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Laboratory Investigation of Micronized Lomashell Powder Effects on Asphalt Binder and Mix Performance

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Abstract

Lomashell, a mineral stone derived from oyster shells and skeletons, is widely available in Iran and across the globe. Typically used for livestock feed due to its high calcium content, its production generates a considerable amount of discarded fine grains. This research focused on incorporating micronized Lomashell as additive for asphalt pavement to enhance performance and environmental sustainability. The impact of this powder on the rheological and physical properties of two common asphalt binders was evaluated. Moisture resistance, rutting, and permanent deformation of Lomashell-enhanced asphalt mixtures were also examined. The results indicate significant improvements in rheological properties and dynamic shear rheometer parameters upon Lomashell addition. Moisture sensitivity was enhanced, as demonstrated by the indirect tensile strength test. Adding 7% of this material to the asphalt mixture enhanced indirect tensile strength by 12% compared to control. Furthermore, utilizing the Hamburg wheel-tracking device (HWTD), it was observed that inclusion of this powder enhanced resistance against permanent deformation, as evidenced by the rutting resistance index (RRD) values. Effective high-speed shear mixing is emphasized for binder modification, as revealed by scanning electron microscopy analysis. These findings highlight Lomashell's positive influence on the overall performance and durability of the asphalt mixtures, reducing rutting and enhancing resistance against permanent deformation. Utilizing this powder as asphalt additive holds promise for improving functionality and addressing environmental concerns, contributing to sustainable infrastructure development.

Keywords: dynamic shear rheometer test; hamburg wheel-tracking device; indirect tensile strength (ITS); micronized lomashell; scanning electron microscopy (SEM).

Introduction

Lomashell (Shelly limestone) is a type of cream to white colored mineral and is formed from the accumulation of stone and the hardening of shells and skeletons of oysters and huge layers of scallop fossils. Basically, this mineral is a limestone made from oyster fossils. Unlike other types of limestone around the world that originated mainly through the lithification of loose carbonate sediments, it is an uncommon variety of limestone made from the shells of extinct mollusks and other marine animals and with a destructive origin. The destructive particles that make up this stone are composed of the crushed remains of shells of marine organisms, which often contain calcite or aragonite [1]. In the third geological period (Cenozoic), from the accumulation of the remains of the bodies of marine organisms, such as corals, scallops and mollusks, short and long walls with valuable deposits were created in the depths of the sea. Over time, with the retreat of the sea, these walls appeared on the surface of the earth and were eroded by wind and rain over millions of years to provide humans the opportunity to utilize the valuable stone resources in them. Significant and viable deposits of Lomashell rocks, totaling more than one billion tons, are situated along both the northern and southern coastlines of Iran. Figure 1 shows the geographical location of one of the Lomashell mines on the southern coast of Iran [2]. Moreover, similar deposits can be found across the globe, including in regions like Florida and North Carolina in the United States, as well as along the coastlines of Australia, Brazil, Mexico, and the United Kingdom. Due to its calcium content, Lomashell is used in the livestock and poultry industries, especially poultry farms.



Figure 1 Geographical location of Lomashell mine in the south of Iran [2].

After extraction and processing, due to the lack of toxic elements and the abundance of calcium carbonate in it compared to limestone, this mineral rock is mainly used in the form of granules with sizes of 3 to 5 mm for consumption in the livestock and poultry feed industries. Particles with dimensions below 1 mm are deposited without special use, while laser granulation analyses performed on these materials have shown that a large portion of these materials are below 500 microns. Due to a lack of use, the majority of this precious material is currently disposed in landfills. Asphalt cement is a complicated chemical substance with viscous and elastic characteristics that change with temperature and loading time. Traditional pavement materials can hardly meet the road traffic load requirements, which are increasing day by day, or resist harsh weather conditions and repeated loading. Hence, ensuring safety, reliability, and environmental compatibility necessitates the utilization of additive materials of exceptional quality [34]. As a result, over time, different types of modifier additives have been introduced by scientists, to boost pure bitumen's efficiency. The characteristics of the bitumen and asphalt mixture can still be improved by using conventional modifier additives. These properties include moisture sensitivity, rutting, fatigue life, etc. However, due to the rapid development of material technology, researchers are interested in the application of new materials for the modification of binder and mixtures, especially waste and eco-friendly materials [5-7]. For this reason, many researchers have recently been drawn to the idea of incorporating biowaste materials into asphalt. Many attempts have been made to utilize waste products from various sources to use as modifier or filler in the production of hot mix asphalt. Materials that include waste from construction and demolition sites have been considered, e.g., coal waste ash and rice husk ash [8], waste glass [9], eggshell [10], coconut shell [11], sea and fish shell [12], palm shells [13-14], etc. It has been discovered that these materials include recoverable fractions that could be valuable as pavement material, making it less expensive to maintain streets and highways, use less energy, and save important landfill space if these materials are removed from the waste stream and recycled for use in highway applications.

