Journal of Engineering Science and Technology
Vol. 17, No. 2 (2022) 0820 - 0838

EFFECTIVENESS OF MICRO- AND NANO-SILICA AS MODIFIERS IN ASPHALT CONCRETE MIXTURE

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Abstract

Pavements are sometimes subjected to failure due to over-increasing traffic loading and weather conditions; numerous modified asphalt mixtures were utilized to improve their performances. This study compares the performance of asphalt concrete mixtures used in road pavements by using Nano silica (NS) and micro silica (MS) additives to find the best modifier. The characteristics of asphalt binder containing NS and MS of 0, 2, 4, 6 and 8 percent by its weight; have been investigated in terms of rheological properties through the Superpave™ tests. A further laboratory evaluation was achieved to distinguish the performance characteristics of the used bitumen mixes based on Marshall stability and flow, indirect tensile strength, wheel tracking, and moisture susceptibility tests. The results indicate that NS is considered the best modifier that has accomplished maximal stability, higher tensile strength, minimum flow, and lower rutting depth while the other modifier with accomplished lower rates of mix performance. Moreover, the NS's existence significantly impacted the increase in asphalt binder's complex viscosity (η*) and complex modulus (G*). This, in turn, results in the improvement of the rutting resistance. It has been concluded that introducing the NS to the asphalt binder may improve the rheological characteristics of the asphalt binder. The addition of the NS material has a better impact than the MS on the several asphalt cement and mix characteristics and may be used to get the durable pavements, especially at 6% NS, thereby, they decrease the lifecycle costs of pavement.

Keywords: Asphalt mixes, Bitumen, Micro silica, Modified asphalt, Nano silica, Rheological properties.

1. Introduction

Asphalt pavements suffered from different distresses due to deficiencies in their elastic characteristics, heavy traffic loading, and the weather's impact on the properties of the asphalt binder [1]. There is a growing necessity for the improvement of the performance characteristics of the asphalt binders to minimize the rate of the mechanisms of failure [2]. The most failure types in the asphalt pavements include cracks, fatigue and rutting which resulted from the shortage in the mix characteristics and/or the excessive traffic loads [3]. To improve the performance of the mixture; binder modification plays a key role [4]. Recently, nanotechnology has regularly been merged into modified bitumen [5]; it is considered the likely alternative to significantly improve the effectiveness and durability of construction materials. Nano-technology is making new devices, materials, and systems at molecular level which is related to molecular and atomic interactions mainly affecting macroscopic material characteristics [6]. Nanomaterials are determined as materials which have no less than one dimension with the length scale of 1nm - 100nm. These materials frequently show characteristics that entirely differ from those shown by the same products with larger dimension values [7]. The nanoparticles may be very encouraging to enhance the characteristics of the asphalt binders [8, 9]. The nanoparticles have the physicochemical interactions and reactivity which are mainly higher when comparing with other particles of the same material at macro-scale [10].

The most nanomaterials used as asphalt binder modification are titanium dioxide, carbon nanotubes, nano-clay (i.e., organic montmorillonite), nano silicon oxide, nano calcium trioxocarbonate, and nano zinc oxide [5]. Amid the nanomaterials, the Nano silica NS is a promising material for designing and preparing new functional materials due to its high stability and surface area [11-13]. As a result of these supportive properties, the NS can be utilized as an asphalt modifier for improving asphalt performance [14, 15]. Further than the form and dimension of silica particles are appropriate for applications in bitumen binder mostly as the surface area of interaction is greater than traditional fillers. Consequently, the present analysis could help in knowing the performance characteristics of composite modified bitumen mixes for enhancing the design of bitumen mixes with relevant performance. Similar to nanomaterials, micro-materials also have the potential to modify bitumen materials. Micro materials have dimensions in the scale of micrometres (10^{-6} m) [16]. In this research, the impact of NS on the characteristics of the bitumen binder and the mix is compared with the effect of MS on the same properties.

Nanoparticles employing is very new-fangled although it has already raised some problems regarding its potential toxicity. Some studies have shown that nanoparticles can cause symptoms such as those caused by asbestos fibers [17]. Therefore it is recommended that nanoparticles be used with the same care used in university for materials of unknown toxicity, i.e., the use of air aspirations to prevent inhalation and gloves from preventing skin contact.

2. Background on the Modifications of Asphalt

The continuous increase in the traffic loads and volume in conjunction with rising costs of asphalt as a by-product of crude oil makes the need for improving asphalt pavements. This can be utilizing asphalt modification [18, 19]. Several trials and efforts have been initiated in the scope of preparing modified asphalt for realizing the technique of modification and the subsequent enhancement in performance [4].

Generally, it was found that the application of nanotechnology to bituminous has extremely beneficial [8] as a key to a completely new world in the field of building and construction materials [20], although more research is also needed in the field of nanotoxicity as when using nanoparticles; extreme caution must be used as much as possible. Recently, the NS has significantly considered highway studies for using bitumen materials with required properties due to their desirable stability, low cost, large surface area, chemical purity, high adsorption, and relevant dispersing capability [12, 13]. The benefit of this material is a cheap manufacturing and the performance benefits [15]. The silica material has extremely reactant with bitumen compared to the traditional fillers, and dispersal capability of the NS particles into bitumen binder [14]. In fact, NS material is widely utilized as an inorganic additive for the enhancement of the characteristics of asphalt binders; Table 1 presents previous studies on improvement of the asphalt binder.

Table 1. Previous studies on nano silica additive.

