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# Research Article

# **Performance Evaluation of Bone Glue-Modified Asphalt**

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Asphalt is one of the primary materials that are extensively used by the pavement industry throughout the world. Its behaviour is highly dependent on the amount of loading and the level of temperature it is exposed to. Asphalt has been modified in the past with different additives to improve its high- or low-temperature properties. In Pakistan, temperature remains high for most of the time of the year; hence, asphalt binders with less susceptibility to higher temperatures are preferred for flexible pavements. Acids, polymers, fibers, and extenders have been used by the researchers to improve high-temperature performance of asphalt mixture. In the present study, a bio material derived from the animal waste, named as bone glue (BG), has been used with the 60/70 penetration grade binder in dosages of 3%, 6%, 9%, and 12% by weight of asphalt binder. The bone glue is produced from a sustainable source. It is a cost-effective and eco-friendly material. Moreover, it produces a durable and nonhazardous asphalt composite. The influence of addition of bone glue on asphalt binder was evaluated using different testing techniques which include consistency tests, rheological analysis, and adhesion tests. Furthermore, different performance tests were conducted on bone glue-modified asphalt mixtures. Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) analysis were carried out to ensure the homogeneity and proper mixing of bone glue in asphalt binder. The results from the tests reveal that bone glue stiffens the asphalt binder hence enhancing its high temperature performance. Bone glue dosage of 9% by weight of the binder was found to be the optimum dosage based on the rheological and performance analysis.

#### 1. Introduction

In Pakistan, most of the transport infrastructures consist of flexible pavements. Asphalt binder is the primary material used in the construction of flexible pavements. Asphalt binder being a viscoelastic material is quite sensitive to temperature and traffic loading. Asphalt behaves as an elastic solid under lower temperatures and higher loading frequencies, while at higher temperatures and low loading frequencies, it behaves as a viscous fluid. In Pakistan, one of the most common types of failure is rutting because of very high temperature during summer. Furthermore, the traffic loading from slow-moving loaded trucks increase the viscous behaviour of asphalt hence making it sensitive to rutting [1, 2].

In order to improve the performance of flexible pavements, pavement engineers are continuously in pursuit of advance and more efficient asphalt modifiers. These

modifiers enhance the durability and sustainability of pavements by improving high- and low-temperature performance. Flexible pavement performance can be enhanced through change in aggregate gradation and/or source [3, 4], modification of binder through polymers [5], styrene-butadiene-styrene (SBS) [6, 7], crumb rubber [8, 9], Elvaloy [10], polyethylene [11–13], lime [14–17], and costly nanomaterials [18, 19].

Poly phosphoric acid (PPA) has been extensively used as bitumen modifier and has been reported to improve high-and intermediate-temperature performance of asphalt [20]. Studies conducted on nanomaterials, such as nano-TiO<sub>2</sub>, nano-SiO<sub>2</sub>, and nano-ZnO, have shown that bitumen modified with nanoparticles results in significant improvement in high-temperature performance of asphalt [21, 22], but it is considered as an expansive solution.

Polymer-modified asphalt has been used in almost every part of the world, specifically in advanced countries. The

price of polymer and extent of energy used for the modification is a big concern for the contractors and researchers. Polymers require high temperatures (65°C to 190°C) and a longer period of time (60 to 200 minutes) to give homogeneous blend when mixed with bitumen or asphalt mixture. Besides the preparation of modified asphalt, its durability is another big concern. Apart from the concerns discussed above, the use of polymer modification of asphalt is also expensive. So, its use is limited specifically in developing countries.

In the past, bone glue has been used to improve the rheological properties of neat binder [23]. A procedure has been introduced for mixing BG with asphalt binder [24]. Bone glue is naturally a protein-based gum prepared from collagen which is generated from animal skeletons and their meat excrete. Collagen is network of existing available proteins. The major amount of collagen protein is available in animal bones that consist of about 20 to 35 percent protein of the whole body [25].

Raw material for BG is animal waste. It is easily available in developing country like Pakistan; however, its preparation is very restricted due to limited application. It is generally used by the wood industry for production of furniture. Successful application of BG as asphalt binder modifier could result in a cheap alternative to other expansive modifiers. Currently, it is about \$0.8–\$1.9 per kg as per local market bidders of Pakistan, and the price can be further reduced if ordered in bulk. According to a survey conducted in 2013, only twenty plants are manufacturing BG in Pakistan for fulfilling the local wood glue requirements [24]. The production of BG is very limited compared to synthetic glue that is considered as the modern form of glue. The features of synthetic glue have cut down the application of BG in local areas.

Different researchers used BG to improve the properties of asphalt binder; however, to author's best knowledge, no comprehensive study is available on BG asphalt mixture performance.

To characterize the performance of binder for a specific region and temperature, pavement industry in the developed countries is using performance grading (PG) rather than old penetration grading. The main aim of using BG is to produce PG-70 [26] as suggested by Mirza et al. [27] considering Pakistan's environmental conditions.

