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Influence of Natural Zeolite and Paraffin Wax on Adhesion Strength Between Bitumen and Aggregate

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Abstract

Asphalt mixture that is used for the construction of flexible pavements is mainly composed of two constituents i.e. bitumen and aggregate. Sturdy adhesion among bitumen and aggregate is the sign of durability of asphalt pavements. Adhesion is considered as one of the most important factors for sustainable asphalt pavement. This is the motive why its miles utmost important to deeply understand the phenomenon of adhesion considering the effect of alternate in temperature, moisture conditions. In this study softer binder 80/100 was selected that has less adhesion compared to hard pen grades. Limestone aggregates which is commonly used for the construction of asphalt pavements has also been selected. Two types of modifiers (Zeolite and Paraffin Wax) were selected because of the extensive use in asphalt foaming and the polymer modified asphalt mixtures as temperature reducing agent. To investigate the strength of adhesive bond, Bitumen Bond Strength (BBS) was performed at different temperatures, in dry, and wet conditions. To quantify the effect of modifiers on penetration grade and softening point conventional testing is performed. For performance grading, the PG test was performed using Dynamic Shear Rheometer. The comparisons were developed among pull of tensile strength at dry and after 72hrs water conditioning while preserving the temperature at 25℃.To check the effect of temperature BBS is performed at 15 °C. The results illustrate that 2% zeolite shows best results in terms of adhesion and performance grade while Paraffin wax has less adhesion and poor performance grade.

Keywords: Bitumen; Aggregates; Adhesion; Pen Grade; Softening Point; BBS; DSR; Performance Grade.

1. Introduction

Most of the pavement are constructed in the world are asphalt pavements. During the life period, these pavements must bear climate impact in terms of moisture, temperature and loads. The major reason for pavements deformation is moisture damage [1]. Introduction of moisture into asphalt pavements leads to adhesion loss between the binder and aggregates. The deformation of asphalt pavements mainly is due to adhesion loss [2]. Diffusion of water into pavement layers weakens the adhesive bond between aggregate and bitumen. The occurrence of stripping is due to debonding, which is caused by water ingress. Studies have done on properties of asphalt pavements shows that the mechanical properties of flexible pavement depend on the bond between the binder and aggregate. The strength of the interface bond between binder and aggregate defines the life of pavement and its potential to withstand against heavy loading and climate condition.

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The moisture damage is not only causing the reduction in stiffness but also strength of asphalt pavements as well as leads to various types of distresses like stripping, ravelling, hydraulic scour, rutting and alligator cracking. So, it is important to quantify the adhesion properties and effect of water on interfacial bond in asphalt mixtures [3, 4]. Factors like aggregate mineralogy, chemical properties of binder and surface of aggregates control the moisture damage in asphalt pavements [5]. According to studies, aggregate with basic nature shows more adhesion than acidic aggregates and have greater resistance against moisture damage [6]. 54$ billion are spent on maintenance of road pavements in America because of moisture damage. About 82% of American Transportation Agencies recommend the use of adhesion promoters in low-performance job mix formulas [7-9]. Zhang has used the Rediset [10] Nano-charcoal and coconut shell ash [11] and, Hamedi and Tahami (2018) has used Zycosoil as adhesion promotor [2]. There are two types of modification against moisture damage: additives are mixed in bitumen or aggregates are modified. Because of ease in modification in bitumen, method of mixing of anti-stripping agents in bitumen is adopted [2]. The zeolites are used in asphalt foaming technologies as water containing group. The zeolites consist of microporous aluminosilicate minerals. The crystalline structure of zeolites continuously releases the water at 400°C without damaging their structure [12]. The pen grade and softening point are also improved by zeolite. When zeolite is added in asphalt mixtures, it decreases the mixing temperature. Because of this property, there is the saving of energy. Moreover, by the addition of the zeolites, the potential of asphalt pavements is increased against permanent deformation [13].

The polymer-modified asphalts are developed to reduce rutting, fatigue and thermal cracking which leads to reduce maintenance costs. High working temperature is required for polymer-modified asphalt mastic. At the same time, high temperature makes bitumen brittle material which has a bad impact on performance. High working temperature required more energy and cause the emission of bitumen fumes and CO_2 than conventional HMA works. To meet the allowed working temperature conditions and emission, the wax is used to decrease mixing temperature and emission as flow agent [14, 15]. Numerous test methodologies were developed by researchers to investigate adhesion between bitumen and aggregates. The EN 1297-11 provides three methods to quantify affinity in asphalt mixture. These tests are rolling-bottle-test, static water storage and detachment in boiling water. Whereas, in the Lithuanian standard LST 1362.23 – boiling water test method is used. The Rolling-bottle testing is considered most suitable but the mechanical strain by rolling process shows bad impact of accuracy of results. There is no differentiation of results in static water storage, and the boiling water test doesn't provide accurate results [16]. The Pneumatic Adhesion Tensile Testing Instrument (PATTI) instruments which is known as most suitable device for newly proposed Bitumen Bond strength test. It is a portable device and can be used easily for adhesion testing. Procedure to perform this test is quite easy and quick [10].