Reference	Modification Type	Tests	Improvement
Yao et al. [14]	4%and 6% NS by wt. of binder	Rutting, aging, and fatigue cracking dynamic modulus, and flow number	A substantial enhancement has been found
You et al. [6]	2%and 4% Montmorillonite Nanoclay by wt. of binder	Rotational viscosity, dynamic shear complex modulus was	Increased
	4%Nano clay	complex modulus	Increased by 196%
	2%Nano clay	complex modulus	Increased by 184%
Yang and Tighe [8]	NS	Antiaging resistance, rutting fatigue cracking	Enhanced
Abutalib et al. [21]	2, 4 and 8% micro silica MS by wt. of binder	Asphalt aging index The aging index	Decreased up to 4% Negatively affects after 8% MS
Mostafa [5]	7% Optimum NS	Penetration Viscosity	Decrease by 7.13% Increased by 8.33%
Al-Taher et al. [22]	7.0% NS	Penetration viscosity Marshall Stability flow Stiffness and ITS rutting resistance	Decreased Increased 25% increase Decreased Increased Increased by 40%
Al-Taher et al. [23]	MS or SF	penetration viscosity Marshall Stability Marshall Stiffness	Decreased Increased Increased Decreased

3. Problem Statement and Objectives

The continuous increasing traffic loads and volume in conjunction with the rising cost of asphalt as a by-product of crude oil, makes the need to improve durability, safety and efficiency of asphalt pavements imperative. In Iraq pavements are subjected to different distresses due to heavy traffic and climatic changes. The improvement of the performances of bituminous pavements is necessary for reducing these distresses. Normally, some common materials are selected to improve the performance of asphalt mixtures. However, the overall performance of asphalt mixtures has not been significantly increased compared to the control asphalt. Therefore, it is permissible to use the other materials/nanomaterials to modify the asphalt to perform the asphalt mixtures better.

In recent years, the use of nanomaterial to improve bitumen binder and mix performance has become popular. It deals with dimensions in the scale of nanometres (10^{-9} m). Nanomaterials provide exceptional properties to materials such as high strength, better conductivity, etc. Hence, nanomaterials such as nano carbon tubes, nanoclay, nanosilica, nanopolymers, etc. are used in bitumen applications to improve the mechanical performance and heat resistance. Similar to nanomaterials, micromaterials also have the potential to modify bitumen materials. Micro materials have dimensions in the scale of micrometres (10^{-6} m). Likewise, the hypothesis is to use these materials for asphalt modification. Based on the literature review, these micro- and nanomaterials were barely used in the modification of asphalt mixtures. Silicon dioxide (SiO_2), also known as silica is an inorganic material, most commonly found in nature as quartz and in various living organisms, it is the main constituent of sand in many areas all over the earth. Silica nanoparticles have been utilized in industrial applications for reinforcing the elastomers as a rheological solute. The main benefits of Nanosilica are the low production cost and high-performance features. Therefore, in this research, the microsilica (MS) and nanosilica (NS) were selected and added to the control asphalt. The modified bitumen binders and aggregates have been blended and compacted in accordance with to the superpave specifications.

The objectives of this research are investigating MS and NS materials as modifiers to asphalt mixture and for conducting a comparative study to inspect the rheological properties of asphalt binder and mechanical performance of modified bitumen mixes. Different samples with (2, 4, 6, and 8)% MS and NS materials by wt% of control binder were prepared to enhance the whole efficiency of the improved bitumen binders and mixes. The asphalt mixture performance tests indirect tensile strength (ITS) and wheel tracking test (WTT) rutting tests have been employed to assess the effectiveness of asphalt mixtures and to find the optimal modifier.

4. Materials and Experimental Tests

4.1. Materials

The materials utilized in the present study are locally available including bitumen, aggregates, filler and additives. Bitumen binder of 60/70 grade of penetration, which has been obtained from Al-Duarah refinery southwest of Baghdad, was used to prepare the modified binder blends. The physical properties of the supplied base binder are shown in Table 2. The coarse and fine aggregates have been utilized to prepare bitumen mix samples; crushed coarse aggregate having physical properties given in Table 3 is highly used in pavement construction whereas limestone is used as filler material.

Table 2. Asphalt binder physical properties of 60/70 grade of penetration.

Property	Test approach [ASTM]	Results	ASTM Specification
Penetration @25˚C 100g, 5s, 0.1 mm	D5	66	60 - 70
Softening Point, ˚C	D36	47	46 - 54
Kinematic viscosity @135 ˚C, C.st.	D2170	358	≥ 320
Absolute viscosity @ 60 ˚C, Pa.s	D2171	210	-
Retained penetration (%)	D5	50	-
Mass loss (%)	D1754	0.12	0.5
Ductility @ 25ºC, 5cm/min, cm	D 113	+100	+100
Flash point, ˚C (Cleveland Open Cup)	D92	255	230 min.
Specific gravity	D 70	1.017	-
Solubility	D2042	% 99.9	99 min.

Table 3. Coarse and fine aggregate physical properties.

Property		Test methods Coarse Aggregates > 4.75		Results	Specification
Sieve Size		4.75 mm	9.5 mm	12.5 mm	ASTM C127
Specific gravity	Bulk	2.651	2.585	2.570	
	Apparent	2.674	2.591	2.582	
Absorption%		0.32	0.09	0.18	
Wear% (Los Angeles abrasion)		ASTM C131,		21.3%	30 max.
Soundness (Loss byNa_2SO_4),%		ASTM C88,		3.2%	10-20 max.
Angularity,%		ASTM D5821		97%	95 min.
Flat and elongated particles, %		ASTM D4791		0.90%, 2.50%	10 max.
Fine Aggregates properties (Crushed Sand < 4.75)					
Bulk Specific Gravity		C 128-04		2.643	-
Apparent Spec. Gravity		C 128-04		2.616	-
Water Absorption %		C 128-04		0.19	-
Equivalent Sand (Clay Content %)		ASTM D2419,		89.6%	45 min
Deleterious Material,%		ASTM C142		1.12	3 max.