The use of biowaste materials in micro and nano sized form has great impact for the advancement of asphalt pavement technology in the fields of materials design, construction, characteristics, laboratory tests, and modeling. The micro and nano particles needed for pavement materials should be non-hazardous, low-cost products that are widely available in high volume [15-16]. Micro and nano scale materials have become increasingly popular due to their special characteristics, such as low sensitivity to high temperatures, high flexibility, large surface area, strong resistance to strain, and low electrical resistance. As a result, researchers and engineers have begun to incorporate this technology into pavement materials as well as modified bitumen and asphalt mixtures containing a variety of particles [17-18]. Studies have shown that nanomaterials derived from bio-waste have the ability to enhance the cohesion and adhesion qualities of asphalt as well as the attributes of bitumen to impede rutting and fatigue failure [19]. Another study [20] looked at the effect of eggshell powder on asphalt mixtures and discovered that the resilient modulus and dynamic creep values for SMA produced by 4% eggshell powder were the highest and lowest, respectively. Another study [21] examined the utilization of nano sized agricultural waste ash from three sources: rice husk, sugar cane bagasse, and wheat straw ash. The results revealed that these materials were found to be superior in terms of moisture damage and rutting performance when compared to other materials. A study on the use of nanowood ashes [22] found that it led to a marked improvement in terms of rutting and moisture resistance performance but had little impact on fatigue resistance when added to modified materials with higher concentrations. In another study [23], the effect of microsilica on asphalt was investigated, and the outcomes demonstrated that the addition of 6% microsilica ameliorated the physical characteristics of asphalt and improved its characteristics in hot weather

conditions. In another study [24], nano-CaCO₃-modified mixtures outperformed the control samples in terms of fatigue and rutting performance as well as moisture damage resistance.

The possible use of micronized recycled polypropylene (RPP) to enhance asphalt performance was evaluated in Ref. [25], which showed that RPP enhanced the water resistance of the asphalt binder. Another study [26] utilized groundnut shell ash (GSA) as a modifier for asphalt binder. The findings revealed that the asphalt binder modified with a maximum of 10% GSA was superior to unmodified asphalt in terms of fatigue and thermal cracking. The modification also caused the penetration grade to drop and the ductility to increase as well as improving the viscosity, softening point, and rutting factor.

The goal of the present preliminary investigation was to evaluate for the first time the viability of using Lomashell as a performance-improving addition to asphalt pavement. On this basis, asphalt behavior was studied in relation to the impact of this powder. The main issue with employing bio-waste materials as modifier in the bitumen matrix is its poor adhesive bonding. One strategy for enhancing the asphalt mixture's resistance to fatigue, rutting, and thermal cracking is asphalt binder modification. The majority of additives reduce rutting but have little effect on fatigue resistance and, in particular, resistance to low temperature cracking. In this research, by considering the huge amount of available Lomashell stone and the fact that only granule sizes of this material between 3 and 5 millimeters are available for use in the livestock and poultry feed industries, and smaller grains with dimensions below one millimeter have no special use, the novel concept of employing waste Lomashell to increase asphalt's efficiency is proposed, addressing the issues of resource waste and environmental pollution. The use of Lomashell powder in micronized dimensions can be effective in improving the physical, rheological, and mechanical properties of binder and asphalt, so we decided to use this waste material in our research. Most previous research was done on micro and nano materials, and bio-waste materials that do not have abundant and cheap resources. This research used micronized powder of Lomashell, which is a stone with a naturally destructive origin and consists of the crushed remains of shells of marine organisms.

Materials and Methodology

Asphalt Binder

Throughout this study, two types of binder commonly used in various regions, AC 60-70 and AC 85-100, were used for the control samples as well as for modified bitumen production. Table 1 lists the specifications of the base bitumens.

