

Research Article

The Effect of Wetfix and Nanohydrated Lime Additives on Bitumen Aging and the Cohesion and Adhesion Failure Mechanisms of Hot Asphalt Mixtures

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Aging due to sunlight and the passage of time is an effective element in the occurrence of moisture damage in hot asphalt mixtures. Still, the effects of this parameter are scarcely considered in the experiments examining the moisture damage potential. Accordingly, this study investigated the effect of aging on the performance of hot asphalt mixtures and examined the improvement of moisture damage performance by using antistripping additives via mechanism and functional tests. Two types of aggregates (limestone and granite) with different degrees of moisture sensitivity, two types of bitumen (PG64-16 and PG58-22) with different performances, and two additives (Wetfix liquid additive and nanohydrated lime) as bitumen modifiers were used. Bitumen samples and asphalt mixtures were subjected to short- and long-term aging. The pull-off test was performed to explore the aging effect on different failure mechanisms (cohesion and adhesion), and the indirect tensile stiffness modulus (ITSM) test was conducted to study the asphalt mixture's performance against moisture damage. The results specify that aging, in terms of hot asphalt mixture hardening in dry and wet conditions, decreased the MSR (resilient modulus wet to resilient modulus dry ratio), and this decline was greater in long-term aging. The pull-off test results exhibited that aging, especially in the long term, decreased the asphalt mixture's adhesive strength in dry and wet conditions; this decline in adhesion was greater in the wet than in the dry state, and this difference decreased the wet-to-dry adhesion strength ratio (the pull-off ratio). The additives relatively improved the yield modulus of the asphalt mixture, but their effect was greater in the wet state. A comparison of the pull-off test results in cohesion and adhesion failure demonstrated that Wetfix was more effective in improving bitumen–aggregate adhesion, whereas nanohydrated lime was more effective in enhancing bitumen adhesion. The resilient modulus (M_r) ratio in wet-to-dry conditions indicated that nanohydrated lime had better effects on the overall performance of the aged asphalt mixture against moisture damage. To investigate the effect of additives on the performance of asphalt mixtures, a t -test was performed in all modes of control, short-term aging, and long-term aging. The findings showed the effect of Wetfix and nanohydrated lime on increasing the modulus of elasticity of the samples.

1. Introduction

1.1. Background. The premature failure of asphalt pavements is partially due to the damage caused by environmental factors, e.g., ultraviolet (UV) rays and moisture. Moisture causes the loss of asphalt mixture strength and durability, leading to what is called moisture failure. Bitumen and aggregates are the main components of asphalt mixtures, and their physical and chemical properties directly affect asphalt mixtures' performance. Incompatibility between bitumen and aggregates is one of the fundamental reasons

for moisture damage in asphalt mixtures. Moisture damage usually occurs in two cases: (1) a lack of adhesion between the bitumen and aggregates, and (2) a lack of adhesion between bitumen molecules in the presence of water [1]. The substitution of bitumen with water on the aggregate surface is called stripping. This phenomenon, which is dependent on the chemical structure of the bitumen and aggregates and the relevancy between this two, causes the loss of the bond between bitumen and aggregate in the presence of water. This loss occurs due to the weak adhesion between the bitumen and aggregates and the tendency to form a bond between

water and aggregates. Therefore, the characteristics of each asphalt mixture component greatly affect the structural performance of asphalt pavements [2].

In addition to the negative impacts of moisture on asphalt mixtures, UV and the movement of hot air in asphalt mixture voids can cause bitumen aging, which is another factor aggravating the occurrence of moisture damage. The main aging factor is thermal oxidation aging. The thermal-oxidative aging of bitumen occurs from the mixing stage to the end of pavement life. There are two phases for the thermal-oxidative aging of bitumen: one is short-term aging and the other is long-term aging [3]. Short-term aging occurs during asphalt mixture construction, while long-term aging happens during the pavement's service life. Hardening caused by the passage of time in asphalt mixtures is an inevitable process occurring due to oxidation and UV [4]. The physico-chemical characteristics of the oxidized asphalt mixture lead to the appearance of small cracks, which will eventually turn into larger cracks, allowing water to more easily penetrate the asphalt mixture and accelerating moisture damage [5].

A key failure factor in asphalt mixtures is the stripping of the aggregate surface from the bitumen. Moisture damage and bitumen aging in the first stage separate the bitumen coating from the aggregate surface. Antistripping additives, e.g., liquid antistripping additives and various nanolimes, are usually incorporated to overcome this phenomenon [6].

1.2. Literature Review. Punith et al. [7] examined the impact of extended aging on the moisture sensitivity of foamed warm mixed asphalt (WMA) mixtures that contained moist aggregates. The authors found that prolonged aging enhanced the moisture resistance of WMA mixtures, irrespective of the use of an amine-based antistripping agent (ASA). Furthermore, the moisture conditioning and source of aggregates had a significant effect on moisture resistance, regardless of the foaming technology, ASA, and moisture content of aggregates.

Ma et al. [8] investigated the effect of moisture on the aging behavior of asphalt binder. Pressure aging vessel (PAV) test and penetration grade tests were conducted to fully evaluate the moisture aging effect of binder. The findings show that moisture conditions can accelerate the aging of asphalt binder and shorten the service life of asphalt binder.

Yusoff et al. [9] conducted a study on the effectiveness of incorporating nanosilica into polymer-modified asphalt (PMA) mixture. The findings indicate that nanosilica reduces moisture susceptibility and improves the strength of asphalt mixtures. Additionally, PMA mixed with nanosilica particles exhibits enhanced fatigue and rutting resistance and a reduction in aging index values, particularly observed in long-term aging.

Li et al. [10] studied the moisture susceptibility of asphalt mixtures and binders with Sasobit warm mix additive compared to hot mix asphalt. Aging duration and temperature affected moisture susceptibility, as shown by freeze-thaw splitting strength ratios and residual Marshall stability. Asphalt binder's surface free energy increased with aging time and temperature.

Rahmani et al. [11] examined the thermodynamic principles underlying moisture damage in asphalt mixtures. Their study found that aging of the asphalt binder increased debonding energy, adhesion, and cohesion free energy. The correlation between debonding energy and moisture sensitivity was 0.77 and 0.65 for aged and controlled specimens, respectively.

Kakar et al. [12] examined the impact of moisture and aging on asphalt binder adhesion failure through pull-off tension testing. The researchers utilized a chemical surfactant-based additive to modify the asphalt binder and tested it against pull-off tension force with limestone aggregate substrates. The findings indicated that the percentage of adhesion failure rose under moisture conditioning, as well as with binder aging, particularly when subjected to long-term aging.

Omar et al. [13] conducted a study on the impact of aging on the chemical and strength properties of nanoclay-modified bitumen and asphalt mixture. The results of ITS tests demonstrated that mixtures containing nanoclay exhibited greater tensile strength and resistance to aging. Therefore, it can be concluded that incorporating nanoclay as an additive in bitumen modification enhances its resistance to aging, which consequently improves the strength of the asphalt mixture.

Ma et al. [14] examined the influence of moisture and oxygen on bitumen, which is regulated by their respective transport and reaction mechanisms. The aging process caused by oxidation and the movement of moisture within bitumen can have a considerable impact on the durability of pavements. By combining the effects of moisture and oxygen, it becomes feasible to forecast the long-term performance of pavements. The objective of this review was to develop models that can anticipate pavement performance over extended periods in relation to moisture-induced damage.

Zhang and Hoff [15] conducted a comparative study to investigate the impact of thermal-oxidative aging and salt solution aging on bitumen performance. Results indicate that both types of aging had comparable effects on the oxygen content, physical, low-temperature, and high-temperature properties of bitumen; however, they exhibited distinct changes in morphology.

Notani et al. [16] investigated the effectiveness of waste toner as a modifier for bituminous materials in terms of short-term aging and resistance to moisture sensitivity. The results revealed that incorporating small amounts of waste toner into the binder improved its resistance to short-term aging. Furthermore, binders modified with 12% waste toner exhibited a notable enhancement in moisture resistance for both asphalt binders and mixtures.

Ali et al. [17] aimed to assess the impact of various additives and aging on the moisture-induced damage performance of asphalt mixtures. The results indicated that the addition of WMA and ASA improved the resistance of asphalt mixtures to moisture-induced damage, as evidenced by the SFE method. However, thermal degradation of binder constituents and additives caused significant changes in indirect tensile stiffness (ITS).

