

Research Article

Studying Engineering Characteristics of Asphalt Binder and Mixture Modified by Nanosilica and Estimating Their Correlations

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The objective of this research was to investigate rutting and fatigue distresses in asphalt containing 2, 4, 6, and 8 percent of nanosilica (NC) and to find out the correlation between engineering properties of the modified binder and mixture asphalt. In order to study the effect of NC on the rutting and fatigue properties of modified binders, the multiple stress creep recovery (MSCR) and linear amplitude sweep (LAS) tests were carried out. The Marshall stability, dynamic creep, and four-point bending beam fatigue tests were used to evaluate performance characteristics of the mixtures. The binder and mixture tests all indicated an improvement of fatigue and rutting resistance using NC as a modifier. Furthermore, some statistical correlations between engineering properties were developed successfully.

1. Introduction

Binder plays an important role in preventing common distresses associated with asphalt mixture such as fatigue cracking and rutting. For instance, binder with enough adhesion and cohesion can significantly hinder the segregation and separation of aggregates from the pavement surface [1]. In order to improve the behavior of asphalt binder at different temperatures, many types of additives have been used. Among them, nanomaterials have been used by many researchers for their high surface area and capability of creating powerful networks in asphalt binder, culminating to an increase in the mixture's resistance to permanent deformation [2]. Performance characteristics of binders and asphalt mixtures have been affected to some extent due to the addition of nanoparticles such as nanoclay, nanolime, carbon nanofiber, and carbon nanotube [2, 3].

The effect of nanoclay as an asphalt binder additive on the mechanical properties of the asphalt mixture demonstrated a considerable increase on rutting resistance and resilient modulus of asphalt concrete (AC) samples. However, the additive has no considerable effect

on low-temperature fatigue resistance of the modified sample [4].

Amirkhanian et al. studied the effects of carbon nanoparticles on the performance characteristics of asphalt binder. The viscosity, performance grade (PG), creep and creep recovery, and frequency sweep tests were carried out on modified binder. Experiment results indicate that the addition of the carbon nanoparticle was effective in increasing viscosity, failure temperature, complex modulus and elastic modulus, and as a result, rutting resistance of the binder [5].

Yao et al. used NC as a binder modifier. They added NC to an SBS-modified asphalt in 4 and 6 percent by the weight of the modified base binder. Experiment results showed that the value of viscosity at high temperatures decreased slightly; in fact, at low temperatures, modified binder with NC behaves similar to control binder samples; furthermore, NC improved binder antioxidation characteristics. The rutting and fatigue cracking performance of asphalt binder modified by NC was improved [6].

Among the advantages of NC are its functional features and low-cost production. NC is one of the new minerals which include potential useful features, such as huge surface

area, good distribution, high absorption, high stability, and high percentage of purity.

Today, researchers are looking for binder tests that not only could demonstrate the mixture's performance-related characteristics of both modified and unmodified binder but also are easy and quick to conduct. Insufficiency of the performance grade (PG) binder specification as one of the common methods to evaluate binder performance, especially when it is modified or rejuvenated by additives, has been proved by many researchers [7, 8]. To address this issue and as a way to find a better performance-related test method, the LAS and MSCR tests were introduced to evaluate fatigue and rutting performance of the binder, respectively. The LAS test showed a good correlation with long-term pavement performance (LTPP) field fatigue cracking data [9]. Furthermore, unlike the existing SHRP test method, MSCR captures the nonlinear behavior of rutting phenomenon and correlates fairly well with field rutting data [7].

In this research study, the binder is modified by 2, 4, 6, and 8 percent of NC, and two important distresses of asphalt, rutting and fatigue, are evaluated through the LAS, MSCR, 4-point bending beam, and dynamic creep tests. Finally, some correlation between binder and mixture test results was developed successfully.

2. Materials

The asphalt binder used in this study was AC-60/70, provided by Pasargad Oil Company, Tehran. The characteristics of the binder are presented in Table 1. The required aggregates to produce the sample are taken from Asb-Cheran Mine located in Roudehen in the north of Tehran. Rock dust is used as the filler in the production of samples. The characteristics of the aggregates are presented in Table 2. The gradation of aggregates is according to AASHTO M323 and presented in Table 3 and Figure 1. NC used in this research study has a purity of more than 99%. The maximum diameter of the particles is 10 nm, and the surface area is 600 m²/g. Its bulk density is less than 0.10 g/cm³, and the true density is 2.4 g/cm³.

3. Sample Preparation

NC is added to the asphalt binder by 2, 4, 6, and 8 percent of the original binder's weight. A high-shear mixing device is used to mix NC and binder with 4000 rpm for 2 hours at 135°C. The SEM images of the modified binder with 4 percent NC in three magnitudes are shown in Figure 2. Accordingly, particles' diameters are roughly between 50 and 150 nanometer.