Property	Test method	AC 85-100	AC 60-70
Specific gravity at 25 °C (gr/cm ³)	ASTM D70	1.01	1.02
Viscosity at 135 °C (mm ² /s)	ASTM D2170	255	420
Penetration at 25 °C, 100g, 5 s (deci-mm)	ASTM D5	95	61
Softening point, ring and ball (°C)	ASTM D36	45	50
Flash point, Cleveland open cup (°C)	ASTM D92	296	300
Ductility at 25 °C at 5 cm/min (cm)	ASTM D113	110	120
Solubility in trichloroethylene, (%)	ASTM D2042	99.6	99.6
Mass loss upon heating (%)	ASTM D6	0.05	0.03

Table1 Specifications of the base binders.

Aggregate

The aggregates utilized in this study were rated using the FHWA 0.45 Power gradation, which is shown in Table 2 and Figure 2 [27].

Sieve(mm)	19	12.5	4.75	2.36	0.3	0.075
Lower–upper limits	90-100	90	-	23-49	-	2-8
Passing (%)	95	90		43		6

 Table 2 Aggregate gradation.



Figure 2 Gradation graph.

Lomashell (Shelly limestone)

Lomashell, or Shelly limestone, refers to a mineral stone that is formed by the accumulation and hardening of stones and the shells and skeletons of oysters. A sample of Lomashell rock and its micronized powder is shown in Figure 3.



Figure 3 Lomashell rock and its micronized powder.

The chemical composition, physical characteristics and a scanning electron microscopy (SEM) image of Lomashell are provided in Tables 3 and 4 and Figure 4.

Chemical	Lomashell
compounds	powder
CaCO ₃	93.1
SiO ₂	1.18
Al2O ₃	0.42
MgO	0.83
Fe ₂ O ₃	0.31
K ₂ O	0.20
MnO ₂	0.22
P_2O_5	0.74
SO ₃	0.15
TiO ₂	0.04
LOI	2.81

Table 3	Chemical	composition	of	Lomashell.
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Material	Density (gr/cm³)	Specific Surface Area (cm ² /gr)	Water Content (%)
Lomashell	1.93	6000	0.1

Table 4 Physical characteristics of Lomashell.

Figure 4 SEM image of Lomashell powder.

As depicted in Figure 4, micronized Lomashell powder has an asymmetrical block structure with clear corners and edges. It also has some voids in it, which can enhance asphalt adsorption. Due to its structural pattern, the particles can be blended more efficiently with the asphalt, altering its characteristics [28]. In this research, the Lomashell stones were first thoroughly cleaned with deionized water (DI water) to get rid of excess alkalinity and chloride, after which they were then dried for 24 hours in a drying oven (T = 105 °C). A dry ball mill was used to grind the dried stones into the desired micronized sizes. Micronized powder with particle sizes of 10 to 20 microns, i.e., in the sub-nanometer range based on ASTM C115, was used. To ascertain the effects of this powder in bitumen and asphalt, addition of 3%, 5%, 7%, and 10% by weight of binder to the mixture was considered.

Sample Preparation

Figure 5 schematically illustrates the sample preparation process. The original bituminous material was heated to 160 °C before being blended with modified powder and manually stirred for 10 minutes. In a short period of time, the corresponding temperature rose to 170 °C, and for 40 minutes, high-speed shear mixing at 170 °C with a revolution of 5000 r/min was performed. The use of a high-speed shear mixer in this study was specifically chosen to achieve a more uniform dispersion and distribution of the micronized powder within the binder matrix [23]. This research employed the Marshall Mixture design based on the ASTM D1559 standard to identify the optimal asphalt content of the mixture. To increase the accuracy of determining the optimal binder content, three Marshall samples were created from each asphalt mixture. The optimal amount of bitumen was found to be 5.5%, void spaces between the aggregate (VMA) were 14%, and the asphalt-to-void ratio (VFA) was 4%. To investigate the influence of the Lomashell on the binder and the asphalt mix properties, the binder content and aggregate amount in both the control and the modified samples were maintained constant [29].



Figure 5 Schematic diagram of preparing Lomashell-modified asphalt.

