase 2 of the experimental plan, mineral fillers were added to the asphalt binders to evaluate their influence on moisture resistance. The analysis of the results was split into several groups, each aiming to quantify the effect of adding a mineral filler to an asphalt-aggregate combination.

5.2.1. Group One: Effect of Filler Type on the Interaction between the PG70 Binder and Limestone Aggregate

The experiments included in group 1 are as shown in Table 10. The analysis aims to evaluate the effect of limestone, lime, and RCA fillers on moisture resistance. The fillers are mixed with the PG70 binder and coupled with the limestone aggregate.

Table 10 Experiments Included in Group 1

Combinations				Replicates	
Binder	Aggregate	Filler	Extension Rate	Dry	Wet
PG70	Limestone	Limestone Lime RCA	20 mm/min	3	3

5.2.1.1. Strength

Figure 35 shows that the type of mineral filler used has a significant effect on the measured strength. The samples fabricated with the Limestone mastic displayed a high strength when tested in the dry condition, but they also lost the biggest percentage of that strength after moisture conditioning. When the samples were tested with the lime mastic, they displayed a similar strength in the dry condition to that with the limestone mastic and, on average, exceeded that strength when testing after moisture conditioning. This indicates the ability of lime modification to improve moisture resistance by inhibiting the loss in strength. As for the samples fabricated with the RCA mastic, the strength when testing in the dry condition was significantly lower when compared to the lime and limestone mastic. After moisture conditioning, the variability of the results drastically increased and, on average, strength decreased by 8.2%. It is important to point out that the variability in results of the RCA mastic could cause problems when it comes to its predictability and durability under changing traffic conditions if incorporated into a pavement section in the field. The data on the variation in strength after moisture conditioning is detailed in Figure 36 and show that the lime and RCA mastics perform significantly better than the limestone mastic.