The Marshall method was used to determine the stability, flow, and optimum binder of all asphalt samples (ASTM D2726 and ASTM D1559). Percentage of optimum binders obtained were 5.5, 5.3, 5.2, 5 and 4.9 at mixtures with 0%, 2%, 4%, 6% and 8% of NC content, respectively. The samples were compacted using a Gyratory compactor for the dynamic creep, indirect tensile strength, and resilient modulus tests. Samples used in fatigue tests were originally fabricated as slabs with dimensions of 5*30*40 cm using wheel track compactor. All the samples made at optimum binder and 4% air

TABLE 1: Physical properties measured of bitumen.

Parameter measured	Test method	Test value
Specific gravity at 25°C (g/cm ³)	AASHTO T228	1.01
Penetration at 25°C (0.1 mm)	AASHTO T49	60
Softening point (R&B) (°C)	AASHTO T53	56
Viscosity at 120°C (centistokes)	AASHTO T201	1055
Viscosity at 135°C (centistokes)	AASHTO T201	361
Viscosity at 160°C (centistokes)	AASHTO T201	170
Ductility at 25°C (cm)	AASHTO T51	>100

void. Then, they were sawn to the prismatic beams with dimensions of 38.5 mm × 63.5 mm × 50 mm, considering the AASHTO T321 standard [10].

The binder performance characteristic tests have been carried out on aged samples. Prior to the multiple stress creep recovery (MSCR) and linear amplitude sweep (LAS) tests, all the modified binder samples, as well as the 60/70 base binder, were aged in the rolling thin-film oven (RTFO) in order to represent a short-term aging condition.

4. Experimental Design

4.1. Multiple Stress Creep Recovery (MSCR). This test has been used to measure the percent of recovered strain (R) and unrecovered strain (j_{nr}) of asphalt binders. The elastic response of the binder under the shear stresses can be calculated by this test methodology. The aged samples in the RTFO process are used in this test method. In order to conduct the MSCR test, the dynamic shear rheometer (DSR) is used. The binder sample is put under a 0.1 kPa shear stress for a 1-second duration, followed by a 9-second rest period at the temperature of 60°C. This loading repeats for 10 cycles. Then, after the completion of the first ten cycles, a similar procedure will be applied to the sample with a stress level of 3.2 kPa. According to the ASTM D-7405-10a standard, at each 0.1 sec interval, the relevant output should be recorded [11].

4.2. Linear Amplitude Sweep (LAS) Test. This test was proposed by Johnson and Hintz to investigate the fatigue resistance of asphalt binders [12]. According to the AASHTO standard (AASHTO-TP 101-12-UL), the binder samples of 8 mm thickness are tested in the dynamic shear rheometer (DSR). All DSR tests are conducted on RTFO aged samples. The test is carried out under the strain-controlled mode with linearly increased load amplitudes from 0.1% to 30% strain in a total time of 310 seconds [13].

In the viscoelastic continuum damage (VECD) analysis, the binder fatigue performance parameter N_f can be calculated by

$$N_f = A_{35} \times \gamma^B, \quad (1)$$

where N_f is the number of cycles to failure, A_{35} is the damage intensity corresponding to 35 percent reduction of undamaged $|G^*| \sin \delta$, and B demonstrates the binder sensitivity to applied strain level. The parameters A_{35} and B are experimentally defined, and γ is the applied shear strain.

TABLE 2: Properties of used aggregates.

Properties	Method	Requirement	Values
<i>Coarse aggregate</i>			
Los Angeles abrasion (%)	AASHTO T96	30 max.	20
Water absorption (%)	AASHTO T85	5 max.	0.8
Bulk specific density (g/cm ³)	AASHTO T85	—	2.654
Flat and elongated (3-1) (%)	ASTM D4791	20 max.	12
Soundness (sodium sulfate) (%)	AASHTO T104	15 max.	4.8
Crushed content (one face) (%)	ASTM D5821	100 min.	100
Crushed content (two faces)	ASTM D5821	90 min.	100
<i>Fine aggregate</i>			
Water absorption (%)	AASHTO T84	—	1.4
Bulk specific density (g/cm ³)	AASHTO T84	—	2.617
<i>Mineral filler</i>			
Bulk specific density (g/cm ³)	AASHTO T84	—	2.702

TABLE 3: Gradation of the aggregates used in the study.

Sieve sizes	US	3/4"	1/2"	No. 4	No. 8	No. 50	No. 200
Sieve sizes	Metric	19	12.50	4.75	2.36	0.3	0.075
Passing (%)	HMA gradation	100	95	60	40	15	5

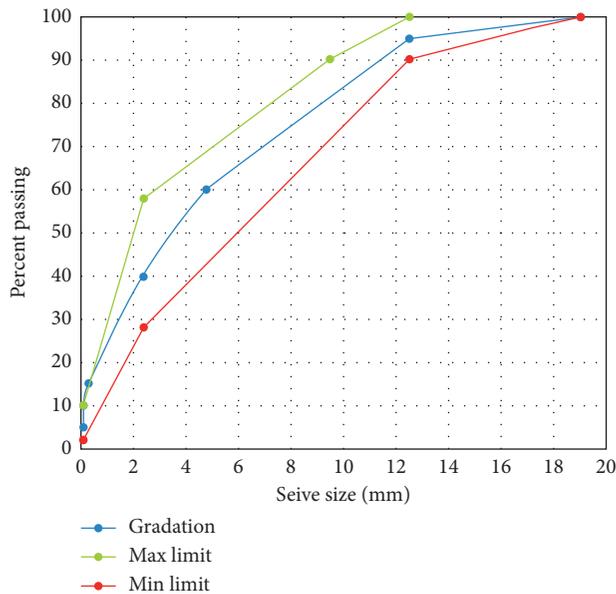


FIGURE 1: Gradation of designated aggregates.