Experimental Procedures

Empirical Rheological Tests

Empirical rheological tests on the asphalt binder involved the use of laboratory testing equipment to measure the viscoelastic characteristics of the asphalt binder. These properties describe the way in which the asphalt influences the binder's ability to deform and flow under applied stress and are crucial for predicting how asphalt pavements will behave under different stress and temperature conditions. To assess the impact of Lomashell on the rheological parameters, a number of tests were carried out, including those for penetration (ASTM-D5), softening point (ASTM-D36), ductility (ASTM-D113), and viscosity (ASTM-D2170) in accordance with the standards mentioned.

Rheological Characterization Analysis

To ascertain how the binders treated with Lomashell behave viscoelastically, a set of rheological testing protocols by dynamic shear rheometer (DSR) was applied using the ASTM D7175 standard. By providing the complex shear modulus ($|G^*|$) and phase angle (δ), which are both used to assess fatigue cracking resistance and rutting resistance, the DSR quantifies the viscoelastic behavior of the binder. An asphalt binder is only elastic when the phase angle is zero, and it is purely viscous when the phase angle is 90 degrees. A stiffer asphalt binder is associated with increased |G*| at high temperatures [30-31].

Indirect Tensile Strength Test

In this research, we used the modified Lottman test according to AASHTO T283 to examine deterioration as a result of moisture entering the mixture. The resistance to moisture was determined by the tensile strength ratio (TSR) method. For each mixture, three samples were created in both wet and dry conditions. During the testing process, three repetitions were carried out for each experiment; the average results of the three repetitions were reported as the outcome. To begin with, the samples were saturated between 70% and 80% with a vacuum pump device. Subsequently, after saturation, they were placed in a plastic bag containing 10 ± 0.5 mm of water. Then, they were kept in a freezer at a temperature of -18 °C for at least 16 hours. Then, the samples were placed in a Marshall water bath at 60 °C for 24 hours. In the last stage of processing and before the test, the samples were placed in a water bath at 25 °C for 2 hours. The wet samples are what they are called. The test was conducted by loading the samples at a rate of 1.5 cm (2 inches) per minute until the sample broke. The load at failure time was noted. Using Eq.(1), the indirect tension strength was calculated (25 °C):

$$ITS = 2F / t\pi d$$

(1)

where ITS stands for indirect tension strength (kPa), F stands for failing force (kN), t stands for the thickness of the sample (m), and d stands for the diameter of the sample (m). By calculating the resistance of the asphalt sample mix to moisture using Eq. (2), we can determine the average ratio of the indirect tension strength of the wet and the dry samples:

$$\Gamma SR = (ITS_{wet} / ITS_{dry}) \times 100$$

(2) In this equation, TSR is the ratio of indirect tension strength (%), and ITS_{Wet} and ITS_{Dry} represent the average amount of indirect tension strength of the wet and the dry samples (kPa), respectively [32].

Hamburg Wheel-Tracking Device Test

Hamburg wheel-tracking device (HWTD) was employed to assess the vulnerability of the mixtures to premature failure. This susceptibility arises from various factors, such as deficiencies in the aggregate structure, insufficient stiffness of the binder, moisture damage, and inadequate adhesion between the asphalt binder and aggregate in the asphalt mixture. According to the relevant specification outlined in AASHTO T324-19, the Hamburg wheel tracking test can be conducted in both air bath and water bath environments and the water bath environment's temperature can be controlled within the range of 25 °C to 70 °C. The Hamburg wheel tracking device used in this study exerted a wheel load of 705 N with the loading frequency of the steel wheel ranging from 36 times per minute to 70 times per minute. In this study, the wet condition was considered in order to evaluate the

effect of the Lomashell powder on the mixture. Each set of specimens underwent loading for a maximum of 20,000 passes or until the midpoint of the specimen was deformed by 12.5 mm.

The Hamburg wheel tracking test typically employs two key indices, namely the total rutting depth (TRD) and the number of wheel passes (N_{max}), to evaluate the ability of the asphalt mixture to withstand permanent deformation [45]. In general, a smaller TRD or a greater N_{max} indicates a higher level of rutting resistance in the asphalt mixture [33].

$$RRD = TRD/N_{max} \times 10^3$$

(3)

Eq. (3) defines the rutting resistance index (RRD), which takes into account both the rutting depth and the number of wheel passes recorded at the conclusion of the Hamburg wheel tracking test. A higher value of RRD indicates poorer resistance of the asphalt mixture to permanent deformation.