Jameel et al. [18] conducted a study on the impact of aging on the adhesion of bitumen and aggregates, as well

as moisture damage in asphalt mixtures. The results of the BBS test indicated an improvement in pull-off tensile strength (POTS) under both dry and wet conditioning. Specifically, the unaged and rolling thin-film oven test (RTFOT)-aged binders exhibited a cohesive failure pattern under dry conditioning, while the PAV-aged binder showed an adhesive failure pattern. Under wet conditioning, all three binders displayed cohesive failure patterns. It was found that aging enhances the resistance to moisture damage and bond strength of the binder.

Yang et al. [19] investigated chemorheological, mechanical, morphology evolution and environmental impact of aged asphalt binder coupling thermal oxidation, UV radiation, and water. Thermal oxidation and UV radiation had the most significant aging acceleration effect.

1.3. Research Motivation. The purpose of this paper is to investigate the effect of Wetfix and nanohydrated lime (NHL) additives on bitumen aging and the cohesion and adhesion failure mechanisms of hot asphalt mixtures. Specifically, we aim to explore the relationship between aging and moisture damage of asphalt samples. This study is important because considering both effects of aging and moisture damage in both adhesion and cohesion view of asphalt samples in both parts of adhesion and cohesion is a new method. However, there is some research on the negative effects of moisture and aging on asphalt pavement. This paper differs from previous works in that it focuses on the adhesion and cohesion failure of asphalt pavement. Additionally, we will be using two additives, namely, one is Wetfix and the other is NHL. By doing so, we hope to provide a more understanding of the relationship between these two additives. Ultimately, our goal is to provide recommendations for a better way to improve asphalt durability.

1.4. Statement of the Problem and Objectives. Moisture damage is an important factor in asphalt pavement destruction and usually occurs via two mechanisms of cohesion and adhesion failure. The causes of moisture damage are divided into two internal and external factors; internal factors depend on the characteristics of the materials and the asphalt mixture mix design, whereas external factors depend on conditions such as traffic load and climate that affect the mixture. A factor related to both internal and external factors is aging. This study by using two nondestructive tests, including the pull-off test and indirect tensile stiffness modulus (ITSM) tests, innovatively examined the effect of two antistripping additives (Wetfix and NHL) on the performance of aged and unaged asphalt mixtures against moisture damage and the type of cohesion or adhesion failure by using two types of aggregates (granite and limestone) and two types of bitumen (PG64-16 and PG58-22). The objectives of this research were:

- (i) Studying the effect of Wetfix and NHL on aged and unaged asphalt mixtures in dry and wet conditions.
- (ii) Evaluating the simultaneous impact of aging and moisture on asphalt mixtures' resistance to moisture damage.

- (iii) Investigating the effect of bitumen modifiers on the mechanisms of bitumen cohesion failure and bitumen–aggregate adhesion failure in dry and wet conditions.

2. Experimental Plan

To perform laboratory work, aggregate, bitumen, and anti-stripping additives were first prepared, and Marshall samples were constructed for the mixing plan to obtain the optimal bitumen. The control samples and the samples modified with Wetfix and NHL were then fabricated in dry and wet conditions (AASHTO T283 standard). The content of the additives was selected based on the previous study. To investigate the impact of aging on asphalt mixtures' performance, aged asphalt mixture samples were prepared and compared with controls (AASHTO R30). The ITSM test was performed to investigate the effect of aging on moisture sensitivity (ASTM D4123), and the pull-off test was conducted to examine the bitumen–aggregate adhesion mechanism (AASHTO TP91-15, AASHTO2015). Figure 1 shows the flowchart of laboratory work.

2.1. Materials

2.1.1. Aggregates. Two types of aggregates with different potentials against moisture damage, i.e., limestone (hydrophobic) and granite (hydrophilic), were incorporated to fabricate the asphalt samples. The ASTM D3515 standard was employed for grading the stone materials. An X-ray fluorescence (XRF) test was performed to determine the mineral components of the aggregates (Table 1). The physical characteristics of the aggregates are presented in Table 2.

2.1.2. Bitumen. Two types of bitumen, PG64-16 and PG58-22, which are used in rainy climates, were utilized to examine different composition modes (Table 3).

2.1.3. Additives.

(1) Wetfix. Wetfix is an antistripping liquid that improves the adhesion between bitumen and aggregate by creating thermal stability. The percentage of this material for HMA and WMA is usually 0.1%–1% of the bitumen weight, chosen according to the type of bitumen and aggregates [20]. The characteristics of Wetfix are listed in Table 4. A target percentage of 0.4% of bitumen weight was utilized for different combinations. The liquid was mixed with the bitumen via a mixer. The bitumen was initially heated up to 160°C, and Wetfix was then gradually added to it. The mixture was stirred for about 5 min at 9,000 rpm until the ingredients were completely mixed. The same mixing method was performed for unaged and aged samples, so that the effect of aging would be the same on both types of samples [21]. Wetfix is shown in Figure 2.

(2) Nanohydrated Lime. NHL (electron microscope image in Figure 3) was also utilized. This material has more than 90% CaO and less than 3% calcium carbonate. Its specific mass is 2.24 g/cm³, its acidity is 12.4, and it is considered a relatively strong base.

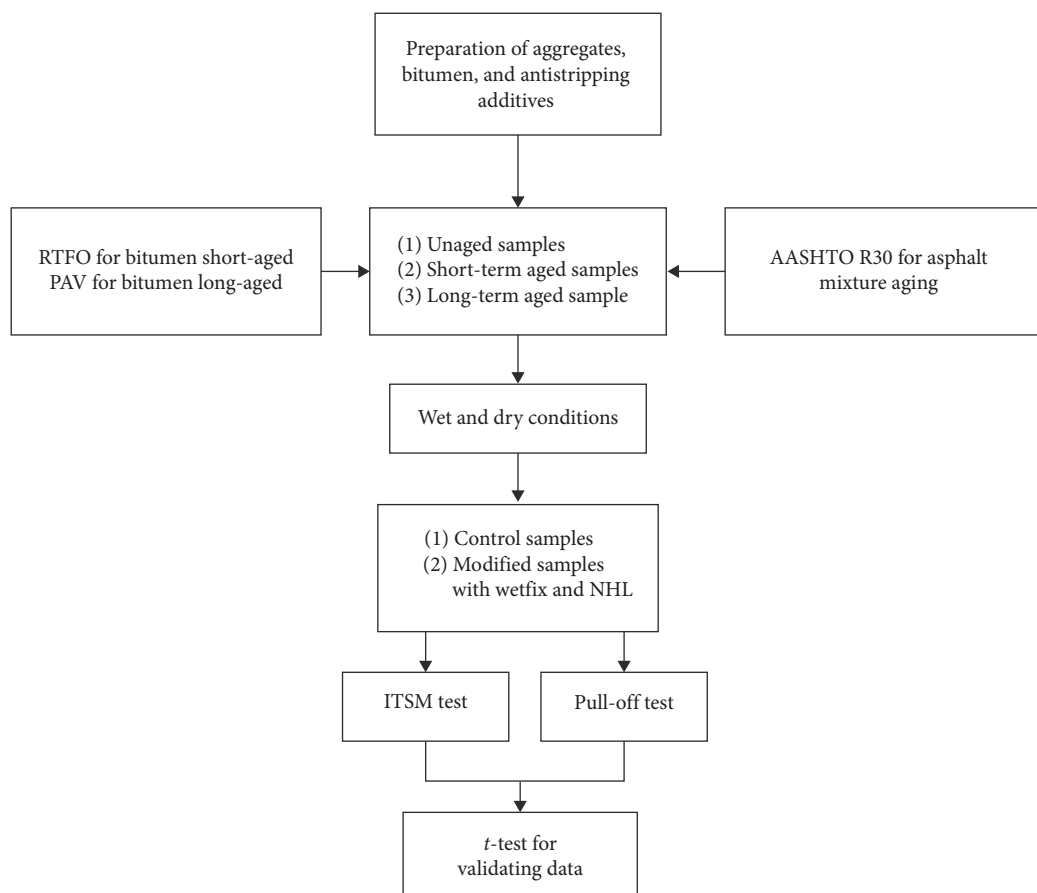


FIGURE 1: The process of performing laboratory activities.

TABLE 1: Minerals forming the aggregates.

Aggregates	Silicon dioxide, SiO ₂	Aluminum oxide, Al ₂ O ₃	Ferric oxide, Fe ₂ O ₃	Magnesium oxide	Calcium oxide
Limestone	4.8	0.9	0.3	1.8	62.6
Granite	54.1	13.3	2.7	0.8	4.7

TABLE 2: Physical characteristics for both types of stone materials.