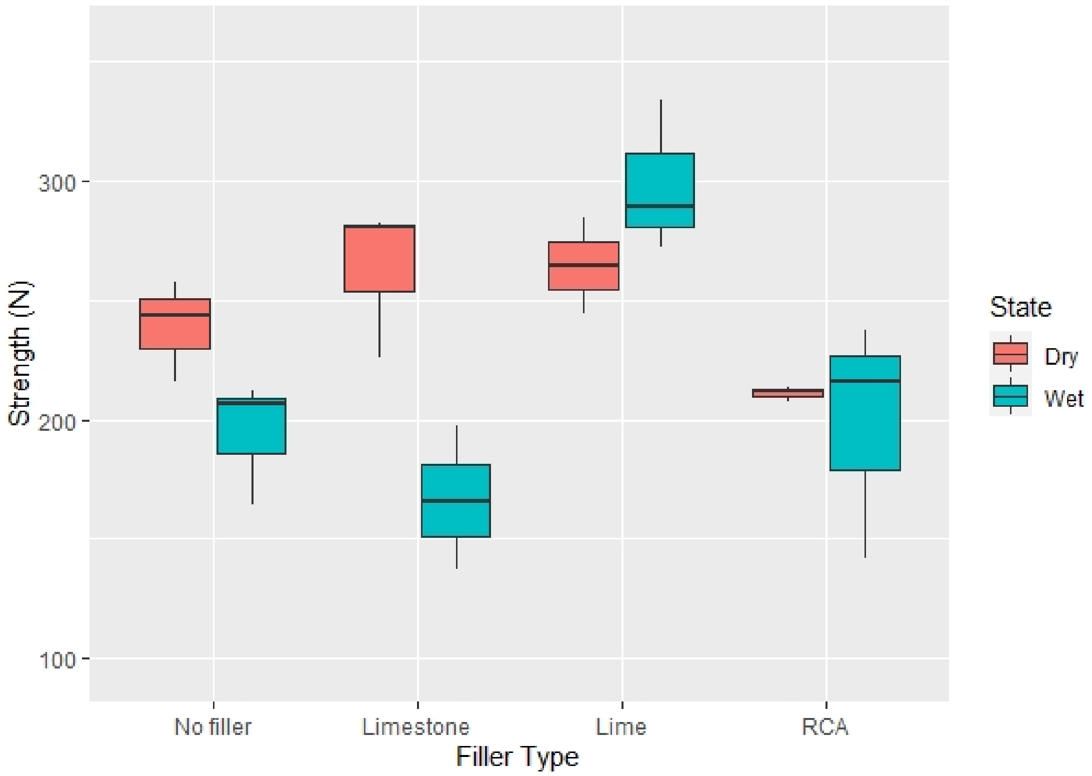


Figure 35 Boxplot Summary of Effect of Filler Type on Strength

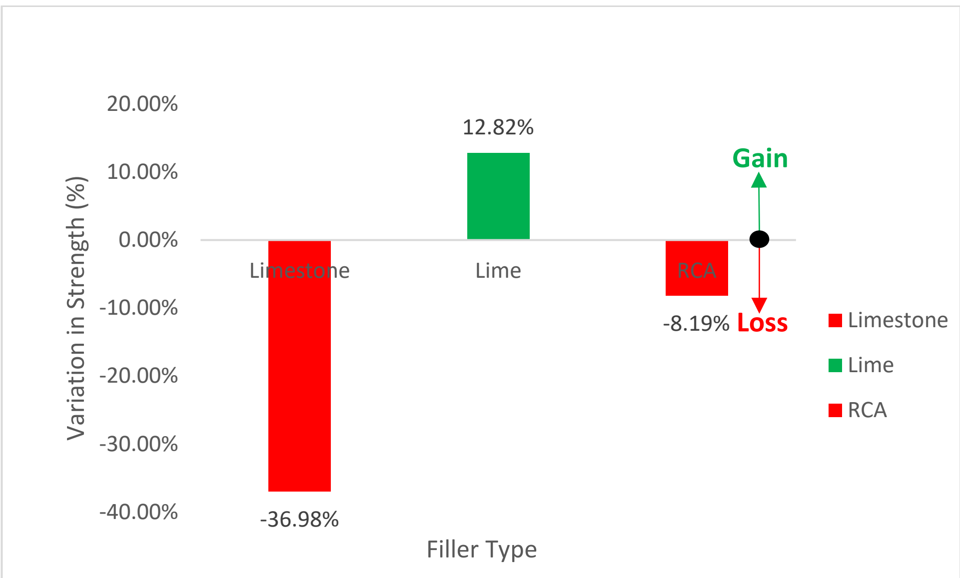


Figure 36 Effect of Filler Type on Variation in Strength after Moisture Conditioning

5.2.1.2. Mode of Failure

Mixing lime filler with the PG70 binder leads to a significant reduction in moisture susceptibility. The mode of failure of the samples fabricated with the lime mastic, shown in Figure 37, further supports the anti-stripping effect of the lime filler as a mixed mode of failure rather than fully adhesive failure. On the other hand, the samples fabricated with limestone and RCA filler fail in adhesion as the mastic film completely strips off the aggregate surface.

It may be concluded that the RCA improves moisture resistance in term of the lost strength after moisture conditioning, but sacrifices toughness due to the decreased overall strength, and reliability due to the large variability after moisture conditioning.

