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# Moisture Damage Assessment of Asphalt Mixture using Eatons Reagent

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**Abstract:** *The main purpose of this research work was to create a correlation model called a moisture damage index, that could predict moisture damage probability in both qualitative and quantitative test regimes. To determine the effect on moisture damage of asphalt mixture, chemical-based modifiers and single aggregate sources, such as calcium carbonate, were selected. Eaton's Reagent, i.e., Phosphorous Pentoxide in Methane Sulphonic Acid, is used to make modified bitumen in this research. Eaton's Reagent is made in a laboratory with a 1:10 synthesis formula and a unique technique. Base binders are changed with varied amounts of Phosphorus Methyl Compound (PMC) by weight of optimum binder content i.e 0.25%, 0.50%, 0.75% & 1.00%. To establish a moisture damage index, the dosage of Eaton's Reagent was optimised using both qualitative and quantitative measurements. This reagent interacts strongly with the asphaltene fraction of bitumen rather than the maltenes portion, resulting in a higher service temperature than the base binder. Asphalt mixtures were prepared and tested using performance testing to verify the effect of phosphorous methyl compound on their performance. The boiling water test was simple to administer and took less time, while the rolling bottle test was found to be more reliable than other qualitative tests. Although Marshal stability was easy to perform in quantitative tests, while the Hamburg wheel track test were shown to be more effective in determining moisture susceptibility with a reasonable level of confidence. A testing regime comprises of qualitative as well as quantitative tests to ascertain relative accuracy of each test on moisture damage.*

**Keywords:** *Eaton's Reagent; Asphalt mixtures; Loss of adhesion; moisture damage; limestone*

## I. INTRODUCTION

Moisture damage refers to the loss of strength, stiffness, and durability of asphalt mixtures when exposed to water. Moisture damage affects the performance of asphalt mixtures as a result of cohesion and adhesion loss at the binder and binder-aggregate interfaces, respectively. As a result, damage to the asphalt mixture occurs in both circumstances, resulting in a lower rigidity modulus [1]. Water can reach the bitumen-aggregate interface through diffusion mechanisms across the bitumen film, seep through gaps and fissures in the bitumen film if any exist, or enter directly through the partially coated aggregate surface as soon as it comes into contact with the asphalt mixture [2]. Another effect of water interaction with the asphalt mixture is the softening of the bitumen film without debonding between the aggregate and the bitumen. The loss of stiffness can be reversed by removing the moisture, however there is a problem with applying load when the stiffness is low. When a load is applied under low stiffness circumstances, as previously described, the pavement deteriorates quickly and the mechanism can become irreversible [3]. Moisture can also cause indirect damage to road pavements by hastening the deterioration of existing distresses such as cracking, rutting, and potholes in the presence of moisture.

Cumulative deterioration in different pavement layers causes asphalt pavement breakdown. Moisture damage is one of the most complicated, but it is most typically seen in asphalt pavements owing to stripping. It is the breakdown of aggregate adhesion to the asphalt binder, or cohesion within the asphalt binder, that causes the asphalt mixture to lose stiffness, strength, and durability [4, 5]. Water penetration within the asphalt binder and aggregate contact causes stripping. As a result of water causing molecular diffusion through the asphalt film and onto the aggregate surface, the asphalt aggregate bond is disrupted [6].

Water penetration within the asphalt binder and aggregate contact causes stripping. As a result of water causing molecular diffusion through the asphalt film and onto the aggregate surface, the asphalt aggregate bond is disrupted. Moisture damage is commonly thought to be the cause of asphalt pavement premature breakdown. As a result of moisture damage, the serviceability of asphalt pavement is severely harmed. As a result, rutting, ravelling, and cracking in asphalt pavement are more prone to occur [7]. Increases in asphalt film thickness, compaction, and drainage can lower the risk of moisture damage. According to Solaimanian, the most popular method for preventing moisture damage is to combine anti-stripping modifiers with asphalt binder [8].

Sulphur, methane, and phosphorus compound are chemical modifiers (MPC). Liquid anti-stripping agents are essentially amine-based compounds.

The adhesion qualities of asphalt mixtures can be improved with these compounds [9]. Sulphur has been found to be a useful asphalt binder modifier for improving the basic physical and performance qualities of asphalt mixtures. Sulphur in asphalt mixture performance is a cost-effective way to reduce asphalt binder use and, as a result, greenhouse gas emissions [10]. MPC asphalt cement powder, also known as 'MAC Powder,' is a multi-reagent asphalt binder that is made by mixing MPC asphalt cement powder with conventional asphalt binder [11]. It has much better adhesion than neat asphalt binder [12].

Aggregate surface texture, porosity, cleanliness, aggregate surface charge and energy, aggregate mineralogy, aggregate adsorption sites and polarity, asphalt binder chemistry, and the interaction between asphalt binder and aggregate are all factors that affect moisture susceptibility, according to many researchers [13]. It is critical to include aggregate and asphalt binder with varied polarities in the study of polar interactions on adhesion failure or debonding process. For aggregates, this is accomplished by selecting material from various sources and compositions for the preparation of the asphalt mixture, whereas for asphalt binder, it is accomplished by modifying the material [14].

Carboxylic acids are found in asphalt binder. These are polar and stick to dry aggregate very well. Surfactants in asphalt binder are sodium and potassium salts of carboxylic acids that lose their binding when water infiltrates. In hot mix asphalt (HMA) pavements, stripping can be caused by a variety of mechanisms, including spontaneous emulsification, detachment, displacement, hydraulic scouring, and pore pressure [15]. Furthermore, the key transit modes of stripping are permeability (infiltration from the surface), vapor diffusion, and capillary rise [16].