4.3. Marshall Stability and Quotient. The test was carried out according to ASTM D1559. Before the test, all samples were put in 60°C water for 30 minutes. Marshall stability is the peak resistance load obtained during a constant rate of the deformation load sequence, and Marshall flow is a measure of deformation of the asphalt mix determined during the stability test. The Marshall quotient equals the ratio of the Marshall stability to the value of the Marshall flow. The value of the Marshall quotient indicates the resistance of asphalt mixtures against permanent deformations and the value of rutting [14].

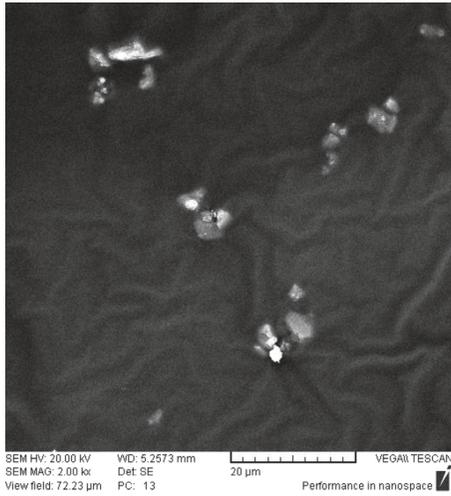
4.4. Resilient Modulus. Resilient modulus is one of the important parameters in the pavement design procedure. The measurement of this parameter is in the form of pavement response under dynamic loads and the corresponding strains associated with them. The value of resilient modulus is measured based on the ASTM D4123 [15] standard. This test is conducted at temperatures of 5°C and 25°C, and the minimum numbers of loadings are 100. The value of resilient modulus (M_r) can be obtained by [16]

$$M_r = P \frac{(\mu + 0.27)}{(t\delta_h)}, \quad (2)$$

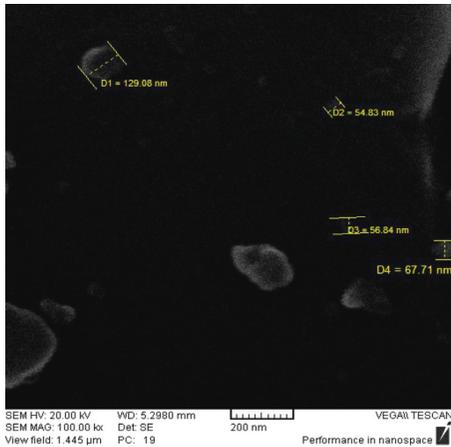
where P is the maximum dynamic load (N), μ is Poisson's ratio, t is the specimen length (mm), and δ_h is the horizontal recoverable deformation (mm).

4.5. Dynamic Creep Test. In the present research, the dynamic creeping test has been used to evaluate the rutting property of the asphalt mixtures. The output creep curve of the test is made of three areas. In the present article, the flow number (F_n) parameter is used as representation of rutting resistance of asphalt mixtures, which is the number of cycles the creep curve enters from the second to the third phase [17].

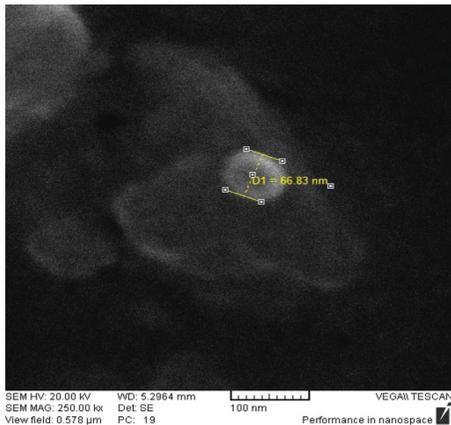
4.6. Four-Point Beam Fatigue Test. The fatigue life of the asphalt mixtures is evaluated by the 4-point bending beam test under AASHTO-T321 standard specifications. A constant sinusoidal loading was applied on beam specimens at constant strain levels of 600, 800, and 1000 microstrains until 50 percent reduction of initial stiffness.



(a)



(b)



(c)

FIGURE 2: SEM images of NC with asphalt binder: (a) 20 μm , (b) 200 nm, and (c) 100 nm.