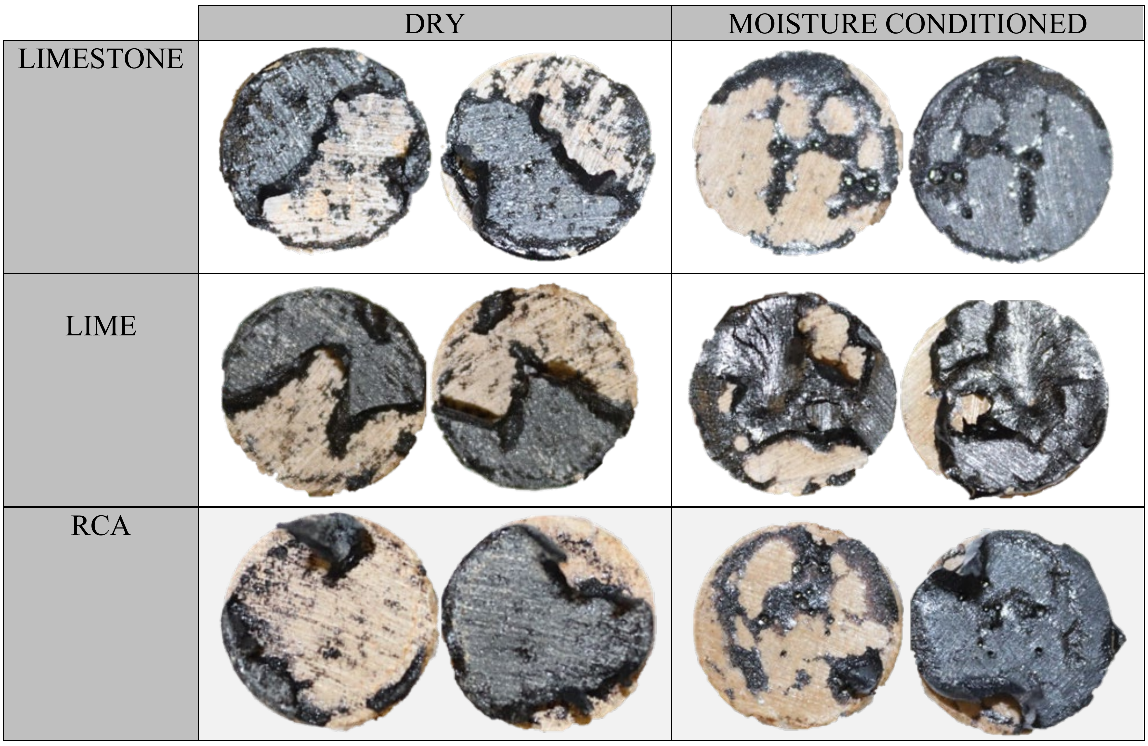


Figure 37 Mode of Failure of Samples with Different Mastics

To target cohesive and adhesive failure separately, the limestone mastic prepared with the PG70 binder and coupled with the limestone aggregate was further tested at 5, 20, and 80 mm/min. Likely due to the increased stiffness of the mastic, the dominant mode of failure was adhesive at all extension rates as shown in Figure 38. When testing the PG70 neat binder at 80 mm/min in phase 1 of the study, a drop in strength was seen and attributed to the weakness of the binder in withstanding the high stresses. When testing the PG70 binder with limestone filler, the strength did not drop at 80 mm/min, but the mode of failure remained partly cohesive. Figure 39 shows the different behavior when testing at different extension rates between the PG70 neat binder and its limestone mastic. Figure 39 also shows that while the limestone mastic led to an increase in strength in the dry condition, the strength when testing after moisture conditioning did not improve. This led to a bigger percentage of strength being lost when testing at the same extension rate for the PG70 limestone mastic compared to the neat binder as shown in Figure 40. A similar observation was made by Al Basiouni Al Masri et al., 2019. The authors used the surface free energy concept to evaluate the moisture susceptibility of asphalt-aggregate system and noted that the moisture susceptibility of the PG70 neat binder is better than that of its limestone mastic. A possible explanation to this observation is that while the addition of limestone filler increases the cohesive strength of the binder by making it stiffer, the limestone filler particles create more space for water infiltration at the asphalt-aggregate interface. The extra space may cause the adhesive bond to deteriorate faster since in Figure 39, the strength after moisture conditioning did not significantly vary between different extension rates.

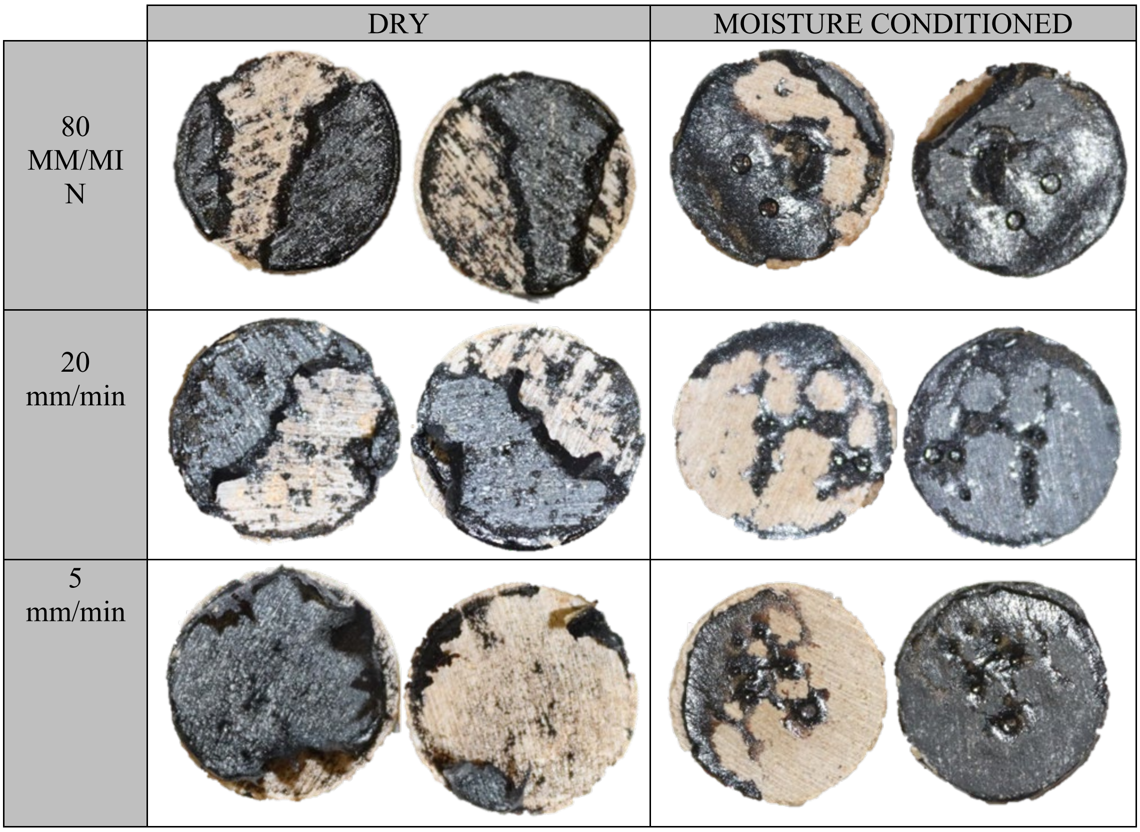


Figure 38 Mode of Failure for Samples with Limestone Mastic and Limestone Aggregate