## II. PROBLEM STATEMENT

Moisture damage has a significant impact on the performance of asphalt pavements. Moisture has major effects on Hot Mix Asphalt (HMA) over time. Moisture damage in asphalt can lead to deterioration of asphalt pavements in the form of cracking, potholes, and rutting that effect the ease and safety of travel along roads paved with HMA. Currently, there are many laboratory test methods i.e., tests on loose mixtures (qualitative tests), tests on compacted mixture (quantitative tests), pull off tension test, Fourier transform infra-red and surface free energy methods have been used to access moisture damage. From the literature review, it is observed that asphalt mixture performance is mainly dependent on the aggregate or binder properties. This research study tends to correlate the qualitative test results with quantitative test results using Eaton's Reagent and aggregate minerals to access moisture damage resistance of asphalt mixtures.

## III. OBJECTIVES OF RESEARCH STUDY

The overall aim of the study is to evaluate the effect of Eaton's Reagent on moisture damage assessment of asphalt mixtures. The following is the list of objectives to achieve the specific aim.

- 1) To study the effect of aggregate on adhesion properties of asphalt mixtures.
- 2) To analyze and compare the tendency of moisture damage of different testing methods.
- 3) To develop moisture damage index between qualitative and quantitative test regime results in accessing moisture damage.

## IV. MATERIALS AND ASPHALT MIX DESIGN

To achieve the study's goals, a three-phase research study was created. Material selection was completed in the initial phase. We selected single aggregate quarries, asphalt binder, and various types of asphalt binder modifiers (dosage of 0.25%, 0.50%, 0.75% & 1.00% by weight of optimum binder content). The petrography of rock sources was used to choose aggregate quarries. In Pakistan, aggregate quarries, such as Margalla, which includes calcium carbonate, are readily available. Specimens, i.e., asphalt mixture design, were prepared in the second phase. Both qualitative and quantitative testing were carried out on the specimens. Both qualitative and quantitative moisture sensitivity tests were performed in the laboratory during the last phase of the experimental study. The static water immersion test, total water immersion test, boiling water test, and rolling bottle test are all examples of qualitative tests. The Marshal immersion test and Hamburg wheel track test are all quantitative tests. Finally, all of the results from laboratory tests were statistically analysed. In the experimental programme, the results of both qualitative and quantitative tests were compared. Scope of research study has been given in Fig 1.

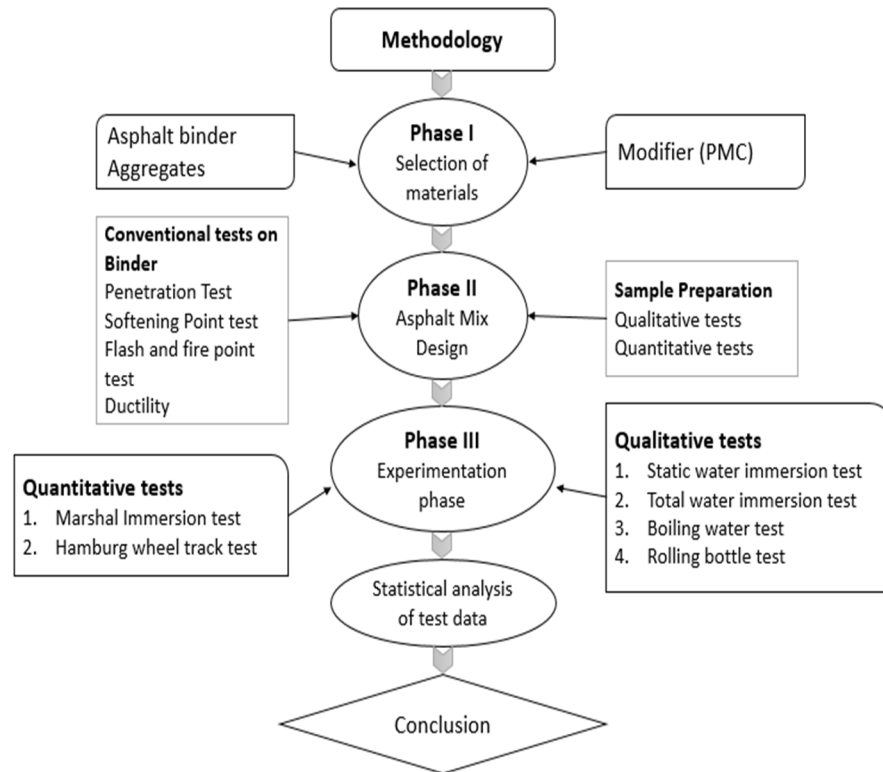


Fig 1: Scope of research study

### A. Aggregates

The petrography of rock sources was used to choose aggregate quarries. The amount of silica in a rock determines whether it is acidic or basic (SiO<sub>2</sub>). Some silica-rich rock sources exhibit acidic behavior and contain minerals such as quartz, biotite, and feldspar. The basic nature of the rock source has a low concentration of silica and minerals such as olivine and pyroxene. To determine the influence of minerals on moisture damage, the aggregate quarries locally available in Pakistan, namely Margallah, which contains calcium carbonate, were chosen to prepare asphalt mixture. The aggregate quarries' locations are depicted in Fig 2. To prepare specimens for quantitative analyses, NHA-B gradation was utilized see Fig 3.



