

Evaluation of the moisture damage susceptibility and the rutting resistance of natural bitumen mixture: An experimental investigation

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ABSTRACT

The concerns of oil depletion and the growing demand for new and eco-friendly alternatives for crude oil asphalt (COA) has prompted the utilization of natural bitumen (NB) in pavement field. Iraq has many deposits of NB and the most common types are lake bitumen (LB) and Gilsonite bitumen (GB). This research investigated the performance improvement of LB using GB as a modifier. The modified blends were designed by partially replacing 30%, 35%, and 40% of the weight of LB with GB. Physical and chemical testing were conducted on bitumen specimens, including specific gravity, solubility, softening point, ductility, penetration, viscosity, and x-ray diffraction (XRD). In addition, indirect tensile strength test and wheel tracking test were applied on optimal NB-modified mixture and control mixture prepared with COA. On the basis of the results, modified LB with GB significantly reduced fluidity, increasing rotational viscosity at 135 °C approximately by 1.7, 2.6, and 4.3 times for the blends with 30%, 35%, and 40% GB, respectively. Also, the stiffness of modified blends was increased, for instance, the penetration value of modified LB with 35% GB reduced by 67.4% compared to base LB (unmodified). Modified LB with 35% GB is the optimal blend, as it complies with the specification limits for COA. XRD analysis demonstrated that LB has an amorphous structure similar to COA and unlike GB, which has a crystalline structure. Also, modified LB samples have a crystalline structure, indicating the dominant influence of GB on the chemical properties of LB. Compared to the control mix, the NB-modified mix showed a higher TSR value and a lower rut depth with improving rate of 13.9 % and 53.9%, respectively. In conclusion, the GB modifier with optimal content of 35% makes the LB mixture more resistant to moisture damage and less susceptible to rutting failure.

Keywords: crude oil asphalt, Gilsonite, lake bitumen, moisture damage, natural bitumen, rutting.

INTRODUCTION

The disproportionate consumption of the natural-materials resources urged the pavement engineers to search for new alternatives, such as recycling the waste plastic to produce artificial aggregates and use it as a partial replacement for both coarse and fine natural aggregates [1]. In addition, asphalt binder is one of the essential components of flexible pavement, because its properties and bonding with other components (aggregates and additives) directly affect paving performance. This binder is obtained from crude oil refining processes [2]. In fact, asphalt production industry recently faces major challenges, concerning

about approaching oil peak, fluctuations in crude oil prices, and the continuous depletion of crude oil resources. Correspondingly, natural bitumen (NB) has acquired much attention, especially in the recent years, used as a modifier or a stand-alone binder material in asphalt mixtures production because NB can enhance the performance and cost efficiency of road pavement [3]. Also, there is growing interest in the world to explore the adoption of green, natural, and eco-friendly materials in the construction sector which is crucial especially for saving energy and reducing environmental pollution [4].

NB deposits are spread across many countries in the world, where the total volume of

NB reserves worldwide is about 1,856,853 billion barrels [5]. The United States of America leads the countries with the largest reserves of NB, and the rest of the reserves are distributed in Canada, Iran, Iraq, Saudi Arabia, Russia, Venezuela, China, Australia, Mexico, Albania and the Philippines. NB is also found in other countries, such as Madagascar, Buton Island in Indonesia, Romania, Kazakhstan, France (Gard and Tham), Switzerland (Traver Valley), Italy (Ragoza), Nigeria and Greece [6]. Iraq is considered one of the most important countries rich in these deposits. There are three forms of NB in Iraq: rock bitumen, lake bitumen (LB), and Gilsonite bitumen (GB). This research focused on using LB and GB in the manufactured of asphalt mixture instead of local petroleum asphalt.

LB is a black, viscous liquid, and a thick bitumen that is extracted from surface deposits, often called sulfur springs in Iraq, globally known as bitumen or asphalt lakes. These lakes or springs can vary significantly in area. They are typically formed when crude oil transfers from the deep underground and forced up to the Earth's surface with water and other mineral materials, usually along fault lines. Due to the environmental effects, the lighter hydrocarbons of crude oil are evaporated after settling on the surface, leaving behind a sticky, thick, and viscous bitumen. This bitumen has been used since ancient times for several applications such as ship coating and sealing boats channels. Currently, large quantities of LB are extracted annually from the bitumen lakes in Iraq and used limitedly in the building sector [7].

LB comprises a relatively high content of aromatics (55.67%) and asphaltenes (26.56%) fractions. This may be related to the thermal maturation of organic elements in the natural deposit [8]. Because of its chemical composition and high asphaltene content, LB has high thermal stability, good oxidation resistance, and good adhesion, making it an effective material and a suitable alternative to industrial asphalt [9].

GB is a mineral material includes complex hydrocarbons, such as aliphatic and aromatic compounds. It is a resinous solid hydrocarbon material and is frequently called asphaltum, mineral asphalt, uintaite, mineral pitch, natural hydrocarbon, and asphaltite [10]. It is mostly discovered in particular geological formations. It occurs in dikes or vertical veins through sedimentary rock layers, primarily in the areas with significant tectonic activity. Its natural regions are

mainly related with zones where organic materials were exposed to high temperature and pressure over millions of years. This NB type is found in deposits in many locations around the world, including the United States, China, Turkey, Iraq, Iran, Afghanistan, and Canada. Also, GB is a valuable substance for its unique features, including excellent adhesion, resistance to chemicals and high temperatures, good electrical insulation characteristics, highly resistant to corrosion, good abrasion resistance, as well as high solubility in aliphatic and aromatic solvents [11].

For many years, GB has been utilized in various industrial applications, especially in oil and gas drilling, construction and road maintenance, foundry and steel sector, paints and coatings, waterproofing and sealing industry, agriculture field, concrete industry, and chemical products [12]. There are various formulations and grades of GB, besides, its quality depends on location of mining. In addition, its formulation types includes processed, powdered, and pelletized. Grade of GB is categorized by solubility, softening point, mesh, and content of impurities. Moreover, the GB price is varied and depends on the season which is related to weather condition, oil price, purity level, market conditions, and number of mines under exploration [13]. In the last decade, many successful companies in Iraq, particularly in Kurdistan region and Basra governorate, are responsible and specialized in extraction, utilization, pulverization, packing, and exportation of GB, besides seamless attraction of numerous international investments and companies. This product is packed and offered in three kinds: lump, granulated, and micronized powder.

The current investigation utilized LB with soft grade. This grade of LB differs drastically from the refined asphalt produced through distillation processes of crude oil in terms of physical properties, impurity, and chemical composition. These differences impart the distinct characteristics of LB, such as flexibility, which limits its use as a standalone binder in asphalt pavement. Also, it is more susceptible to moisture damage [7]. Therefore, many researchers have paid attention to modifying LB and making it suitable to use as a sole binder and green alternative to refined asphalt in paving works.

Researchers have applied thermal treatment to modify LB as well as help increase its stiffness and reduce its temperature sensitivity. For example, Ahmed et al. [14] conducted thermal treatment

on LB from Abu-Jeer Lake for different periods as follows: 5, 10, 15, 20, and 25 hours at 163 °C. After applying thermal treatment for 20 hours, LB properties were improved and confirmed to the specification requirements of asphalt binder that used in paving works. Compared to local mixture, Marshall stability was increased by 17.6% and a slight enhancement in the water damage resistance (0.37%) was observed for treated-LB mixture. In addition, Mohsin and Latief [7] studied the effect of thermal treatment on the LB samples collected from five bitumen lakes in Hit city located in the western region of Iraq. It was revealed that thermal treatment is effective in improving the characteristics of LB. The stability of the mixture prepared with treated LB, collected from Mamora Lake, increased by 41.3% compared to control mixture. Meanwhile, the resistance to moisture damage improved by 5.72% and the stiffness index raised by 40.36% for the treated LB collected from Askaree Lake. The best thermal treatment durations for LB from Mamora and Askaree lakes were 17 and 26 hours, respectively.