#### 1.1. Objectives. Following are the objectives of this study:

- (1) The target of this study is to use BG as a modifier in asphalt binder in order to enhance its performance properties
- (2) To check the influence of BG on rheological properties of asphalt binder
- (3) To investigate adhesion potential of bone gluemodified asphalt binder
- (4) To check the impact of BG on moisture susceptibility of asphalt mixture
- (5) To investigate permanent deformation and fatigue behaviour of bone glue-modified asphalt mixture

# 2. Materials and Experimental Plan

2.1. Materials. The 60/70 penetration grade bitumen, which is the most common type used for flexible pavement construction in Pakistan, has been used for this research study. The basic properties of asphalt binder are presented in Table 1.

The bone glue was obtained from a local vender in Pakistan. Bone glue (BG) is a by-product of food and livestock productiveness [23]; the major raw material for the preparation of bone glue is waste of animals.

The aggregates (limestone) were procured form Margalla queries in Pakistan. Some of the properties of Margalla aggregates are presented in Table 2. The NHA (National Highway Authority) class B midpoint gradation specifications were used for the preparation of asphalt concrete. Figure 1 shows the NHA class B gradation used in this study.

2.2. Preparation of BG-Modified Asphalt Binder. The technique for mixing BG developed by Rizvi et al. [24] was followed in the present study. BG was mixed with water before adding it to the asphalt binder to ensure proper mixing. The mixing time and temperature are given in Table 3.

Initially hand mixing was tried; however, it does not ensure complete dispersion/mixing of bone glue in bitumen. So, a high shear homogenizer was used to ensure complete dispersion/mixing of bone glue in bitumen which can be seen through scanning electron microscopy (SEM) analysis results presented in Figure 2.

FTIR (fourier-transform infrared spectroscopy) results for neat and modified asphalt binder are presented in Figure 3. From the results, it can be observed that no new peaks appeared, nor any existing peaks disappeared which shows similarity between neat and BG-modified asphalt binder.

Fourier-transform infrared spectroscopy results of modified asphalt binders show carbon-hydrogen stretching and bending frequency properties of a typical hydrocarbon. FTIR peaks at approximately 2838–2950 cm<sup>-1</sup>, 1360–1760 cm<sup>-1</sup>, and 620–950 cm<sup>-1</sup> can probably be attributed to the C-H symmetric/asymmetric stretch, C-H bending vibrations, and C-H rocking vibrations, respectively [24]. The FTIR spectrum of the bone glue-modified asphalt binders is quite similar to that of the neat binder which is an indication of a homogeneous product.

The homogeneity was further ensured by performing (PG) tests on the sample taken from three different portions, i.e., top, middle, and bottom of the container. A dynamic shear rheometer (DSR) was used to determine high PG temperatures of samples. The results of the DSR (PG) are listed in Table 4 which confirm the homogenous dispersion of BG in asphalt binder. The PG-70 was achieved at 9% dosage, so it was selected as optimum content.

2.3. Preparation of Asphalt Mixture Samples. For preparation of asphalt mixture samples, NHA class B gradation was used. To find optimum binder content, Marshall mix design

TABLE 1: Basic properties of asphalt binder.

Test	Value	Standard
Softening point (°C)	48	ASTM D36
Penetration (0.1 mm, 25°C)	67	ASTM D1586
Ductility (cm) (25°C)	102	ASTM D113

TABLE 2: Physical properties of aggregates [28].

Property	Test result (%)	Limit
Elongation	11	15% (max)
Water absorption	1.03	2% (max)
Los Angeles abrasion	14	15% (max)
Sand equivalent	75	50% (min)
Flakiness	5	15% (max)
Uncompacted voids	48.5	45% (min)
Fractured particles	100	90% (min)

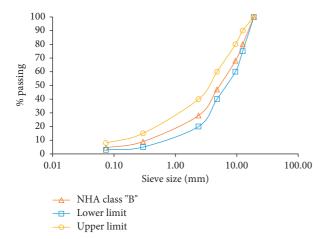


FIGURE 1: Gradation curve of aggregates used for asphalt mixtures.

(ASTM D1559) was used for BG-modified asphalt samples for three percentages (3%, 6%, and 9% by weight of bitumen). 75 blows of standard hammer having weight 4.5 lbs and height of fall 18 inch were applied on both sides of samples as per Asphalt Institute for heavy traffic. 5.5% air voids were targeted as per MS-2 series (3–8%). OBC (optimum bitumen content) for unmodified (0%) and modified samples (3%, 6%, and 9%) was 4.4%, 4.46%, 4.53%, and 4.65%, respectively. The increase in BG dosage resulted increase in optimum binder content. This increase is attributed to the increasing viscosity of the BG-modified asphalt binder, which, in turn, induces a relatively thicker film of asphalt binder around aggregates, hence increasing the optimum binder requirement for the mixture [24].