5. Results and Discussion

5.1. Binder Test

5.1.1. MSCR Test Results. The MSCR test encompasses different outputs. The accumulated strain versus time over

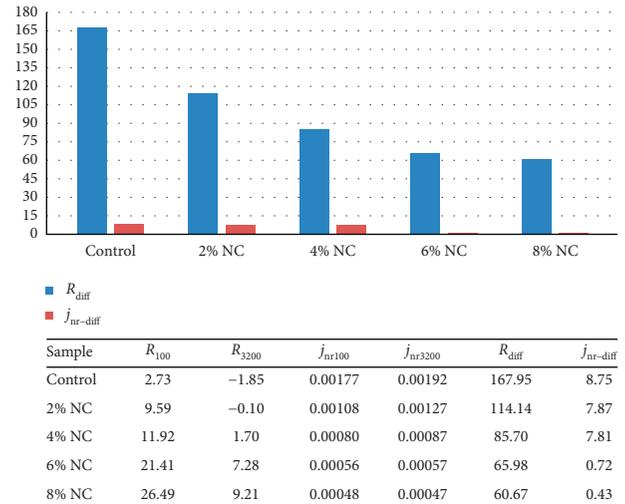


FIGURE 3: Summary of MSCR test results.

a 10-cycle period for the shear stress values of 100 Pa and 3200 Pa is shown in Figure 3. Results indicate a considerable decrease in accumulated strain for the NC-modified binders. An amount of 8% of NC decreases the accumulated strain of the base binder from 1.7% to 0.4% over a 10-cycle period under a 100 Pa shear stress. For the case of 3200 Pa applied shear load, the accumulated strain decreased from 62% to 14%. This decrease indicates a higher resistance of the modified binders against applied stresses and consequently decreases of binder deformation under cyclic loading.

The values of percent recovered strain (R) for base and modified binders are also shown in Figure 3. It can be seen that, by adding NC, there is a considerable increase in recovered strain in the modified binders. Under a 100 Pa load, the recovered strain for base and modified binders with 8% of NC is 2.73 and 26.94, respectively. The recovered strain under 3200 Pa shear stress for the modified binder containing 8% of NC is 9.21, while the value for the base binder is less than zero. This negative R value is due to the fact that the binder has no recovered strain during the unloading process. Since any increase in percent recovered strain contributes to an increase in elastic response of the binder, adding NC causes an increase in recovered strain of the binder and improves the elastic response of it.

The permanent deformation characteristics of binders can be quantified by the j_{nr} parameter. A less value of j_{nr} indicates lower permanent deformation of the binder. The j_{nr} values under the 100 Pa and 3200 Pa shear loads for all base and modified binders are shown in Figure 3. It can be seen that adding NC can decrease the j_{nr} values, and as a result, the permanent deformation of the modified binders decreased. The stress sensitivity of the binder can be described by the $j_{nr-diff}$ and R_{diff} values. The less $j_{nr-diff}$ and R_{diff} values indicate less stress sensitivity, and the binder has similar behavior under the 100 Pa and 3200 Pa shear loads. The $j_{nr-diff}$ and R_{diff} values are also shown in Figure 3. The results indicate a considerable decrease in the $j_{nr-diff}$ and R_{diff} values. Therefore, adding NC can decrease the stress sensitivity of the binder.

TABLE 4: Linear amplitude sweep (LAS) test results.

Parameters	0% NC	2% NC	4% NC	6% NC	8% NC
A_{35}	33,200	49,334	57,990	134,765	146,928
B	-1.92	-2.09	-2.13	-2.35	-2.45

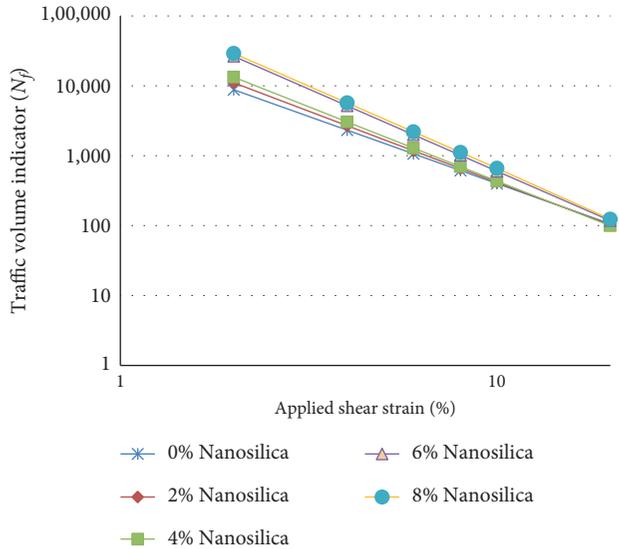


FIGURE 4: Fatigue models from the VECD analysis.

5.1.2. *LAS Test Results.* The results of linear amplitude sweep (LAS) tests are analyzed based on the theory of viscoelastic continuum damage. Utilizing this theory, A_{35} and B parameters should be calculated to assess the fatigue life of the asphalt binder. A higher value of A_{35} indicates better performance of the binder. Results showed that modified binders with NC had higher A_{35} . The A_{35} values for unmodified and modified binders are presented in Table 4.