Nejres et al. [15] studied the potentiality of using synthetic low-density polyethylene polymer (LDPE) and waste eggshell powder (WEP) as a LB modifier. It was concluded that the rheological characteristics of LB were improved by the incorporation of LDPE and WEP. The results of chemical tests revealed that NB has a colloidal system of SOL type. The preferable percentages of modifiers are 8% and 15% for LDPE and WEP, respectively.

Furthermore, the properties of LB can be improved by mixing it with crude oil asphalt (COA) at varied ratios. In the investigation of Ahmed et al. [16], COA was modified with two grades of LB (55% hard grade and 45% soft grade) in adding proportions of 20, 40, 60, and 80% to enhance the mechanical characteristics and water damage resistance of conventional mixture. The optimal ratio of LB is 80% which achieved the best performance for modified mix in terms of increasing the Marshall stability as well as stiffness index by 23.5% and 6%, respectively, compared to control mixture. Also, a slight enhancement in the resistance to moisture damage for modified mixture was observed since the tensile strength ratio was raised only by 0.57%.

Two studies evaluated the impact of the limestone filler (LSF) on the characteristics of LB. The first study was conducted by Abdul-Jaleel et al. [17]. The main components of LB

was investigated in this study. Also, modified LB with LSF, at 5%, 15%, 25%, and 35%, was evaluated. LB was collected from Abu-Jeer Lake, located in Hit city, west of Iraq. The separation results reveal that LB contains 10.2% asphaltene and 89.19% Maltene. Modification LB with LSF reduced the penetration, increased the softening point, and improved the workability of binder. The optimal content of LSF was 35%, providing the best behavior. The second study was carried out by Mohammed et al. [18], who added LSF to LB in four ratios: 25%, 30%, 35%, and 40% by the weight of LB. Five samples of LB were collected from five different lakes. LSF is an effective modifier and has a crucial role in enhancing the characteristics of LB, especially the resistance against temperature changes. The LSF-modified LB from Mamora and Askaree lakes achieved the best behavior in terms of stability, stiffness index, and resistance to moisture damage. The optimal percentage of LSF was 40% for all LB samples except for LB collected from Atffa Lake, which was 30% because this lake produced a stiffer LB.

Although the literature extensively investigates the potential of using modified LB as COA modifier or as alternative to COA in road pavement construction, using GB as LB modifier to enhance its properties has not been previously evaluated. Utilizing GB to modify LB can be more effective than thermal treatment, because it may adversely affect the rheological properties of LB due to aging and could cause environmental pollution since this treatment increased the emissions of CO₂. Additionally, the high-cost of thermal treatment would increase the price of LB more than COA [14]. Also, GB is available in high quantities with a low price and limited use as well as it is known for its good affinity with COA and ease of use, unlike other modifiers [13].

In most cases, GB is used as a modifier to improve the rheological characteristics of COA [19]. For example, GB was used as an alternative modifier to styrene-butadiene-styrene (SBS) polymer in asphalt modification. COA with penetration grade 160/220 was modified with GB at ratios of 20, 35, and 50% to investigate the alterations in the rheological and conventional characteristics of COA. The experimental results indicate that modified asphalt with 20% GB presented similar performance with the asphalt modified with 4% SBS. Adding 20% and 35% of GB improved the workability of asphalt binder, providing lower mixing and compaction temperatures.

The best ratio of GB was 35% since the asphalt mix demonstrated a greater performance in resisting the impact of heavy and repeated loads. However, modification with more than 20% GB will negatively affect the resistance of asphalt mix to cracks at low temperature. Furthermore, modification COA with 35% GB is cheaper than modification with 3% SBS and 4% SBS by 17% and 21%, respectively. This is because GB from Iraq is 20% cheaper than CAO while SBS is more expensive by 5–6 times. Also, the modification process with GB is not complex and can be done in asphalt tanks with mixing apparatus unlike modification with polymers, which needs additional blending equipment [20–21]. Thus, using GB in asphalt modification is more cost-effective than polymers.

Therefore, this research bridged this gap by studying the utilization of GB in three contents (30%, 35%, and 40% by weight) as modifier for LB. The effect of GB stiffness is anticipated to offer an effective solution for using LB in road construction by enhancing the pavement performance especially the resistance against rutting distress and water damage. These distresses are the most prevalent failure types in Iraq roads. They not only reduce the service life of asphalt pavement, but also cause further issues, like premature failure, increased repair rate, raised maintenance costs, and finally reduced ride safety for road users [22]. For example, based on the statistics of the traffic accidents in Iraq, about 6.8% of these accidents are because the hazardous conditions generated from road distresses especially rutting failure [23].

RESEARCH METHODOLOGY

The experimental research had two main aims. The first aim was to assess the physical and chemical characteristics of LB modified with GB and the tests program flowchart for the binder level was outlined in Figure 1a. The second aim was to examine the moisture damage susceptibility and rutting resistance of modified mixture and compare the results with those of control mixture (COA mixture). Figure 1b illustrates the flowchart of the tests program for the mixture level.

Three modified blends were prepared with 30%, 35%, and 40% of GB as partial replacements for base LB, as detailed in Table 1. These blends were designed to examine the stiffening

impact of GB on LB. Additionally, CAO, LB, and GB were tested to compare their properties with modified blends. Three replicates for each sample and average results were recorded. Error bars were used to validate the variability of results.

MATERIALS

For this research, the raw materials utilized in design bitumen mixtures, including COA, LB, GB, coarse and fine aggregates, as well as mineral filler, are presented with all details in Table 2. As it is shown in Figure 2, Mamora Lake is selected to collect LB sample. GB used in this research as a modifier material and it is chosen for its distinct characteristics and potential to enhance the properties of LB. Figure 3 illustrates the physical form of COA, LB, and GB samples.

Also, the aggregates, including coarse, fine, and mineral filler, were tested to ascertain their physical properties according to the requirements limits established by Iraqi specification [24]. The design gradation for the aggregates utilized in this study for surface course Type III-A, as presented in Figure 4.

PREPARATION AND TESTING METHODS

Modification process

Before starting LB modification, measuring the water content in NB samples, particularly in LB, is mandatory. Because NB binders are naturally occurred and combining them with water is expected. Therefore, the ASTM D-95 standard was used to determine the water content, and it was found that the water contents for LB and GB (by the weight of bitumen) are 26% and 0.447%, respectively. Water must be removed to achieve the best compatibility between LB and GB. However, GB was utilized immediately without any prior treatment as its water content was extremely low. In contrast, due to high water content, LB was kept in steel containers (500 ml) and subjected to heat treatment for two and a half hours at 110 °C in controlled oven to remove water, as reported by [7].