#### 2.4. Experimental Methods

2.4.1. Storage Stability. The high-temperature storage stability of BG-modified asphalt binder was studied by performing the storage stability test as per ASTM D5892. The storage stability is estimated in terms of separation index (SI), which is the softening point difference of top and bottom portions of the sample. According to ASTM D5892,

the sample will be regarded as storage stable if SI is less than 2.2°C.

2.4.2. Consistency Tests. The influence of BG addition in the asphalt binder was assessed by performing penetration, ductility, and softening point tests as per ASTM D1586, ASTM D113, and ASTM D36, respectively.

2.4.3. Rheological Testing of Asphalt Binder. An Anton Paar dynamic shear rheometer (DSR) was used to study the rheological properties of asphalt binder at high and intermediate temperatures. Frequency sweep, performance grading, and multiple stress creep recovery (MSCR) tests were carried out as per AASHTO T 315 [29]. A DSR plate of 25 mm geometry was used for testing samples at temperatures higher than 46°C while 8 mm geometry was used for testing samples at temperatures less than 46°C.

The limiting strains in the frequency sweep test as per superpave criteria were 10% and 0.45% for neat and BG-modified asphalt binder, respectively. The gap between parallel plates was kept 2 mm and 1 mm for 8 mm and 25 mm geometries, respectively. This test was conducted at six different temperatures (22°C, 34°C, 46°C, 58°C, 70°C, and 82°C) while the frequency was kept between 0.1 and 10 rad/s. Rheological properties were studied by determining the following parameters; phase angle  $(\delta)$ , rutting factor  $(G^*/\sin\delta)$ , and complex shear modulus  $(G^*)$ .

The multistress creep recovery test was performed at a temperature of 58, 64, and 70°C as BG-modified asphalt binder in our case has a maximum PG of 70. Plate geometry of 25 mm was used with stress ranging from 0.025 to 25.6 kPa at each stress level in ten cycles. For the MSCR test, a constant creep stress was applied for 1 second followed by a relaxation period of 9 seconds. At the end of 10 cycles, recoverable and nonrecoverable strain percentages were calculated [26].

2.4.4. Rolling Bottle Test. To measure the moisture susceptibility of neat and BG-modified asphalt binders, the rolling bottle test was performed as per BS EN 12697-11. The samples were prepared using 170 g of aggregate (passing through 8 mm sieve and retained on 6.3 mm sieve) and 8 g of asphalt binder, conforming the full coating of bitumen to aggregate. The samples were placed in bottles filled with deionized water. The bottles were placed in machine rotating at 60 rpm, and coating of samples was checked after 6, 24, and 48 h.

2.4.5. Bitumen Bond Strength Test. PATTI (Pneumatic Adhesion Tensile Testing Instrument) was used to check the adhesion of bitumen with aggregate after dry and moisture conditioning in terms of bitumen-aggregate bond strength (BBS) according to ASTM D 4541. For sample preparation, asphalt binder samples and limestone aggregate plates  $(15'' \times 6'' \times 1.5'')$  were heated at  $150^{\circ}$ C to ensure proper adhesion of stubs with aggregates. For each unconditioned and conditioned specimen type, five samples were tested.

Table 3: Mixing time and temperature for preparation of modified binders [24].

BG-modified blend	Mixing temperature	Mixing time	Physical appearance
3% by weight of binder		18 minutes	
6% by weight of binder	130°C	32 minutes	Settled free of foam and bubbles
9% by weight of binder	130 C	40 minutes	Settled free of foam and bubbles
12% by weight of binder		51 minutes	

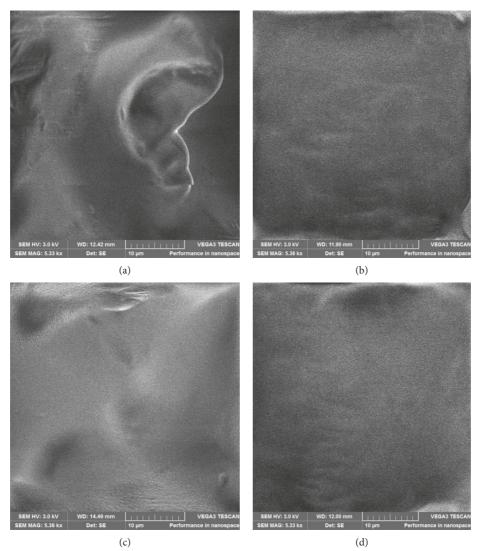


FIGURE 2: Continued.

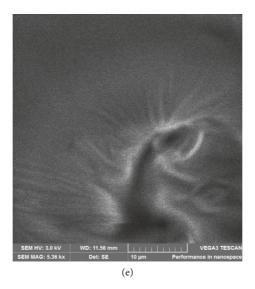


FIGURE 2: SEM images of neat and BG-modified bitumen samples. (a) Neat binder. (b) 3% BG by weight of binder. (c) 6% BG by weight of binder. (d) 9% BG by weight of binder. (e) 12% BG by weight of binder.