Scanning Electron Microscopy (SEM) Test

In this research, a scanning electron microscopy (SEM) was done to look at the microstructure characteristics, surface morphology, and Lomashell particle dispersion in asphalt binder. The working distance was around 15 to 18 mm, and the accelerating voltage was 5 kV in SE mode [34]. For testing, the modified binder samples (7.0% Lomashell, by weight) were divided into tiny circular pieces [35].

Results and discussion

In this section, the impact of the micronized Lomashell modifier on the empirical and fundamental rheological properties of the asphalt binder at various temperatures and the moisture susceptibility of the mixtures are discussed.

Asphalt Binder Empirical Rheological Tests Results

On the unmodified and the modified binders with various amounts of Lomashell, penetration, softening point, ductility, and viscosity tests were conducted. The Lomashell contents used in the tests were 3%, 5%, 7%, and 10% by weight of the binder. Figure 6 illustrates the results. As can be seen, various percentages of Lomashell had an advantageous influence on the binder's rheological properties. The penetration diminished at 25°C and the softening point value increased as the percentage of Lomashell increased. When the proportion of Lomashell exceeded 5%, however, the variance slope gradually got smaller.

As can be seen in Figure 6(a), penetration initially decreased and then tended to stabilize with 10% increasing modifier. Lomashell decreased penetration by 13% for AC 60–70 and by 11% for AC 85–10, which indicates that the hardness of the Lomashell increased the bonding between the materials and led to increased consistency and hardness of the modified asphalt. The ductility at 25 °C decreased rapidly at first, then slowly; by adding 10% of Lomashell, it decreased by about 28%. The change in ductility trend suggests that Lomashell decreases the temperature sensitivity of bitumen, which is in line with previous research findings. According to the findings, Lomashell can boost the low-temperature extendibility of modified binders, but increasing the Lomashell dose may compromise the modified asphalt's low-temperature properties. The substance that is added to improve its properties, known as the 'modifier', can absorb certain light components; however, if the amount of these components is too high, the absorption process may harm the asphalt. The softening points of the Lomashell modified binders increased by about 11% and 26% for AC 60–70 and AC 85–10, respectively, which indicates that the bitumen had better high temperature endurance with the modified binders. Also, the viscosity at 135 °C increased by about 6%, which shows Lomashell has a low ability to absorb heat; it loses more energy when heated, causing the asphalt binder to soften at a higher temperature, and at a slower rate [36].



Figure 6 Conventional properties of binders samples with different Lomashell percentages: (a) penetration, (b) viscosity, (c) ductility, (d) softening point.

Dynamic Shear Rheometer Test (DSR) Results

Asphalt binder is a very temperature-sensitive material; consequently, in order to research the rheological properties at different temperatures, a DSR test was implemented at 10 rad/s with controlled strain at 12% and temperatures of 55 °C, 60 °C, 65 °C, and 70 °C. Figures 7 and 8 show the measured G* at various temperatures for two different binders with varying Lomashell contents.

Figure 7 Complex shear modulus of unmodified and modified samples with different percentages of Lomashell.

The complex shear modulus ($|G^*|$) measures how resistant a material is to deformation when it is sheared. $|G^*|$ is obtained by subtracting the maximum shear stress by the maximum strain. When the shear modulus value increases, the material's deformation resistance improves as well. Due to the fact that bituminous materials become more fluid at higher temperatures, they are more prone to deformation at the same stress level. Therefore, in order to ensure that asphalt pavement is resistant to high-temperature deformation, a higher complex shear modulus ($|G^*|$) is necessary [37]. A diagram of shear modulus changes against various temperatures is displayed in Figure 7. The G* of both binder materials decreases as the temperature rises, as observed, because the elastic part of the asphalt binder influences how well the elastic properties of the material are reflected at low temperatures. The results show that the use of Lomashell increased the values of the shear modulus and improved the properties of the modified binders at all temperatures. The sample containing 7% powder had the highest value for the shear modulus, and the $|G^*|$ of the modified asphalt binder was better than that of the unmodified asphalt in the temperature range of 55 °C to 70 °C. The phase angle is a measure of the ratio of the lost to the stored modulus and is susceptible to the physicochemical composition of the binder; it reflects the bitumen's viscoelastic response to temperature variation. The phase angle of bitumen increases

with rising temperature. Bitumen with a higher phase angle has poor performance and less elastic recovery [38]. According to Figure 8, the phase angle increased with increasing temperature, and increasing the additive percentage caused the phase angle to decrease. The samples containing different percentages of Lomashell showed the lowest phase angle value at different temperatures. Therefore, the elastic recovery in these percentages was better compared to control. The modified bitumen became less sensitive to temperature as the amount of powder in it increased. The addition of micronized Lomashell powder alters the interaction between asphalt molecules, leading to improved rebound stability at the same temperature; therefore, modified asphalt with this material has better elasticity than matrix asphalt.