To prepare modified binders, the GB concentrations as partial substitutes for 30%, 35%, and 40% of the weight of base LB were blended, resulting in three modified blends (70LB/30GB,

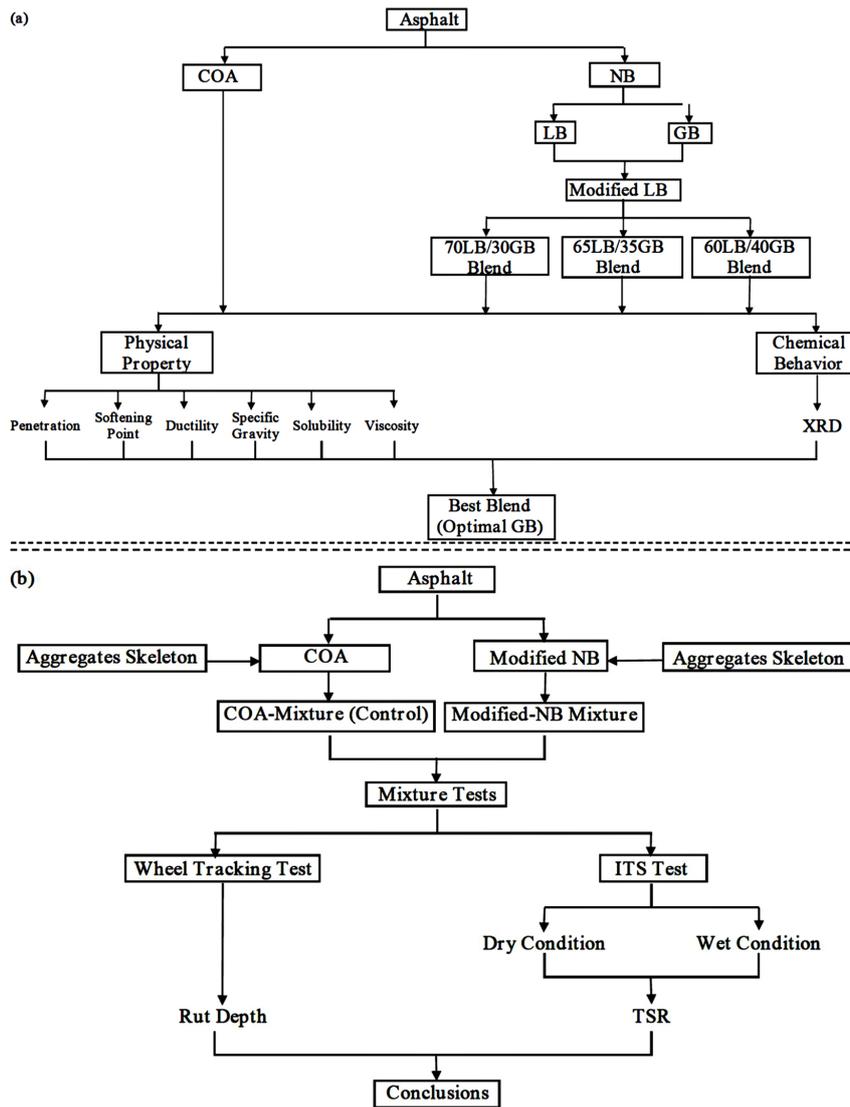


Figure 1. Flowchart of the experimental program: (a) binder level, and (b) mixture level

Table 1. Bitumen blends symbols and modification variables

| Blend symbol | Description |
|--------------|--|
| COA | Crude oil asphalt |
| LB | Raw lake bitumen |
| GB | Raw Gilsonite bitumen |
| 70LB/30GB | Mixing of 70% lake bitumen and 30% Gilsonite bitumen |
| 65LB/35GB | Mixing of 65% lake bitumen and 35% Gilsonite bitumen |
| 60LB/40GB | Mixing of 60% lake bitumen and 40% Gilsonite bitumen |

65LB/35GB, and 60LB/40GB). Before blending, base LB and GB were heated to 135 °C and 165 °C for two hours, respectively, to ensure a flowing condition. For modification, GB was gradually added to LB sample, then the modified

blend was mixed for one hour at a temperature of 180 °C and a rate of 1000 (revolutions/minute) by using laboratory-high shear mixer to achieve uniform blend [21].

Asphalt tests

The physical properties of COA, GB, base LB, and modified LB were assessed using a set of standardized tests. Consistency and temperature susceptibility of asphalt were measured employing the penetration test (ASTM D-5) and the softening point test (ASTM D-36), respectively. The temperature susceptibility of binders was assessed by computing the penetration index (PI) which is obtained using the following equations:

$$PI = \frac{20 - 500A}{1 + 50A} \quad (1)$$

Table 2. Details of materials used in the study

| Material | Type | Supplier | Characteristics |
|-------------------------|-------------------------------------|---|---|
| Crude oil asphalt (COA) | Penetration grade 40–50 | Dora petroleum refinery, located in Baghdad, center of Iraq | Black color and a high viscous nature |
| Lake bitumen (LB) | Soft grade | Mamora lake bitumen, located in Hit city, Anbar, west of Iraq | Thick form, sticky texture and black liquid |
| Gilsonite bitumen (GB) | Lump | Gilsonite mine, located in Ramadi city, Anbar, west of Iraq | Dull black color, breakable material, and high asphaltene content |
| Coarse aggregate | Crushed stone | Nibaie quarry, located in Baghdad, center of Iraq | Rough-surfaced with 95% crushed and the particle size between 4.75 and 12.5 mm |
| Fine aggregate | Natural river sand and crushed sand | Nibaie quarry, located in Baghdad, center of Iraq | Angular grains and the particle size between 0.075 and 4.75 mm |
| Mineral filler | Limestone dust | Lime factory, located in Karbala, center of Iraq | This filler has a specific gravity of 2.7 and 90% of its particles have a size less than 0.075 mm |



Figure 2. Mamora lake bitumen

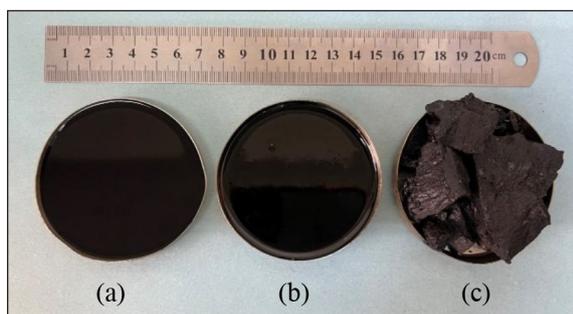


Figure 3. Asphalt types: (a) COA (b) LB, and (c) GB

$$A = \frac{\text{Log } P - \text{Log } 800}{T - T_{RB}} \quad (2)$$

where: P is the penetration in 0.1 mm; T is the penetration testing temperature in 180 °C, which is typically 25 °C; and T_{RB} is the softening point temperature in 180 °C.

Following the standard method ASTM D-113, the ductility test was performed at 25 °C to assess the tensile characteristics of bitumen samples. The specific gravity was measured, according to test method ASTM D-90. Also, method ASTM D-2042 is followed to perform solubility test which is a crucial measurement to assess the content of impurities in NB samples. Additionally, viscosity test was performed to examine the workability by measuring the viscosity at three temperatures, 115, 135, and 155 °C, using a rotational viscometer as per ASTM D-7945. Overall, these tests provided an exhaustive evaluation of the impacts of GB on the physical properties of LB-base binder.

X-ray diffraction test

X-ray diffraction (XRD) technique help to understand NB composition relevant to its application as an asphalt binder in road pavement. It is a non-destructive analytical test utilized to study the atomic structure of crystalline solids, and is one of the common standard laboratory methods. Figure 5 shows the XRD device. This technique depends on the interference of elastically propagated X-ray waves when they collide with atoms arranged in a specific crystal lattice, which leads to the appearance of diffraction patterns that reflect the arrangement of the atoms within the material. The principle of operation of the device depends on fixing the sample and the detector system on angle measuring arms, in addition to using tubes made of copper or silver anodes as a source of X-rays. When X-rays are directed at the material, they propagate at sharp angles according to Bragg’s law, where interference of X-ray waves occurs when the path difference between the propagating waves is equal

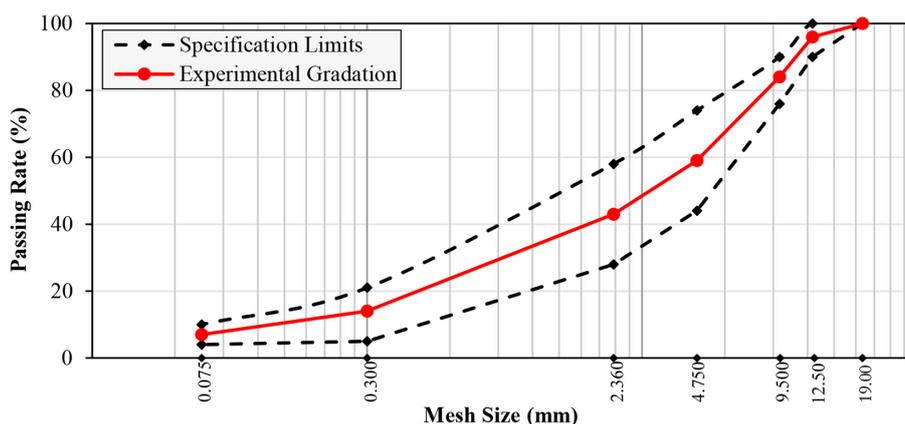


Figure 4. Experimental gradation align with specification limits for surface course



Figure 5. XRD device

to an integer number of wavelengths. The XRD technique can provide quantitative data about the diffraction intensity, while the locations of the peaks and their distribution in the diffraction patterns are analyzed to calculate the crystal size and other structural properties.