Fig 2: Pakistan map showing aggregate quarries location

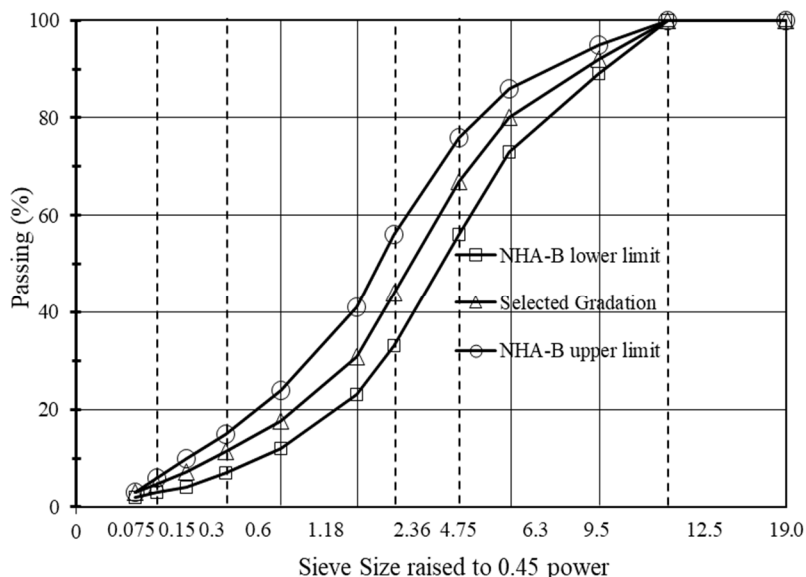


Fig 3: Gradation Curve of Aggregates

It may be noted from Fig 3 well graded aggregate particles has been used to prepare the specimens for quantitative tests. The particle size distribution of aggregate such that the smaller particles should fill the voids between larger particles. So, coarse and fine aggregates be well graded.

Percentage of constituent's elements of the aggregate quarries have been given in Fig 4. Petrography results of all these selected aggregate quarries were taken from Pavron Consultant Islamabad Pvt. Ltd. Petrographic analysis of the selected aggregate quarries was performed according to ASTM C295. The samples were tested under low as well as high magnification

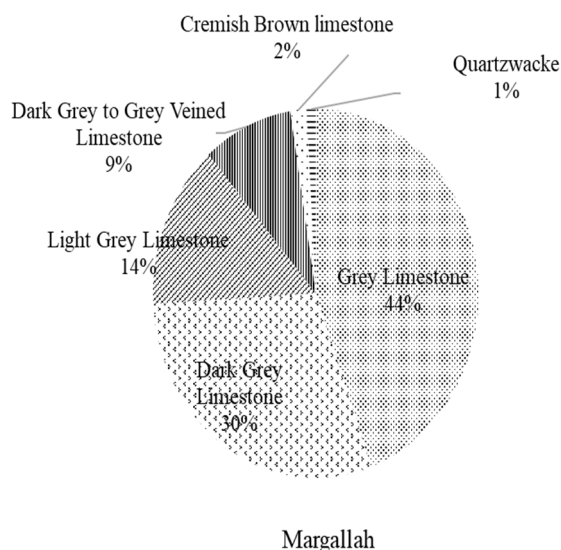


Fig 4: Percentage Elemental Composition of Aggregate Quarries

Margalla aggregate quarries have a nearly identical elemental composition, i.e., maximum limestone, as shown in Fig 4. Calcium carbonate are basic in nature aggregate source.

In the laboratory, various experiments were carried out to determine the physical and mechanical properties of aggregate. TABLE 1 summarizes the physical and mechanical properties of aggregates.

TABLE 1: Physical Properties of Margalla Aggregate

Test Title	Standard	Margalla	Limits by NHA
Type of Rock	----	calcium carbonate	-----
Fractured particles (%)	ASTM D 5821	100	95 % (min)
Flakiness Index (%)	BS 812.108	4.75	11 % (max)
Elongation index (%)	BS 812.109	6.5	10 % (max)
Los Angeles abrasion (%)	ASTM C 131	24	30 % (max)
Water absorption (%)	ASTM C 127	0.95	2 % (max)
Sand equivalent	ASTM D 2419	75	50 % (min)
Soundness	ASTM C 88	4.7	12 % (max)

It may be noted from TABLE 1 that fractured particles of aggregate source are 100% with less than 15% of flakiness and elongation index. The aggregate source has loss angles abrasion value less than 30%.

**B. Asphalt Binder**

Asphalt binder used in this research study was taken from Attock Oil Refinery (ARL) Pakistan having 60/70 pen grade. This asphalt binder source has been primarily used for flexible pavements in Pakistan. The four generic functions of the chosen asphalt binder are based on molecular size and chemical reactivity that is Saturates, aromatics, resins, and asphaltenes. Aromatics account for 40-65% of the total volume of asphalt binder. As a result, asphalt binder is nonpolar and acidic in nature. Laboratory tests results of asphalt binder were within the specified limit.