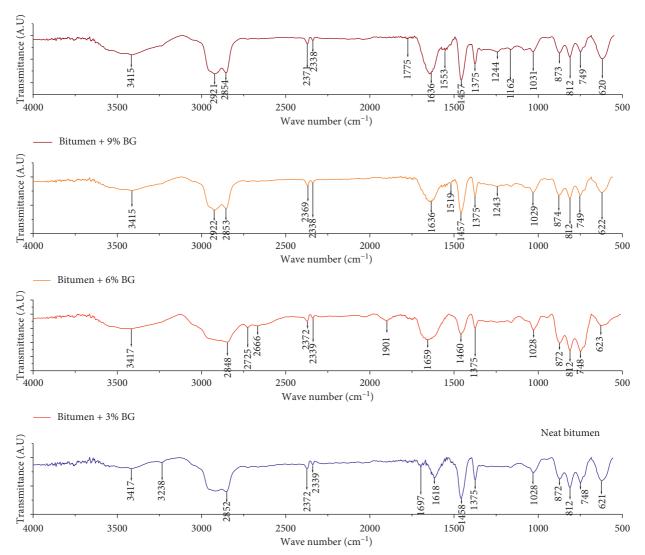


FIGURE 3: FTIR results of neat and modified binder.

Sample container 3% BG 6% BG 9% BG Top portion 65.4°C (PG-64) 67.2°C (PG-64) 69.8°C (PG-70) Middle portion 64.7°C (PG-64) 67.8°C (PG-64) 69.6°C (PG-70) Bottom portion 65.1°C (PG-64) 67.3°C (PG-64) 70.1°C (PG-70)

TABLE 4: Performance grades showing homogeneous dispersion.

Moisture conditioning was done by placing the samples in water for 24 hours at 25°C.

The value of burst pressure at the point of stub detachment is recorded. The BBS in terms of pull-off tensile strength (POTS) is calculated form burst pressure using the following equation:

$$POTS = \frac{\left(BP \times A_{g}\right) - C}{A_{ps}},\tag{1}$$

where  $A_g$  is the contact area of the gasket with the reaction plate,  $A_{ps}$  is the pull-out stub area, C represents piston constant, and BP is the burst pressure.

The values of  $A_g$ , C, and  $A_{ps}$  are 4.06 in<sup>2</sup>, 0.286 in<sup>2</sup>, and 0.193 in<sup>2</sup>, respectively, for the stub type we have used in the present study, i.e., F-4.

2.4.6. Dynamic Modulus Test. A superpave gyratory compactor was used to make the cylinder-shaped sample (150 mm diameter and 170 mm height) at a mixing temperature of 160°C. To achieve the desired height, 600 kPa pressure was applied to the sample. A sample having 150 mm height and 101.6 mm diameter was extracted from the compacted sample for dynamic modulus testing. After preparing the samples, the dynamic modulus of asphalt was determined by Cooper NU-14 test according to AASHTO TP 62-03. Loading of 195 kPa and 53 kPa in the form of sinusoid with varying loading frequency of (25, 10, 5, 1, 0.5, and 0.1 Hz) was applied at 40°C and 55°C correspondingly.

2.4.7. Cooper Wheel Tracking Test. The rutting of asphalt mixtures was assessed using a Cooper wheel tracker in accordance with BS EN 12697-25. The roller-compacted slabs (300 mm  $\times$  300 mm  $\times$  150 mm) with a target air void of 5.5% were prepared. The rut depth was recorded at 10,000 loading cycles with the application of 700 N wheel load at a frequency of 26.5 rpm. All tests were performed at 40°C and 55°C to satisfy the environmental conditions prevailing in the country.

2.4.8. Four-Point Bending Beam Fatigue Test. The fatigue of asphalt mixtures was studied through the four-point bending beam fatigue test inconformity with AASHTO T321. The roller-compacted slabs (380 mm  $\times$  250 mm  $\times$  63 mm) with a target air void of 5.5% were prepared. Beam samples having a size of  $380 \times 50 \times 63$  mm were then trimmed from each slab. The test was conducted in strain-controlled approach on HMA beam samples in repetitive four-point loading at 300 microstrain ( $\mu\varepsilon$ ). All tests were performed at 20°C with sinusoidal load at a frequency of

10 Hz. The number of loading cycles to failure is considered as fatigue life of asphalt mixture.

# 3. Findings and Discussions

3.1. Thermal Susceptibility and Penetration Index of Selected Binder. The performance of BG-modified asphalt binder in terms of temperature susceptibility can be assessed by determining its penetration index (PI). The penetration index depends on penetration and softening point values of the asphalt binder. The penetration value represents the stiffness of asphalt binder at moderate temperature. A lower penetration value indicates a stiffer binder. It is clear from Figure 4 that increasing the dosage of BG in bitumen makes the binder stiff due to decrease in the penetration value. The observed decreases in penetration values were 7%, 13%, and 22% with the BG dosage of 3%, 6%, and 9%, respectively. The softening point test represents the deformation characteristics of bitumen at high temperature. The observed increases in softening point values were 2°C, 5°C, and 7°C for BG content of 3%, 6%, and 9%, respectively. The increase in softening point and decrease in penetration might be due to high surface energy and presence of intermolecular forces in the bone glue that makes bitumen stiff [30].