The instrument records the diffraction pattern, which appears as peaks at certain angles (2θ), allowing the identification of the crystalline phases in the material shown in the diagram. XRD patterns of asphalt typically show four main peaks, (γ) gamma peak, (002) grapheme peak, (100) peak and (110). These four peaks are detected approximately at $2\theta = 20^\circ, 25^\circ, 44^\circ$ and 80° , respectively. The (γ) peak results from the diffraction of the saturated hydrocarbon in the asphaltene fraction, while the (002) peak arises from the diffraction of the aromatic hydrocarbon in the thick ring. The crystalline structure of asphalt is definitely connected to the peak rate [25].

Indirect tensile strength test

In this research, the resistance of asphalt mixtures against moisture damage distress is assessed by determining the tensile strength ratio (TSR) following the standard method ASTM D-4867. Marshall samples, with diameter of 101.6 mm and height of 63.5 mm, were used in this test. In addition, the samples prepared were with the optimum asphalt content of 5% and 5.3% for COA mixture and modified-NB mixture, respectively. For each mixture, six samples were prepared with air voids between 6% and 8%. These samples separated into two sets: unconditioned set and conditioned set, three samples for each set. For each sample, measure and record the height in mm and the air void in % after sample has reached room temperature (25°C).

The unconditioned set was submerged in water bath for twenty minutes at $25\pm 1^\circ\text{C}$. To compute the indirect tensile strength (ITS), these samples

were placed horizontally on the metal base then tested by applying a vertical loading through two metal strips located below and above the sample until they fractured using a compression machine at a loading rate of 50.8 mm/minute. In turn, the conditioned set were subjected to saturation by using a vacuum pump at a partial saturation level of 55% to 80%. After that, these saturated samples were exposed to one freezing cycle for 16 hours in freezer at -18 °C, then subjected to one thawing cycle for 24 hours in water bath at 60 °C. Before fracturing the samples with the compression machine, the conditioned set were placed in water bath for one hour at 25±1 °C. The specimens after performing ITS test for conditioned and unconditioned sets are presented in Figure 6. TSR is computed by divided the average ITS for the conditioned set (ITS_c) by the average ITS for the unconditioned set (ITS_u). ITS and TSR were calculated by applying Equation 3 and Equation 4, respectively.

$$ITS (kPa) = \frac{2000 P}{\pi T D} \tag{3}$$

$$TSR (\%) = \frac{ITS_c}{ITS_u} \times 100 \tag{4}$$

where: P is the maximum compressive load needed to fracture the specimen (N); T is specimen thickness in mm, and D is the specimen diameter in mm.

Wheel tracking test

The wheel tracking test was adopted in this research to evaluate the resistance of mixture against rutting distress. It simulates the impact of traffic loading on asphalt pavement. The rut depth or depression is measured in this test, which is a crucial performance indicator to evaluate the ability of bitumen mixture to resist permanent deformation over time.

This test was performed on two mixtures: control and modified LB. Therefore, two slabs, measured 300 × 400 × 50 mm, were prepared and designed at optimal binder content of 5% and 5.3% for COA and optimal modified LB, respectively, following the standard requirement EN 12697-33. A Dyna compaction device was used to prepare the slabs by compacting the loose asphalt mixtures through applying specific loads. The samples were left in the slab mold to cool at standard temperature (25 °C) for 24 hours and then extracted.

According to the standard method EN 12697-22, the wheel tracker or Dyna-Track was used to measure the ruts depth at 60 °C by performing a standard wheel load of 80 psi (800 N) that moving back and forth across the surface of slab sample for 20,000 passes (10,000 cycles). Before performing the rutting test, the slab was insulated in a wheel tracking machine for 4 hours at 60 °C. Figure 7 shows the procedure of this test.

Finally, the dynamic stability (DS) of the slab specimen was computed. DS is a common parameter utilized to measure the resistance of bitumen

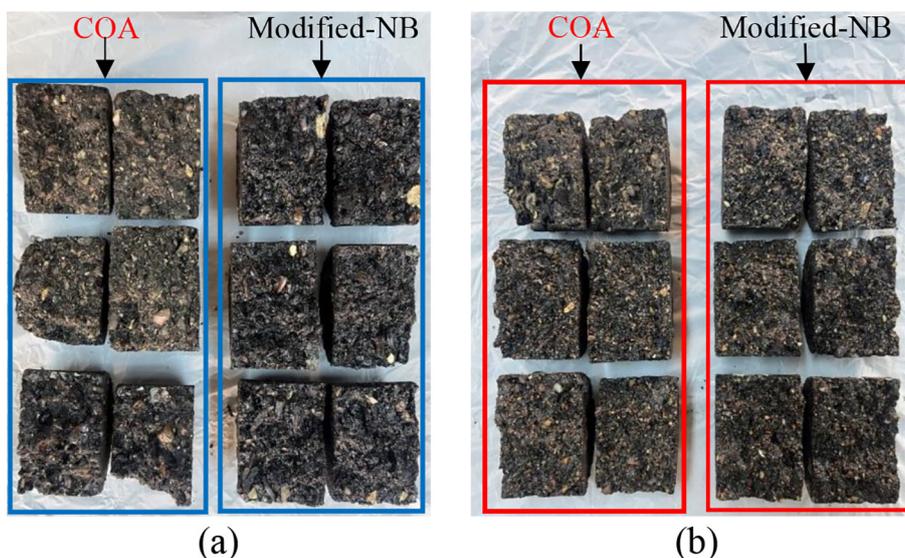


Figure 6. Specimens after ITS test: (a) dry condition, and (b) wet condition

mixes against permanent deformation resulting from repeated traffic loads. It is expressed as the number of cycles required to induce a permanent deformation of 1 mm during the last 25% of the wheel tracking test. This parameter is essential for evaluating the behavior of asphalt mixture, as it is directly related to its resistance to rutting, a major defect affecting road safety and service life [26]. Equation 5 was used to calculate DS value [27].

$$DS \text{ (cycle/mm)} = \frac{t_2 - t_1}{d_2 - d_1} \times N \times C_1 \times C_2 \quad (5)$$

where: d_1 is the rut depth at time t_1 in mm, t_1 is the time in minutes at 7500 cycles; d_2 is the rut depth at time t_2 in mm, t_2 is the time in minutes at 10,000 cycles; N represents the speed rate of the wheel tracking and it is around 42 cycle/minute; C_1 is the machine type parameter, which is typically 1.0, and C_2 is the specimen coefficient, which is typically 1.0.