The increase in the absolute value of B indicates that, by increasing the level of strain, the fatigue life will decrease in a higher rate. Moreover, any decrease in the absolute value of the parameter B indicates the reduction in the rate of fatigue life. The B values for unmodified and modified binders are presented in Table 4. Results show that adding NC can increase the binder sensitivity against loading level strain.

The trend of fatigue life of asphalt binders based on the VECD theory is depicted in Figure 4. Values in the Y-axis are the number of cycles to failure (N_f), which are indicative of traffic loads passing on the pavement, and the X-axis demonstrates applied shear strain. Results show that adding NC causes an increase in N_f values for lower shear strain levels. The N_f values for modified binders with 6 and 8 percent of NC are almost identical at low shear strain levels.

5.2. Mixture Performance Analysis

5.2.1. *Marshall Stability, Flow, and Quotient Tests.* Table 5 summarizes the engineering properties of the mixtures. The maximum increase of the Marshall stability, flow, and quotient is for the mixtures containing 8% of NC, which are

improved by 31%, 12%, and 35%, respectively. The more the amount of Marshall quotient, the stronger the asphalt mixture is against permanent deformation [14].

5.2.2. *Resilient Modulus Test.* Resilient modulus (M_r) is an important factor in designing the pavement. Results of M_r show that, at 25°C temperature, the M_r value is 1.37 times greater than the base binder when the NC content is 8%, and at 5°C temperature, the M_r value is 1.24 greater than the unmodified binder when the NC content is 8%. Accordingly, NC improved fatigue resistance. Other research test results on polymer-modified asphalt showed that adding NC increases fatigue resistance at intermediate temperatures conducting the same test [18].

5.2.3. *Dynamic Creep Test.* Dynamic creep test results indicate that asphalt mixtures modified with NC have higher resistance against permanent deformation in comparison to control samples. Such an increase in NC can raise the flow number, which is a parameter of resistance against rutting. Increasing the amount of NC by 8% led to an almost 71% increase in the flow number. This finding is in a good agreement with other researchers [6, 18].

5.2.4. *Four-Point Beam Fatigue Test.* In the 4-point bending beam test, it was observed that, by increasing the percentage of NC, the variation trend of N_f in all three strain levels identically increased. Adding 8% of NC to the base binder increased the fatigue life of asphalt mixtures to the amount of 52%, 92%, and 65% under 600, 800, and 1000 microstrains, respectively.

Further details and discussions about engineering performance of NC-modified mixture are presented in another article by the same authors [19].

5.3. *Regression Analysis.* A series of linear regression models between binder test results, which are considered as independent, and mixture test results, which are considered as dependent, are developed. Linear regression is a statistical method that defines the relationship between two independent variables [20]. Summary of R -squared values and equations for each correlation is presented in Table 6. Independent values did not have strong correlation with each other, so it was possible to develop a reliable regression model and avoid the multicollinearity problem. Examples of two correlations of three independent variables are shown in Figure 5. On the other hand, the independent and dependent variables showed strong correlations with R -squared values close to 1. Furthermore, for each correlation, there is a fundamental relation between independent and dependent variables, which makes them comparable. For example, N_f and A_{35} correlation is investigated because both represent the fatigue performance; one for mixture and the other for binder. So, the same characteristics are correlated to see if it is possible to estimate one using the other. Also, MSCR test results are proved to have a good correlation with the rutting depth obtained from the field

TABLE 5: Summary of mixture test outcomes.

		Control	Mixture + 2% NC	Mixture + 4% NC	Mixture + 6% NC	Mixture + 8% NC
Average Marshall stability (KN)	—	10.23	11.30	12.10	12.73	13.40
Average Marshall flow (mm)	—	4.2	4.32	4.41	4.55	4.72
MQ (KN/mm)	—	2.44	2.62	2.74	2.80	2.84
N_f (4-point bending beam)	@600 $\mu\epsilon$	106,092	120,477	141,428	151,623	158,685
	@800 $\mu\epsilon$	76,738	85,541	113,843	139,781	147,428
	@1000 $\mu\epsilon$	26,246	33,888	38,133	42,355	43,414
Resilient modulus	@25°C	3520.2	3707.5	3976.2	4420.6	4828.5
	@5°C	11,884.6	12,826.2	13,255.3	13,988.7	14,711.5

TABLE 6: Summary of correlation equations and regression coefficients.