PI can be estimated using following equation [31]:

PI = 
$$\frac{20 - 500A}{1 + 50A}$$
, where  $A = \frac{\log(800) - \log(\text{Pen at } T)}{\text{SP} - 25}$ ,

where T represents the penetration test temperature, i.e., 25°C, Pen at T represents the penetration value at desired temperature T (0.1 mm), SP represents the softening point of asphalt (°C), and Pen at SP represents the penetration at the softening point and assumed as 800 (0.1 mm) [32].

A higher value of PI indicates lower temperature susceptibility. Most of the asphalt binders have PI values in the range of -2 and +2. Asphalt binders having PI values less than -2 are highly temperature susceptible and at low temperatures will be brittle, causing transverse cracking [33].

The PI values for BG-modified asphalt binders are presented in Table 5 and Figure 5. The PI results are within normal PI range, i.e., -2 to +2. The PI value of bitumen increased with the addition of BG which indicates decrease in the bitumen temperature susceptibility.

The ductility value gives an idea about tensile properties of asphalt binder. From Figure 6, it is clear that increase in BG content causes reduction in ductility values. The observed decreases in ductility values were 20%, 27%, and 39% with the addition of BG content of 3%, 6%, and 9%, respectively. The reduction in ductility was due to increase in stiffness of bitumen with the addition of BG which could reduce the fracture resistance of the binder when subjected

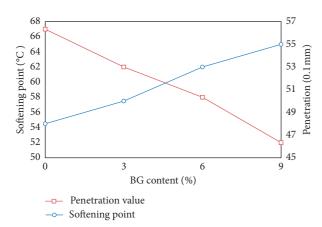


FIGURE 4: Effect of BG addition on penetration and softening point values.

TABLE 5: Penetration index (PI) of BG-modified bitumen.

Sample	Neat binder	3% BG	6% BG	9% BG
PI	-1.02156	-0.6873	-0.1157	0.07226
A	0.04683	0.04443	0.0407	0.03957

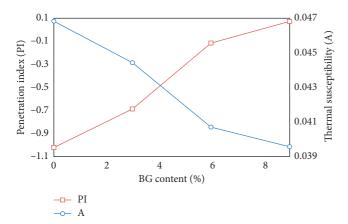


FIGURE 5: Effect of BG addition on penetration index and thermal susceptibility.

to tensile stress [34]. It may be important to study the fatigue behaviour of BG-modified asphalt.

3.2. Storage Stability Analysis. The storage stability of BG-modified asphalt binder is evaluated in terms of separation index (SI), which is the softening point difference of bottom and top portions of a test tube. The separation index values are listed in Table 6. The results show that the BG-modified asphalt binder is storage stable as separation index values are less than 2.2°C as per BS EN 13399:2017.

3.3. Rheological Characterization of BG-Modified Asphalt Binder. A dynamic shear rheometer was used to investigate the rheological properties of asphalt binder at intermediate to high temperatures for different frequencies. Figures 7–9 show the master curve developed at  $58^{\circ}$ C for  $G^{*}$ ,  $G^{*}$ /sin  $\delta$ ,

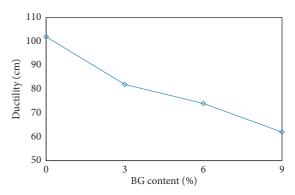


FIGURE 6: Ductility of bitumen with varying content of BG.

TABLE 6: Separation index (SI) of BG-modified bitumen.

Softening point (°C)	Neat binder	3% BG	6% BG	9% BG	
Top portion	48.4	49.1	52	54	
Bottom portion	48.9	49.8	53.1	55.5	
Difference	0.5	0.7	1.1	1.5	

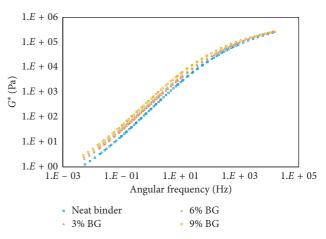


FIGURE 7: Master curve for  $G^*$  at 58°C.

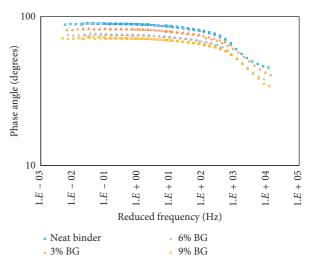


FIGURE 8: Master curve for  $\delta$  at 58°C.