RESULTS AND DISCUSSION

Physical properties of asphalt

The physical properties of COA, LB, GB, and modified blends are outlined in Figure 8. It was noted that the inclusion of GB significantly

impacts the physical properties of LB-modified binder. The outcomes obtained from consistency tests confirm that LB has a soft grade, as its penetration and softening point were 132 and 36.2 °C, respectively. This bitumen grade is not compliant with the specification limits of asphalt binders for paving, as listed in Table 3, which specified the penetration range between 40 and 50 (0.1 mm), thus, base LB cannot be used as a sole binder in asphalt paving works. Therefore, to enhance the LB characteristics for asphalt pavement uses, it is modified with GB at three partial replacements. GB has zero penetration and 158.5 °C softening point, consequently, these properties make GB a promising modifier for LB by improving hardness and exhibiting environmental advantages.

The penetration results demonstrate significant changes since the penetration measures for NB-modified blends are 51, 43, and 31 for 70LB/30GB, 65LB/35GB, and 60LB/40GB, respectively, compared to 132 for unmodified LB, as provided in Figure 8a. The lower penetration values for modified blends indicate a notable increase in stiffness, mostly because of high contents of GB, which performs as a reinforcement agent for the soft consistency LB. This rising in hardness improves the strength of LB against elastic deformation, making it more acceptable and stable for high-temperature uses. Likewise,

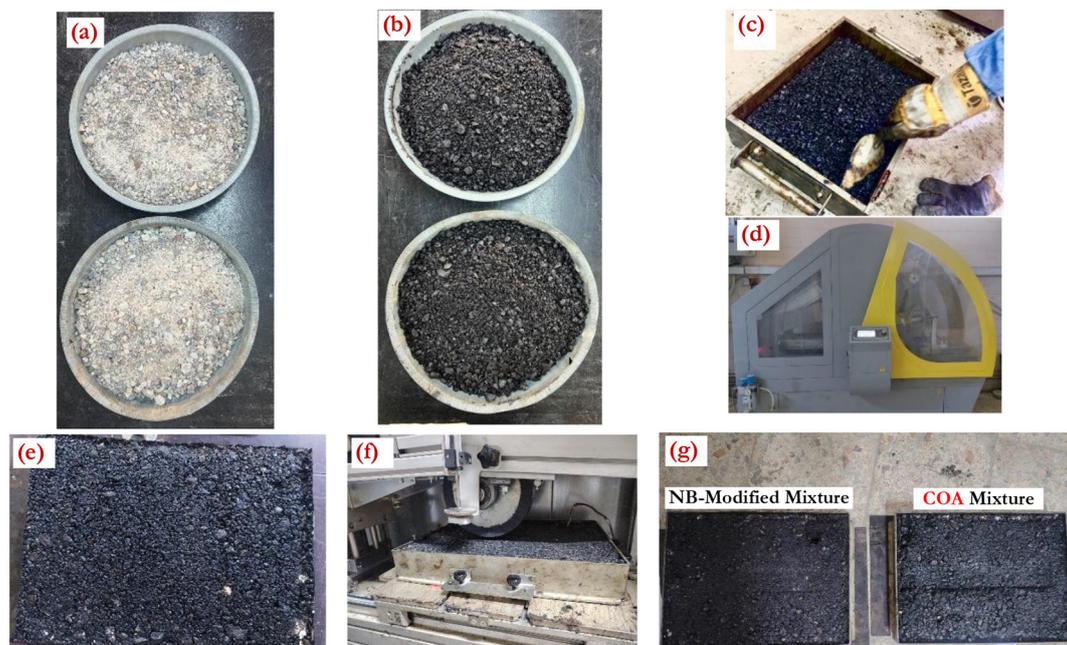


Figure 7. Wheel tracking test procedure: (a) aggregate mix; (b) loose-asphalt mixtures; (c) preparing slab specimen; (d) roller compactor; (e) compacted slab; (f) compacted slab in wheel track machine; and (g) slabs after test

compared to base LB, a considerable improvement in softening points was observed in modified blends (Figure 8b).

In addition, sensitivity to temperature changes were evaluated for modified blends through determining the PI, which is a heat sensitive index and a dimensionless number. PI can be computed based on the values of the penetration and the softening points [28]. The results of PI, as presented in Figure 8c, indicates that base LB has lower PI (-3.324), implying more sensitive to temperature. In turn, the PI for the GB sample cannot be computed since it has zero penetration. The modified blends have higher PI values than base LB, making their consistency more stable and less susceptible to temperature changes. Also, it can be observed that 65LB/35GB and 60LB/40GB blends have the PI values higher than that of COA and this means that these modified binders can be good alternatives to COA in paving applications. The increase in PI values is related to the presence of sulfoxide groups in GB,

making modified LB more resistance against temperature susceptibility [29].

Obviously, according to the ductility test results that presented in Figure 8d, a noticeable alteration in the flexibility of LB with the addition of GB was achieved. The modified blends exhibit considerably lower ductility, compared to 172 cm for unmodified LB. This is due to the hardening effect of GB, which strengths and improves the stiffness of the soft consistency LB. The ductility values for 70LB/30GB, 65LB/35GB, and 60LB/40GB blends are 131 cm, 109 cm, and 93 cm, respectively, progressively reducing with raising GB ratio. This reduction indicates that the high content of asphaltene in GB contributes to raising hardness and decreasing the ductility of the modified samples. Nevertheless, the ductility values for 70LB/30GB and 65LB/35GB blends complied with minimum requirement of asphalt binder used in paving works (see Table 3).

Trichloroethylene is an organic solvent used in solubility test to measure the amount of

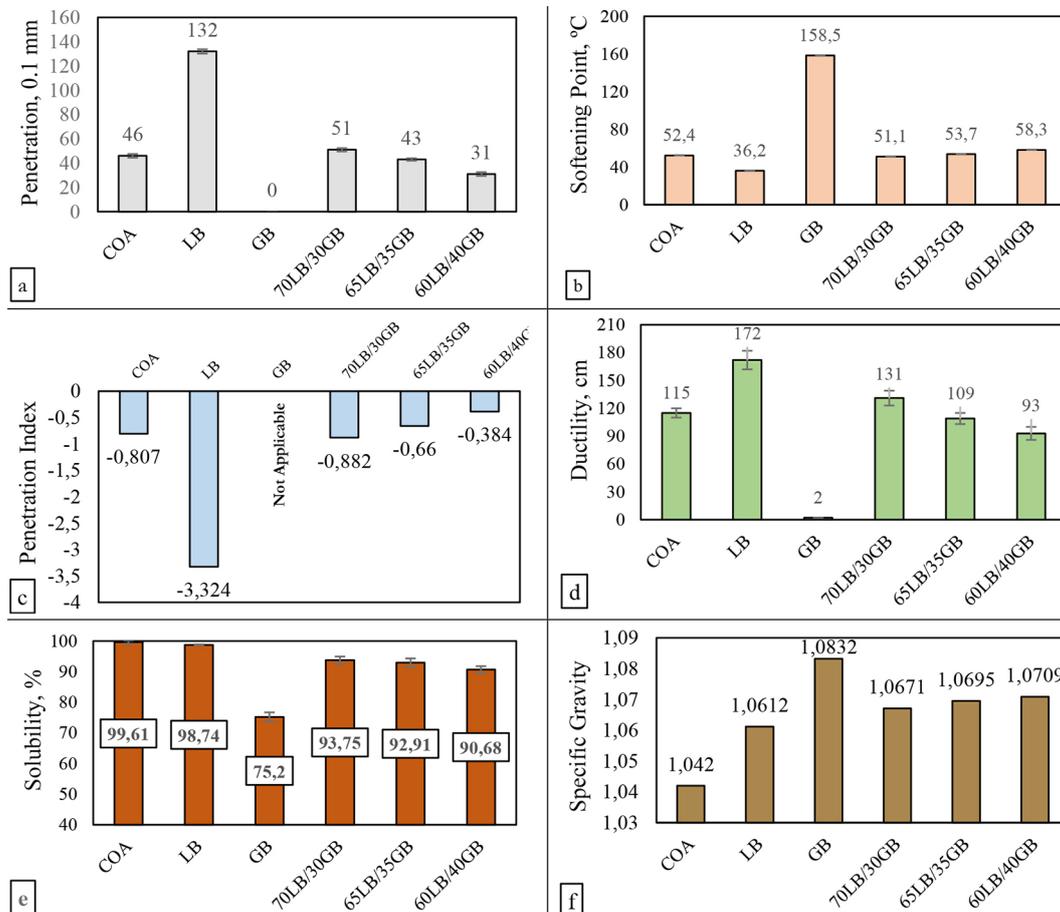


Figure 8. Physical properties results for different tested binders: (a) penetration, (b) softening point, (c) penetration index, (d) ductility, (e) solubility, and (f) specific gravity