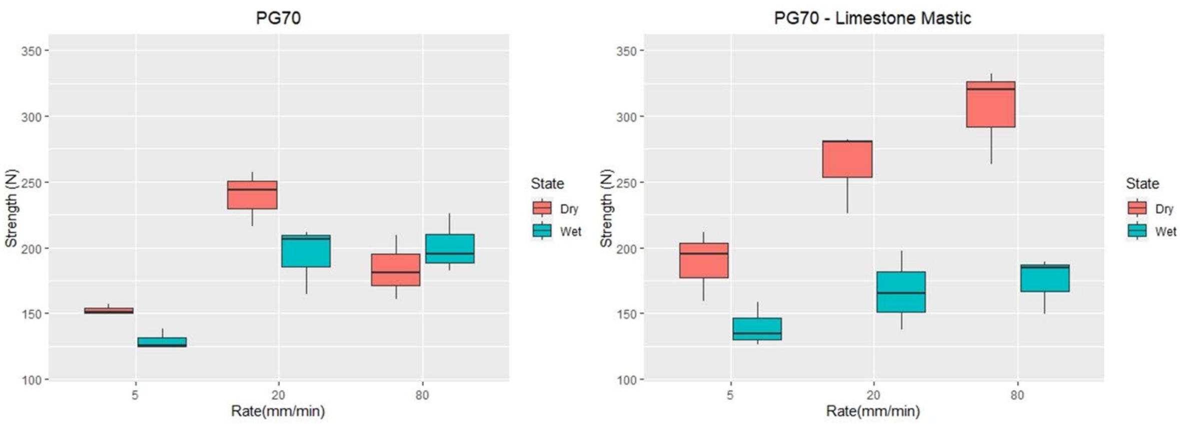


Figure 39 Strength at Different Extension Rates for PG70 Neat Binder and its Limestone Mastic

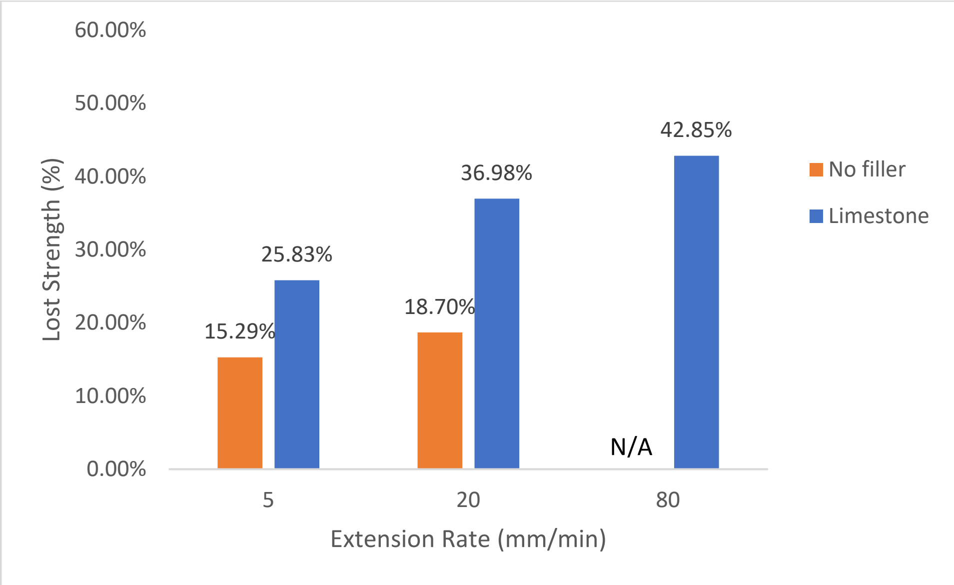


Figure 40 Effect of Moisture Conditioning on Samples with PG70 Neat Binder and its Limestone Mastic

5.2.2. Group Two: Effect of Lime Filler on the Interaction Between the PG76 Binder and Limestone Aggregate

The results of phase 1 of the experimental plan showed that the PG76 binder displayed a stripping potential when coupled with the limestone aggregate. The analysis of the data in group 2 aims to evaluate the effect of lime as a filler on moisture resistance when used with the PG76 binder and limestone aggregate. The experiments included in group 2 are shown in Table 11.

Table 11 Experiments Included in Group 2

Combinations				Replicates	
Binder	Aggregate	Filler	Extension Rate	Dry	Wet
PG76	Limestone	No filler	20 mm/min	3	3
PG76	Limestone	Lime	20 mm/min	3	3

5.2.2.1. Strength

Adding lime to the PG76 binder improved strength when testing in the dry condition and after moisture conditioning as shown in Figure 41. It is interesting that the addition of lime did not limit the loss of strength after moisture conditioning, which was the case with the PG70 binder (Figure 42). As a result, the loss in strength remains the same when comparing the PG76 neat binder to its lime mastic as shown in Figure 43. It seems that the incorporation of lime with the polymer-modified binder improves the adhesive bond with the limestone aggregate but does not limit moisture damage.