Nano silica NS and micro-silica MS were used as additives to improve the bitumen for preparing the nano-composite modified binders. NS material in a white coloured powder is nano-structured silicon dioxide SiO_2 polymorphs and was utilized to improve the bitumen binder. It has a large surface area, better adsorption and better stability. It frequently found in nature as quartz and in different living organisms. In many areas all over the earth, silica is the main constituent of sand, while primarily; it is produced from silica precursors. Silica has been defined as a very complicated and one of the most abundant materials, which exist as a complex of many minerals and synthetic products. Silica nanoparticles have been utilized in industrial applications for reinforcing the elastomers as rheological solute. The main advantage of this nanomaterial is the low production cost compared to its high-performance prospect. The properties of NS particles utilized here are indicated in Table 4. The chemical composition of NS is listed in Table 5.

Table 4. NS particles properties.

Physical Property	Value	Physical Properties	Value
Appearance	White powder	Purity	> 99.9%
Density	2 g/cm^3	Loss of ignition	≤ 6%
Average size of the particles	15nm	PH value	6.50-7.50
Specific gravity	2.2 – 2.4	Specific surface area	100 ± 25 m^2/g

Table 5. Chemical composition of NS.

R_wp	6.64	Anhydrite	0.24	Hematite	0
Alite_Sum	0.0	Calcite	0.28	Thenardite	0
Lime	0.06	Mulite	0.0	Rutile	0
Periclase	0.09	Magnetite	0.0	Si_amorph	99.15
Quartz	0.19	-	-	-	-

The second additive micro-silica MS or silica fume is an amorphous polymorph of SiO_2. It is obtained as a by-product of silicon or ferrosilicon alloy production. The bulk density of silica fume ranges from 130 – 600 kg/m^3. The average particle size is 440 nm. Its specific gravity is 2.2. It has a greyish powder appearance. The Chemical composition of the used SF minerals is indicated in Table 6. For obtaining a suitable interlocking of the aggregates, a dense gradation plot for the surface layer with a maximal nominal size of 19 mm has been used for the asphalt mix preparation. Gradation of aggregates was utilized in the current study consistent with the Superpave specification and the limits of the Iraqi specification SCRB/R9 [24] as illustrated in (Fig. 1). Before blending with the bitumen, the aggregates have been dried for as a minimum 120 minutes at a 165˚C temperature. Depending on the viscosities of micro- and Nano-modified bitumen, the blending and compaction temperatures have been about 165˚C and 155˚C, respectively, a total of approximately 66 samples have been utilized and compacted by Superpave Gyratory Compactor (SGC). The binder content of 4.5% was then utilized in all the mixes. Four sets of modified test specimens have been prepared by using the modified asphalt with the determined (OAC) % and to add different percentages of micro-silica MS or silica fume (2, 4, 6, and 8% by weight of asphalt). Three specimens are prepared for each set. Each of the prepared specimens is tested to find or measure the mixture properties.

Table 6. Chemical Properties of SF.

Parameters	Test Value
Silica (SiO_2, % by mass)	89.9
Lime (CaO, % by mass)	7.85
Total Sulphur Content (SO3, % by mass)	0.58
Alumina (AL_2O_3, % by mass)	Nil
Magnesia (MgO, % by mass)	4.03
Iron Oxide (Fe_2O_3, % by mass)	Nil

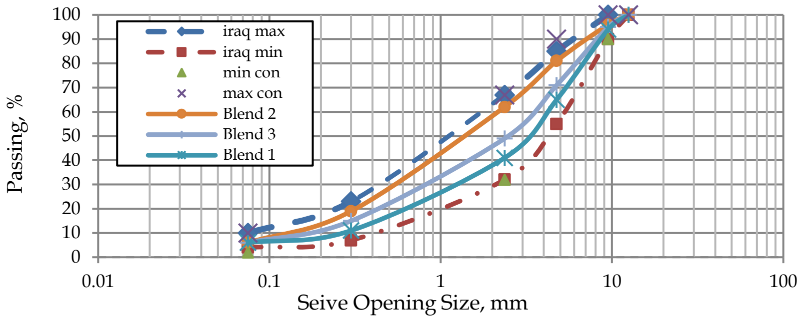

Fig. 1. Trail selected aggregate gradation blends.

4.2. Binder modification

The first step in the experimental program was preparation of modified bitumen. In this process, to get the modified asphalt a 500g of base asphalt AC 60/70 Pen. The grade was heated to 160˚C, and a specified number of NS and MS by binder weight has been separately mixed with the bitumen. The NS material has been mixed with base binder at various concentration values (2%, 4%, 6% and 8%) by wt., with the use of a shear mixer at a 2000rpm rate for one hr. then modified asphalt (NSMA) samples were produced [25]. A like mixing pattern, temperature, rotation speed and mixing time have been done by other researchers [26, 27]. For easiness in referring to every one of the samples, they were termed utilizing the following abbreviation: NEAT, NSMA2%, NSMA4%, NSMA6% and NSMA8%.

It has been identified that in the case where the NS disperses and melts in the control bituminous binder, the surface of the binder had a small number of the floating bubbles. Whereas, mixed, the binder samples have been saved at a temperature of 163˚C and combined with the use of a shear rate of 2000 rpm for 60 min duration.

To prepare the modified binder with silica fume or MS, asphalt cement has been heated in oven at a 150˚C temperature. After that, the suitable amount of asphalt is heated into a steel beaker. Then, the required quantity of SF (2 to 8% by weight of asphalt) is added to bitumen. The beaker is positioned on a hot plate to keep the mixing temperature at least 150 ˚C (according to its viscosity), and the bitumen and SF are mixed carefully until the binder become homogenous. The MS material has been added to control binder at the concentration levels of MSMA2%, MSMA 4%, MSMA6% and MSMA8% by weight of the binder. Different requirement test methods were implemented on produced samples to investigate the influence of these additives on characteristics of bitumen binders. It is noteworthy that base binder is blended without additive for the same blending conditions to avoid any varied aging degree between unaged prepared binders by blending process.