insoluble material, then to determine the purity of bitumen. Purity level can significantly affect the performance of binder. The solubility percentages (Figure 8e) exhibit a significant decrease, with modified blends demonstrating reduction of 5% to 8% compared to unmodified LB. This is due to the impurity of GB which has a lower solubility of 75.2%. In general, the NB samples are less soluble in the trichloroethylene solvent, because they are naturally occurring, as well as the content of ash and insoluble materials in their structure is anticipated, while COA has a 99.61% solubility, satisfying the minimum requirement of 99% and indicating its high purity. To clarify this, the quality of COA, especially purity property, is controlled through careful monitoring of the distillation process of crude oil.

In general, NB binders have a relatively higher specific gravity than the COA binder. According to Figure 8f, the specific gravity outcomes exhibit a gradual increase in the density of all modified blends compared to base LB, which has a specific gravity of only 1.0612. Notably, 60LB/40GB has the highest GB ratio, demonstrates the higher specific gravity, with an increase of nearly 0.91%, reflecting the impact of the higher molecular weight of GB. This reflects the contribution of GB to LB density through its high carbon content and complex molecular structure.

Overall, considering all physical properties results, it can be stated that the 65LB/35GB blend (modified LB with 35% of GB) is the optimal blend, since its properties consistent with that of COA.

Workability assessment

The viscosity test was performed to assess the workability of bitumen by measuring its resistance to flow. The viscosity results for COA, base LB, and modified LB with 30% (70LB/30GB blend), 35% (65LB/35GB blend), and 40% (60LB/40GB blend) of GB, were presented in Figure 9. Additionally, the individual error bars indicate one standard deviation. As it is clearly seen, the viscosity values measured at 115 °C, 135 °C, and 155 °C for COA were

higher than those of base LB, attributing to its soft structure. For instance, at 135 °C, the viscosity of COA and base LB were 722 mPa.s and 481 mPa.s, respectively, meaning that the viscosity of COA was greater than that of LB by 50%.

After modification, the viscosity of LB increases consistently with the addition of GB at all tested temperatures. Particularly, the blend with high GB content (60%) at 135 °C has a rotational viscosity more than base LB by 4.3 times. This is because GB acts as a stiff modifier and enhances the fluidity of LB by stiffening its structure. In addition, LB exhibits viscosity higher than COA after modification with GB at all temperatures. Nevertheless, the viscosity of modified LB binders (70LB/30GB, 65LB/35GB, and 60LB/40GB) at 135 °C remains lower than the permissible viscosity requirement of 3000 mPa.s. This complies the requirements of the Superpave specification (ASTM-D6373). Hence, modified LB ensure sufficient fluidity and provide acceptable workability for asphalt pavement construction applications.

A higher content of GB (more than 35%) can significantly raise the stiffness and viscosity of LB, thus, the mixing and compaction temperatures must be raised to comply with the design requirements during pavement construction. This leads to premature aging of LB and can increase the construction costs, energy consumption, and harmful emissions.

Statistical analysis

One way ANOVA was conducted to assess the statistical significance for the independent variable (binder type) on the dependent variables (penetration, softening point, and viscosity). The null hypothesis suggests that there is no difference in any of the dependent variables across the investigated binder types. A 95% confidence level was chosen for the analysis, where p value less than 0.05 indicates a significant difference in the dependent variables leading to the rejection of null hypothesis, in other words, the variations observed in the binder performance were not due to random experimental error but revealed differences due to the binder composition. The

Table 3. Specification limits for the physical properties of asphalt

| Property | Penetration at 25 °C [0.1 mm] | Softening point [°C] | Penetration index [unit less] | Ductility [cm] | Solubility [%] | Specific gravity [unit less] |
|--------------|-------------------------------|----------------------|-------------------------------|----------------|----------------|------------------------------|
| Limits, [24] | 40–50 | Not limited | Not limited | ≥ 100 | ≥ 99 | Not limited |

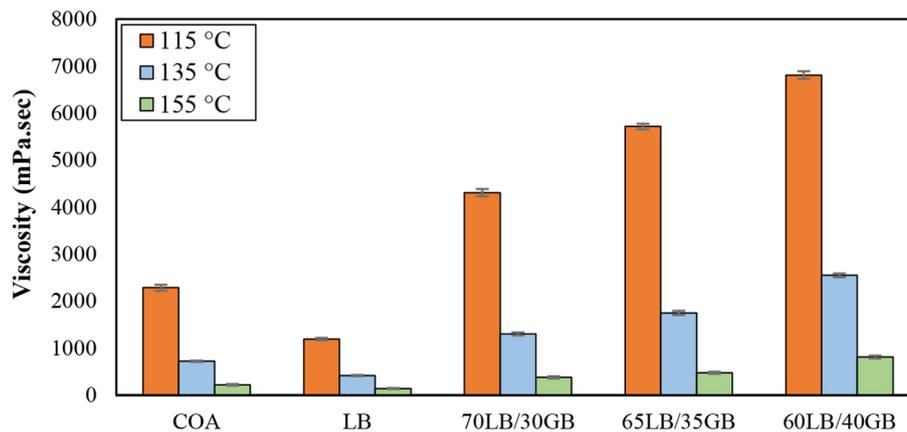


Figure 9. Viscosity data for COA, base LB, and modified LB

results of ANOVA analysis presented in Table 4 confirm that the p-values for all the three performance parameters were below 0.05, confirming that the binder type had a statistically significant effect on each physical property. Moreover, as per the presented data in Table 4, the lowest p-values (or highest F-values) were recorded for the softening point, indicating a notable difference between the binders containing GB and those containing LB. The obtained findings suggest that binder composition strongly influences consistency, temperature susceptibility and workability (flow characteristics).

XRD spectra analysis

XRD was conducted to investigate the crystallographic structure of LB and GB, then it was compared with COA, focusing on the relative intensity, width (d), and position of peaks. Typically, (d) value pertaining to the peak width, signifying the crystallite size. The presence of a peak revealed that the atom was organized in a periodic array, thus confirming its crystalline structure. The periodic arrangement of atoms was capable of diffracting X-rays, resulting in a diffraction pattern, and conversely. The variation in the diffraction pattern produced the peak in the graph. To clarify the differences in the peaks of NB and COA specimens, the graph patterns of

the COA, LB, and GB generated from the XRD test are presented in Figure 10a.