Test	Standard	Limestone	Granite	Specification
Specific gravity (coarse aggregates)	ASTM C 127			
Bulk (g/cm ³)		2.612	2.654	–
SSD (g/cm ³)		2.643	2.667	–
Apparent (g/cm ³)		2.659	2.692	–
Specific gravity (fine aggregates)	ASTM C 128			
Bulk density (g/cm ³)		2.618	2.659	–
SSD (g/cm ³)		2.633	2.661	–
Apparent (g/cm ³)		2.650	2.687	–
Specific gravity (filler)	ASTM D 854	2.641	2.657	–
Los Angeles abrasion (%)	ASTM C 131	25.6	19	Max 45
Flat and elongated particles (%)	ASTM D 4791	9.2	6.5	Max 10
Sodium sulfate soundness (%)	ASTM C 88	2.56	1.5	Max 10–20
Fine aggregates angularity (%)	ASTM C 1252	46.65	56.3	Min 40

TABLE 3: Specifications of bitumen.

Parameter	PG64-16	PG58-22	Test method
Density in 25°C	1.02	1.03	ASTM D70-76
Degree of penetration at 25°C	68	91	ASTM D5-73
Softening point (°C)	51	48	ASTM D36-76
Ductility (25°C, 5 cm/min) (cm)	105	112	ASTM D113-79
Flash point (°C)	262	248	ASTM D92-78
Weight loss (%)	75	75	ASTM D1754-78
Purity (%)	99.5	99.5	ASDM D2042-76
Viscosity (Pa·s)	0.776	0.576	ASTM D2170
Rotational viscosity at 135°C (Pa·s)	0.305	0.260	ASTM D4402

TABLE 4: Specifications of Wetfix.

Material	Wetfix
Appearance at 20°C	Brown liquid
Density in 20°C (kg/m ³)	961
Flowing temperature (°C)	≤0
Flash point (°C)	>180



FIGURE 2: Wetfix used in this research.



FIGURE 3: Nanohydrated lime powder used in this research.

Based on most studies, the percentage of nanoadditives to improve asphalt mixtures' performance is about 0.6%–1.2% [22]. Herein, 1% NHL was used for bitumen modification. A mixer with a speed of 12,000 rpm was used to mix the

TABLE 5: Characteristics of nanohydrated lime.

Material	Nanohydrated lime
Structure	Calcite
Particle form	Cubic
Mass density (gr/cm ³)	2.24
Specific surface area (m ² /gr)	34 ± 1
Particle size (nm)	≈40
Volume specific mass (gr/cm ³)	0.55
Water amount (%)	≥0.3
Acidity range	12.4

bitumen with NHL. Bitumen was heated up to 155°C, and NHL was gradually added to it; it was mixed for 15 min, so that the nanomaterial particles would be homogeneously dispersed in the bitumen. The characteristics of the NHL are given in Table 5.

2.2. Asphalt Mixture Combinations. To evaluate the resilient modulus, resilient modulus tests were performed in all three conditions of control, short-term aging, and long-term aging for both dry and wet states. The pull-off test was also conducted in all cases to check the bitumen's resistance against the adhesion failure of bitumen and aggregates and the cohesion failure of bitumen particles. Specifications of the tests used in this research are shown in Table 6.

Two types of bitumen (PG64-16 and PG58-22), two types of aggregates (limestone and granite), and two types of additives (Wetfix and NHL) were incorporated. The specifications of the combinations of materials are given in Table 7.

2.3. Experimental Methods. To fabricate asphalt samples, the mix design was adopted to obtain the optimal bitumen percentage. The aging tests of bitumen and asphalt mixtures were then performed, and finally, the resilient modulus was calculated according to the modified Lottman test.

2.3.1. Mix Design. To fabricate Marshall samples, three series of mixtures weighing 1,200 g with four types of stone materials having bitumen percentages of 4, 4.5, 5, 5.5, 6, and 6.5 were prepared (ASTM D1559-89) [23]. Three samples were fabricated for each bitumen percentage; to simulate heavy traffic, each side of every cylindrical sample was hit 75 times.

TABLE 6: Specifications of the tests used in this research.

Row	Test	Aging (UV/thermal oxidation)	Field condition	Purpose
1	Resilient modulus by indirect tensile stiffness modulus	–	Dry	Resistance of asphalt mixture in dry conditions
2		Short-term		
3		Long-term		
4		–	Saturated by freeze–thaw cycle	Resistance of asphalt mixture in wet conditions
5		Short-term		
6		Long-term		
7	Direct tensile by pull-off (cohesion)	–	Dry	Resistance of bitumen against cohesion failure in dry condition
8		Short-term		
9		Long-term		
10		–	Applied moisture cycle	Effect of moisture on bitumen resistance against cohesion failure
11		Short-term		
12		Long-term		
13	Direct tensile by pull-off (adhesion)	–	Dry	Resistance of bitumen–aggregates against adhesion failure in dry condition
14		Short-term		
15		Long-term		
16		–	Applied moisture cycle	Effect of moisture on bitumen–aggregates resistance against adhesion failure
17		Short-term		
18		Long-term		

TABLE 7: Characteristics of asphalt mixtures used in this research.

Row	Aggregates	Bitumen	Additives	Percentage (%)
1	Limestone	PG64-16	–	–
2			Wetfix	0.4
3			Nanohydrated lime	1
4		–	–	
5		PG58-22	Wetfix	0.4
6		Nanohydrated lime	1	
7	Granite	PG64-16	–	–
8			Wetfix	0.4
9			Nanohydrated lime	1
10		–	–	
11		PG58-22	Wetfix	0.4
12		Nanohydrated lime	1	

Mixing and compaction temperatures were determined for different bitumen percentages using the temperature–viscosity diagram. The optimal bitumen percentage was calculated based on the MS-2 guidelines of the Asphalt Institute. The optimal bitumen values in each combination are shown in Table 8.

2.4. Aging Test of Bitumen and Asphalt Mixtures. The bitumen and asphalt mixture samples were subjected to aging. Aged bitumen samples were used for the pull-off test, and aged asphalt mixture samples were utilized to test the moisture sensitivity of asphalt mixtures.

2.4.1. Asphalt Pavement Aging. AASHTO R30 was employed to induce aging in the asphalt mixtures (R30 AASHTO 2016) [24]. According to this standard, the samples were subjected

to processing in three conditions: control, short-term aging, and long-term aging.

In control samples, the bitumen and aggregates were mixed at 163°C and were then placed in the oven for 2 hr. As for short-term aging, the samples were placed in the oven for 4 hr at 135°C in a nondense manner. The samples were taken out and stirred every 1 hr to undergo uniform aging. After 4 hr, the samples were compacted by a Marshall hammer.

For long-term-aged samples, the samples were first compacted and then transferred to the oven with a mold where they remained for 5 days at 85 ± 3°C. Subsequently, the oven was turned off and the samples were allowed to reach room temperature. After applying the aging conditions, the samples were placed in dry and wet conditions to create and test the six combinations.

TABLE 8: Optimum percent of bitumen.

Bitumen type	Aggregates type	Optimum (%) of bitumen
PG64-16	Limestone	5.2
PG64-16	Granite	5.3
PG58-22	Limestone	5.8
PG58-22	Granite	5.7

2.4.2. *Bitumen Aging.* To age the bitumen for the pull-off test, the RTFO (ASTM D2872) in the short term, and the PAV (ASTM D6521) method in the long term were performed (ASTM D2872-22 and ASTM D6521-22) [25, 26].

The RTFO method simulates bitumen aging during the asphalt mixture manufacturing process. Briefly, 35 ± 0.5 g of bitumen was poured into containers with specific dimensions and placed inside the preheated oven. The oven temperature was set to 163.5°C , and the dishes were placed in oven for 85 min.

The PAV method simulates the long-term aging of bitumen that occurs during the pavement service life. The bitumen samples were aged for 6 days at $60\text{--}80^\circ\text{C}$ and under a pressure of 2,070 kPa. These conditions were obtained by simulating the aged bitumen on-site through coring.

2.5. *Moisture Sensitivity Tests of Asphalt Mixtures.* After determining the optimal bitumen percentage, samples were fabricated for moisture sensitivity tests according to the combinations listed in Table 7. To this end, the samples were subjected to moisture conditions (AASHTO T283). Based on this test, to simulate the conditions in the pavement site [27], the samples should be compacted between 6% and 8% of the air void. Therefore, asphalt samples with an air void of 6%–8% were made (ASTM D 4867). According to the combinations of this test, three dry and three wet samples were fabricated for each combination mode (ASTM D 4867). Due to the use of the Marshall method, samples with 45 and 75 blows were fabricated; after obtaining the percentage of void according to the interpolation method, the number of blows necessary to make the sample with a 7% air void percentage was calculated. The test to determine the resilient modulus was then performed on these samples.