The temperatures for blending and compaction that suggested by the binder supplier were 164˚C and 154˚C, respectively, which have been utilized for mixes production. Since the silica effect resulted in higher viscosity values, the blending and compaction temperatures may be greater. Though, to better recognize the influence of modification alone and permit a better comparison amongst implemented mixes, the same blending and compaction temperatures have been utilized for all mixes.

4.3. Testing program

A group of tests was implemented on asphalt samples with MS and NS with 4 various ratios to investigate the performance characteristics of HMA produced with both kinds of stated additives. Asphalt tests are softening point, penetration and viscosity test and the Superpave binder tests are PAV, RTFOT, BBR and DSR tests. HMA tests have been (indirect tensile strength test ITS, Marshall Test, and Wheel Tracking Test WTT).

The Marshall Test method as per ASTM D 6927-06 is used in this research to determine asphalt mix characteristics. The aggregates (coarse, fine and mineral filler) are prepared, and the required asphalt cement (60/70) is estimated. Optimum Asphalt Content (OAC)% is calculated by preparing three HMA sample sets, which have been produced at 3.50%, 4.0%, 4.50%, 5.0% and 5.50% asphalt content and compacting samples with 75 blows with a standard hammer. Similar sets of modified test specimens are prepared using the modified asphalt with the determined (OAC) % and adding different percentages of MS and NS. The ITS test has been utilized to define the structural characteristics of a bituminous mix and assess the moisture susceptibility of asphaltic mixes as per AASHTO (T322-03). This test includes loading a cylindrical sample with the compressive load acting along and parallel to the vertical diametrical plane. Such configuration of the loading is developing rather uniform tensile stress perpendicular to the applied load direction. Such tensile strength results in causing the failure of the specimen by the rupturing and splitting along vertical diameter. The values of the ITS (St) are specified through the use of this Eq. (1):

$$St = 2P/(\pi HD), \text{ (kg/cm}^2\text{)} \quad (1)$$

where: P represents applied load in kg, H represents the height of the specimen in cm, and D represents the diameter of the specimen in cm, and this may be specified with decreasing the indirect tensile strength loss after conditioning samples in the water at a temperature of 60˚C for 24 hours. In the present research, Wheel Tracking test WTT test has been utilized to quantify the rutting resistance of micro- and nanomodified asphalt mixes according to AASHTO T324-04. The test is conducted with a temperature of testing 60˚C with 10000 test cycles. The sample air voids set to 4±0.5% and the dimensions was 40 mm length by 30 mm in width and the load of wheels on rubber hoses was 700±5 kPa. The test samples' performance is associated with the field pavement performance. A lab compactor was constructed to get slab specimens. This test measures the rutting depth RD and number of passes or time to the final RD. The test is conducted on both of control and modified specimens.

5. Results and Discussion

5.1. Consistency tests of modified asphalt

Consistency tests are carried out on both control and the modified binder to measure penetration value, softening point, ductility and absolute viscosity values. Figure 2 shows the result of testing on modified bitumen. Results of penetration test indicate that the penetration value decreased for all modified samples. Nanosilica record the lowest penetration values, the penetration is decreased by (4.5, 10.6, 15.1 and 30.3%) for (2, 4, 6, and 8%) respectively. In the case of microsilica modification, it was observed that penetration decreases steadily for microsilica-modified bitumen, it was decreasing consistently by (3%, 7.5%, 12% and 16.6%) for (2, 4, 6, and 8%) respectively. However, at 8% the bitumen is becoming stiffer due to increase in concentration of nanosilica. However, the softening point was increasing up to 8%. Viscosity test results indicate that the viscosity increases for all modified specimens using the different additives. Adding nanosilica achieves the highest viscosity values; the viscosity increases by (52, 171, 233% and 395%) for (2, 4, 6, and 8%) respectively. Microsilica increases the viscosity value by (28.5%, 114%, 166 and 210%) for (2, 4, 6, and 8%) respectively. As shown in Fig. 2, bitumen complex viscosity rises with the increases in the nanosilica concentration. A nanosilica modified asphalt binder with high viscosity can develop a thicker film that surrounds the aggregates that increase cohesive strength. This, in turn can promote pavement durability.

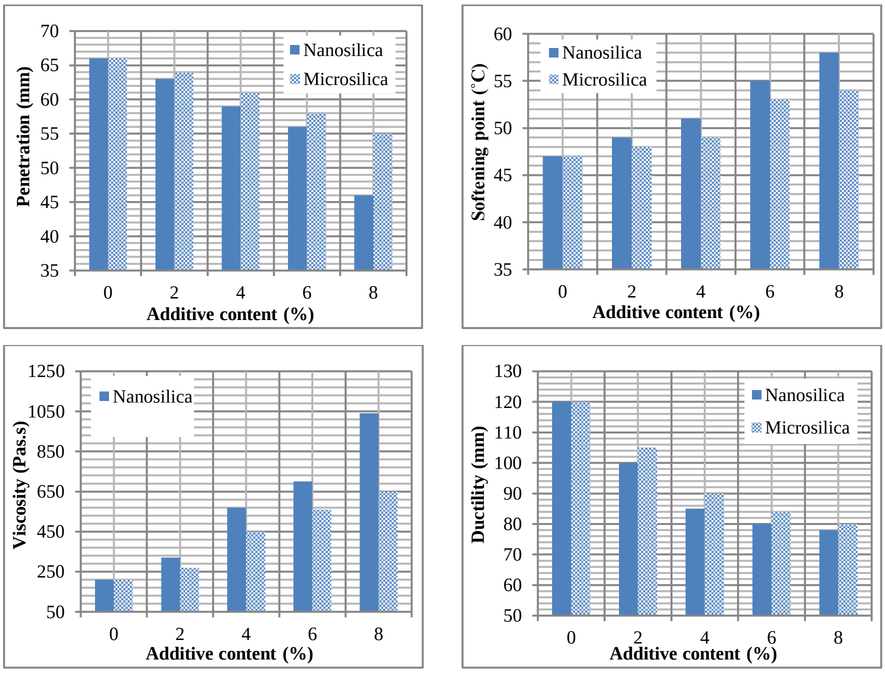

Fig. 2. Physical laboratory tests of modified asphalt.