The waxes are used in PMAs as flowing agent and Zeolites are used in asphalt foaming as water agents. Due to environmental limitations, waxes and zeolites are extensively used in asphalt pavements to reduce energy emission and CO2. Because of their use in asphalt pavements, it is necessary to investigate their effect on adhesion. The major aim of this study is to quantify the effect of Waxes and Zeolite on adhesion between bitumen and aggregate

In Pakistan, flexible pavements are facing severe issues of adhesion failure especially in Rainy and high-temperature areas. Due to adhesion loss, the maintenance cost of pavements has been increased as well as service life is decreased too. To sort out this problem, this study was done by using modifiers. So that this research will help highway agencies to uses this research in asphalt pavements.

The material that is used in this study is easily available throughout Pakistan. The modifier Natural Zeolite and Paraffin wax are cheap and commonly available. There is ease of mixing of these additives in bitumen. The methodology of investigating adhesion strength is quite simple and portable. This test can be performed at any desired temperature into a controlled temperature chamber. The outcomes of this study are very helpful and adaptable in the field. By using this research, the maintenance cost of asphalt pavements will minimize.

2. Materials

2.1. Bitumen

80/100 was selected as Binder. The Penetration Test (BS EN 1426) and Softening Point Test (BS EN 1427) was performed to verify the pen grade and calculate softening point of this bitumen. According to results, the Pen Grade was 87 and the softening point was 44°C. The name of bitumen was given "B" in this study.

2.2. Aggregates

As limestone shows better adhesion due to its basic nature that's why limestone was selected. Lime-stone is selected to investigate how the Zeolite and Wax modified bitumen affect adhesion bond with basic aggregates because the limestone is considered a suitable aggregate with respect to adhesion. The properties of this aggregate are presented in the following Table 1.

Table 1. Aggregate nature and type

Materials	Tye	Component	Rock Type	Nature
Aggregate	Lime Stone	Calcite 65%, Silica 30%	Fossiliferous Limestone	Basic

Figure 1. Crushed limestone

2.3. Additives

Two additives were selected based on their use in Asphalt technologies. One was Natural Zeolite (Z) which easily available in Pakistan a 2nd is Paraffin Wax (P). The chemical composition of zeolite is given in Table 2.

Table 2. Chemical composition of zeolite

Chemical Component	Quantity (%)
Silica as SiO_2	67.72
Aluminum as Al_2O_3	16.29
Sodium Oxide as NaO_2	1.49
Iron as Fe_2O_3	0.62
Potassium Oxide as K_2O	3.68
Calcium Oxide	3.14
Manganese Oxide	0.01
Magnesium Oxide	0.00

The Paraffin Wax is colourless or white material extracted from petroleum, coal or shale oil. The Waxes are hydrocarbons containing twenty to fourth atoms with formula $C_nH_{2n+2.}$ These additives were used in 0.5, 2 and 3.5 percent by weight of bitumen.

3. Experimental Methodology

Figure 2 shows the flowchart methodology which has been followed in this study while performing laboratory testing.