Figure 8 Phase angle of unmodified and modified samples with different percentages of Lomashell.

The graph in Figure 9 shows the outcome of a frequency sweep test, which replicates how changing traffic speeds would affect the complex shear modulus of both standard base bitumen and bitumen modified with Lomashell at a temperature of 65 °C. As indicated by the test findings, the complex shear modulus $|G^*|$ of the asphalt binder increased with increasing frequency; this is because as soon as it becomes cold, the asphalt binder's fluidity decreases as its elastic content increases, resulting in a larger modulus in the high-frequency zone, which corresponds to the low-temperature region [39].

Figure 9 Complex modulus master curve of unmodified and modified samples.

As can be seen in Figure 9, the $|G^*|$ of the binders with Lomashell powder compared favorably to the unmodified asphalt at the same temperature. When the frequency increased, the amount of asphalt binder $|G^*|$ also increased; this shows that the addition of the modifier powder made the material harder and decreased shear deformation at high temperatures. The pavement rutting factor ($|G^*|/Sin$) is a measure of bitumen rutting resistance. This index was used to compare the resistance of the modified and the original binder to distortion over time. The graph of changes in the rutting parameter against temperature for the binders containing Lomashell with different percentages is shown in Figure 10. The results show that with the increase in the powder proportion, the rutting factor of the specimen increased. The use of this material will probably increase the interaction between the binder and the particles, forming a long chain bond between them. It seems that these conflicts cause an increase in the resistance of bitumen chains against external forces and resistance against deformation and rutting, therefore, the addition of this powder improves the rutting factor. When the temperature was between 55 °C and 70 °C, 10% Lomashell powder was added to the AC 60–70 asphalt binder. The value of ($|G^*|/sin$) increased when compared to matrix asphalt.

Figure 10 Rutting factor for neat and modified samples.

The use of larger amounts of Lomashell caused a decrease in the slope of improvement of the physical properties, which is probably due to the adhesion of particles to each other and the increase in their accumulation, which leads to the creation of stress concentration points and places prone to cracking and rutting.

Indirect Tensile Strength Test Results

In this research, the indirect tensile strength was measured for untreated (dry) and treated (wet) samples at 25 °C after 1, 3, and 5 freezing-thawing cycles. This was done to simulate the reduction of asphalt mixture resistance in more realistic environmental conditions. The experiment was designed so that three repetitions were conducted, and the final value was provided for each test using the average results of the three repeats. Figure 11 illustrates the average values of indirect tensile strength for the wet and the dry asphalt mixture samples containing different percentages of Lomashell. The adherence of bitumen and aggregate decreased as the percentage of water in asphalt mixtures increased, resulting in lower ITS values for the wet asphalt samples compared to the dry asphalt samples. In addition, when water in the voids of the asphalt mixture turns into ice during freezing-thawing cycles, the link between the bitumen and the aggregate is weakened and broken. As shown in Figure 11, the level of indirect tensile strength of the mixtures that included micronized Lomashell powder was higher than that of the control mix. As the frequency of freezing-thawing cycles rose, the modified mixtures performed better than the unmodified ones. The addition of 3%, 5%, 7%, and 10% of this material in the asphalt mixture improved the indirect tensile strength of the modified samples by 9%, 11%, 12%, and 10%, as compared to the control sample, respectively. Since surface tension has a significant impact on how well the binder coats the aggregate, the inclusion of micronized Lomashell powder improves the interaction between the bitumen and the aggregate by increasing the related factor [40].

Figure 11 Average values of indirect tensile strength for the wet and the dry asphalt mixtures.