5.2. Superpave bitumen binder tests

The rheological properties of bitumen binders have been determined utilizing the Superpave grading system to investigate the variations in the effectiveness grade of base bitumen binder [28]. The performance-graded binder specification (PG) for Superpave control the quality of binder for the pavement failures as permanent deformation, low-temperature cracking, and fatigue cracking. Those failures have been governed using a variety of apparatus pieces to the governor utmost of measured physical characteristics. These apparatuses are bending beam rheometer (BBR), dynamic shear rheometer (DSR), rotational viscometer (RV), rolling thin film oven (RTFO), and pressure aging vessel (PAV). Validation of the PG has been achieved for all the modifier asphalts and the result of these findings are presented in Figs. 3 to 9.

The Rotational Viscometer RV measurement is mainly conducted on non-aged bitumen and value within 3Pa.s at 135˚C as per pumping purposes' specifications. Viscosity is measured using the Brookfield viscometer as per ASTM D 4402. Fig. 3 indicates that the viscosity increases mainly for both MS and NS comparing with the control asphalt, it is increased by (35, 62.5, 85, and 117.5%) with adding (2, 4, 6 and 8%)MS, respectively. For NS additive gives higher viscosity values than MS.

Dynamic Shear Rheometer (DSR) tests were implemented on the original and modified binders per ASTM D 7175. The stiffness parameter ($G^*/\sin\delta$) has been utilized to assign binder stiffness in the Superpave. The measurement of asphalt characteristics is performed in three conditions (the non-aged asphalt, asphalt aged with RTFO, and asphalt aged with the RTFO-PAV) at the specific temperature values. The ($G^*/\sin\delta$) value for neat bitumen at a specified temperature (70˚C) has been specified with the use of the DSR; it has been discovered that neat asphalt implemented an average DSR value of more than 1kPa at this temperature. Therefore, the conversion to a thermal degree has been followed (76 °C as Superpave degree at each 6 °C degrees). The outcomes indicated that the local neat bitumen's ($G^*/\sin\delta$) value was 0.821, which is lower than the permitted specification (1kPa). This indicates that it has failed at that degree of the temperature. The neat binder has been then tested by the DSR preceded by the RTFO at 70°C where the ($G^*/\sin\delta$) value has been noticed to be 2.53 which is more than the minimal permitted bound for avoiding rutting (2.2kPa). Nevertheless, at 76° C, the ($G^*/\sin\delta$) value has been 1.32, meaning that it has failed at this degree of the temperature, too.

For investigating characteristics of asphalt with different ratios of MS and NS under two degree of temperature, Figs. 4 and 5 indicate that modified bitumen binders commonly revealed higher values of ($G^*/\sin\delta$) compared with base asphalt, demonstrating better resistance to the rutting of the pavement, the ($G^*/\sin\delta$) value increased by 7.7% and 13% for 4%MS at 70 and 76 °C. In contrast, it increased by 12% and 17.7% for 4%NS at 70 and 76 °C, respectively. On the other hand, similar behavior is noticed for the neat asphalt, in the case of RTFO test for binder; among different ratios; asphalt with NS has ($G^*/\sin\delta$) value higher than MS as indicated in Figs. 6 and 7. Notably, as the ratio of NS increases, the value of ($G^*/\sin\delta$) increases for both degrees of temperature. However, the Variation of rut factor ($G^*/\sin\delta$) for NS and MS modified samples has increased at 70 °C test temperatures; the Nanosilica has improved the stiffness and elasticity of base binder at the high temperature. Thus; superior performance of the Nano-silica modified samples has been noticed.

For the purpose of resisting fatigue cracking, the asphalt binder has to be elastic however, not very stiff. Which is why the complex viscous portion G*×sin δ, has to be a minimal (≤5,000 kPa). In Figs. 8 and 9, it is clear that an increase in the value of G*×sin δ for NS and MS modifiers which demonstrates the enhancement from the resistance to the fatigue cracking (as it has been stated by Superpave bitumen specifications), the effects of MS and NS ratios on the value of G*×sin δ which have minor influence for MS and significant once for NS.

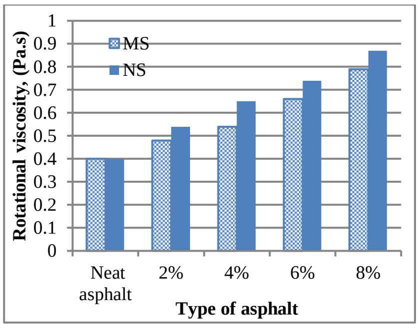

Fig. 3. Impact of type of asphalt on rotational viscosity at 135°C.

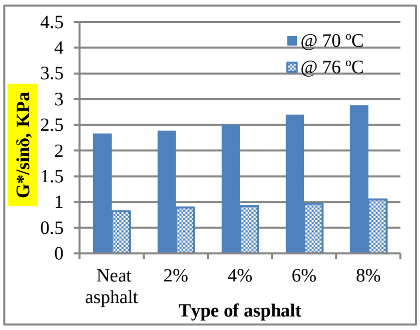

Fig. 4. Effects of MS ratios on DSR values for original binder.

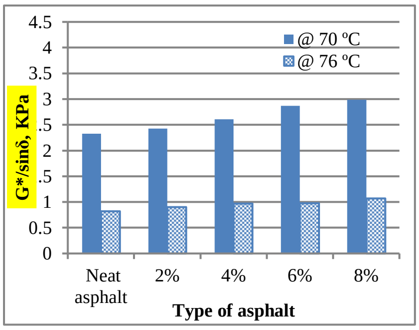

Fig. 5. Effects of NS ratios on DSR values for original binder.