The samples were saturated between 70% and 80% by a vacuum pump under a pressure of 35 kPa. Each aggregate type had a different time to reach the desired saturation percentage due to the variety of aggregates. The saturation percentage of the sample was calculated based on the following equations:

$$Pa = 100 \times \frac{Gmm - Gmb}{Gmm}, \quad (1)$$

$$Va = \frac{Pa \times E}{100}, \quad (2)$$

$$J = B - A, \quad (3)$$

$$S = \frac{100 \times J}{Va}, \quad (4)$$

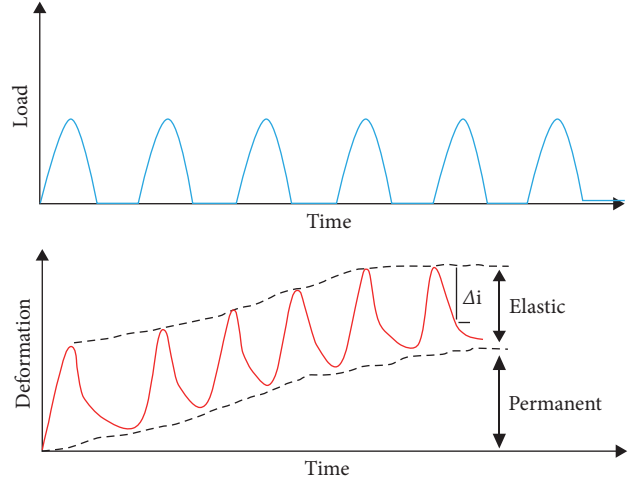


FIGURE 4: Loading and deformation in the resilient modulus test.

where Pa is air void in the asphalt mixture (%), Va is the void volume of the asphalt mixture (cm^3), E is the sample volume (cm^3), B is the weight of the saturated sample with a dry surface after saturating the sample with a vacuum pump (gr), and S is the sample saturation percentage (%).

The samples were saturated and then transferred to an isolated plastic bag. This bag was then placed inside another plastic bag containing 10 mL of water and stored in a freezer at -18°C for 16 hr. After removal from the freezer, the samples were submerged in a 60°C water bath for 24 hr. Following this, they were left at 25°C for an hour before being prepared for the tensile modulus test [28]. ITSM and MSR were determined based on Equations (5) and (6), respectively [29]. Finally, the MSR parameter (the ratio of wet samples' Mr to dry samples' Mr) was calculated.

$$Mr = \frac{P(\nu + 0.2734)}{\delta t}, \quad (5)$$

where P is the maximum load applied (N), m is Poisson's ratio, t is the length of the sample (mm), and d is the horizontal recoverable deformation (mm).

$$\text{MSR} = \frac{\text{ITSM}_{\text{wet}}}{\text{ITSM}_{\text{dry}}} \times 100. \quad (6)$$

2.6. *ITSM Test.* The ITSM test to determine the resilient modulus in asphalt mixtures in the indirect measurement mode is a well-known stress–strain measurement test and one of the most important tests to determine pavement characteristics. This method is used to determine the indirect tensile strength via the Nottingham device (ASTM D4123). The force is applied linearly and widely along the sample diameter; the loading cycle is 0.1, and the rest cycle is 0.9. The loading cycle is depicted in Figure 4. The samples prepared in the Marshall mold had a diameter of 10.1 cm and a height of 6.8 cm. The loading method on each sample was as

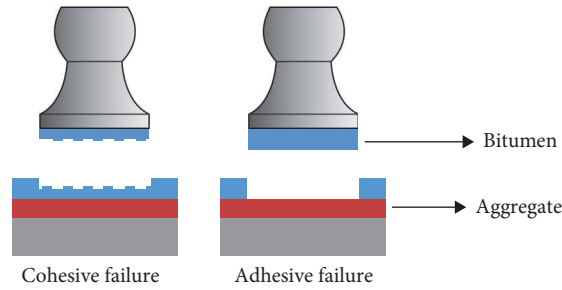


FIGURE 5: Two type of failure in pull-off test.

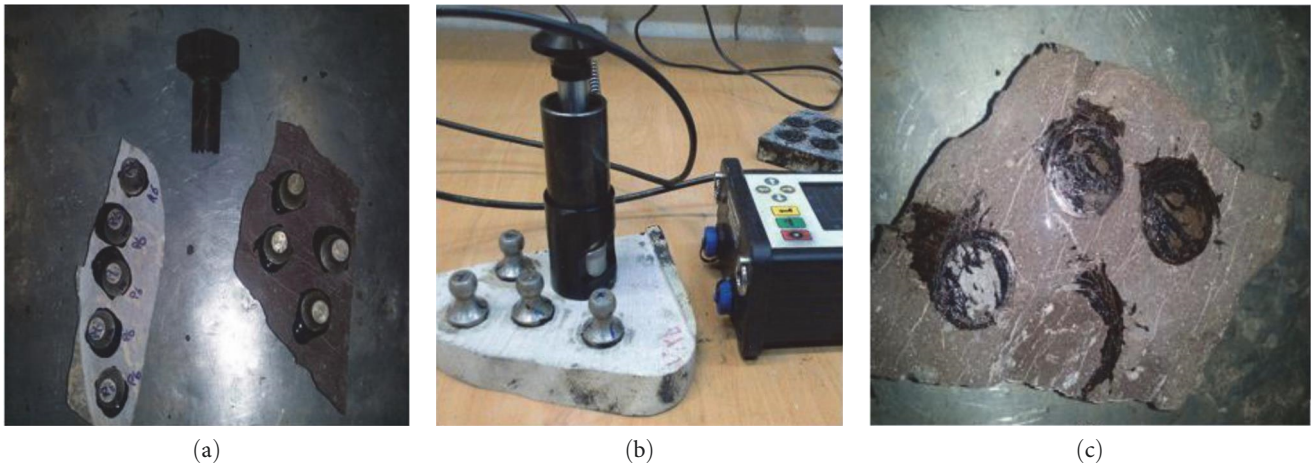


FIGURE 6: Pull-off instruments and mechanisms: (a) relation of preservative to bitumen sample; (b) connecting the device to the preholder; (c) adhesion failure produced in the pull-off test.

follows: loading was performed once in a specific direction; then, the sample was taken out of the machine, rotated 90° around the center, and loaded on the machine for the second time. Because the deformations of this test remain in the elastic range, this test is considered nondestructive. Poisson's ratio is assumed to be 0.35.

2.7. Pull-Off Test. This test measures the bonding and tensile strength of the bitumen adhered to a sheet. The air adhesion tester is used to determine the pressure necessary to separate the samples under moisture conditions at 25°C. The adhesion strength for the aggregate-bitumen combinations was measured (ASTM D4541), and mixture performance was predicted in the laboratory (ASTM D 7234) [30].

Each bitumen-aggregate sample was placed in a 60°C water bath for 1 hr. The thickness of the bitumen membrane was such that rupture occurred at the bitumen-aggregate contact surface. Thus, the bitumen thickness was considered to be 0.5 mm based on previous studies [31]. There are two types of failure in the pull-off test, as shown in Figure 5. Finally, based on the type of failure, the test results were evaluated and compared with other tests. In this experiment, the tension rate was around 66 kPa/s. Before the adhesion process, the desired aggregate surface was placed in the ultrasonic device for 1 hr. The preservatives surface was then cleaned from any pollutants, dust, or oxidation using a tissue soaked in acetone because the pollutants could affect the test

results. Before the stretching process, a circular groove the size of the preretainer diameter was created using a special tool to remove the adhesion factor of the bitumen layer. Figure 6 depicts the pull-off test device and a sample of aggregates with a surface prepared for the pulling process.

$$\text{POTS} = \frac{(\text{BP} * A_G) - C}{A_{PS}} \quad (7)$$

where POTS is pull-off tensile strength (kPa), BP is burst pressure (kPa), A_G is the contact area of the gasket to the reaction plate (mm^2), C is piston constant (provided by the manufacturer), and A_{PS} is the area of the pull-off stub (mm^2).