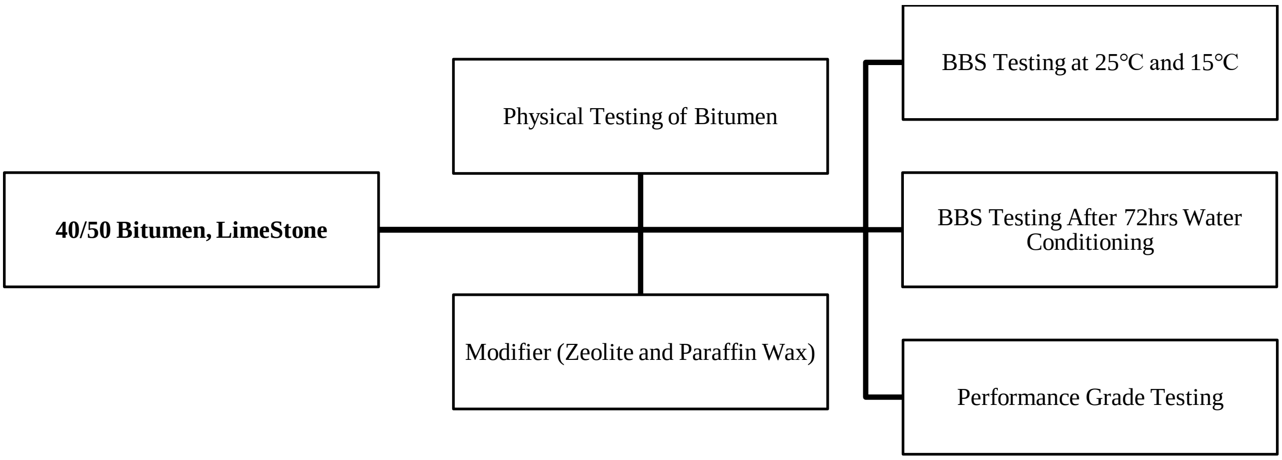

Figure 2. Flowchart of research methodology

3.1. Physical Properties Testing

The softening point was determined under British Standard (BS EN 1426) test. That is commonly known as Ring Ball Test. To find penetration grade, Penetration Test was performed under BS EN 1427. The results are presented in Table 3.

Table 3. Physical properties of B bitumen with and without additives

Description	Units	Virgin	0.5%Z	2%Z	3.5%Z	0.5%P	2%P	3.5%P
Penetration at 25°C	1/10 of mm	87	74	61	57	94	107	134
Softening Point	°C	44	47	49	50	43	41	39

The results illustrate by increasing dosages of Z in bitumen the softening points are increased and pen grade decreased. This means that Z has made the B stiffer than virgin B. While in the case of P, as the dosages are increased the bitumen B becomes softer than Base bitumen.

3.2. Mixing of Additive into Bitumen

To mix additives into bitumen, 200 gr of bitumen was heated at 130 ℃ to 150 ℃ until it converted in liquid form. Then prescribed ratios of additives were added into liquid bitumen. For homogenous mixing of bitumen, the mixture was put under stirrer with rpm 500 to 700 for five minutes. The homogenous mixture was poured into cans with a proper seal. Then these cans were kept in a dark place at 25 ℃ [9].

3.3. Bitumen Bond Strength Test (BBS)

Basically, BBS is a modification of the PATTI test. This test is performed under specification of (ASTM D-4541). This test is used to determine the bond strength between aggregate and binder in dry and after water conditioning. The BBS device consists of a metallic pull-off stub, reaction plate, pressure hose, piston, and a portable pneumatic adhesion tester. In this test the metallic stubs are pulled off by pressure. If the applied stress is greater than the cohesive and adhesive strength of bond, the failure occurs. Equation 1 is used to calculate the Pull of Tensile Strength (POTs).

$$POTs = \frac{(BP - A_g) - c}{A_{ps}} \tag{1}$$

Where BP is burst pressure in kPa, A_g is contact area between reaction plate and gasket, A_{ps} is area of pulled-off metallic stubs and c is the piston constant. The outer and inner diameters of metallic stubs are 22 mm and 20 mm respectively with 800 µm edge thickness. The systematic diagram is given in Figure 3.

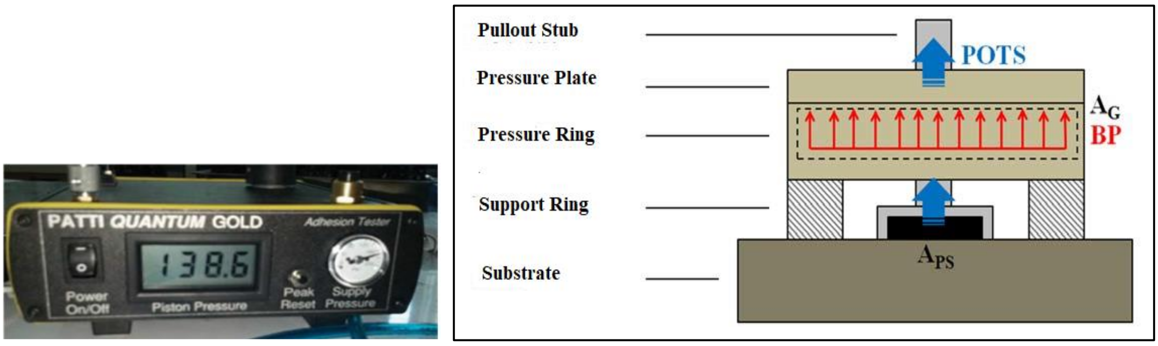

Figure 3. Test assembly of bitumen bond strength test

For sample preparation, the limestone substrates of size 300 mm × 150 mm × 25 mm were selected as shown in Figure 4. To clean this plate and to remove impurities, it was washed with de-ionized water and then place into oven at 60°C for 60 minutes to evaporate surface water. The metallic stubs first cleaned with acetone and then they were heated in oven at 75 °C. The bitumen was heat at 160 °C for 5 minutes to convert it into liquid form. Then bitumen was poured onto stubs surface with spoon and then firmly put on substrate [17].

Figure 4. Lime-stone substrate

After that, the specimens were kept in a dark and dry place to cool down at 25°C. This test was performed at different temperatures 15°C and 25°C to observe the effect of temperature on adhesion. To investigate the effect of water, the samples were kept in d-ionized water for 72 hrs. After performing a test, POTs were calculated using Equation 1. The BBS specimen is shown in Figure 5.