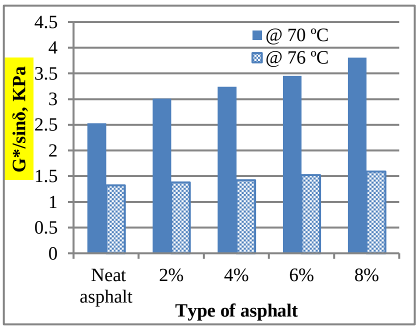

Fig. 6. Effects of MS ratios on DSR values after RTFO test.

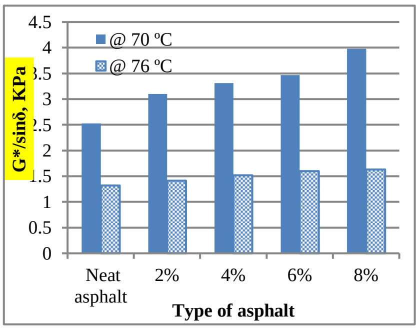

Fig. 7. Effects of NS ratios on DSR values after RTFO test.

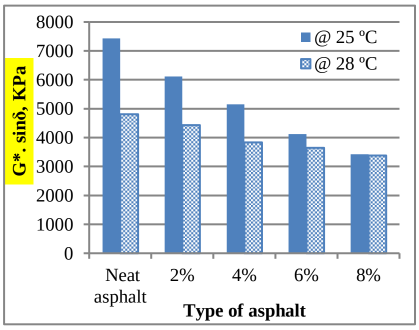

Fig. 8. Effects of MS ratios on DSR values after PAV test.

Figure 10 shows the effects of MS and NS ratios on mass loosing test, it is observed that mass loss decreased with increasing of microsilica and nanosilica additives. The PAV aged binders have been used for the Bending Beam Rheometer (BBR) .test. The results indicated that low local bitumen temperature has been -16, demonstrating that bitumen stiffness (S) has been successful (in other words, S < 300 MPa). The testing has been achieved on (-6) and (m-value) and percentage of variation in the stiffness to the time (in other words, the curve slope) has been <0.3. This confirms the stress release of the shrinkage and expansion that happens in bitumen concrete pavement because of various temperature values between winter and summer, day and night. Figures 11 and 12 illustrate how the influence of asphalt type on BBR values for different types of binders under various temperatures. Similarly, the effect of MS and NS ratios has been implemented; it is observed that m-value and creep stiffness increased for all modified asphalt binders and at 6% NS addition all of those values offer maximum values as indicated in Figs. 13 and 14. An enhancement in the m-value and stiffness exists in nanosilica modified specimen is higher compared to those in microsilica modified specimens, therefore it may be concluded that the low temperature cracking as well could be regulated.

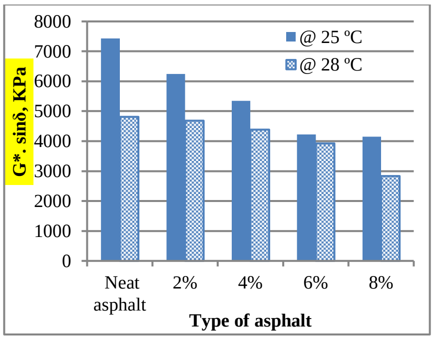

Fig. 9. Effects of NS ratios on DSR values after PAV test.

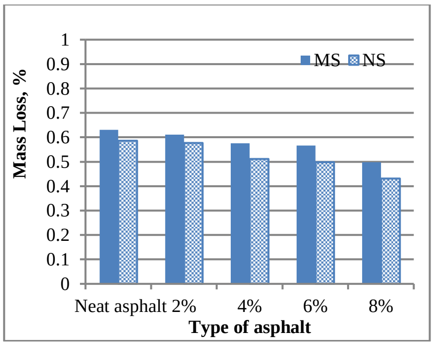

Fig. 10. Effects of MS and NS ratios on mass loosing.

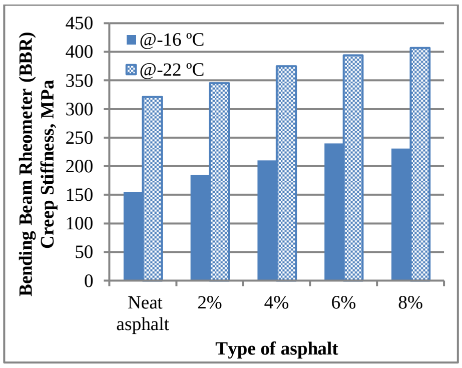

Fig. 11. Effects of MS ratios on BBR values for PAV binder.

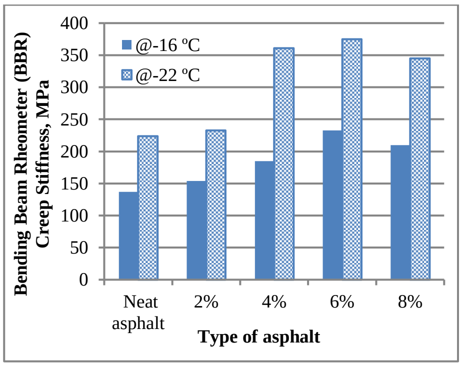

Fig. 12. Effects of NS ratios on BBR values for PAV binder.

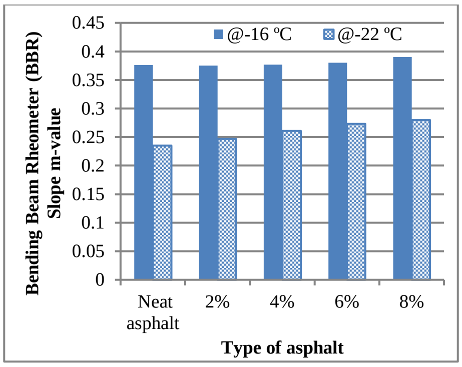

Fig. 13. Effects of MS ratios on BBR values for PAV binder.