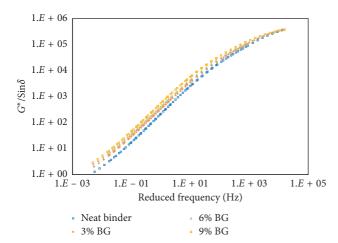


FIGURE 9: Master curve for  $G^*/\sin\delta$  at 58°C.

and  $\delta$ , respectively. Figure 7 shows the values of  $G^*$  (complex shear modulus) at different frequencies. It can be observed that the stiffness of asphalt binder increases with the addition of BG and gives maximum value at 9% when examined at high temperature. Furthermore, the addition of BG makes the asphalt binder stiff causing more resistance against permanent deformation.

Bitumen is a viscoelastic material that exhibits elastic behaviour at lower temperature and viscous behaviour at high temperature. The phase angle ( $\delta$ ) is used to illustrate the bitumen elastic or viscous behaviour. Figure 8 shows gradual increase in phase angle values with the decrease in loading frequency or increase in temperature. The addition of BG resulted a decrease in phase angle at all loading frequencies, and this reduction is more prominent at 9% BG content with a decrease of 19% in the phase angle at 10 Hz. This decrease in the phase angle is an indication of the increase in elastic behaviour of bitumen [35]. Thus, the results show that BG has improved the elastic behaviour of asphalt binder; hence, the chances of permanent deformation at higher temperatures are low.

The most important problem in asphalt pavements is deformation of the pavement in the wheel path due to heavy traffic at high temperatures, which is called rutting. The resistance against permanent deformation at high temperatures is evaluated in terms of rutting factor, i.e.,  $G^*/\sin\delta$  and can be used in the performance analysis of pavement [35]. Figure 9 indicates that the BG-modified asphalt binder has high rut resistance in comparison to neat asphalt binder, which means increase in resistance against permanent deformation and elastic behaviour of asphalt binder at high temperatures with the addition of BG.

The premature failure in flexible pavement at high temperature is due to mix rutting, which is the main problem faced by the pavement industry of Pakistan. Considering this factor, in high temperature areas of a country, PG-70 is recommended [27]. The influence of BG on PG of the asphalt binder is presented in Figure 10. The addition of 3% BG by weight of the asphalt binder increases failure temperature form 63.3°C to 65.7°C and improves

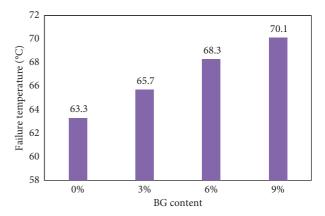


FIGURE 10: Influence of BG on performance grade (PG).

PG-58 to PG-64. The addition of 9% BG has increased failure temperature of the asphalt binder from 63.3 to 70.1°C hence bumping the grade to PG-70, which means that 9% BG-modified asphalt binder can meet the performance grade requirement for most of the temperature zones of Pakistan.

3.4. Creep Compliance of BG-Modified Asphalt Binder. The creep recovery behaviour of BG-modified asphalt binders was assessed by performing the MSCR test at varying temperatures (58°C, 64°C, and 70°C). However, the effect of different stress levels on  $J_{nr}$  values of asphalt binder was checked at 70°C as PG-70 is the desired performance grade. It is clear from Figure 11 that the increase in stress level increases the  $J_{nr}$  value, and this increase continues gradually up to a stress level of 25.6 kPa. The neat asphalt binder has highest  $J_{nr}$ , and 9% BG-modified asphalt binder has the lowest  $J_{nr}$ . This is due to the stiffness of asphalt binder by addition of BG by enhancing its tendency to recover upon release of stress. So, the asphalt binder with higher  $J_{nr}$  value will has less propensity to return back to original position and vice versa. The addition of 9% BG resulted a decrease in  $J_{\rm nr}$  by 69.3%. The decrease in  $J_{\rm nr}$  is due to the presence of intermolecular forces in BG which makes the asphalt binder stiff [36].

# 3.5. Moisture Susceptibility Analysis

3.5.1. Bitumen Bond Strength Test. The pneumatic adhesion tensile testing instrument (PATTI) was used to check the influence of BG on bitumen-aggregate bond strength, and the results are presented in Table 7 with failure pattern. The bitumen bond strength (BBS) is described in terms of pull of tensile strength (POTS). Figure 12 shows the POTS values for dry and moisture conditioned samples (24 hours at 25°C). For the dry conditioned samples, addition of BG resulted 54.76% increase in the POTS value, while for the moisture conditioned samples, the decrease in the POTS value was 48.33% and failure plane shifted from within the binder to bitumen-aggregate interface.

The BG-modified binder shows a poor performance in comparison to the neat binder after 24h of moisture conditioning, and 9% dosage has lowest POTS values. The

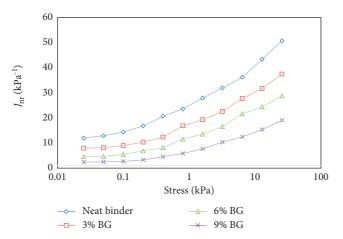


Figure 11: Effect of stress level on  $J_{nr}$  values of the asphalt binder at 70°C.

TABLE 7: POTS (psi) of unmodified and BG-modified binders and failure pattern.