Dependent variable (y)	Independent variable (x)	Equation	R^2
N_f @ 600	A_{35}	$y = 0.3763x + 103,883$	0.8097
N_f @ 800	A_{35}	$y = 0.5729x + 64,290$	0.9086
N_f @ 1000	A_{35}	$y = 0.1008x + 29,046$	0.8712
Dynamic creep	j_{nr100}	$y = -85,5191x + 3066.6$	0.9626
Dynamic creep	j_{nr3200}	$y = -760,429x + 3040$	0.9829
Marshall	j_{nr100}	$y = -309.13x + 2.978$	0.9915
Marshall	j_{nr3200}	$y = -272.86x + 2.9663$	0.9976
M_r at 5	R_{100}	$y = 114.28x + 11,676$	0.9816
M_r at 20	R_{3200}	$y = 55.483x + 3289.8$	0.9755

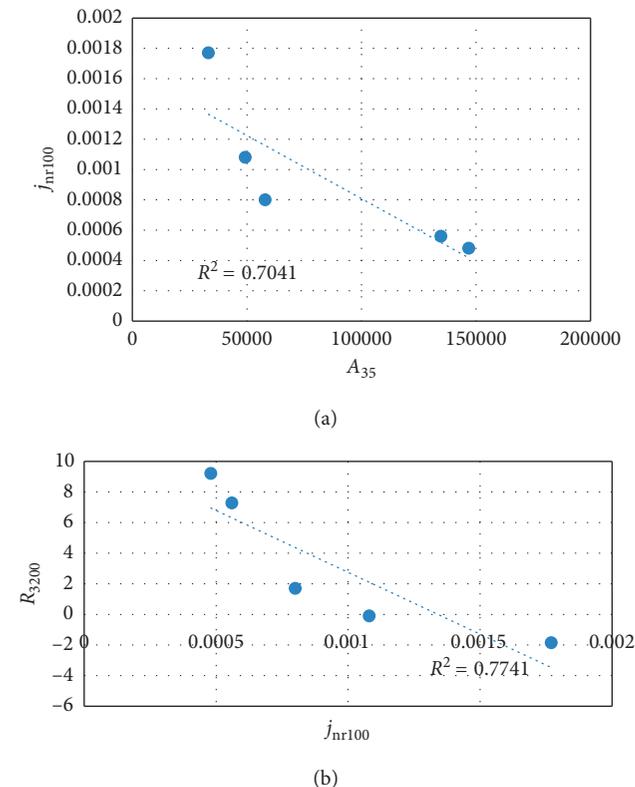


FIGURE 5: Correlation between independent variables (a) A_{35} vs. J_{nr100} and (b) J_{nr100} vs. R_{3200} .

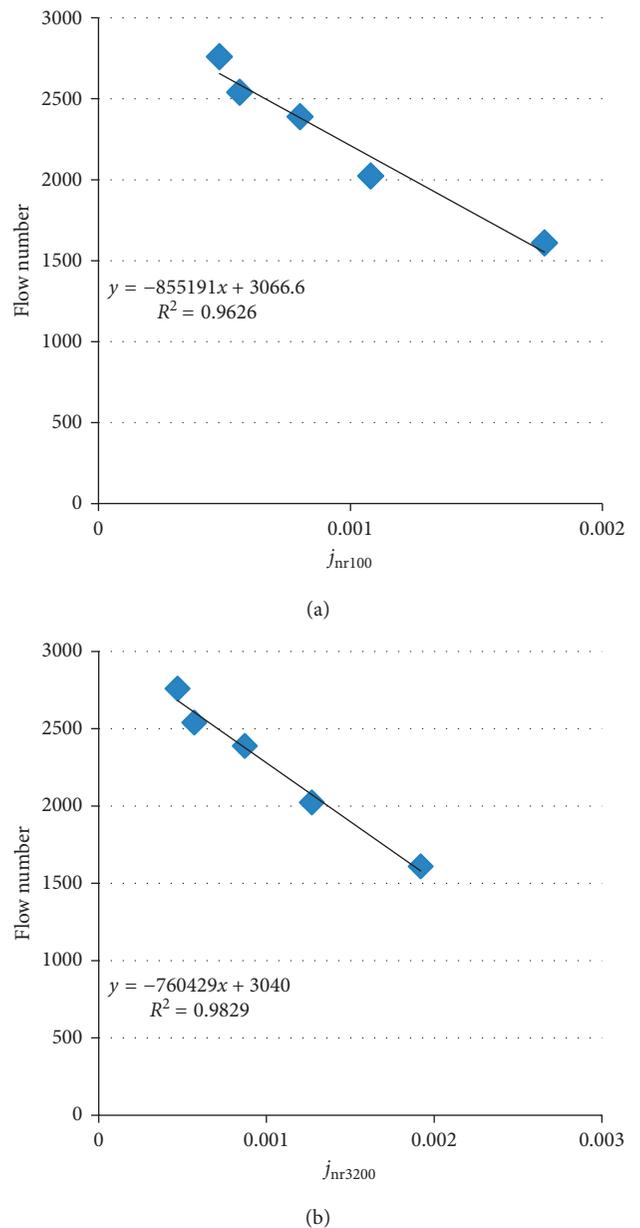
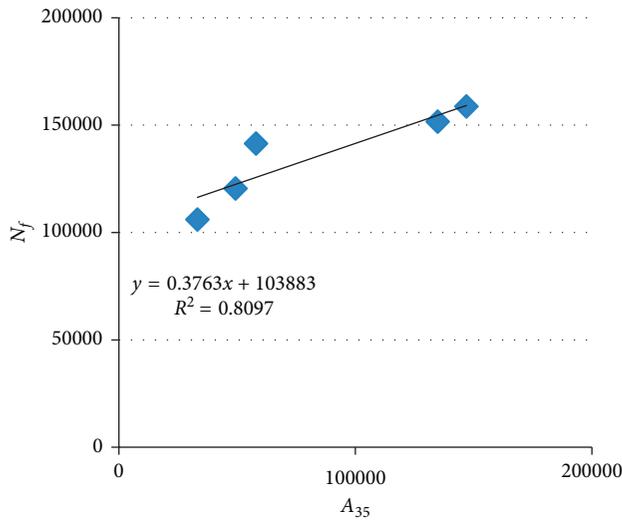
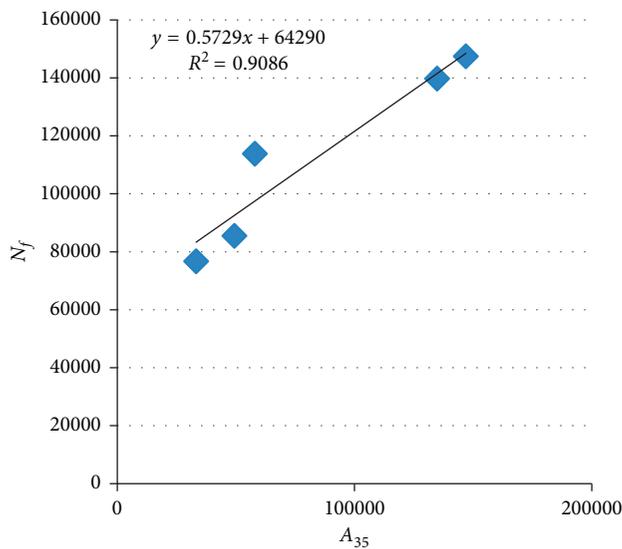