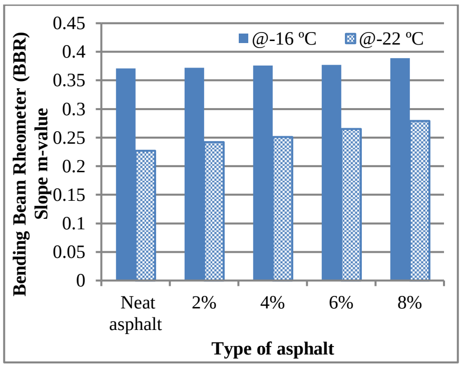

Fig. 14. Effects of NS ratios on BBR values for PAV binder.

5.3. Marshall stability and flow properties evaluation

From the results it was found out that the optimum bitumen content for control mix is 5%. For nanosilica modified mix it was 4.9% and for microsilica modification it was 4.8%. Figure 15 shows the relationship between the obtained stability and the additive contents at the OAC. The Marshall Stability increases as the percentages of MS increase up to 6% and then it decreases. Results indicate that the maximum stability of modified mixture using NS and SF improved by 21% and 19% respectively. Accordingly, NS is the best additive in improving the stability of asphalt mixtures.

Figure 16 displays the relationships between the flow and MS contents and also with NS content of asphalt mixtures compacted at the optimum asphalt content (OAC). Results indicate that the mix flow is firstly decreases with an increase in MS contents up to 2% and then it increases with the increase in MS accordingly. The maximum flow value is obtained at 8% MS. The high flow values mean high flexibility that increases the ability of HMA pavement for the deformation without the cracking. Figure 16 shows that a considerable reduction in the Marshall flow with the use of the NS at the OAC, it has been found that the decrease of the flow with the increase in the NS content up to 3.0% by wt. of the content of the asphalt.

Then it increased with increasing the content of NS. All modified mixture values have lower values as compared to the control mixture value. The first decrease results from gaining densification that resulted in increasing stability and decreasing the flow accordingly. After the mixture reaches the maximum densification and stability, any addition of NS resulted in making mixture has the tendency of having higher flexibility and increasing its tendency of being deformed, which results in making modified mixtures more resistance to deformation under traffic loads.

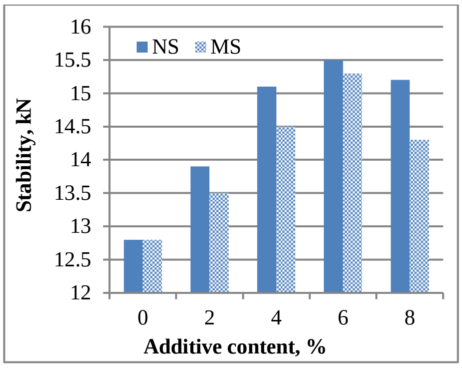

Fig. 15. Impact of the addition of the MS and NS on Marshall stability at OAC.

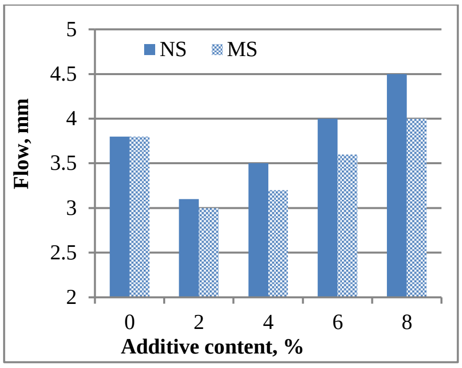

Fig. 16. Impact of the addition of the MS and NS on Marshall flow at OAC.

5.4. The indirect tensile strength (ITS) test

The ITS is a very good approach in measuring bitumen mixes strength once it exposed to the tension. Figure 17 demonstrates ITS results for the unconditioned as well as the conditioned modified mixtures. The conditioned samples have been noticed to have somewhat lower values of the ITS compared with the unconditioned samples. A decrease in the conditioned samples' ITS may result from the loss of the adhesion in the mixture or the cohesion loss in modified binder, resulting from longer sample exposure to the moisture. The results indicate that the ITS value increases for 2% microsilica modified mix from 700 to 753 kPa, representing an improvement by about 7.5%. The presence of MS in the modified mix makes it to be denser and resists load. The cementation effect of the modified specimens increases the ITS values. It can also be noticed that the mixture having 6% microsilica demonstrate the maximal ITS increase that has been approximately 83% higher compared to control mixes. Conversely, it can be observed that there is a drop in the ITS values in the mixes with 8% microsilica. The decrease in value of the ITS might be associated with reducing the stiffness of the mixtures because of high microsilica concentration.

Regarding the sensitivity to water, it has been observed that all mixes that have been prepared with NS modified binders represented better TSR values. In addition to that, nano-modifications had a considerable impact on ITS dry and wet. As observed, the composite mixtures of the nanosilica have higher values of the ITS than the micro-silica mixes, which presented that the nano-silica enhances the composite mixes' tensile resistance. It is found that the addition of 8% NS to asphalt mixture indicates the maximum increase in ITS enable the mixture to resist the tensile stresses.

The results of TSR test are illustrated in Fig. 18. It is observed from outcomes that there has been an agreement over TSR increase after modifying the mixes that have been prepared with both modifications. Tensile strength ratios (TSR) for mixes produced with microsilica with the addition of (2%, 4%, 6%, and 8%) are (86%, 87.9%, 96%, and 87.5%) respectively. The TSR for the mixtures that have been modified with the Nanosilica are (89%, 87.1%, 94.3%, and 97.2%) respectively.