Replicates	24 h dry conditioning			24 h moisture conditioning				
	Neat binder	3% BG	6% BG	9% BG	Neat binder	3% BG	6% BG	9% BG
1	7.78 (C)	12.25 (C)	13.91 (C)	16.94 (C)	6.21 (A)	4.83 (A)	4.5 (A)	3.32 (A)
2	7.57 (C)	11.83 (C)	13.98 (C)	16.64 (C/A)	5.46 (A)	4.76 (A)	3.89 (C/A)	2.98 (A)
3	7.73 (C)	12.07 (C)	13.65 (C)	16.38 (C)	6.1 (A)	4.7 (C/A)	3.87 (A)	3.13 (A)
4	7.45 (C)	11.75 (C)	13.82 (C/A)	16.53 (C)	6.23 (A)	4.85 (A)	3.95 (A)	3.10 (A)
5	7.49 (C)	11.59 (C)	13.67 (C)	17.52 (C)	6.02 (A)	4.92 (A)	3.88 (A)	3.13 (C/A)
Avg	7.60	11.90	13.8	16.8	6.00	4.8	4	3.1

C = cohesive failure; A = adhesive failure; C/A = 50% cohesive 50% adhesive failure.

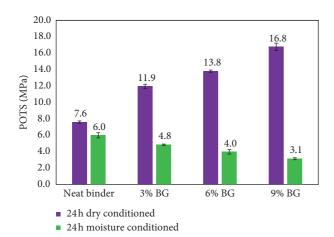


FIGURE 12: POTS of dry and wet conditioned samples.

decrease in POTS values can be attributed to the moisture sensitive behaviour of BG [37]. From results, it is concluded that BG-modified samples behaves poorly under moisture conditioning.

3.5.2. Rolling Bottle Test. The rolling bottle test was performed to check the moisture susceptibility of loose coated asphalt mixture. Figure 13 shows the relationship of percentage coating of modified and unmodified asphalt binder at durations of 6 h, 24 h, and 48 h. It is clear from the results that percentage coating was decreased with the addition of

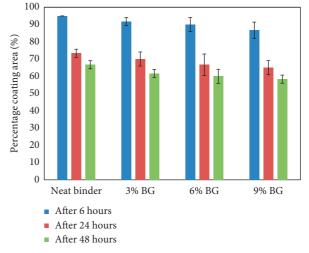


FIGURE 13: Loss of coating for various BG percentages at different durations.

BG. Hence, it can be concluded that the BG-modified asphalt binder is sensitive to moisture and gives a poor moisture damage performance.

### 3.6. Permanent Deformation Analysis

3.6.1. Dynamic Modulus Test. The resistance of asphalt mixture against permanent deformation is evaluated in

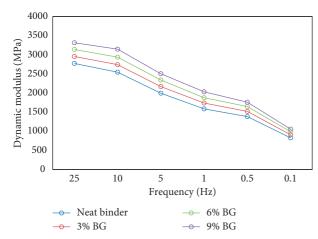


FIGURE 14: Dynamic modulus graph at 40°C.

terms of dynamic modulus. The asphalt mixture having high values of dynamic modulus will have more resistance against permanent deformation and vice versa. The results of dynamic modulus are presented in Figures 14 and 15. All tests were performed at 55°C and 40°C considering climatic conditions in Pakistan. The results indicate that dynamic modulus of asphalt mixture decreases with the increase in temperature and decrease in loading frequency. This could be attributed to temperature-dependent behaviour of asphalt as the increase in temperature makes the asphalt soft, causing decrease in dynamic modulus [38]. Moreover, 9% BG-modified asphalt shows better performance at both temperatures and loading frequencies.

Increase in dynamic modulus can also be due to the fact that BG-modified asphalt binder produces interactive forces with aggregates. BG fills the gap among aggregates causing improvement in dynamic modulus of asphalt [39]. The sustainability of pavement depends upon its service life and also on the functional and structural ability of pavement in terms of performance measure [40]. Improved pavement performance leads to longer service life and lower maintenance cost, subsequently conserving resources. Based on performance testing results, it can be concluded that introduction of BG in asphalt can be helpful in improving the service life of pavements in Pakistan.

3.6.2. Cooper Wheel Tracker Test. The effect of BG on rut depth was studied through Cooper wheel tracker test. The rut depth was recorded at 10,000 loading cycles as per BS EN 12697-25. The test results at 40°C and 55°C are presented in Figures 16 and 17. It is clear from results that rutting resistance increased with the addition of BG. The neat asphalt binder gave the maximum rut depth values against the standard ten thousand wheel passes. With the addition of 3%, 6%, and 9% BG, a decrease in rut depth of 0.41 mm, 0.82 mm, and 1.28 mm was observed at 40°C. At 55°C, a decrease of 1.99 mm, 2.65 mm, and 3.2 mm rut depth was observed for 3%, 6%, and 9% BG-modified asphalt mixture, respectively. The performance of 9% BG-modified asphalt was observed to be better at both temperatures after 10,000 passes. The

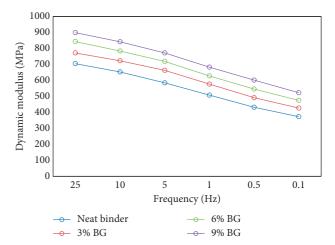


FIGURE 15: Dynamic modulus graph at 55°C.