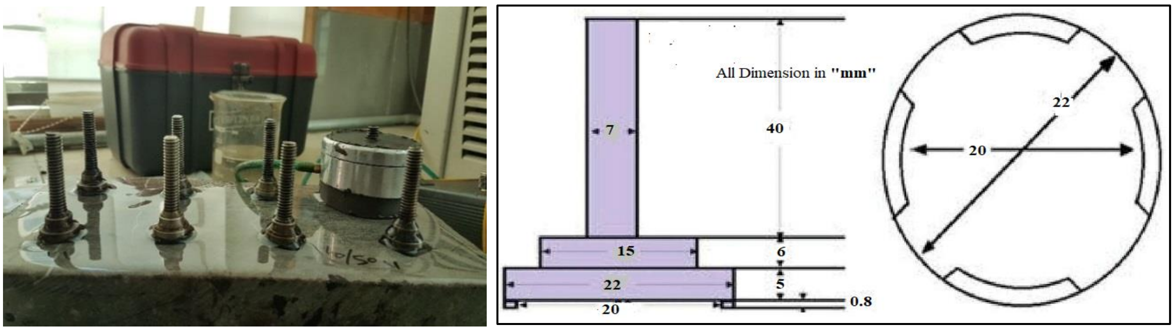

Figure 5. Pull of test specimen and pull of stub for BBS test

3.4. Performance Grade Testing

Penetration grading or viscosity grading have limited ability to fully characterize asphalt binder to use in Hot Mix Asphalt (HMA) pavements. To overcome this problem, performance grade was developed. In performance grading system, the bitumen is categorized according to their failure temperature. This characterization is more accurate than conventional Penetration Grading. This reliability of PG Testing enhances the quality of the binder which is used in HMA pavements.

The core theme behind this testing is to correlate the HMA asphalt binder properties with the conditions under which it will be used. High-Temperature Grade: To calculate the upper limit of performance grade, Dynamic Shear Rheometer was used. The test was performed under specification of AASHTO M320. In DSR testing, high-temperature grade is measure in strained-controlled mode. Where strain was kept 10% and frequency was fixed at 10 rad/s. Complex shear modulus (G*) and phase angle (δ) were measured in response to a sinusoidal stress for each sample [18]. The results have been summarized in Table 4.

Table 4. Summary of DSR test results for virgin and modified binders

Sample	52°C	58°C	64°C	70°C	76°C	82°C	Failure Temperature
G*/sin(δ) kPa	---	---	---	---	---	---	---
Virgin 80/100	4.43	2.46	1.10	0.536	---	---	64.8°C
0.5% P	3.41	1.70	0.670	---	---	---	62.4°C
2% P	2.89	1.42	0.610	---	---	---	61.1°C
3.5% P	2.77	1.21	0.565	---	---	---	59.6°C
0.5% Z	4.83	2.47	1.05	0.542	---	---	64.9°C
2% Z	5.19	2.75	1.15	0.581	---	---	65.8°C
3.5% Z	5.52	2.92	1.25	0.614	---	---	70°C

4. Results and Discussion

4.1. Results

The specimens were prepared under the specification of ASTM-4541 and the test was performed in a dry condition at 25°C. To investigate the effect of temperature, the samples were also tested at 15°C. All tests were performed in the temperature-controlled chamber. To examine the effect of moisture, the samples were conditioned by keeping in de-ionized water for 72 hrs at 25°C. For each combination of bitumen and aggregate, 4 specimens were tested for their average results. The POTs were calculated by using Equation 1.

In Figure 6 the results show that 2% of Z Bitumen has the highest pull of tensile strength (POTs), which is 509 psi than that of virgin B with POTs 400 psi. The results illustrate with an increase of dose of Z, the adhesion increases but after 2% of Z further addition of Z decrease the adhesion. While P shows the decrease in adhesion as the quantity of P increase in B. 3.5% of P bitumen has lowest POTs that is 156 psi. The 0.5% of P bitumen shows a slight decrease in adhesion in dry condition. To quantify the effect of moisture the BBS was performed at 15°C on 80/100 Bitumen with lime-stone.