FIGURE 6: Flow number of mixture versus (a) j_{nr100} and (b) j_{nr3200} .

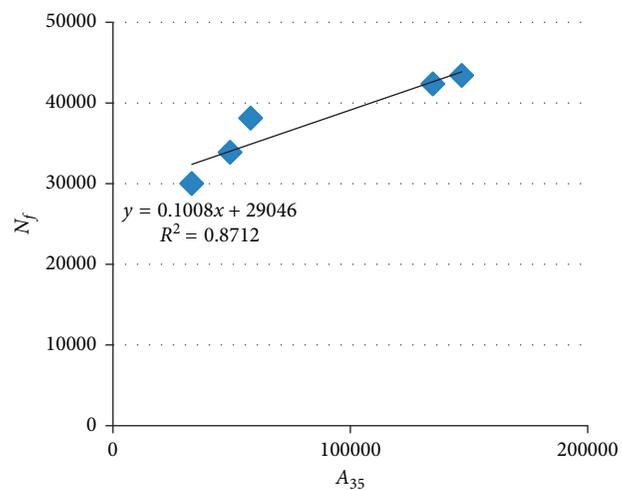
measurements [7]. Therefore, in this research, flow number, MQ, and M_r as parameters showing rutting susceptibility of mixture, are also correlated with MSCR test results.



(a)

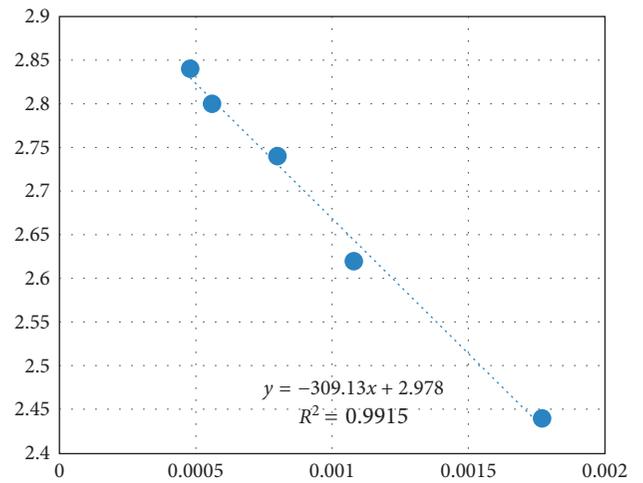


(b)

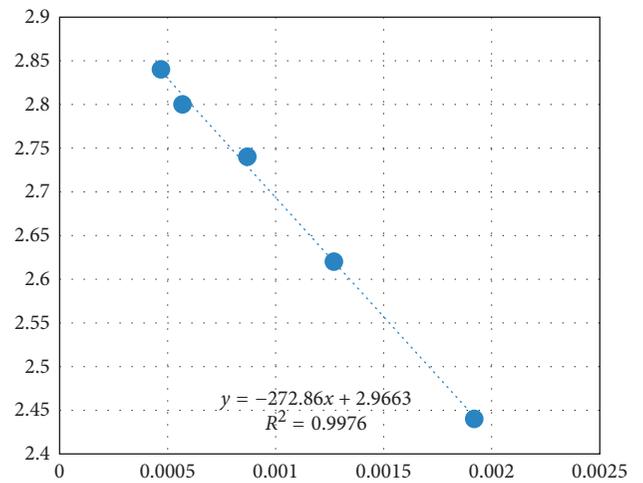


(c)

FIGURE 7: Fatigue life of mixture (N_f) at three strain levels: (a) $600 \mu\epsilon$, (b) $800 \mu\epsilon$, and (c) $1000 \mu\epsilon$ versus A_{35} .