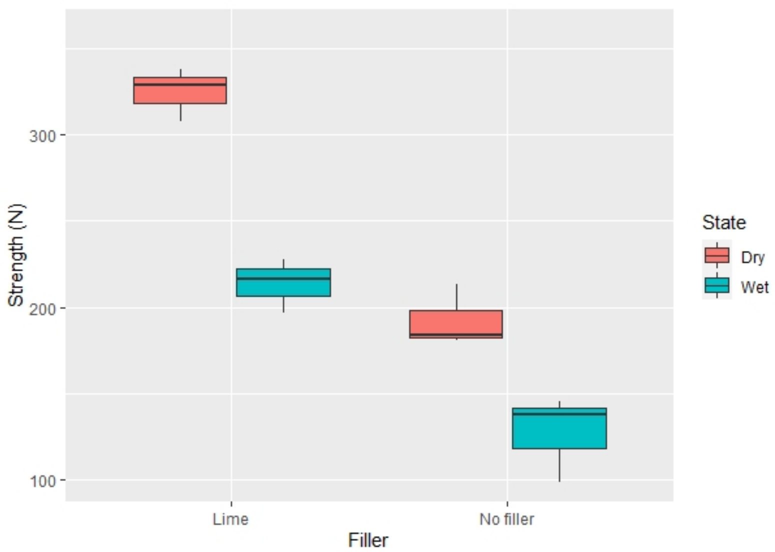


Figure 41 Effect of Adding Lime to the PG76 Binder

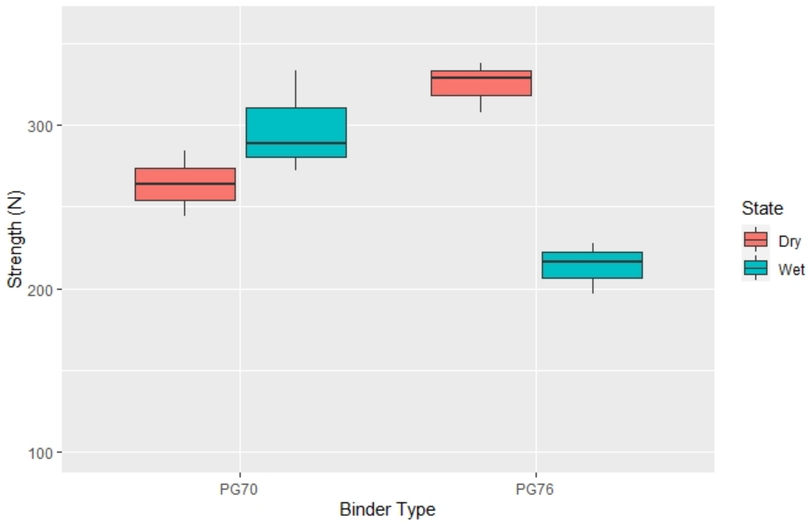


Figure 42 Effect of Lime Mastic on Strength with PG70 and PG76 Binders

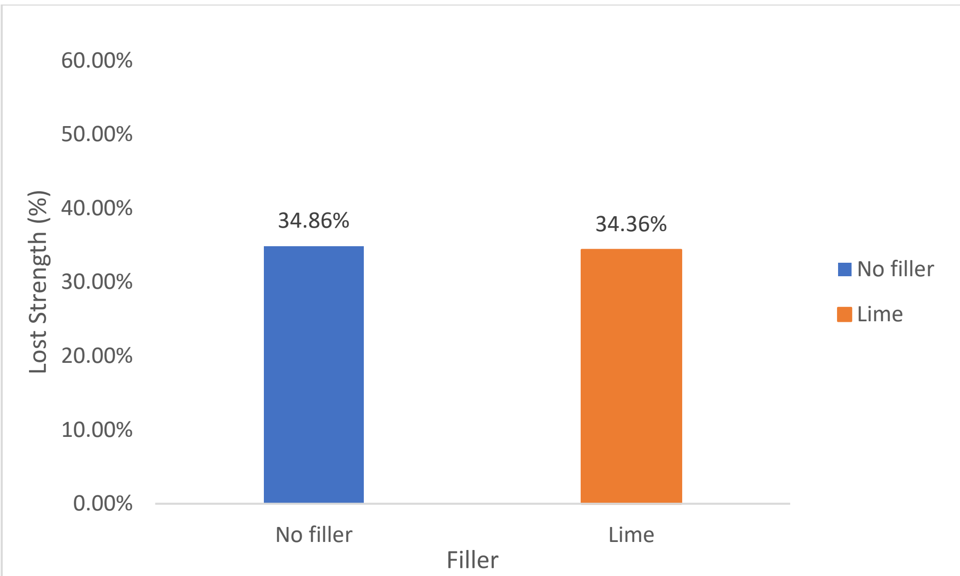


Figure 43 Effect of Lime on Loss of Strength after Moisture Conditioning

<u>5.2.2.2. Strain at Maximum Strength</u>

The improvement in adhesion is apparent when examining the strain at maximum strength in Figure 44. When the PG76 binder was tested without any fillers at different extension rates in phase 1 of the experimental plan, the recorded strain at maximum strength increased in the dry condition, but remained around 0.4 when testing after moisture conditioning. After adding the lime filler to the PG76 binder, the samples displayed an increase in the strain at maximum strength and a minor loss after moisture conditioning. It can be concluded that the lime modification improves adhesion with the limestone aggregate which allows the mastic film to deform rather than detach from the aggregate surface. The increased deformation is shown in Figure 45.