3. Results and Discussion

3.1. Results of the Elasticity Modulus Test (M_r). Figure 7 displays the simultaneous effect of Wetfix and NHL on the modulus of elasticity in bitumens and aggregates in the base state. Wetfix in the PG64-16 bitumen increased the modulus of elasticity in limestone aggregates by 35% in the dry state and 51% in the wet state in the comparison with base state, but for granite aggregates, this increase was 15% in the dry state and 35% in the wet state (Figure 7). Therefore, the addition of Wetfix to limestone aggregates and the PG64-16 bitumen increased the modulus of elasticity in both wet and dry

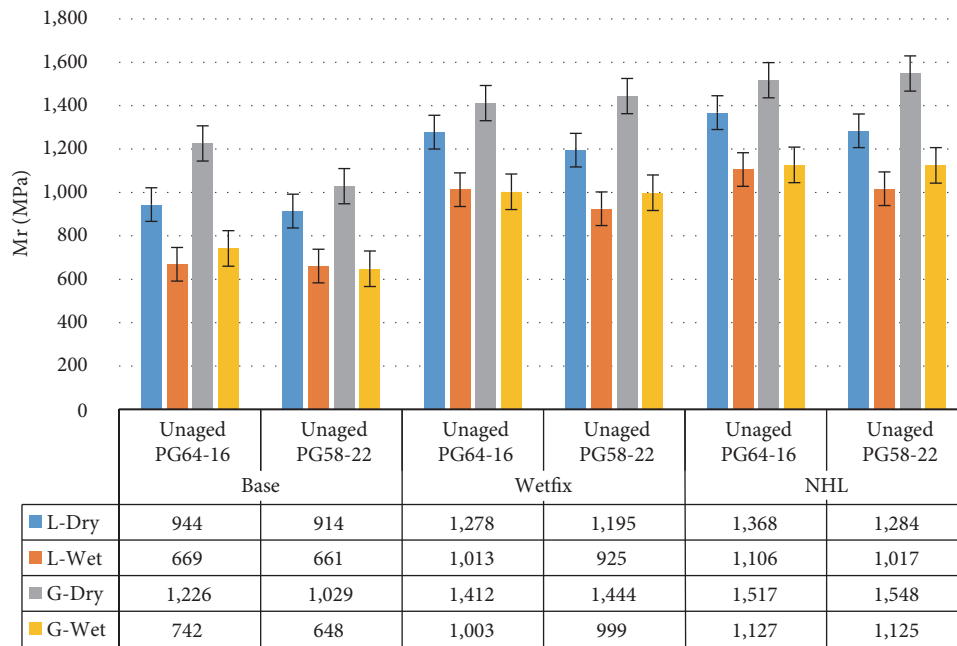


FIGURE 7: The effect of Wetfix and nanohydrated lime on the Mr in the base state.

states more than granite aggregates. Limestone aggregate is more resistant than granite aggregate because of the hydrophobic property of limestone aggregate. A similar result was indicated for wetfix additives in previous research by Arabani and Rahimabadi [6].

However, for the PG58-22 bitumen, the addition of Wetfix raised the modulus of elasticity in limestone aggregates by 30% in the dry state and 40% in the wet state; in granite aggregates, this value was raised by 40% in the dry state and 54% in the wet state. This demonstrates the opposite effect of Wetfix on the modulus of elasticity growth of limestone and granite aggregates in PG58-22 compared to PG64-16 due to the structural differences between the two types of bitumen in combination with different aggregates. Compared to the effect of Wetfix on the modulus of elasticity, with the addition of NHL, the modulus of elasticity in both types of aggregates and both types of bitumen was 7%–12.5% higher than in samples contain Wetfix. This indicates that the effect of NHL on the modulus of elasticity of hydrophilic granite aggregates was more significant.

Figure 8 compares the variation of the modulus of elasticity in short-term aging with Wetfix and NHL. The modulus of elasticity in PG58-22 with limestone aggregates in both dry and wet conditions showed a smaller elevation than in the PG64-16 bitumen; nevertheless, for granite aggregates in both dry and wet conditions, this increase was greater in PG58-22 than PG64-16 due to the degree of penetration. The modulus of elasticity rose in the mixtures containing both types of additives, but the addition of NHL compared to Wetfix caused a greater elevation in the modulus of elasticity, especially in wet conditions (Figure 8). A softer bitumen like PG58-22 has better flexibility and can withstand deformation caused by heavy traffic loads in wet conditions. On the other

hand, a stiffer bitumen like PG64-16 may crack or break under similar conditions.

Figure 9 shows the changes in the modulus of elasticity in the long-term aging cycle with Wetfix and NHL. The modulus of elasticity increased in both types of bitumen and limestone aggregates but slightly decreased in both types of bitumen and granite aggregates in the dry state. For the wet state and limestone aggregates, the modulus of elasticity increased in PG64-16 but remained almost constant in PG58-22. The rise in the modulus of elasticity with the addition of NHL was more significant than with Wetfix, and this difference was greater in wet than in dry conditions.

The comparison of all aggregate composition states in both types of bitumen and the base state, short-term aging, and long-term aging showed that NHL and Wetfix effectively raised the asphalt mixture yield modulus. In this increase, the effect of NHL was greater than Wetfix because it has a high surface area, which allows it to react more quickly with the asphalt binder and other components in the mixture. This suggests the improvement of the mechanical properties of asphalt mixtures in all conditions, even in wet conditions, by both NHL and Wetfix.

Figures 10–12 depict MSR values for the states without additives, containing Wetfix and containing NHL. Figure 9 displays the state without additives, in both types of bitumen and granite aggregates, and all three states (base, short-term aging, and long-term aging). The MSR values in this figure are less than the permissible limit of 70%. However, in limestone aggregates in the base state and short-term aging, the MSR values in both types of bitumen are about 70%, which is due to the use of water-resistant limestone aggregates.

Figure 11 illustrates the MSR values of the mixtures containing Wetfix. The values are within the allowed range in all

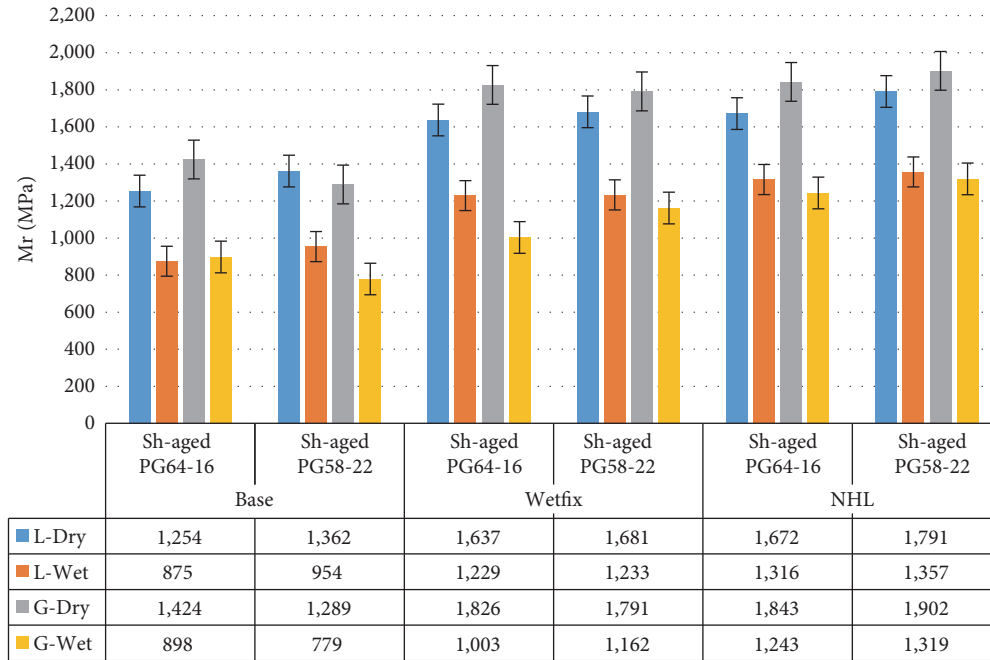


FIGURE 8: The effect of Wetfix and nanohydrated lime on the Mr in short-term aging.