(a)



(b)

FIGURE 8: Marshall quotient versus (a) j_{nr100} and (b) j_{nr3200} .

5.4. *Correlation between Flow Number and j_{nr} .* Results of the flow number of mixture versus nonrecoverable compliance (j_{nr}) are depicted in Figure 6. It is observed that flow number and j_{nr} are inversely correlated, showing a good correlation (R^2 more than 0.9). This indicates that the MSCR binder test result has a close relation with the mixture's response, and using its data could give us a good estimation of the mixture's performance.

5.5. *Correlation between A_{35} of LAS Test and Mixture N_f .* Fatigue properties of the mixture are strongly correlated to those of binders. Therefore, modifying the binder could considerably alter fatigue behavior of the mixture, and the binder test could give us a good estimation of fatigue characteristics of the mixture [21]. In this research, results of the 4-point bending beam and LAS tests were studied in correlation with each other. According to Figure 7, the fatigue life obtained from the 4-point bending beam test is in a fairly well correlation with N_f values for the binder test.

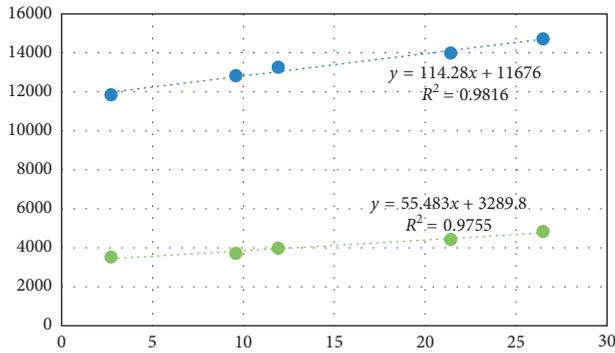


FIGURE 9: Resilient modulus at 5°C and 25°C versus R_{100} .

A_{35} parameter represents fatigue life of the asphalt binder subjected to a rate of 1% strain, which is the recommended value by the Superpave. Plotting A_{35} versus fatigue life of asphalt mixture shows a good relation between them. The best correlation was for the strain level of $800 \mu\epsilon$, and the lowest R^2 was for the strain level of $1000 \mu\epsilon$.

5.6. Correlation between Marshall Q and j_{nr} . The Marshall stiffness index (or Marshall quotient) represents the resistance of the material to shear stress and permanent deformation. Higher MQ means the mixture is stiffer and is more resistant against rutting. The permanent deformation characteristics of binders are quantified by the j_{nr} parameter. The lower value of j_{nr} indicates lower permanent deformation of the binder. Thus, the MQ correlation with unrecovered j_{nr} parameter makes sense as both are indicators of resistance against permanent deformation. As shown in Figure 8, it was observed that the MQ is inversely correlated with j_{nr} . This indicates that as the MQ increases, the unrecovered strain of the binder decreases, which means less susceptibility to rutting. R^2 is too close to 1 for five samples, which is a promising result so as to find the mixture performance using easier and faster binder tests.

5.7. Correlation between M_r and R_{100} . Resilient modulus is an indicator of a material's deflection behavior. In this research, resilient modulus at both 5°C and 25°C is correlated with R_{100} (recoverable deformation of the binder) obtained from the MSCR test. Results are shown in Figure 9. The correlation between resilient modulus at both 5°C and 25°C and R_{100} , had a regression coefficient more than 0.97.

6. Conclusion

- (1) The results of the MSCR test indicate improvement of elastic properties of modified asphalt binders at a high temperature. They also show that NC not only increases rutting resistance in modified asphalt but also reduces its stress sensitivity. Results of the MSCR test are in line with the results obtained from the dynamic creep test, presenting a strong correlation between performance of the binder and mixture. Overall, it can be deduced that the binder

modified by NC contributes to resistance of the asphalt mixture against permanent deformations.

- (2) The LAS test results indicate a considerable increase in the fatigue life of the NC-modified asphalt binder at low strain levels. On the other hand, the fatigue life of modified asphalt binders at high strain levels is a little less than that of the unmodified binders. Furthermore, the results of the four-point bending beam test demonstrate that adding NC up to 8 percent by binder content could considerably improve fatigue resistance, which confirms the results of the LAS test.
- (3) According to the correlation of the flow number, MQ, and M_r with the results of the MSCR test, it could be concluded that the MSCR test results correlate very well with mixture test results related to rutting performance.
- (4) According to a fairly well correlation between binder and mixture test outputs obtained in this research, it is promising to develop a phenomenological relation between characteristics of the binder and mixture using more extensive data and considering important parameters, in future research, so more time and materials are saved using binder tests instead of the mixture tests.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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