- 1) *Chemical Based Modifiers:* Chemical based modifiers methane phosphorus compound.
- 2) *Methane Phosphorus Compound:* Methane Phosphorus Compound is significant because when added to bitumen, it exhibits a chemical reaction comparable to that of polyphosphoric acid. Eaton's reagent (1:10 phosphorus pentoxide in methane sulfonic acid) is a low-cost, commercially available material developed by Philip E. Eaton in 1973 and proven to be a good alternative to poly phosphoric acid, overcoming many of the disadvantages of previous catalysts. Chemical-based modifiers were purchased from Musa G Scientific store in Peshawar.
- 3) *Samples Preparation for Testing:* Because PMC has never been used for asphalt modification before, the best bitumen content was determined by trial and error. As a result, trial dosages of Base binder + 0.25%, 0.5%, 0.75%, and 1.00% were used to examine the effect on asphalt binder. Unlike manual mixing, a High Shear Homogenizer was used to prepare bitumen samples as it ensures thorough mixing of modifier into Bitumen by using principles of fluid friction, wear, crushing & Shear. After heating the binder to 150 °C the mix is subjected to a shearing speed of 900 rpm using a shear mixer, hot plate was used to maintain the temperature of bitumen. This procedure should provide a homogeneous modified binder. To achieve a homogeneous morphology, the sample is stored in a separate closed container in a dark environment after shearing. The samples were given names according on the amount of PMC added to the binder, for example, 0.25% PMC depicts a sample that has been modified by 0.25 % PMC shown in TABLE 2. For all percentages, at least three replicates were created to assess the departure from the mean.



TABLE 2: Samples Prepared for Testing




Sr. No.	Sample	Penetration (0.1mm)	Softening (°C)	Softening (°C)
1	Base binder	63	49	101
2	Base binder + 0.25% PMC	55	53	88
3	Base binder + 0.50% PMC	49	56	79
4	Base binder + 0.75% PMC	44	58	76
5	Base binder + 1.00% PMC	42	59	71

C. Testing program

Asphalt technologists have carried out many attempts to develop laboratory tests to distinguish between good and poor performing asphalt mixes regarding moisture damage. The experiments were divided into two groups in order to determine the moisture damage resistance of an asphalt mixture. First one consists of qualitative testing on loose coated asphalt mixture while second one was quantitative testing on compacted asphalt mixtures. In this research investigation, both qualitative and quantitative testing were used to determine moisture damage assessment. Qualitative testing included static water immersion test (SWIT), total water immersion test (TWIT), boiling water test (BWT) and rolling bottle test (RBT) while in quantitative tests were consists of marshal stability test (ASTM D1559) and Hamburg wheel track test (AASHTO T324). Moisture sensitivity was assessed using both qualitative and quantitative testing. TABLE 3 contains a list of the tests. Two separate test regimes were utilized to investigate moisture sensitivity of asphalt mixtures, as shown in TABLE 3. The static water immersion test, total water immersion test, boiling water test, and rolling bottle test were all performed on the loose coated asphalt mixture. The Marshal immersion test and the Hamburg wheel track test are all performed on compacted asphalt mixtures. In each test, three replicas of each specimen were created. However, in marshal stability test six replicates was prepared of each specimen. Three specimens were prepared for conditioning in water while other three were for without conditioning.

TABLE 3: Laboratory tests used in experimental work

Sr. No	Test Name (Standard)	Test Conditions	Pictorial View
01	Static Water Immersion Test (ASTM D1664)	Temperature 25 <sup>0</sup> C Immersing time =16-18 h 6.3-9.5mm loose coated aggregate sample	
02	Total Water Immersion Test (ASTM D1664)	Temperature 40 <sup>0</sup> C Immersing time = 3 h 6.3 to 9.5 mm loose coated aggregate sample	
03	Boiling Water Test (ASTM D3625)	Temperature 100 <sup>0</sup> C Immersing time =10 min 6.3-9.5 mm loose coated aggregate sample	

Sr. No	Test Name (Standard)	Test Conditions	Pictorial View
04	Rolling Bottle Test (BS EN 12697)	Temperature 25 _____ C Rotations per minute = 60 Rotation time = 6 h, 24 h, 48 h, 72 h, 6.3 to 9.5 mm loose coated aggregate sample	
05	Marshall Immersion Test (ASTM D1559)	Conditioning time and temperature=24h,60C, Specimen Height =63mm Specimen Diameter = 100mm	
06	Hamburg Wheel-track test (AASHTO T324)	Wheel load =705.0 + 4.5 N Pass rate = of 52±2/minute Temperature 50 _____ C wheel passes = 20000, Specimen Height = 63 mm Specimen Diameter = 150 mm	

### V. RESULTS AND DISCUSSIONS

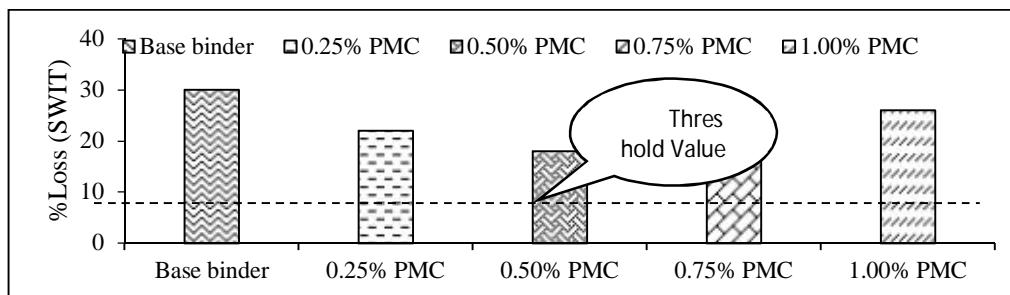
In first phase qualitative tests has been analyzed while second one consists of quantitative test results discussion.

#### A. Basic mechanism (qualitative and quantitative test regime)

To determine the moisture sensitivity of asphalt mixtures, there are two primary test methods. The first is visual observation, which is also known as qualitative determination (tests on loose coated asphalt mixture). This test method takes less time and costs less money. However, qualitative testing does not reproduce mixture qualities, pore water pressure, or driving circumstances in order to determine asphalt mixture moisture sensitivity. Quantitative determination is the second (tests on compacted asphalt mixture). These tests are carried out on asphalt specimens that have been compacted (either in the lab or in the field). This kind of testing takes into account not only the physical and mechanical features of the asphalt mixture, but also the impacts of pore pressure and traffic.