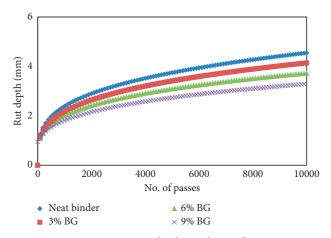


FIGURE 16: Rut depth graph at 40°C.

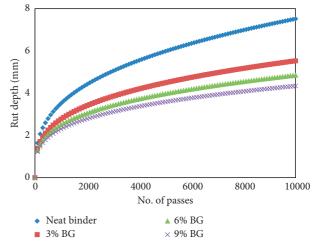


FIGURE 17: Rut depth graph at 55°C.

decrease in rut depth may be due to increase in the surface area of asphalt binder with the addition of BG [41]. So, it is concluded that BG improves high-temperature performance of asphalt by enhancing its resistance against rutting.

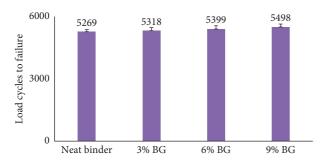


FIGURE 18: Fatigue life of BG-modified asphalt mixture at 20°C.

#### 3.7. Fatigue Life

3.7.1. Four-Point Bending Beam Fatigue Test. Fatigue cracking is a major distress found in flexible pavement particularly in the cold areas, where temperature sometimes reaches below the freezing point. Fatigue cracking is generally observed in the wearing course. The fatigue resistance of asphalt mixture is evaluated in terms of loading cycles in four-point bending beam fatigue test.

The test results are presented in Figure 18. It is clear from results that loading cycles to failure slightly increases with addition of BG. However, no significant improvement in fatigue life of asphalt mixture is observed. The study conducted by Rizvi et al. in 2014 claims enhancement in fatigue life of BG-modified asphalt binder [24]. However, in this study it has been observed that BG has no significant effect in enhancing/compromising fatigue life of asphalt mixture.

#### 4. Conclusions and Recommendations

4.1. Conclusions. This research study points towards the suitability of BG as an effective asphalt binder modifier/additive. It is an economical and environment friendly option. Moreover, bone glue can be produced quite easily in the desired quantities as it comes from animal waste which is a sustainable source, and is commercially available in Pakistan. BG can successfully substitute hazardous chemicals and other uneconomical polymers/binder modifiers, and the local pavement construction industry could benefit a lot from it.

Following are the main conclusions from this study:

- (1) FTIR, SEM, and storage stability test results show that the mixing procedure used for the preparation of bone glue-modified binder ensures complete dispersion of bone glue in the bitumen and results in a homogeneous and storage stable product.
- (2) The addition of bone glue increases the stiffness of asphalt binder causing a decrease in its ductility and penetration values while increasing its softening point. Penetration index values also demonstrate a decrease in thermal susceptibility of asphalt binder once modified with bone glue.
- (3) Complex shear modulus and rut factor values increase after addition of bone glue while phase angle and  $J_{nr}$  values decrease which shows an improvement in elastic behaviour of bone glue-modified binder.

- (4) Addition of 9% bone glue bumped the high PG value of the binder from PG-58 to PG-70, which also satisfies the requirements for most temperature zones of the country.
- (5) Bitumen bond strength and rolling bottle test results show that bone glue-modified asphalt binder is moisture sensitive.
- (6) 9% bone glue-modified asphalt binder results in decrease of rut depth by 28.13% and 42.28% at 40°C and 55°C, respectively. Also, dynamic modulus values enhance at both temperatures. The decrease in rut depth and increase in dynamic modulus may be due to higher stiffness of bone glue-modified asphalt.
- (7) The addition of bone glue in the asphalt mixture shows no significant effect in its fatigue life.
- (8) Based on the experimental analysis, it is concluded that bone glue optimum dosage of 9% by weight of asphalt binder is recommended for enhancement of asphalt properties.
- 4.2. Recommendations. Following are the recommendations for future work:
  - (1) Effect of ageing on bone glue-modified asphalt binder needs to be studied.
  - (2) It has been observed in this research that bone glue is a moisture sensitive material. Therefore, some antistripping agents like hydrated lime should be used with bone glue to counter its moisture susceptibility.

## **Data Availability**

The data used to support the findings of this study are available from the corresponding author upon request.

# **Conflicts of Interest**

The authors declare no conflicts of interest.

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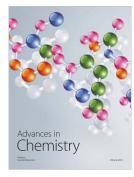


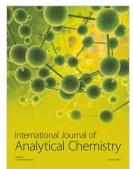














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