The GB specimen has a different graph pattern than the LB and COA specimens which had many sharp, narrow, and distinctive peaks with low counts intensity. Obviously, the maximum 2θ value (peak) was existed at 31.02° with d equal to 2.881 nm, and in range of 2θ between 30° and 100° , there are a crystal phases in the GB structure. Thus, this bitumen was identified as possessing a fully crystalline structure, as the presence of peaks was predominant and was notably diminished. Meanwhile, LB specimen has a pattern approximately similar to that of COA. To clarify, it can be noticed that the XRD graphs pattern for the COA and LB specimens have a remarkable consistency, since only one large and broad peak was observed and existed in each specimen pattern (γ diffraction peaks) which appeared at 21.32° 2θ with $d = 4.165$ nm for COA specimen, and 20.35° 2θ with $d = 4.361$ nm for LB specimen, clearly indicating the amorphous characteristics of the material. The γ peak or band attributes the arrangement and the packing distance between naphthenic rings and aliphatic chains within the binder microstructure [30]. In addition, the XRD pattern, indicative of a mostly amorphous structure, is characterized by broad humps instead of distinct peaks. Also, the elevation of the peaks indicates the presence of amorphous shadows in the

Table 4. ANOVA statistics for the binder types

| Performance parameter | F-value | p-value | Significance |
|-----------------------|-----------|------------------------|--------------|
| Penetration | 4146.31 | 5.49×10^{-19} | Significant |
| Softening point | 57,963.68 | 7.39×10^{-26} | Significant |
| Viscosity | 2050.51 | 1.60×10^{-14} | Significant |

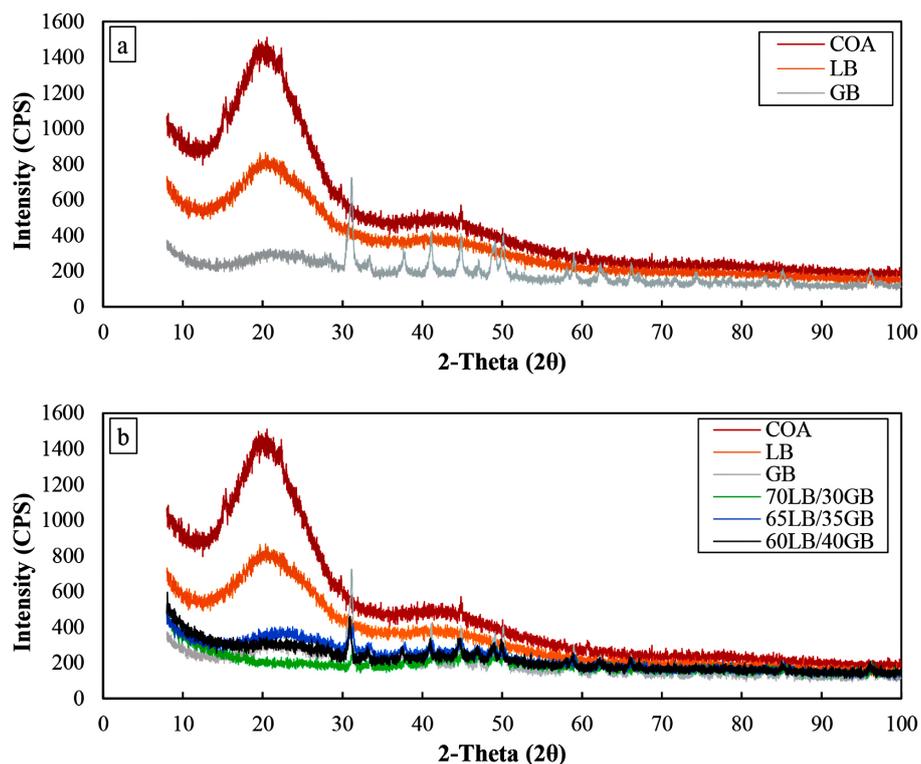


Figure 10. XRD spectrums (a) COA and virgin NB specimens, (b) COA, virgin NB, and modified NB specimens

structure of LB within the 2θ range of 35° to 55° , resembling that of COA. The peak in this range is known as (100) band which links to the aromatic layers within the asphaltene fractions [30]. The presence of (γ) and (100) bands is a clear evidence of the disordered and amorphous matrix of COA and LB. However, a very minor crystal phase is present in the COA structure at 2θ roughly 45° and 65° , whereas the LB specimen exhibits a flat pattern in its profile.

The extensive and planar trend devoid of peaks suggests an amorphous structure resulting from the chemical atomic configuration in the asphalt binder. The crystalline structure illustrated that the ions, molecules, or atoms inside the material were organized in a periodic array, whereas the amorphous structure signified an irregular atomic arrangement.

The XRD graphs of modified specimens (70LB/30GB, 65LB/35GB, and 60LB/40GB) were presented in Figure 10 b. It is clearly visible that these specimens have a crystalline structure similar to that of GB, since sharp and narrow peaks with low intensity appeared in their patterns. In conclusion, the COA and LB specimens possessed the macromolecular structure, since two separate amorphous phases were observed

and sharp as well as large peaks were not recorded. The molecule in the asphalt material was not organized in a periodic array, resulting in the absence of light diffraction and consequently a graph devoid of peaks. In addition, GB has an essential impact on the chemical behavior of LB.

The XRD analysis underscores the interaction between the crystalline and amorphous regions of the COA and NB specimens, significantly influencing their mechanical and physical properties. Amorphous bitumen like COA and LB offers flexibility and stability particularly at lower temperatures, while crystalline bitumen like GB is a stiff and dense asphalt; moreover, it provides rigidity and hardness. The information regarding the differentiation between crystalline and amorphous content facilitates the prediction of NB behavior in diverse situations, providing valuable insights for their application in fields such as construction and research.

Moisture-induced damage susceptibility

Moisture damage is the deterioration of the asphalt mixture due to weakening of the bond between the asphalt molecules and decreased cohesive strength. Considerably, this weakness

leads to decreased adhesion between the bitumen and the aggregate, which in turn leads to separation of the mixture and consequently decreased stability, strength, and stiffness due to decreased cohesion [31]. Therefore, it is necessary to pay attention to the negative effects of water and implement the strategies to minimize them while at the same time maintaining the strength, longevity, and ability to withstand different conditions of the mixture [32].

The outcomes of the ITS test are provided in Table 5. It is clearly that the average ITS values of control mixture are 1169 kPa and 953 kPa for unconditioned and conditioned sets, respectively, and these values are less than those of modified-NB mixture. This outcome complies with the early investigations results [33, 34]. The ITS test showed that GB positively affected the modified LB strength under both conditions (dry and wet), causing an increase in the TSR. Higher TSR values means more resistance to moisture damage. However, TSR for both mixtures is within the required limit, 80% minimum TSR for hot asphalt mixture.

The improvement in TSR is because NB-modified binder was more effective and efficient than COA in producing mastic cover aggregates resulting in improved the adhesion between the aggregate particles and bitumen, making asphalt mix less sensitive to moisture impact. Thus, it can be concluded that GB enhances the adhesion and the cohesion bonds of LB and acts as an anti-stripping agent for aggregates. This finding is aligned with the outcomes of the previous research [35]. Nevertheless, it is expected that a higher dosage of GB (such as 60LB/40GB blend) may negatively affect the adhesion performance of the modified LB due to brittle behavior, flow ability, and fracture mechanism of GB

as reported by the early study [36]. Additionally, although enhancements in the TSR values for the LB-modified mixtures were achieved in previous studies using thermal treatment and modified with LSF [7, 16, and 18], they were not as significant as GB additive. In addition, these modifications can be more expensive than GB.

Rutting resistance

Figure 11 shows the plots of rutting depth with loading cycles for COA (control) and NB-modified mixes. It was observed that the rut depth values increase progressively along with the load cycles. The final rut depth after 10,000 loading cycles would be used as an indicator to evaluate the rutting resistance of asphalt mixtures. The results of rut depth from the wheel tracking test indicated that GB enhances the resistance of NB-modified mix against rutting distress. As it is clearly seen, the NB-modified mixture has lower rut depth than that of the COA mixture, indicating superior resistance to rutting distress.

Figure 12 reflects the change in the final rut depth and DS between the COA and NB-modified mixtures. In addition, it is interesting to note that the final rut depth for the NB-modified mixture was only 2.36 mm, which is relatively small and considered acceptable. The DS of NB-modified mixture is substantially higher than that of the COA-mixture, which is approximately 42% more than the DS of the the COA mixture, supporting the findings of rut depth.