#### B. Qualitative (tests on loose coated asphalt mixture)

All of the tests on the loose coated asphalt mixture, such as the static water immersion test, total water immersion test, boiling water test, and rolling bottle test, were carried out in the laboratory and followed strict guidelines. TABLE 3 summarizes the necessary details of these tests. Fig 5 depicts the results of these testing. In qualitative evaluations, STWIT and TWIT provide better active antistripping against base binder +0.50% of PMC asphalt binder modifiers with calcium carbonate aggregate than other asphalt binder modifiers, as shown in Fig 5. RBT and BWT, on the other hand, exhibit greater adhesion with the basic binder +0.75 % PMC, as demonstrated in Fig 5. Both SWIT and TWIT can be used to determine moisture sensitivity. Although the boiling water test takes less time than the other qualitative tests, the rolling bottle test is superior because it provides better results for determining moisture sensitivity.





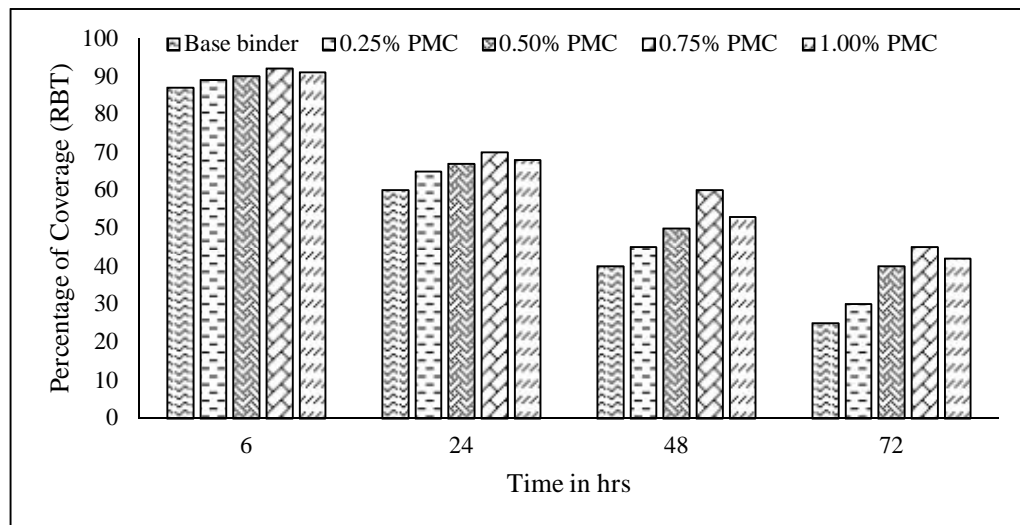
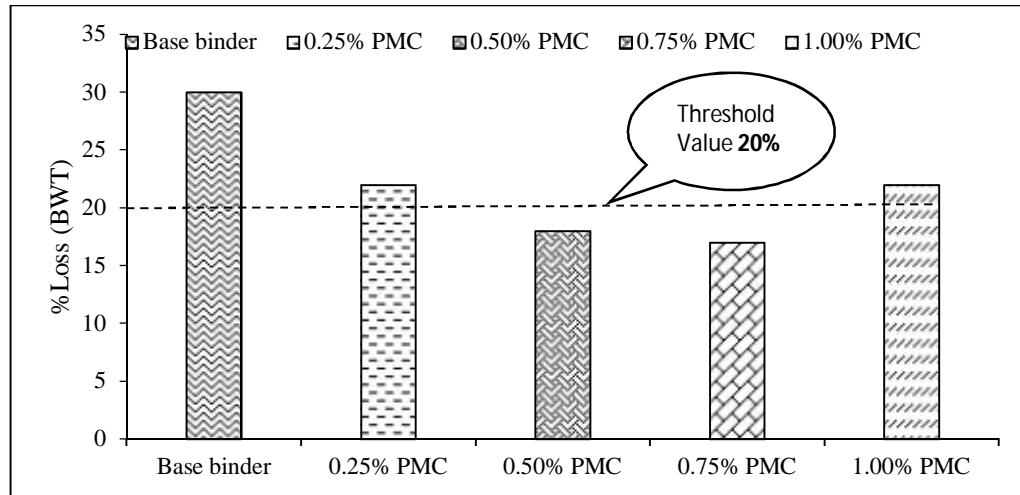
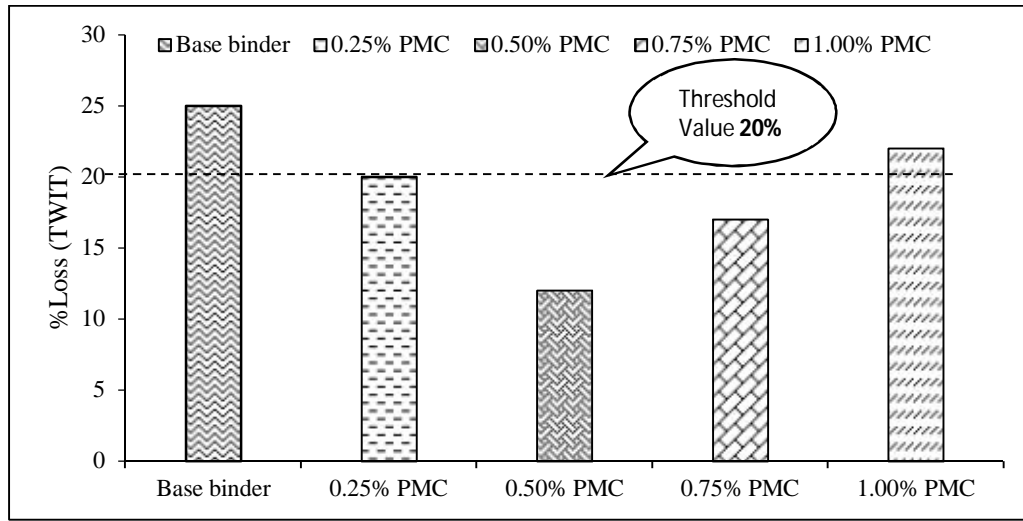