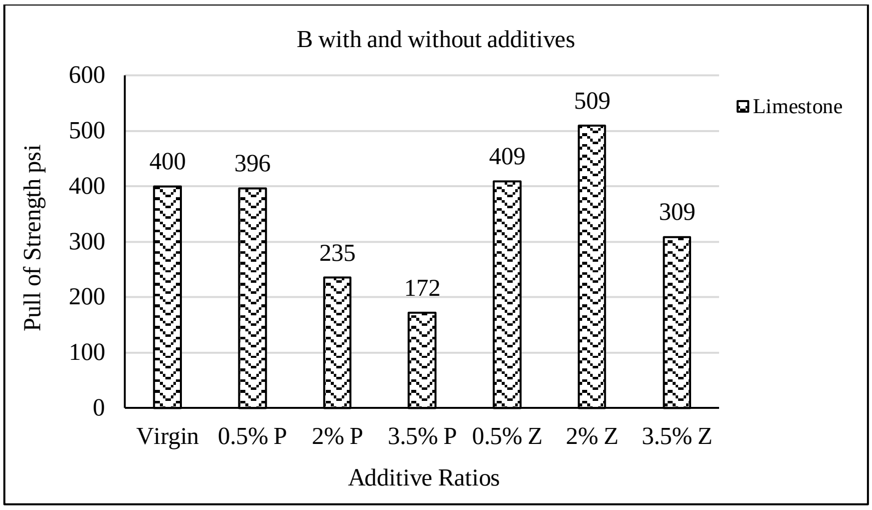

Figure 6. POTs by using B at 25 °C (dry condition)

The results are presented in Figure 7 and it reveals that with a decrease in temperature the adhesion is improved. The 2% of Z bitumen has the highest POTs that are 1545 psi while 3.5% of P bitumen has the lowest POTs that is 707 psi. The virgin has 1397 psi Pull of Tensile strength which is lower than 0.5% of Z modified bitumen but higher than P modified bitumen.

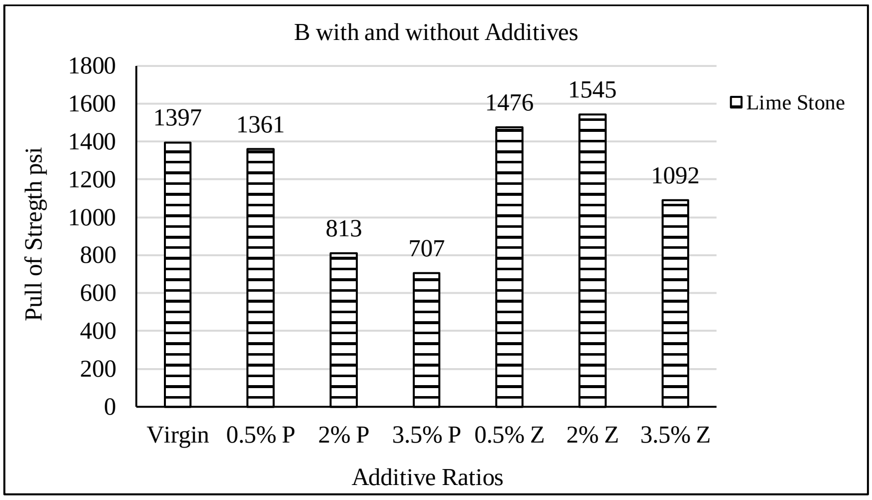

Figure 7. POTs by using B at 15 °C (dry condition)

Figure 8 represents the results of BBS was performed on virgin and modified bitumen with Lime-stone after 72hrs water conditioning. According to results, 2% of Z in bitumen stands at highest position with 361 psi POTs. The virgin B have 261 psi POTs that is higher than other ratios of P and Z, but it is lower than 2% of Z modified B. While 3.5% of P modified B have lowest POTs that are 167 psi.

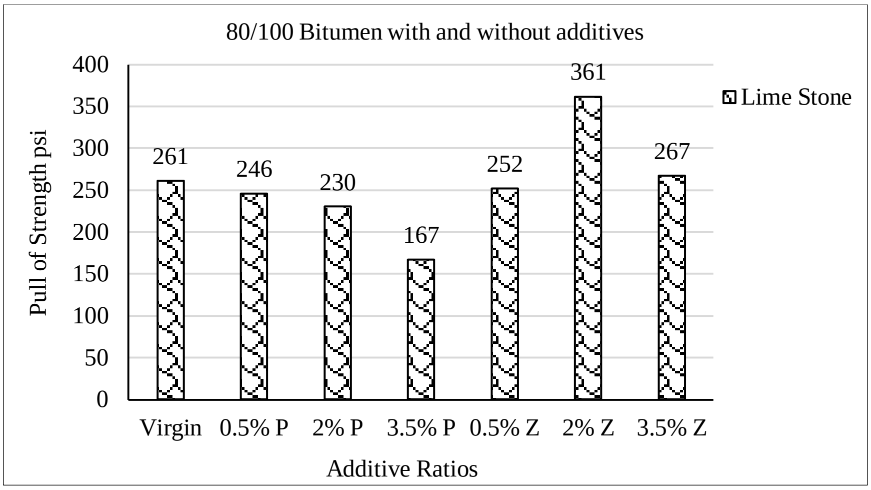

Figure 8. POTs by using B at 25 °C (72hrs water conditioning)