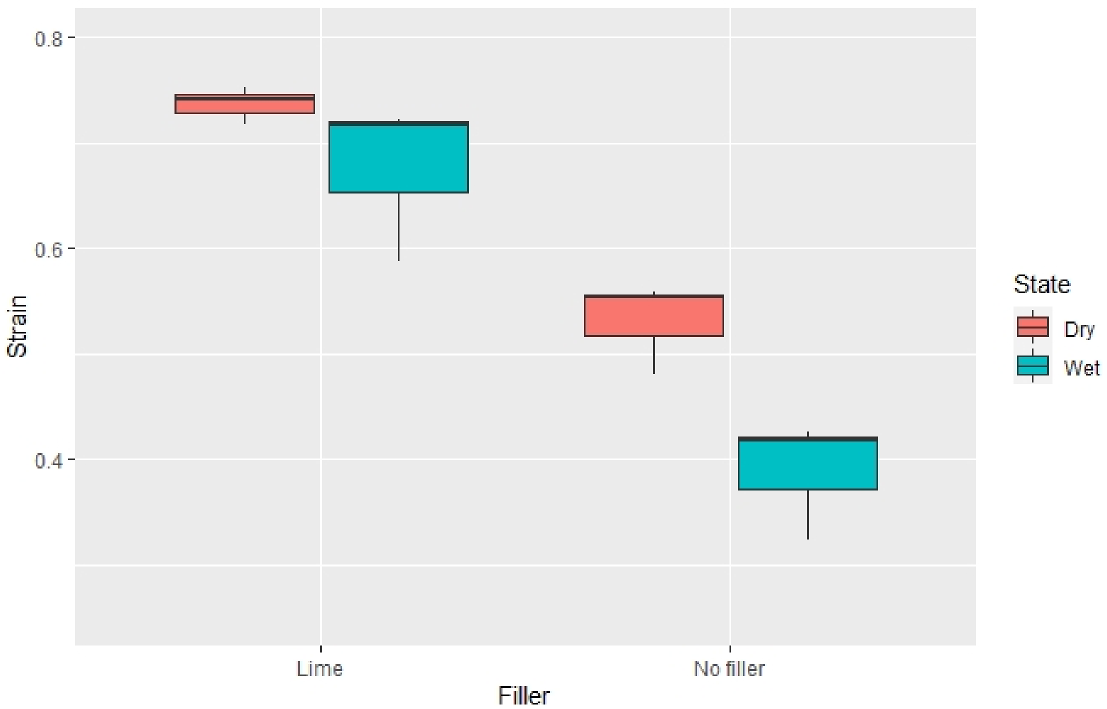


Figure 44 Effect of Lime Modification on Strain at Maximum Strength

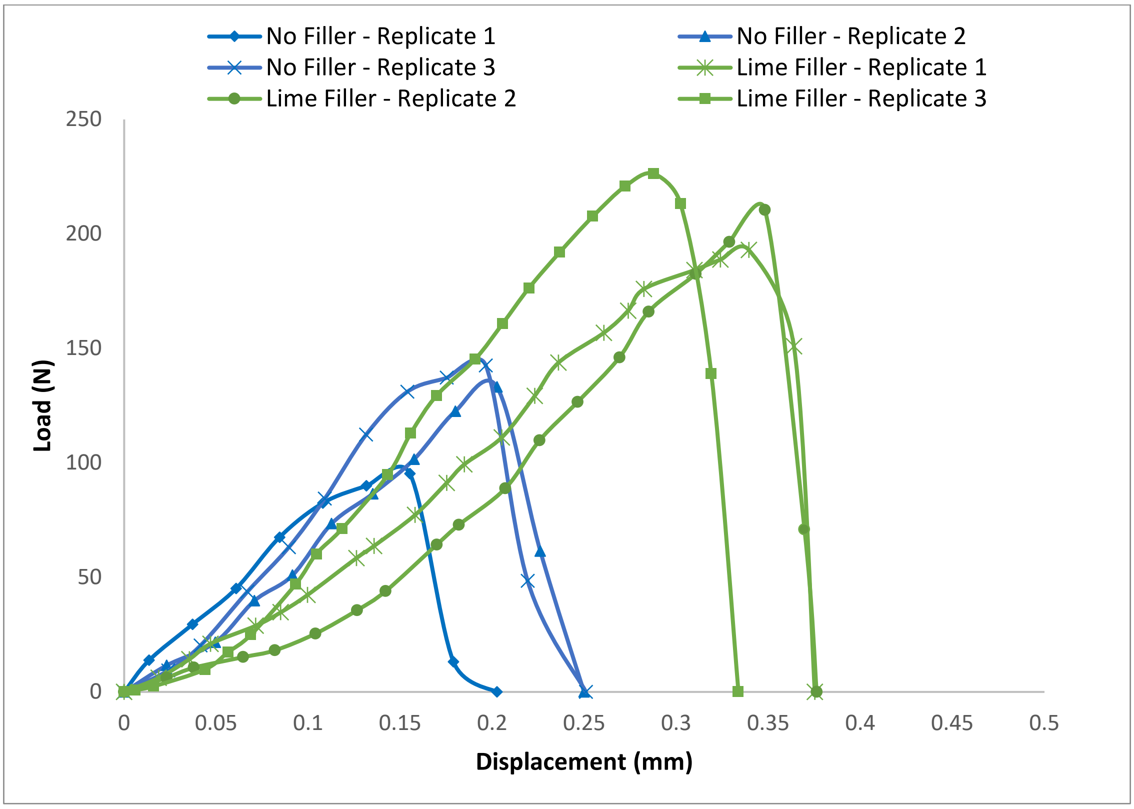


Figure 45 Effect of Lime Modification after Moisture Conditioning

5.2.2.3. Mode of Failure

While the lime modification did improve the strength of the adhesive bond, it did not limit stripping of the mastic. Figure 46 shows that the mode of failure with and without lime modification is adhesive as the binder and mastic film completely strip off the aggregate surface.