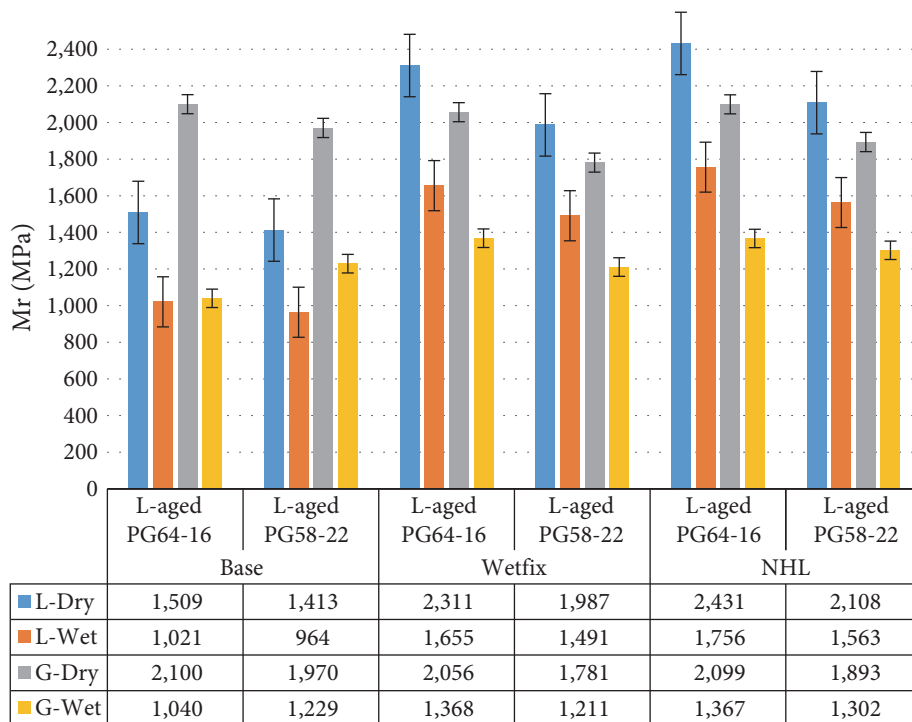


FIGURE 9: The effect of Wetfix and nanohydrated lime on the Mr in long-term aging. L-Dry, limestone dry; L-Wet, limestone wet; G-Dry, granite dry; G-Wet, granite wet.

the samples fabricated with limestone aggregate; yet, in the mixtures made with granite aggregate, after applying short-term and long-term aging cycles, the MSR values are still below 70% even with the addition of Wetfix. This indicates that Wetfix in granite aggregates has less ability to improve the aged asphalt mixture. Thus, with the occurrence of aging,

the modulus of elasticity increases less in the wet than in the dry state. In this case, despite the addition of Wetfix, there is no significant difference in MSR values in short- and long-term aging modes. This shows that although Wetfix significantly increases the modulus of elasticity in dry and wet states, it is not a suitable option to deal with the aging

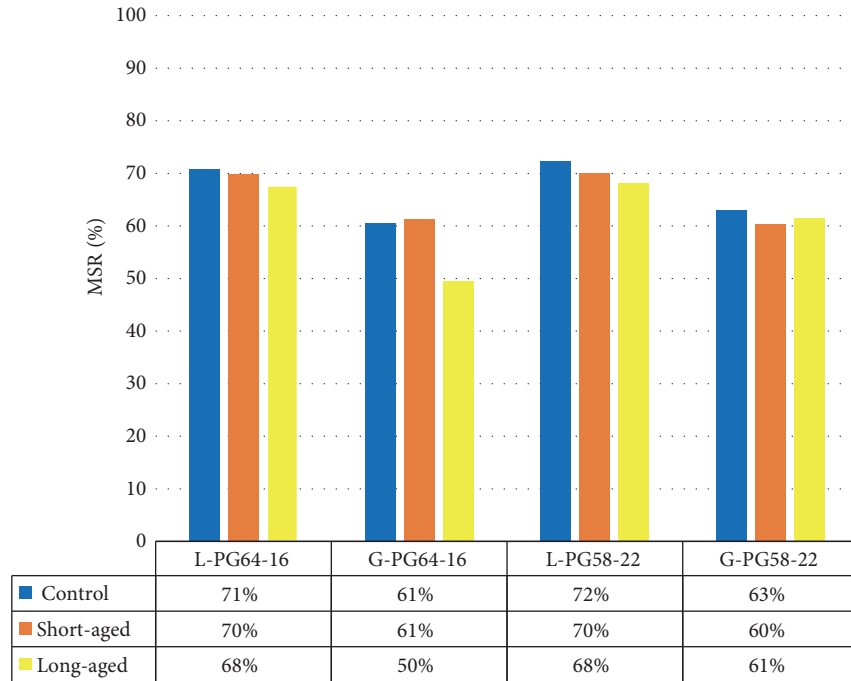


FIGURE 10: Comparison of MSR in all three modes of samples without additives.

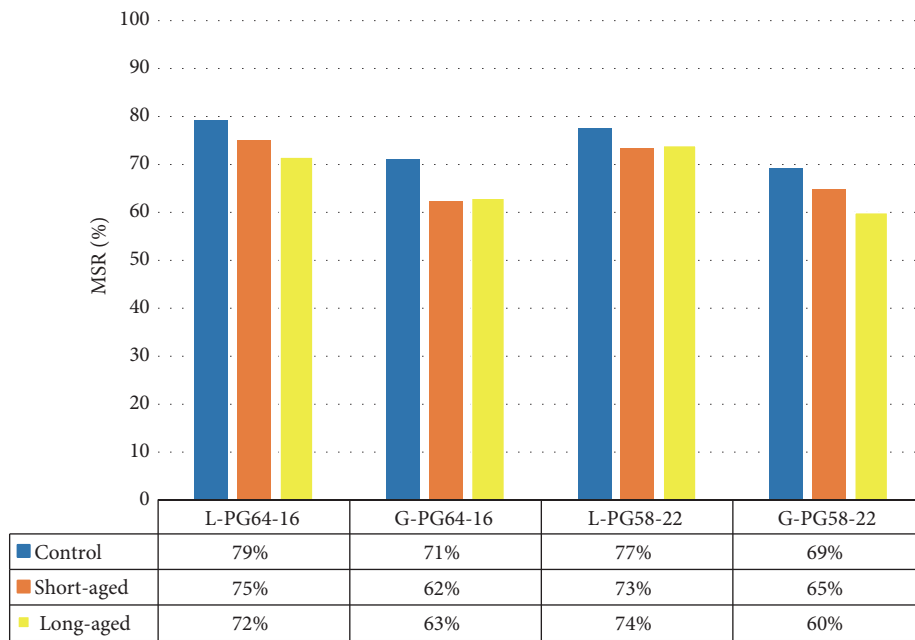


FIGURE 11: Comparison of MSR in all three modes of samples with Wetfix.

phenomenon in terms of the ratio of its effects in wet-to-dry conditions (MSR).

Figure 12 illustrates the values of MSR in asphalt mixtures containing NHL in unaged, short-term-aged, and long-term-aged states. In all dry and wet conditions, the presence of NHL increased the MSR. In aging conditions, in almost all cases except for granite aggregates and PG64-16 bitumen, NHL raised the MSR to be within the allowable range in

short- and long-term aging. This indicates that NHL is a relatively suitable option to deal with the aging phenomenon.

3.2. *Pull-Off Test Results.* Figure 13 shows the values of the pull-off ratio in both types of bitumen (PG64-16 and PG58-22) and both types of aggregates (limestone and granite) in the base state, containing Wetfix and containing NHL. In the bitumen containing NHL in all four bitumen and aggregate

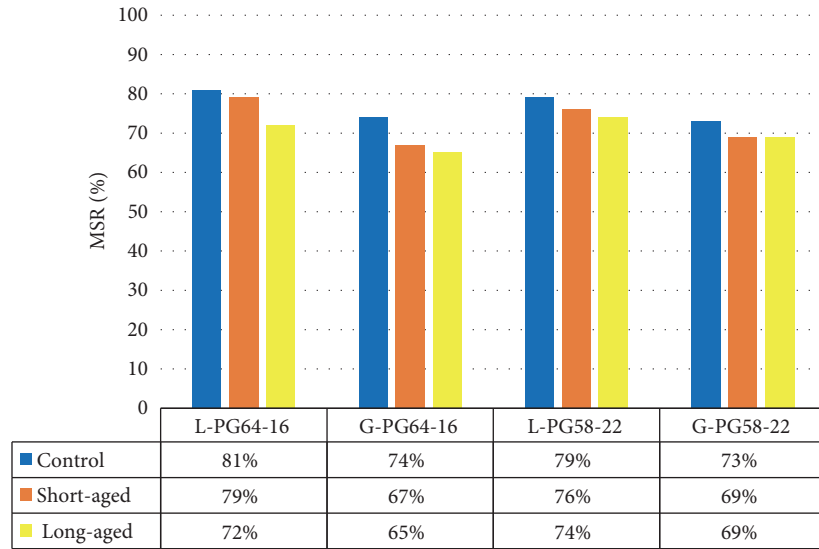


FIGURE 12: Comparison of MSR in all three modes of samples with nanohydrated lime.