4.2. Discussion

Chemical and physical properties, aggregate mineralogy and compatibility of bitumen with type of aggregate in term of polarity and composition are the major factors which governed the adhesion among binder and aggregates. The introduction of modifiers into binder enhances the physical and chemical properties of bitumen. The results are presented in section 0. In the discussion section, the behaviour of bitumen either virgin or modified will be discussed against the adhesive bond between binder and aggregate. In Figure 9, the correlation indicates that with an increase in stiffness of binder, the adhesion increases. When 0.5% of Z was added to bitumen, the adhesion has increased 2.2% than that of virgin B. But, as dose of Z was increased to 2% of Z, the adhesion has increased 20.3% than that of base B. further increment in Z quantity in B shows the 22.75% decrease in adhesion. In the case of P, with an increase in dosage of P into bitumen, adhesion has decreased. For 0.5% of P in B, adhesion has reduced to 1% than that of base B. When 3.5% of P is added into bitumen, the adhesion has dropped to 57% than that of virgin B. The behaviour of Z modified bitumen shows that the adhesion increases due to stiffness till a certain limit and beyond this limit the adhesion start decreasing and this trend is observed in the case of 3.5% of Z. The reason behind this behaviour is that when bitumen becomes stiffer it behaves like a solid brittle material. On the other hand, the introduction of P makes bitumen softer which cause a reduction in adhesion.

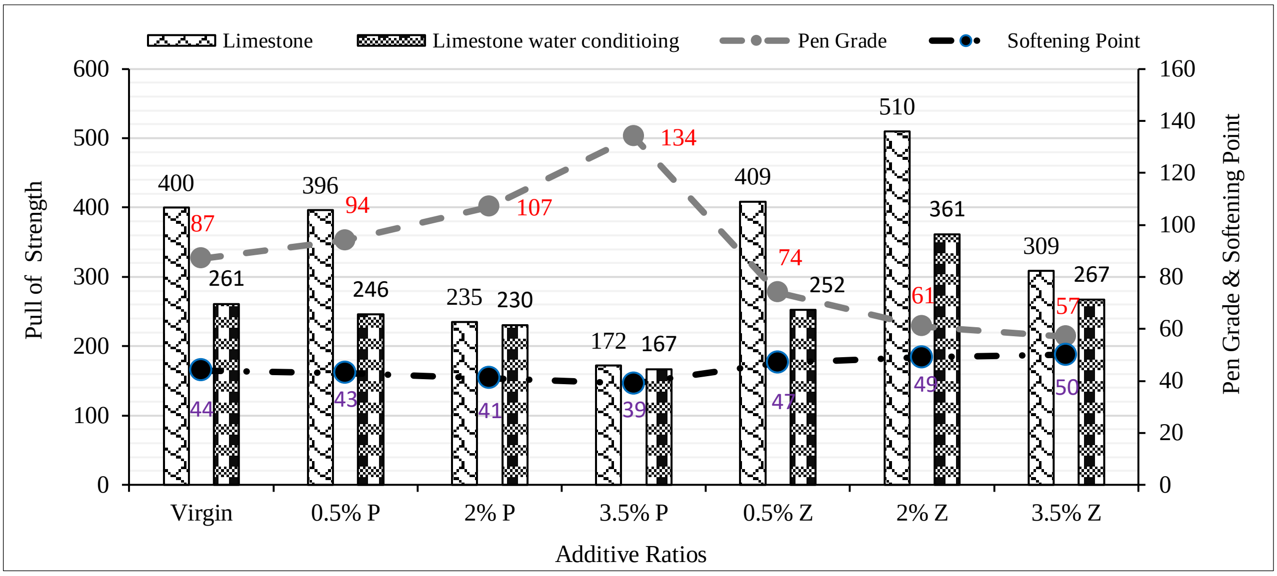

Figure 9. Correlation between BBS results and softening point and pen-grade of B

Effect of moisture: in Figure 10, if BBS after 72 hrs water conditioning is examined, the results illustrate that 2% shows highest resistance against moisture that is 27.7% more than that of base B. While the reduction in adhesion after 72hrs water conditioning for 2% of Z into bitumen with limestone is 29.21% of 2% of Z into bitumen in dry state. The reduction in adhesion for virgin B is 34.75% of virgin B in dry state. If reduction for 2% of Z and virgin B is compared, reduction in adhesion for virgin B is 5.53% more than that of 2% of Z modified bitumen. Now, if results of P modified bitumen are compared before water conditioning and after 72hrs. For 0.5% of P bitumen, the adhesion has dropped to 37.87% after water conditioning. While in the case of 3.5% of P, there is a slight decrease in adhesion that is only 3%. This behaviour shows that 0.5% of P modified bitumen has poor performance against moisture damage.