Fig 5: Qualitative (tests on loose coated asphalt mixture)

1) **Marshall Stability Test:** The test was carried out according to ASTM D1559. Before testing, every mould was maintained in a water bath at 60°C for 30-40 minutes. Prior to testing, specimens were conditioned in a water bath at 60°C for 24 hours to prevent loss of stability. Fig 6 shows the percentage loss of Marshal stability.

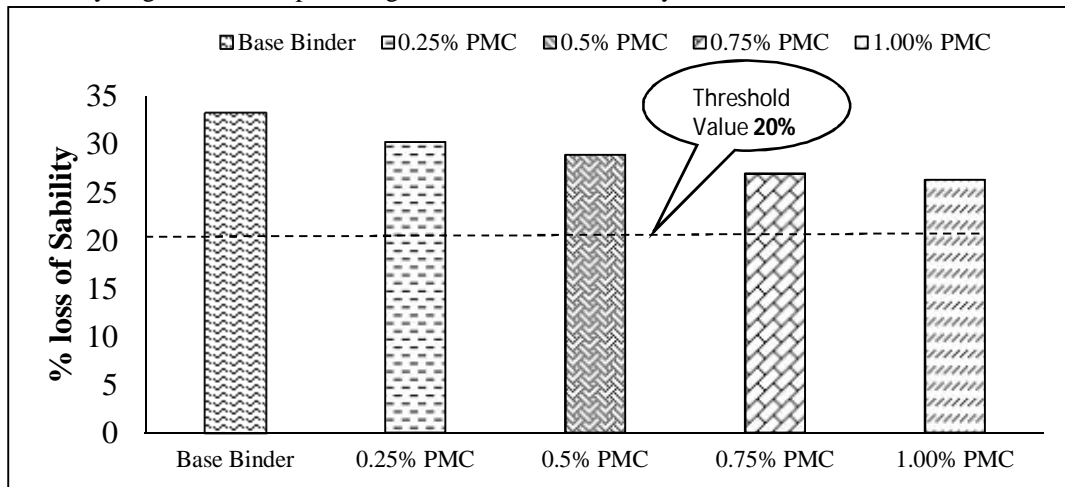


Fig 6: Percent loss of marshal stability against base binder + different percentages of PMC.

As shown in Fig 6, the asphalt mixture includes of a base binder plus various percentages of PMC modifiers. In compared to various percentages of PMC modifiers, Base binder + 0.75% PMC is the most stable against moisture damage.

2) **Hamburg Wheel-track Test:** Hamburg Wheel Track test (HWTT) was used to determine the premature failure susceptibility i.e. moisture damage of asphalt mixture. A wheel having load of 705.0±4.5 N passes at the rate of 52±2 pass per/minute. Results are shown in below Fig 7 with different modified asphalt mixtures.

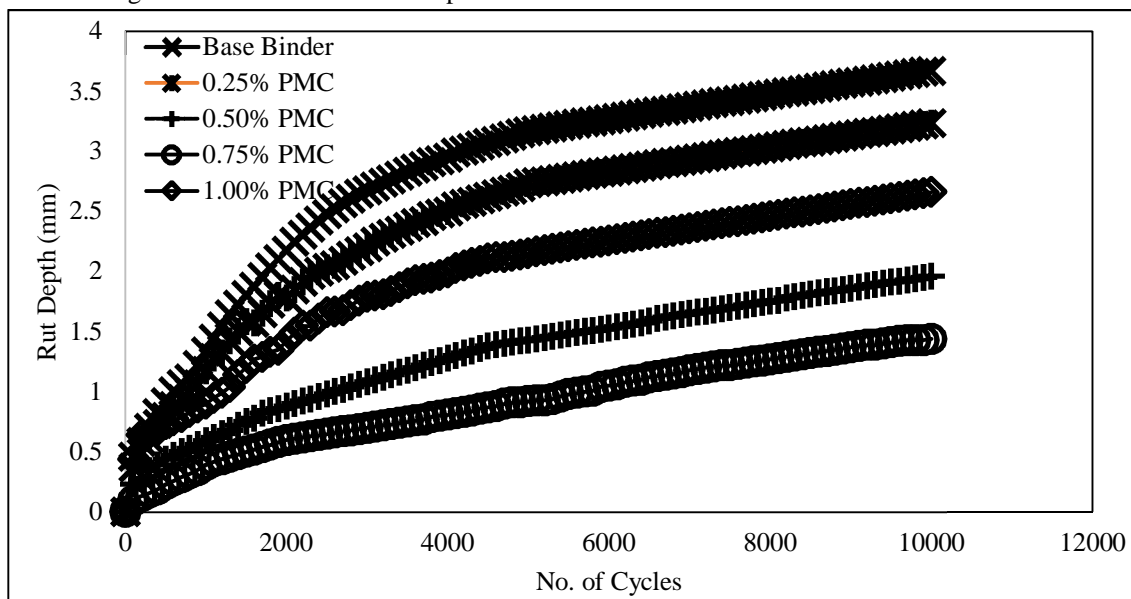


Fig 7: Rut depth of aggregates containing calcium carbonate with base binder + different percentage of PMC

Fig 7 shows Chemical-based modifiers with different percentages of PMC with base binder. Fig 7 shown that Base binder + 0.75% PMC shown maximum stability against moisture susceptibility, as the offer low rut depth against Base binder + 0.75% PMC as compared to others percentages of PMC with base binder. Low rut depth means that at that Base binder + 0.75% PMC have greater stability against moisture. However, Base binder + 0.75% PMC with calcium carbonate shows relatively better adhesion. The aggregate sources i.e., calcium carbonate, are hydrophobic in nature. That is why they are less sensitive to water.