Moreover, the indirect tensile strength ratios (TSR) of the wet and the dry samples for different freezing and thawing cycles are presented in Figure 12. As expected, the values of the ratio of strength of the wet and the dry samples of the asphalt mixtures that include modifiers were superior to those of the control mixes for all freeze-thaw cycles. According to the indirect tensile strength results shown in Figure 11, the strength of the asphalt mixtures decreased for mixtures with different percentages of powder in freezing and thawing cycles. In the first cycle of freezing and thawing, the modified sample showed a 4% increase in the value of the tensile strength ratio compared to the control asphalt mixture. When moisture is present, Lomashell exhibits different behavior, likely due to reactions that occur and result in an increased in the adherence between the binder and

the aggregate. The loss of mixture adhesion or bitumen cohesiveness caused by exposure to moisture could be the reason why the indirect tensile strength of the samples decreased as the number of freeze-thaw cycles increased. The data in Figure 12 suggest that the use of micronized Lomashell as an anti-stripping agent improves the bonding and cohesiveness of the mixture, stops bitumen from rapidly separating from the surfaces of the aggregates, and enhances the mixture's resistance to moisture after freezing-thawing cycles in comparison to samples without additives.

Figure 12 Tensile strength ratio for wet and dry asphalt mixtures.

As shown by the outcomes in Figure 12, increasing the percentage of additives in the asphalt mixtures increased the TSR index when compared to the control samples. This is probably caused by the high surface energy of Lomashell, which strengthens the connection between the aggregate and the bitumen. The lowest value suggested by the ASTM standard for the TSR index is 80%. All asphalt mixtures modified with micronized Lomashell for the first freezing-thawing cycle in this study met this minimum requirement. As a result, addition of this material raises the ITS values in both dry and wet conditions, improving the asphalt mixture's moisture sensitivity. The chemical substance of the Lomashell used in the research is presented in Table 3, which shows that the material is primarily composed of CaCO₃, which has hydrophobic properties and provides resistance against moisture in the mixture. Also, this micronized powder aims to minimize the interfacial tension, a crucial factor influencing the coating of aggregate surfaces with asphalt and prevent the build-up of acid at the interface between the aggregate and the bitumen, resulting in improved moisture resistance of the pavement and ultimately enhancing its durability [39-40]. Finally, increasing the viscosity (as discussed in section 4.1) fosters a powerful bond between the asphalt binder and the aggregate, resulting in a challenging scenario where the binder film struggles to glide smoothly over the surface [41-42].

Hamburg Wheel-Tracking Device Test Results

In this study, a Hamburg wheel-tracking device (HWTD) was employed to assess the performance of two mixtures. These mixtures had identical gradation and binder content, with one utilizing control binders, while the other incorporated binder modified with 7.0% Lomashell. The test was conducted under water at a consistent temperature of 50 °C and standard wheel load (705 N) and a loading frequency of 52 times/mi. Figure 13 presents the permanent deformation characteristics of control and the modified asphalt mixture.

Figure 13 Permanent deformation curves of Control and modified mixture.

Based on the findings obtained from the HWTD, it can be inferred that the inclusion of 7% Lomashell positively impacted the adhesion between the asphalt binder and the aggregate. When the base binder AC 60-70 was

utilized, the mixture displayed a rutting depth of 9.0 mm after 5,110 cycles. However, when the same binder was combined with 7% Lomashell, the same rutting depth was reached after 7,156 cycles. Similarly, for the mixture employing the base binder AC 85-100, a rutting depth of 9.0 mm was observed after 10,476 cycles, while the mixture containing the same binder mixed with 7% Lomashell achieved the same rutting depth after 16,532 cycles. Figure 14 provides a visual representation of the comparative analysis conducted on cylindrical samples before testing on the left side, while the right side displays the same samples after the completion of the testing process. In the left and right portion of Figure 14, the first sample corresponds to AC 60-70, while the second sample represents AC 85-100.

Figure 14 Cylindrical samples (saw-cut) before (Left) and after (right) the HWTD test.

The results reveal a significant improvement of 28% and 36% in rutting performance resulting from the addition of Lomashell. Additionally, the rate of rutting depth (RRD) for the control mixture and the modified mixture using AC 60-70 was found to be 1.76 and 1.25, respectively. Similarly, for AC 85-100, the RRD values were 0.86 for the control mixture and 0.54 for the modified mixture. It is important to note that higher RRD values indicate a reduced resistance of the asphalt mixture to permanent deformation. Therefore, the incorporation of Lomashell contributes to a better resistance against permanent deformation, as evidenced by the lower RRD values in the modified mixtures. The inclusion of micronized powder in the asphalt mixture leads to various beneficial effects, resulting in improved adhesion between the asphalt binder and the aggregate particles. These effects encompass functions such as interlocking, cementing, elastic recovery, and void filling. Collectively, these mechanisms contribute to an overall enhancement in the performance of the asphalt mixture [43].