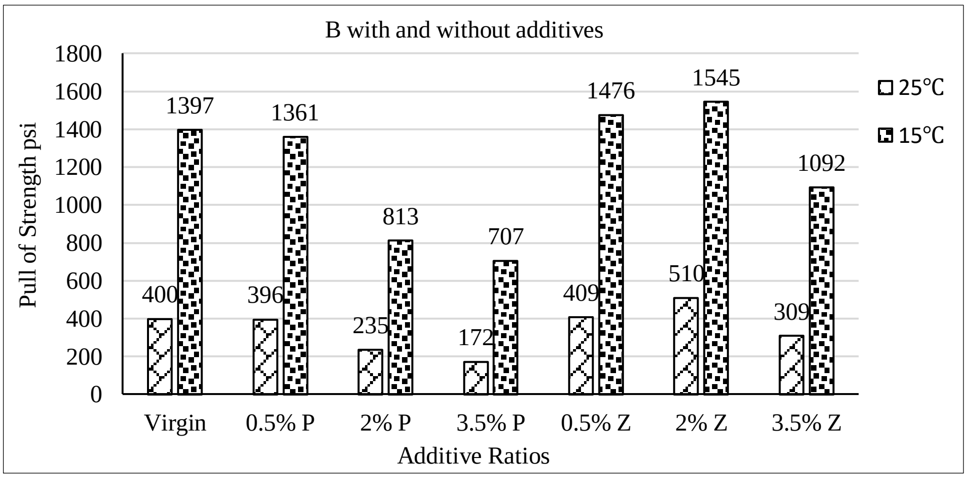

Figure 10. BBS results at 25 °C and 15 of virgin and modified B with lime-stone

In Figure 10, the comparison is developed between BBS results at 25 and 15℃ of virgin and modified Bitumen by using limestone. The core theme behind this comparison is to observe the effect of temperature on adhesion. At 15℃, the adhesive bond of 0.5% of Z bitumen becomes 72.3% stronger than that of at 25℃. For 2% of Z bitumen, the adhesion has increased to 67% than that of at 25℃. For virgin B, the adhesion has increased to 71.3% than at 25℃. For P modification, the decrease in temperature has improved the adhesion. For 0.5% of P bitumen, the adhesive bond has become 70.9% stronger than at 25℃. For 3.5% of P modified bitumen, the adhesion has increased to 75.6% than at 25℃. These behaviours illustrate that at lower temperature the adhesion between binder and aggregate is improved. So, not only physicochemical properties or mineralogy of aggregates the temperature also affect the adhesion. At high temperature, the adhesion decreases.

In Asif Sa et al. (2018) [17] researches Bitumen Bond strength test was performed to evaluate adhesion properties of binder and aggregate. Only virgin bitumen B2 (91 Pen Grade) was tested with different types of aggregates in dry and after 72 hrs water conditioning in that research. While in this research two types of modifier were used to quantify their effect on adhesive properties of bitumen. The pull of tensile strength of B2 with limestone was 345 psi while in present research (B with limestone) that is 400 psi. After 72 hrs water conditioning of B2 specimen, the pull of tensile strength was dropped to 285 psi while in present research that is 261 psi. Due to performing BBS testing at 15℃, 25℃ and after 72 hrs water conditioning with two type of modifier in this study has created greater difference between past study where only virgin bitumen were tested by using time as parameter.

In the above discussion, the adhesion is discussed according to temperature and pen grading. Now adhesion will be discussed according to physicochemical properties and mineralogy of aggregate. The Z has proved itself the best additive in terms of adhesion, as Z has an excess of cations in the form of SiO_2 (67.72%). When it is mixed with bitumen, it increased the polarity of bitumen by increasing positive ions. On the other side, the limestone has excess of anions due to $CaCO_3$ (65%). Because of this polarity difference, the bond between Z modified bitumen and limestone becomes stronger than that of virgin B and limestone. In the results of strong bonding, Z modified bitumen shows great potential against moisture damage. For the high polarity of aggregate, different types of aggregate can be used and to increase bitumen polarity, it is modified with organic and inorganic additives [19, 20]. The phase angle is increased due to enhancement in P content. The increase in phase angle decreases the viscosity of bitumen. This property of P damages the wettability mechanism of bitumen. As the phase angle is increased, the reduction in adhesion occurs. This reduction in adhesion is significant sign of moisture damage [21]. Due to these problems, the P modified bitumen shows lower adhesion than virgin and Z modified B. The viscosity of bitumen has a vital role in stripping. The less viscous bitumen gives better coating, but more viscous bitumen shows more resistance against stripping. So, P modified bitumen has poor performance against adhesion while Z modified has better performance against adhesion.