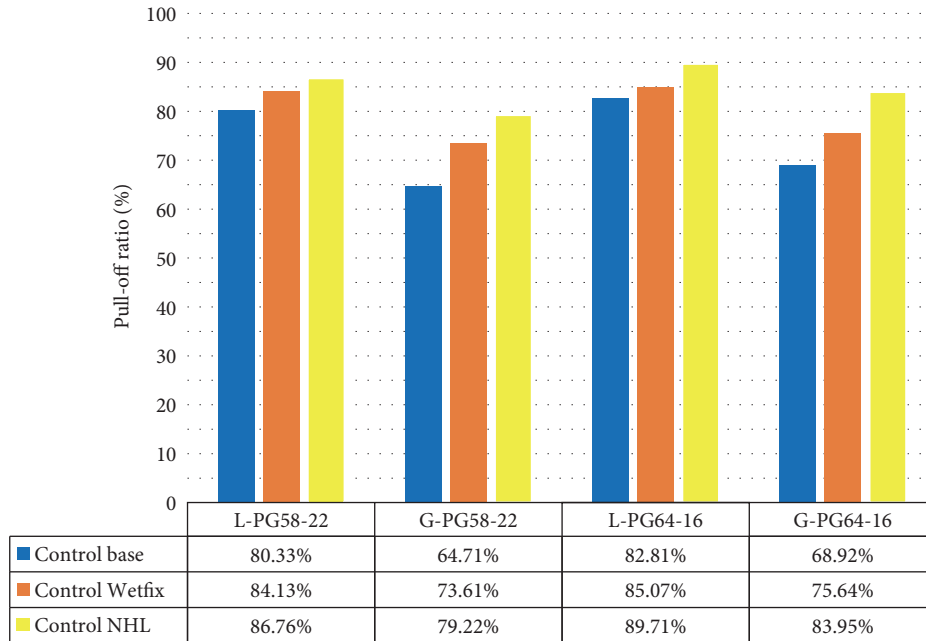


FIGURE 13: Comparison of pull-off ratio in adhesion failure with two types of additives in the control mode.

combinations, the pull-off ratio for the samples in wet conditions greatly increased compared to dry conditions. According to this graph, in the pull-off test results of limestone aggregates, because it has a high surface area, which allows it to react more quickly with the asphalt binder and other components in the mixture, Wetfix had a slight effect on increasing the pull-off ratio, while in the case of granite aggregates, Wetfix had a noteworthy effect on raising the pull-off ratio. Because it can improve the asphalt binder–aggregate adhesion between the particles, reducing the risk of stripping or raveling.

According to Figure 14, after the short-term aging cycle, the performance of additives in improving the pull-off ratio

was greater. This increase was larger in compositions containing NHL than in those containing Wetfix. As for limestone aggregates, the pull-off ratios of short-term-aged samples were within the allowed range (70%–80%), but in the case of granite aggregates, in the combinations without additives and after aging, pull-off ratios were not within the allowed range. This indicates that appropriate modifiers such as NHL should be used in wet conditions to utilize granite aggregates in asphalt mixtures.

According to Figure 15, which depicts the effect of additives on the pull-off ratio after passing the long-term aging cycle, the pull-off ratio improved in granite aggregate compositions and both types of bitumen (PG64-16 and PG58-22)

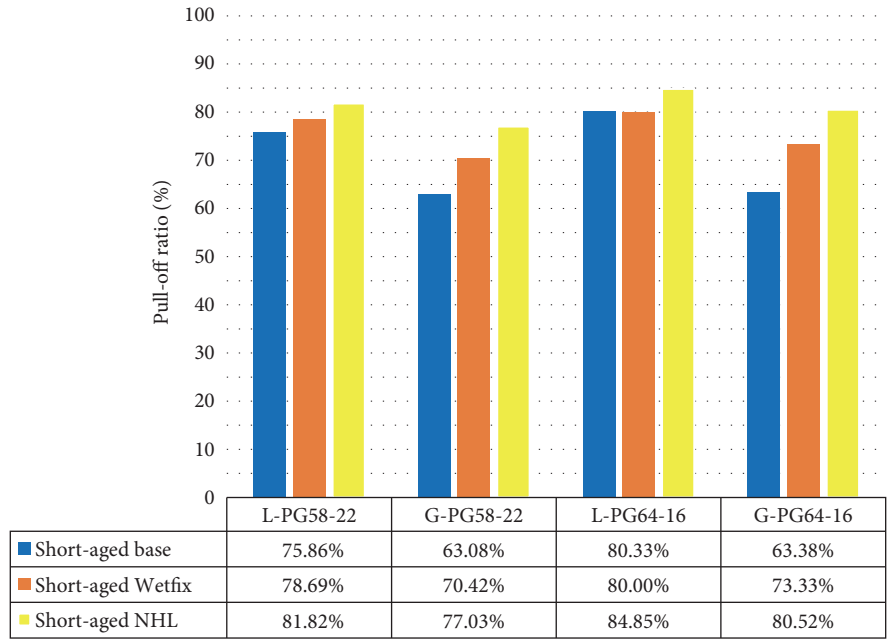


FIGURE 14: Comparison of pull-off ratio in adhesion failure with two types of additives in the short-term aging mode.

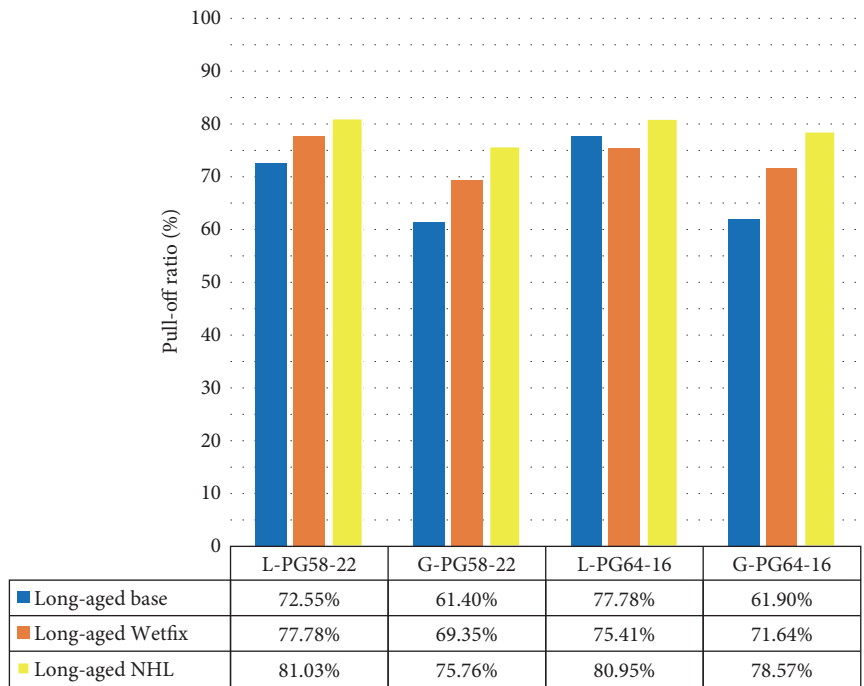


FIGURE 15: Comparison of pull-off ratio in adhesion failure with two types of additives in the long-term aging mode.

containing NHL. In limestone aggregates, the pull-off ratio increased in PG58-22 in both types of additives. The pull-off ratios rose in the samples containing lime aggregates, PG64-16 bitumen, and NHL, but declined in the samples containing Wetfix. This shows that NHL has better effects than Wetfix in long-term aging conditions.

Overall, it can be concluded that the adhesion strength between lime aggregates and bitumen had favorable values in the base state, but this was not the case with granite aggregates

and the values did not reach the permissible limit. However, antistripping additives were effective in improving all conditions. In the short- and long-term aging conditions, the adhesive strength of the aggregates and bitumen further declined, so even lime aggregates that are more resistant to moisture did not perform well in the moisture cycle. Nevertheless, with the use of Wetfix and NHL, the decline in the adhesion strength of bitumen and aggregates decreased. This was confirmed in a research by Arabani et al. [32].