Microstructure Characterization Results of Micronized Lomashell

The SEM images of the unmodified and the modified binder samples (5% and 10% Lomashell, by weight), shown in Figures 11(a) to (c), respectively, display these. As demonstrated in Figure 11(b), the sample containing 5% of micronized powder was well mixed with the asphalt binder, and a homogeneous mixture was created. Lomashell particles can be seen in the image, but in Figure 11(c), due to the increase in the amount of powder to 10%, it can be observed that there is a small amount of clumping in some areas, which indicates that high-speed shear mixing at speeds exceeding 5,000 rpm does not have 100% effectiveness [44].

Figure 15 SEM images of the neat and modified binder samples a) neat b) 5% Lomashell c) 10% Lomashell.

When using a higher percentage of Lomashell, an issue observed was the clustering of micro particles into micro sized clusters, which can decrease the efficiency of Lomashell and increase the shear susceptibility of the asphalt binder. The particles in Lomashell are able to form a stable network structure with the molecules in the asphalt, resulting in an improvement in the resistance to load and thermal stability of the overall structure, as can be seen in Figure 13(b). The lumps formed after the cohesion of particles in Figure 13(c), even under vigorous stirring, because the micronized powder was not sufficiently spread throughout the asphalt. This could explain why the density of this material differed from that of asphalt, resulting in the agglomeration phenomenon; thus, 10% powder addition necessitates shear mixes at speeds exceeding 5,000 rpm.

Conclusions

This study's purpose was to employ micronized Lomashell as additive to improve the mechanical properties of asphalt binder and address environmental problems brought on by its disposal. Lomashell is a mineral stone that is formed by the accumulation and hardening of stones and shells and skeletons of oysters. To achieve this goal, mixtures were created using a Marshall device, and empirical rheological, DSR, indirect tensile strength, and scanning electron microscopy (SEM) were carried out on control samples as well as on modified mixtures containing varying amounts of micronized Lomashell (3%, 5%, 7%, and 10%). This study's findings showed that the modifications with Lomashell performed noticeably better than the control mixtures.

More specifically:

- 1. According to empirical rheological tests, penetration and ductility decreased, which showed that this powder boosted the stiffness and hardness of the binder while the softening point and viscosity increased, which indicates the lower heat absorption efficiency of micronized Lomashell powder.
- 2. DSR frequency and temperature sweep tests revealed that incorporating micronized Lomashell powder into asphalt improved its high-temperature stability, as evidenced by increases in the complex shear modulus (|G*|), decreases in the phase angle, and increases in the rutting factor (|G*|/sin). This implies that adding Lomashell powder as an additive reduces the asphalt binder's susceptibility to changes in temperature and increases its resilience to rutting and permanent deformation at high temperatures. The addition of this micronized powder, increases the ITS and TSR values regardless of whether it's dry or not, thereby improving the moisture sensitivity of the asphalt mixture.
- 3. The Hamburg wheel-tracking device test clearly indicated a substantial enhancement of 28% and 36% in rutting performance as a result of incorporating Lomashell. Furthermore, the rate of rutting depth (RRD) decreased, which demonstrates an improved resistance of the asphalt mixture to permanent deformation.
- 4. The SEM images showed that a difficulty with using a higher percentage of Lomashell is the agglomeration of micro-particles, creating micro-sized clusters, which can diminish the efficiency of this powder while increasing the susceptibility of the asphalt binder to shear. The difference in density between Lomashell and asphalt may be the cause of the agglomeration phenomenon, which needs effective high-shear mixing at higher speed and more time.

Overall, the high-temperature stability and moisture resistance of AC 60–70 and AC 85–100 asphalt binders were improved by adding micronized Lomashell powder. This study indicated the engineering and environmental benefits of using discarded powder as an asphalt modification. Prior to application in field construction, additional mixture testing will be carried out. This testing will include optimization of the mixture design for Lomashell powder content and modified asphalt mixture; it is also important to assess the low-temperature qualities of the modified asphalt binders.

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