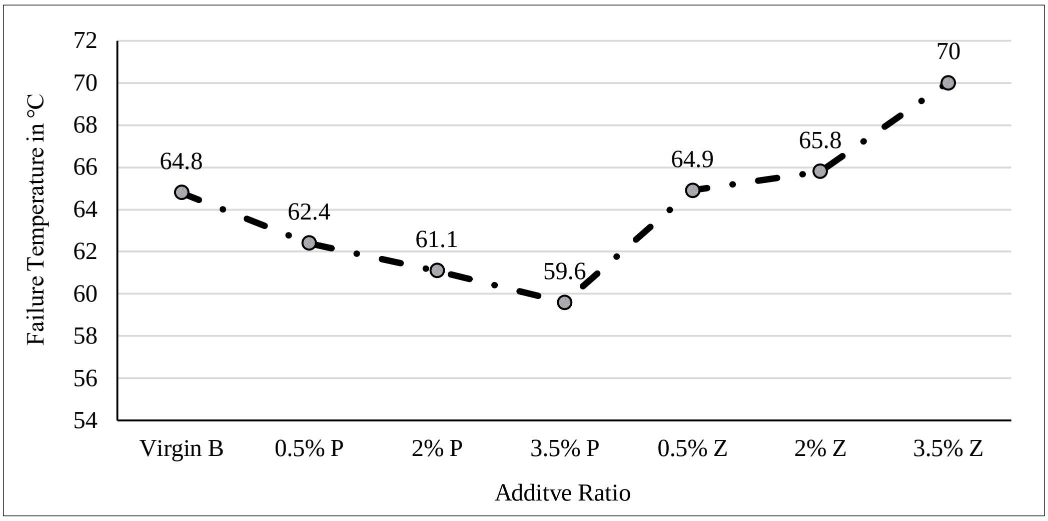

Figure 11. Performance grade of virgin and modified B

Figure 11 is a graphical representation of the performance grading of bitumen. The results show that the modification of P in bitumen decreases the failure temperature. At 3.5% of P in bitumen, the failure temperature has dropped to 8% of virgin bitumen. While when Z is mixed in bitumen the failure temperature has increased. With an addition of 3.5% of Z in bitumen, the failure temperature has raised to 7.4% than the virgin. This raising in failure temperature is a sign of improvement in rutting at high temperatures. And, the fall in failure temperature due modification of P in bitumen reflects the potential to resist fatigue cracking,

5. Conclusions

- Modification of bitumen with Zeolite shows that Zeolite can be used to make bitumen stiff as 0.5% addition of Z in Bitumen change the grade 87 to 74 and 3.5% of Z change the grade 87 to 57. While paraffin wax can be used to make the bitumen soft as 0.5% addition of paraffin wax convert the grade 87 to 94 and 3.5% of paraffin wax improves the grade to 134.
- As many factors control adhesion like viscosity, phase angle, contact angle, surface energy, physiochemical properties, chemical nature of aggregates and bitumen grade. The domination of these factors on adhesion properties is not straight forward. Sometimes one factor dominates while the other dominates under the other conditions. In this study, 3.5% Z should show maximum adhesion in case of limestone with B bitumen, but it shows less. Though it's chemically compatible with limestone at the same time, it makes bitumen stiffer. So, the bond becomes weak.
- The 2% of Zeolite dosage in Bitumen shows maximum adhesion strength with limestone as the POTs increase to 20.3% of virgin POTS. So, when there is moisture damage the combination of 2% Zeolite in B with limestone should be used.
- The modification of Paraffin wax in B shows inverse behaviour. As dosages are increased, the adhesion loss is started. At 3.5% of Paraffin into bitumen the adhesion drops to 43% of the virgin bitumen specimen in dry state and it is dropped to 64% of virgin bitumen after 72hrs water conditioning. So, the use of Paraffin as a flowing agent in Polymer modified Asphalts may affect the adhesion.
- The performance grading is showing that the modification of Zeolite makes bitumen harder. The 3.5% of Zeolite into bitumen changed the failure temperature from 64.8 to 70℃ So, Zeolite modified bitumen is useful in hot areas of Pakistan. Where temperature rises up-to 50°C.
- The results of testing after 72 hrs water condition illustrate 2% of Zeolite in bitumen has increased adhesion (361 psi) as compared to virgin (261 psi) and Paraffin wax modified bitumen (167 psi). Thus, Zeolite is also suitable Rainy Areas of Pakistan for adhesion purposes.
- The performance grading shows that Paraffin wax modified bitumen shows better results at lower temperature 15℃. The addition of 3.5% of Paraffin wax dropped the failure temperature from 64.8 to 59.6℃. Therefore, it can be used in cold areas of Pakistan like Northern Areas.
- The use of PATTI for Bitumen Bond Strength is very helpful. It is a portable device and can be used at any temperature to investigate adhesion either at lower temperature or at higher temperatures as compared to other techniques to investigate adhesion.

6. Acknowledgements

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7. Conflicts of Interest

The authors declare no conflict of interest.

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