A. Statistical analysis

1) Correlation Between Qualitative and Quantitative test Results: A liner equation of asphalt binder has been developed to access correlation between qualitative and quantitative test results. Results are given in Fig & Fig .

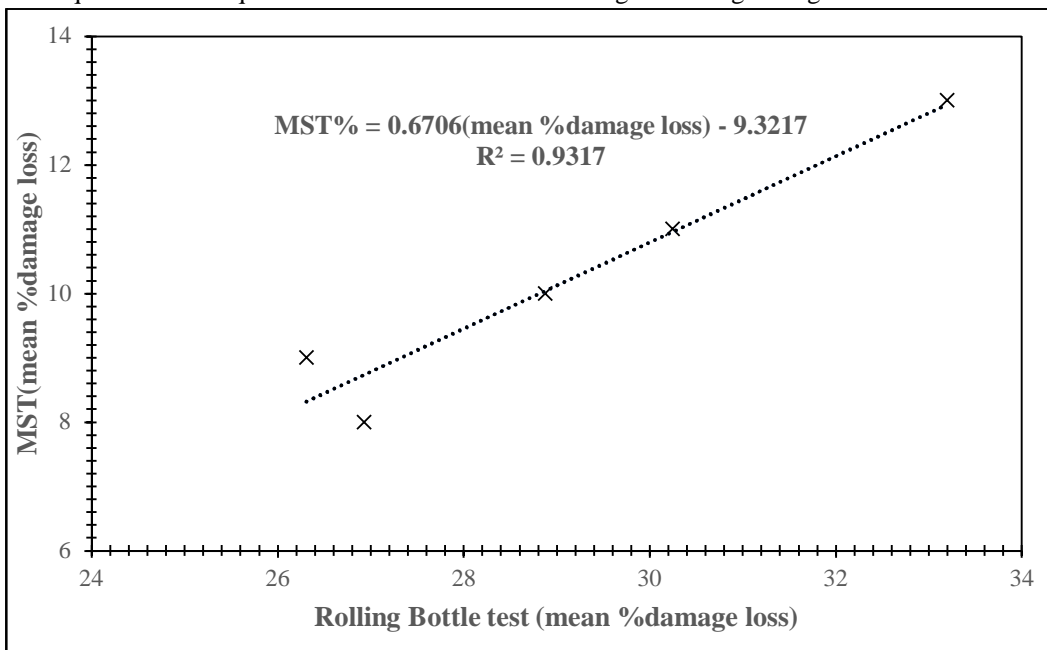


Fig 8: Correlation between rolling bottle test and MST (%)

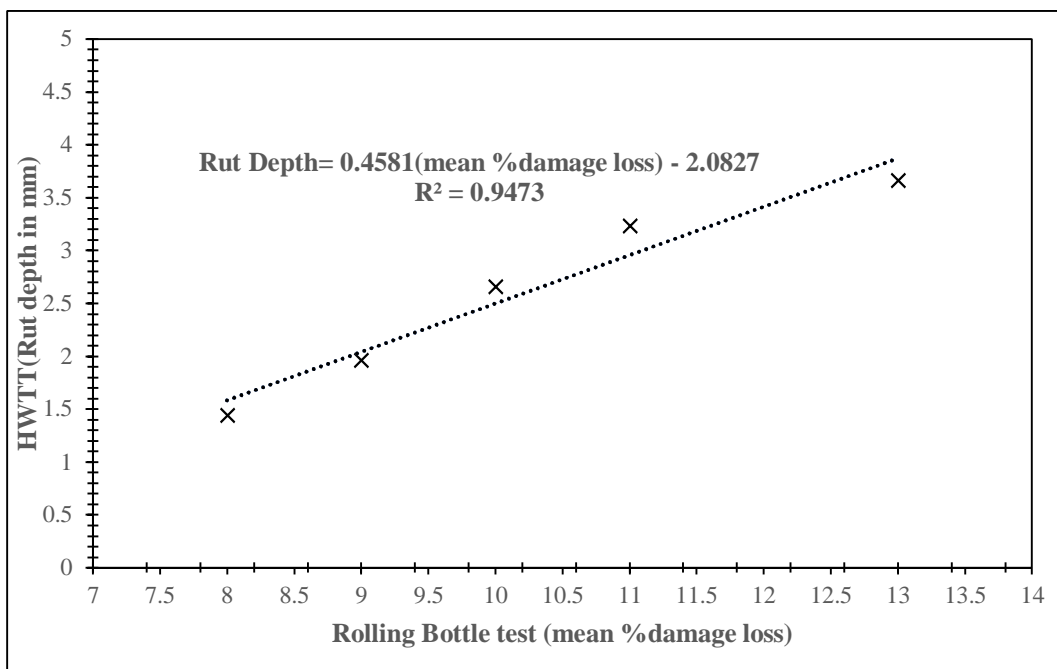


Fig 9: Correlation between rolling bottle test and Hamburg wheel track test results

It may be noted from above Fig & Fig rolling bottle test results are widely acceptable and reliable that is the main reason of the selection of rolling bottle test in qualitative testing. While in quantitative tests, Marshal stability test and Hamburg wheel track test were used. With the help of these liner equations, if we have results of qualitative tests then we can easily access the quantitative test results.