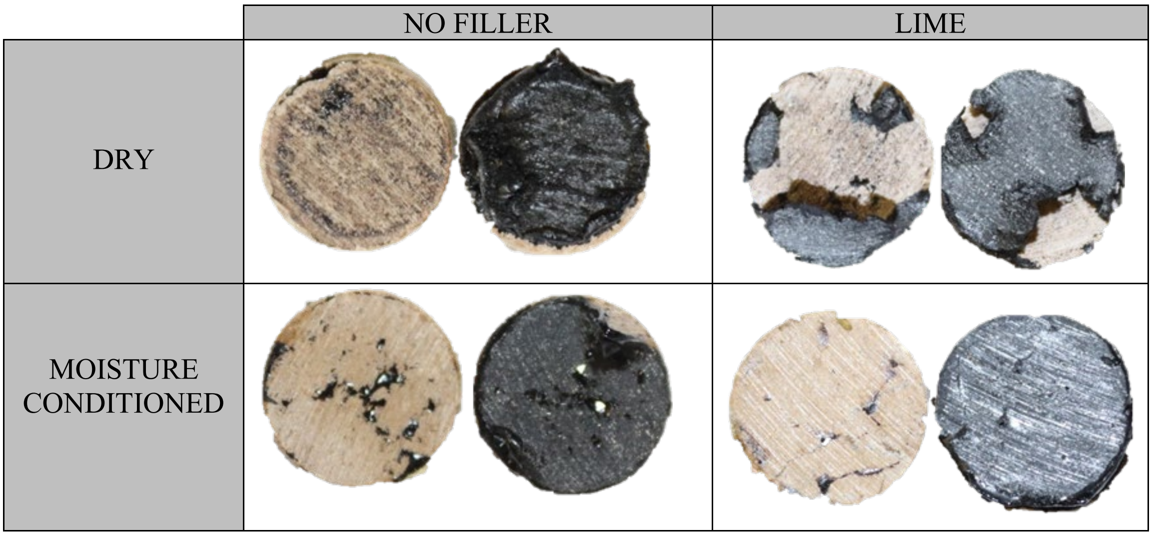


Figure 46 Failure Surfaces When Using the Lime Mastic of the PG76 Binder

5.2.3. Group Three: Effect of Lime Filler on the Interaction between the PG70 Binder and Recycled Concrete Coarse Aggregate

Phase 1 of the experimental plan showed that RCA increased moisture and stripping susceptibility due to its high porosity and moisture absorption. The experiments included in group 3 of phase 2 aim to determine if using hydrated lime mastic decreases the moisture susceptibility of RCA with the PG70 binder.

Examining the strength data in Figure 47, it can be seen that the lime mastic did not have an effect on strength when testing in the dry state. After moisture conditioning, the samples fabricated with the lime mastic exhibited a larger reduction in strength (70%) compared to those with the neat binder (36.7%). Although the lime mastic improved moisture resistance when used with the limestone aggregate, it seems to exacerbate moisture damage when used with the RCA. Figure 48 shows the excessive stripping of the lime mastic with the RCA. It is important to note that the more pronounced adhesive failure when using the lime mastic may be due to the stiffening effect of the addition of lime filler. To verify, that the lime filler exacerbates the stripping potential of RCA with the PG70 binder, it is necessary to compare the performance of lime mastic with limestone mastic.