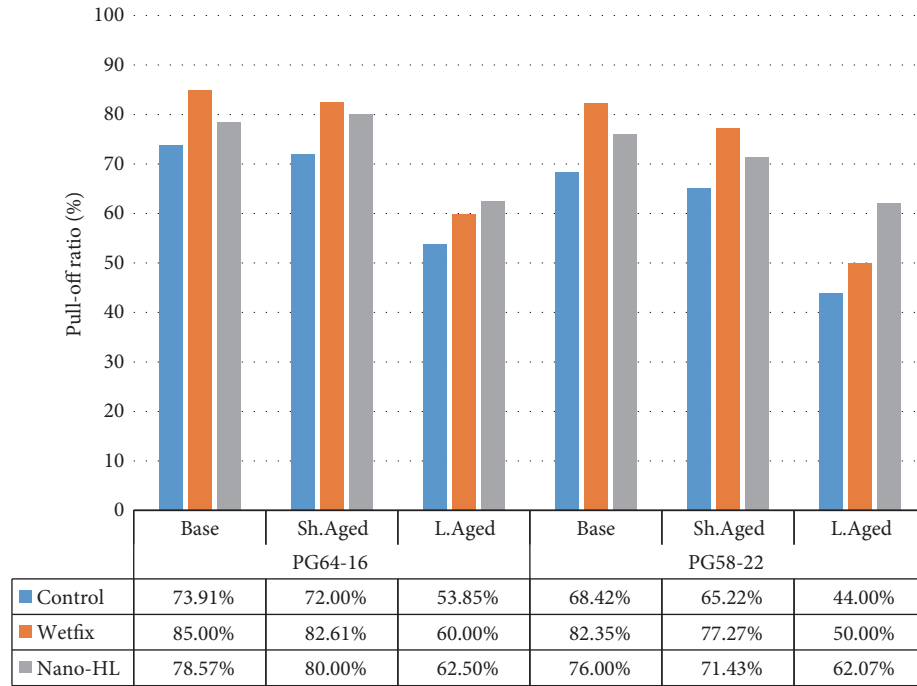


FIGURE 16: Pull-off ratio for cohesion failure.

TABLE 9: *t*-test results on the ITSM for base condition.

Variable	Obs.	State	PG64-16, limestone		PG58-22, limestone		PG64-16, granite		PG58-22, granite	
			<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value
Control Wetfix (0.4%)	66	Dry	-9.5685	0.0000	-6.2802	0.0001	-5.6596	0.0001	-7.1640	0.0000
Control nano-HL (1%)	66	Dry	-12.4587	0.0000	-6.2620	0.0001	-5.1211	0.0004	-7.6064	0.0000
Control Wetfix (0.4%)	66	Wet	-7.7002	0.0000	-5.2513	0.0004	-3.5700	0.0000	-5.6418	0.0002
Control nano-HL (1%)	66	Wet	-6.0524	0.0001	-7.3790	0.0000	-5.7094	0.0002	-8.2155	0.0000

TABLE 10: *t*-test results on the ITSM for short-aged (Sh.A) condition.

Variable	Obs.	State	PG64-16, limestone		PG58-22, limestone		PG64-16, granite		PG58-22, granite	
			<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value
Short-aged Wetfix Sh.A (0.4%)	66	Dry	-4.5696	0.0010	-6.1719	0.0001	-5.2214	0.0004	-8.3007	0.0000
Short-aged nano-HL Sh.A (1%)	66	Dry	-5.7853	0.0002	-8.3540	0.0000	-6.5945	0.0001	-10.1208	0.0000
Short-aged Wetfix Sh.A (0.4%)	66	Wet	-7.5811	0.0000	-4.2964	0.0016	-8.4875	0.0000	-5.5936	0.0002
Short-aged nano-HL Sh.A (1%)	66	Wet	-8.9018	0.0000	-5.3399	0.0003	-9.8697	0.0000	-9.5900	0.0000

Figure 16 displays the cohesion strength of bitumen molecules in two types of bitumen, PG64-16 and PG58-22, using the pull-off test. The pull-off ratios in the unaged state without additives did not greatly differ from each other compared to short-term aging, but these values markedly declined in long-term aging conditions. The reason is that the cohesion between bitumen particles becomes very weak in long-term aging conditions. According to Figure 16, with the addition of Wetfix and NHL, the pull-off ratios increased in the base state. The pull-off ratios of the samples containing Wetfix increased more than those containing NHL. As for short-term aging in the case of PG64-16 bitumen, both types

of additives performed well, but the performance of Wetfix was better in PG58-22 bitumen. As for long-term aging, in the case of both PG64-16 and PG58-22, the addition of NHL had a greater effect on increasing the pull-off ratio than the samples containing Wetfix. The previous study by Rahmani et al. [11] confirms this conclusion.

3.3. *Statistical Analysis Results.* A *t*-test was used to analyze the data and determine if there were significant differences between the means at a 95% confidence level. The results were presented in three Tables 9–11 that listed the *t*-test results for samples under dry and wet conditions. The first Table 9 was

TABLE 11: *t*-test results on the ITSM for long-aged (L.A) condition.

Variable	Obs.	State	PG64-16, limestone		PG58-22, limestone		PG64-16, granite		PG58-22, granite	
			<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value	<i>t</i>	<i>P</i> -value
Long-aged Wetfix L.A (0.4%)	66	Dry	-16.3220	0.0000	-10.0798	0.0000	1.0050	0.3386	4.5463	0.0011
Long-aged nano-HL L.A (1%)	66	Dry	-8.5440	0.0000	-9.8315	0.0000	3.3657	0.0072	-1.7368	0.0000
Long-aged Wetfix L.A (0.4%)	66	Wet	-16.0294	0.0000	-8.7112	0.0002	-3.5700	0.0000	-1.3223	0.0000
Long-aged nano-HL L.A (1%)	66	Wet	-13.6899	0.0002	-9.2630	0.0000	-5.7094	0.0002	-2.8884	0.0162

related to the base samples, while the second Table 10 was related to short-aged samples, and the third Table 11 was related to long-aged samples of asphalt mixtures. Each table was divided into two parts due to the number of composition–construction modes considered for each processing mode. The *p*-value of asphalt samples was less than 0.05, indicating a significant difference between the means at a 95% confidence level. Additionally, it notes that the *p*-values of cohesion for asphalt samples containing additives were less than 0.05, indicating a significant effect of Wetfix and NHL on increasing the modulus of elasticity of the samples. Overall, the results showed that a certain amount of two additives had a significant effect on increasing modulus elasticity in asphalt mixtures.

4. Conclusions and Recommendations

4.1. Conclusions. In current research, the effect of Wetfix and NHL was examined on the aging and moisture sensitivity of asphalt mixtures. The main results of this research are as follows:

- (i) The ratios of the ITSM values of wet/dry mixtures (MSR) containing both Wetfix and NHL were higher than control mixtures for both types of aggregates and bitumen used in this study (15%–51%). These findings suggest that incorporating NHL and Wetfix into asphalt mixtures could enhance their resistance to wet conditions and improve their overall performance.
- (ii) The MSR ratio of the mixtures in the short-term and long-term aging state was about 13% more for limestone aggregates than granite aggregates. Asphalt mixtures containing Wetfix and NHL were found to have MSR values within permissible limits for limestone aggregates, but not for granite aggregates. It is indicated that using NHL and limestone aggregate is an optimum combination against moisture damage.
- (iii) The pull-off test results showed that the adhesive strength of the pull-off ratios increased in all combinations, but this increase was greater in the combinations of both types of bitumen and lime aggregates due to the hydrophobic properties of limestone compared to granite. This improvement was greater in compositions containing NHL than in those containing Wetfix. In short-term and long-term aging, the decrease in the pull-off ratio was more significant. This issue is due to the penetration of water into the

aged asphalt mixture. Additives increased the resistance of the asphalt mixture against adhesion failure (Wetfix: 4%–15% and NHL: 4%–27%).

- (iv) According to the investigation of pull-off ratios regarding the cohesion failure of bitumen molecules, in the base state and short-term aging and in both types of bitumen, the Wetfix additive increased the pull-off ratio more than NHL; however, in long-term aging, the pull-off ratios of the mixtures containing NHL increased a little more than those containing Wetfix. This indicated that as the duration of the aging cycle was increased, the NHL outperformed Wetfix in improving the pull-off ratio.
- (v) In general, the results of this study show that NHL can be a more effective alternative to traditional adhesion promoters like Wetfix in improving the adhesion strength of bitumen. In the improvement of the mixture, cohesion performs the same function. This can lead to better performance and durability of asphalt pavements.

4.2. Recommendations. According to the conducted tests, review and evaluation of their results, and considering previous research, the following recommendations are made:

- (i) Investigation of the field performance of the effect of aging on the moisture deterioration of asphalt mixture. This will provide valuable insights into how aging affects the overall durability of asphalt pavements.
- (ii) A comparison of cohesion failure and adhesion failure due to aging of asphalt mixture from a micro-point of view.
- (iii) Using surface free energy method to investigate the cohesion and also the adhesion between asphalt aggregates for a more comprehensive analysis by this new method containing Wetfix and NHL.
- (iv) The effect of Wetfix and NHL on porous asphalt because of their more sensitivity against moisture damage.

Data Availability

The data used to support the findings of this study are currently under embargo while the research findings are commercialized. Requests for data, 3 months after publication of this article, will be considered by the corresponding author.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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