- 2) **Moisture damage Index:** Moisture damage index (MDI) was established as part of this phase correlation model to forecast moisture damage likelihood in both qualitative and quantitative test regimes. Optimized coefficients were calculated by linear regression between qualitative tests mean percent damage loss (Q1) and quantitative tests mean percent damage loss (Q2) to establish moisture damage index. In qualitative studies, the rolling bottle test was found to be more reliable than other tests [17]. Although the Hamburg wheel track test analyses the combined effect of rutting and moisture susceptibility in quantitative tests.
- 3) TABLE 4 shows the relationships that were used to generate the moisture damage index. Fig 8 shows the results of all of the above relationships. It can be shown in Fig 8 that the percentage damage loss increases as the moisture damage index value rises. The laboratory test findings demonstrate a significant connection with the linear regression model. A higher percent damage loss number indicates that the asphalt mixture has poor adhesion qualities. As a result, asphalt mixtures with higher moisture damage index values are more likely to be damaged by moisture. In total, four qualitative tests and two quantitative tests were used in this study.

TABLE 4: List of relationships used to develop Moisture damage index(MDI)

- i)  $MDI = 0.36*Q_1$  (mean %damage loss) +  $0.64*Q_2$ (mean %damage loss)
- ii)  $Q_1 = 0.19*SWIT$  (mean %damage loss) +  $0.21*TWIT$  (mean %damage loss) +  $0.18* BWT$ (mean %damage loss) +  $0.42* RBT$  (mean %damage loss)
- iii)  $Q_2 = 0.27*MST$  (mean %damage loss) +  $0.31* HWTT$  (mean %damage loss)

Results of all these above relationships has been given in Fig 8.

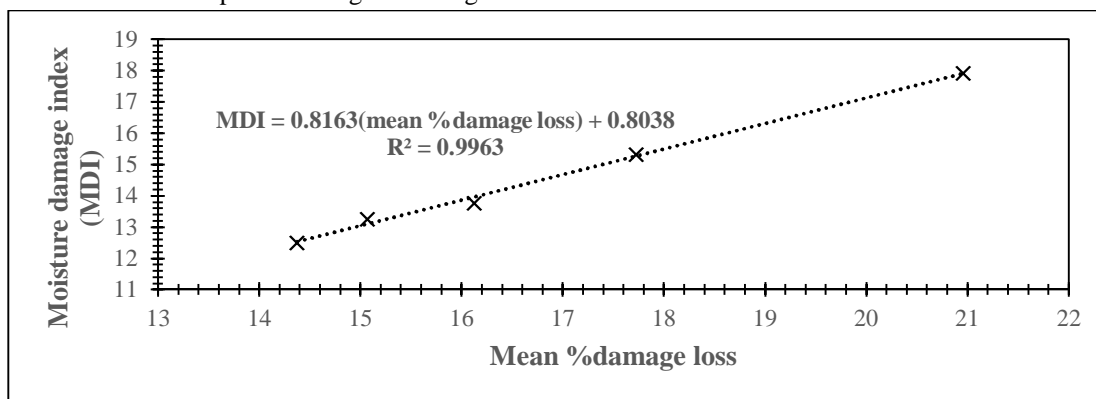


Fig 80: Moisture damage index(MDI)

It may be noted from Fig 8 that values of percentage damage loss increase with the increase of moisture damage index values. The linear regression model shows a strong correlation with laboratory test results. A higher value of %damage loss stands for weak adhesion properties of asphalt mixture. Thus, asphalt mixture with higher moisture damage index values will be more susceptible to moisture damage.

## VI. CONCLUSIONS

Based on all the laboratory test results,

- 1) The Performance of asphalt mixture improved significantly with the addition of Phosphorus Methyl Compound (PMC), accompanied by the enhancement of the rut parameter.
- 2) In moisture damage index there is a strong correlation between laboratory test results. Thus, asphalt mixture with higher moisture damage index values are more susceptible to moisture damage.
- 3) Based on this the base binder +0.75% Phosphorus Methyl compound (PMC) modified asphalt binder is suitable for pavement construction in Pakistan to meet high resistance against moisture.
- 4) The Bitumen aggregate bond strength has improved significantly with the addition of Phosphorous Methyl Compound (PMC). Both cohesive and adhesive bond values increased with increase in Phosphorus Methyl Compound (PMC) up to some extent.
- 5) The statistical analysis shows that the PMC content has a significant effect on rut depth and moisture susceptibility.

- 6) In statistical analysis, base binder +0.75% of PMC test samples of aggregate quarries containing calcium carbonate have less loss due to high polarity, hydrophobic and basic nature of aggregate.

#### VII. RECOMMENDATIONS

Overall, based on all experimental work, this research study proposes using modifier in asphalt mixtures with doses of base binder + 0.75 % PMC and the fundamental character of aggregate sources (limestone) to improve moisture resistance is recommended. However, a more detailed study of PMC with other parameter like rutting and fatigue cracking to find stability of modified asphalt mixture with the same percentages will be recommended to evaluate and select the best option for improving performance of asphalt pavements.

#### VIII. ACKNOWLEDGMENT

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