The significant reduction in the rutting susceptibility for the NB-modified mixture is possibly attributed to three main reasons. Firstly, the high concentration of asphaltenes in GB improves the stability of LB at high temperatures [37]. Secondly, the presence of polar functional and long alkyl

Table 5. ITS and TSR results

| Set type | Control mixture (COA mixture) | | | NB-modified mixture (65LB/35GB mixture) | | |
|---------------|-------------------------------|-------------------|---------------------------|---|-------------------|--------------------------|
| | ITS [kPa] | Average ITS [kPa] | Standard deviation, [kPa] | ITS [kPa] | Average ITS [kPa] | Standard deviation [kPa] |
| Conditioned | 948 | 953 | 5.69 | 1098 | 1107 | 9.54 |
| | 959 | | | 1106 | | |
| | 951 | | | 1117 | | |
| Unconditioned | 1162 | 1169 | 7.02 | 1187 | 1192 | 4.73 |
| | 1170 | | | 1194 | | |
| | 1176 | | | 1196 | | |
| TSR [%] | 81.52 | | | 92.84 | | |

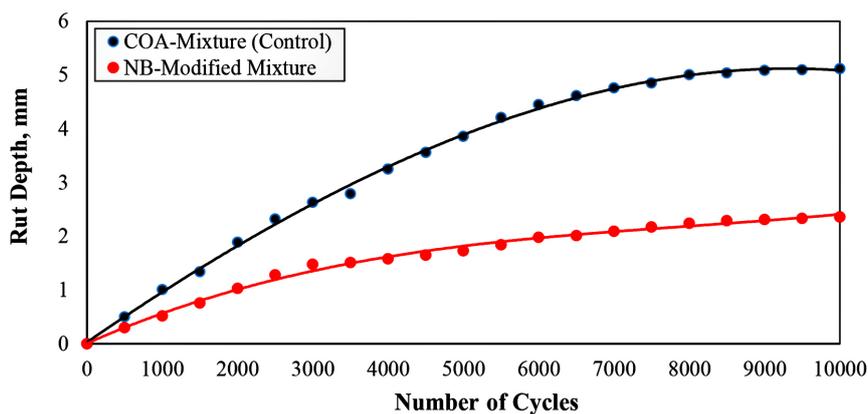


Figure 11. Correlation between rut depth and loading cycles for control and NB-modified mixtures

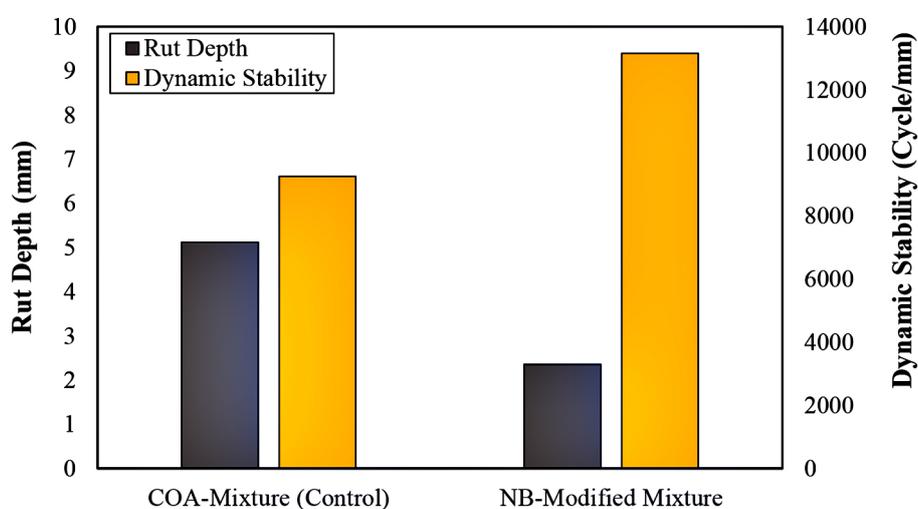


Figure 12. Relation between rut depth and DS for the COA and NB-modified mixtures

groups in GB strengthens the LB structure. These chemical groups enhance the ability of GB to interact and become compatible with LB, contributing to improved cohesion and adhesion bonds in LB matrix [38]. Finally, the softening point of modified NB is greater than that of COA by 1.3 °C and positively affects the high-temperature stability of bitumen mixture. The enhancement in the resistance of NB mixture to rutting is consistent with the conclusions of the early investigation [39].

CONCLUSIONS

A laboratory research has been performed to assess mainly the moisture damage susceptibility and rutting resistance of the modified LB mixture. On the basis of the results and discussion on how GB affects the LB properties, many conclusions have been drawn.

The softening point of the modified LB samples increased when the GB content raised. For instance, incorporating 35% GB revealed a noticeable enhancement in flow resistance of LB since the softening point increased by 17.5 °C. At the same time, adding GB leads to LB stiffening. Additionally, the PI values demonstrated that the modified LB samples are less susceptible to temperature and more favorable for hot weather regions like Iraq. GB can significantly decrease LB ductility due to its stiffening impact and increase the specific gravity of LB binders since GB has a higher specific gravity (1.0832). Considering all properties, the 65LB/35GB blend achieved the best results.

The viscosity of 65LB/35GB (optimal blend) was 1750 mPa.s at 135 °C, which is greater than that of unmodified LB by 2.6 times. Similarly, at 155 °C, the rotational viscosity increased from 142 mPa.s to 478 mPa.s. At the same time, it was

increased from 1193 to 5715 mPa.s at 115 °C. The significant rise in rotational viscosity can be associated to the stiffening effect of GB on the LB. Also, the interaction between LB and GB improved the molecular resistance to flow. Ultimately, the GB modifier was effective in improving the workability and decreasing the fluidity of LB.

On the basis of the XRD analysis, GB has a crystalline structure since many sharp peaks are observed in its pattern. Also, a significant relatively flat XRD pattern graph approximately without sharp peaks was recognized and exhibited for the entire pattern in the COA and LB specimens. This means that COA and LB have poor crystallinity and are predominantly amorphous. This amorphous characteristic, while compromising some structural rigidity relative to more crystalline substances, enhances the flexibility and resilience of the material at lower temperatures. The similarity in the XRD patterns can be ascribed to chemical compatibility between COA and LB. Also, the (002) peak was not observed in the LB graph and this is an evidence that this bitumen has low contents of aromatic fraction, unlike the GB specimen.

Compared with the control mixture, the NB-modified mixture had higher ITS magnitudes especially for conditioned set (wet state), suggesting that GB had great effect on the resistance against moisture damage.

The outcomes of wheel tracking test indicate that the NB-modified mix achieved lower rut depth and higher DS compared to the COA-mix, reflecting a greater ability to resist the deformations caused by traffic loads and environmental conditions. Therefore, the NB-modified mix is more recommended in highly rutted regions.

On the basis of test outcomes and compared to the control mixture made with COA, the NB-modified mixture demonstrated higher performance, especially in terms of moisture damage sensitivity and rutting resistance. However, incorporating more than 35% GB may adversely affect the workability and resistance against low temperature (thermal) cracking for the asphalt mixture due to the excessive increase in the viscosity and stiffness of the LB binder.

In conclusion, GB is an economical choice, since it is less expensive compared to other additives and modifiers (as discussed in the literature) and appropriate alternative for improving the properties of LB. In addition, utilizing local natural materials, such as NB in pavement

construction supports the sustainable development goals by saving energy, urging responsible production and consumption, reducing transportation emissions, and providing new local jobs. Correspondingly, selecting these materials supports local economy and reduces the demand for new resources.

For future studies, it is recommended to inspect the fatigue and thermal cracking performance of modified LB, since adding GB increased the stiffness and reduced the elasticity of mixture at low temperatures. In addition, further research is needed to assess the environmental impacts and the economic benefits of using NB in pavement sector. Finally, it is suggested to investigate the correlation between the behavior of the NB-modified pavement in the field and the laboratory test results to enhance the accuracy of performance predictions.

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