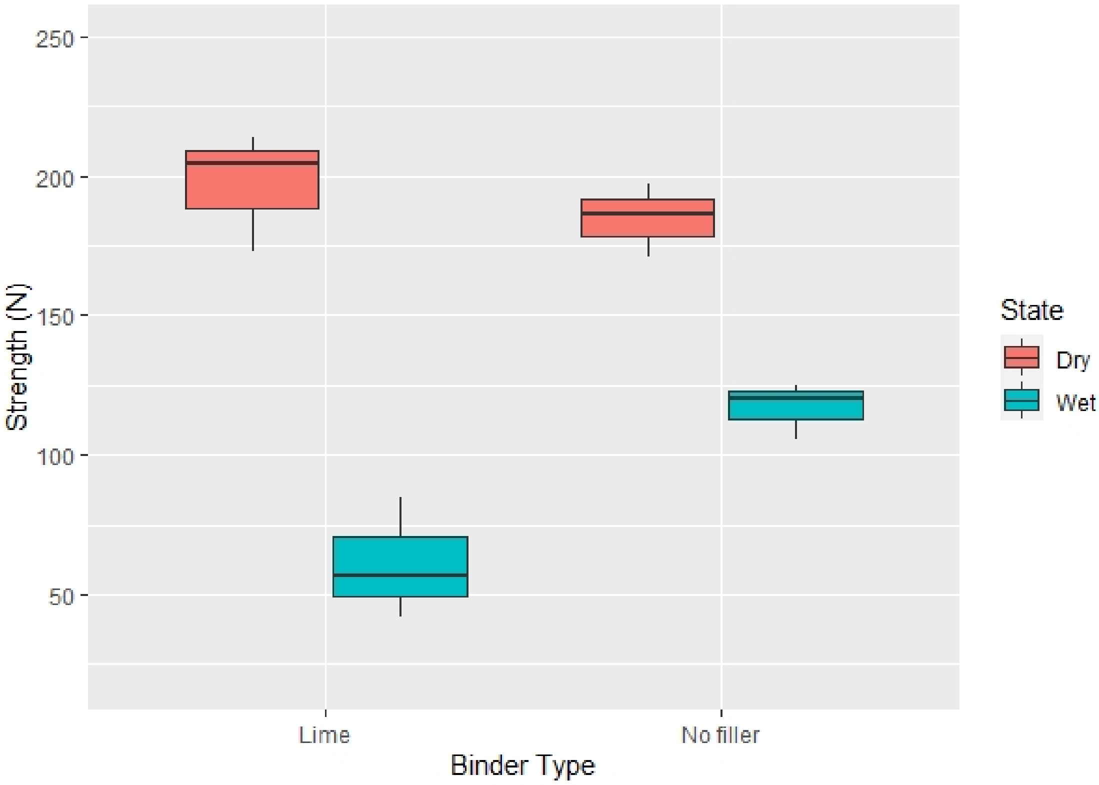


Figure 47 Effect of Lime Modification on Adhesion between PG70 Binder and RCA Aggregate

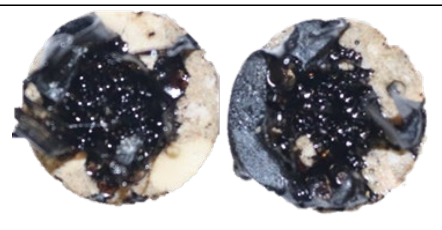
	NO FILLER	LIME
DRY		
MOISTURE CONDITIONED		

Figure 48 Failure Surfaces of RCA Aggregate with PG70 Binder and its Lime Mastic

Having established in group 1 that the incorporation of lime in the asphalt mastic improved the adhesion between the PG70 binder and limestone aggregate, the results discussed in groups 2 and 3 showed that the effectiveness of lime in improving adhesion varies depending on the type of binder and aggregate it is added to. It is well documented in literature that the incorporation of hydrated lime improves the resistance to moisture damage and that it is one of the most widely used anti stripping agents (G D Airey et al., 2008; Khattak & Kyatham, 2008; Nazirizad et al., 2015). The addition of lime to the asphalt mixture improves adhesion in several ways. Lime lowers the surface tension of the bitumen which makes it better able to wet the surface of the aggregate, neutralizes polar molecules on the aggregate surface and in the bitumen which would have formed weak bonds that can easily be displaced by water, and stiffens the binder more than regular filler (Gorkem & Sengoz, 2009; Han et al., 2019; Lesueur et al., 2013). Since lime is a chemically active agent, its performance has also been reported by the aforementioned authors to vary based on the aggregate type as well as the bitumen chemistry and its crude source. Therefore, an inconsistent behavior of lime has been previously documented, but the tests conducted as part of this research effort show that further investigation is required particularly when lime is used with recycled aggregates.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

This research study investigates the effect of different aggregates, binders, and fillers on moisture resistance using an experimental method that is based on the pull-off approach by testing at the binder and mastic scales. The following conclusions are made:

1. The proposed experimental method can evaluate moisture damage for aggregate-binder and aggregate-mastic combinations and can distinguish between different aggregates, binders, and fillers. The method is easy to follow, repeatable, does not require expensive equipment, and allows for easy manipulation of critical parameters such as the extension rate and asphalt film thickness.
2. Evaluating cohesive damaged caused by the action of water may require unique testing conditions such as binder films with high thicknesses, very low extension rates, and metal plates that sandwich the binder film rather than aggregates. The deterioration of the adhesive bond, which was the dominant mode of failure even in the condition which promotes cohesive failure, did not allow to evaluate the damage to the cohesive bond of the binder or mastic.
3. The non-modified binder performs better than the SBS-modified binder in resisting moisture damage but is prone to cohesive failure at high extension rates.

4. Using RCA as coarse aggregate increases the stripping potential and causes, on average, a 17% increase in the lost strength after moisture conditioning when compared to the limestone coarse aggregate.
5. The adopted sample preparation process may not be unsuitable for testing the moisture resistance of RAP. The samples did not conform to the 4:1 representative volume element criterion and exhibited an increase in strength after moisture conditioning due to the blending of the aged and virgin binder over the soaking period.
6. The PG70 neat binder exhibits better moisture resistance than its mastic with limestone filler when tested with the limestone aggregate.
7. Compared to the limestone filler, the use of RCA filler improves moisture resistance in terms of the loss in strength after moisture conditioning. On the other hand, the adhesive bond between the RCA mastic and the limestone aggregate cannot resist high loads and is highly variable. The incorporation of RCA filler in asphalt mixes may be recommended for low volume roads.
8. The effect of lime on moisture resistance is dependent on the type of binder and aggregates used. The incorporation of lime improves the adhesion between the PG70 binder and the limestone aggregate and further exacerbated the stripping potential between the PG70 binder and the RCA aggregate. It may not be recommended to use lime to with coarse recycled aggregates.

CHAPTER 7

FUTURE WORK

Based on the work completed in this study, the following is recommended for future research:

1. Investigate the moisture susceptibility of aged binder samples which better represent the state of the binder in the field.
2. Investigate the moisture susceptibility of binders with different degrees of SBS modification.
3. Further study the moisture resistance of samples fabricated with RCA filler using different aggregates and binders.
4. Conduct surface free energy testing to account for the chemical interaction between mixture components.
5. Correlate binder and mastic level testing with mixture level testing to account for the effect of mix-related factors that could impact moisture resistance.

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