The minor change which has been noticed between the conditioned samples and the unconditioned ones demonstrates that the addition of the nano-silica to asphalt mixes, will not simply allow displacing the asphalt binder which is coated on the surface of the aggregate because of the improvement of the cohesion and adhesion bonds in between asphalt and aggregates. Such improvement will offer better resistance from the damages that are induced by the temperature like the fatigue cracking. It was performed that there is 6% tensile strength increase for microsilica modified mix and 8% increase for nanosilica modification. Hence the modified mix has higher resistance to moisture damage and creep.

5.5. WTT results

The WTT has been carried out on the samples of the asphalt mix that have been prepared at optimal asphalt content, obtained from the Marshall tests. The test has been utilized to find the premature failure susceptibility of HMA caused by weakness in the structure of the aggregate and insufficient stiffness of the binder; it measures the rutting depth and the number of the passes or time to the final rutting depth (at 10000cycle) [29].

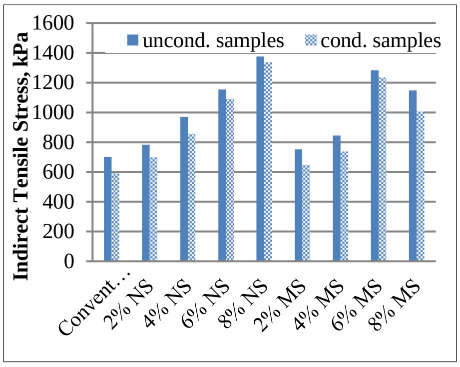

Fig. 17. ITS values for unconditioned and conditioned for each mixture.

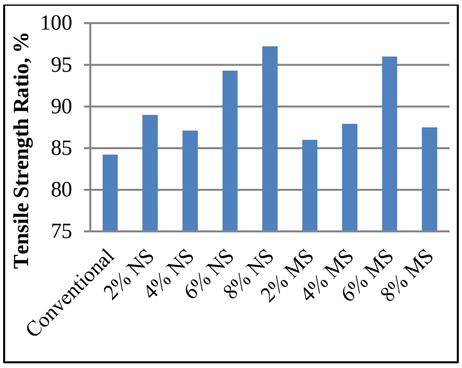

Fig. 18. TSR values for each mixture.

Figure 19 shows the WTT test results for the control as well as the MS modified specimens at a temperature of 50˚C. The observed results have indicated that the rutting depth of modified specimen reduces by about 58.33% at 6% addition of MS. In general, the rutting depth is increased with increasing with a number of cycles for both control and modified mixtures. This indicates that adding 6% MS to the asphalt mix improves the rutting resistance and reduces rutting depth by 58.33%.

Figure 20 shows the results of WTT testing for the control as well as the NS modified mixes. The results indicated that the depth of the rutting for modified mixtures is, in general, lower compared to the ones of the control mix. However, the behaviour of the two mixtures is alike, with low depths of rutting for NS modified mix by approximately 68.05%. This means that the nano-modifications result in improving the resistance of the mixture to permanent deformations.

The testing MS rut de6t ths are higher compared to the ones of bitumen mixtures with NS materials. Testing rut data of modified and control asphalt mix with 8% is lower than that of 6% modified bitumen mix. An 8% content of the MS and NS in control bitumen mix may reduce the rut depth values in modified mixes by a mean value of 50% and 58%, respectively. The greater NS concentration in the mix of the asphalt could probably lead to lower rut depths in modified mixes of the bitumen. Consequently, the bitumen mixes modified by the Nanosilica have higher resistance to the permanent rutting and deformation.

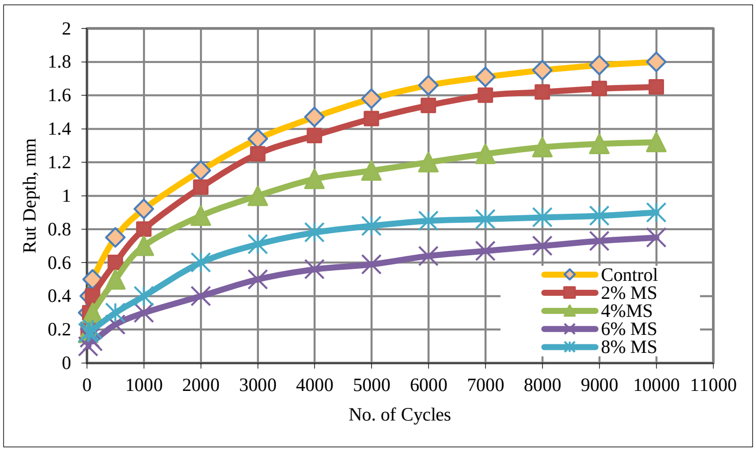

Fig. 19. Impact of the addition of the MS on rutting depth.

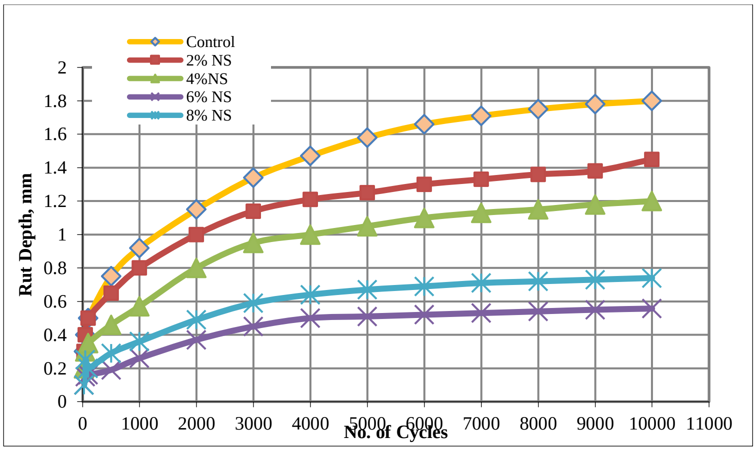

Fig. 20. Impact of the addition of the NS on the rutting depth.

6. Conclusions

The present research has been focused on impacts of the use of the micro-silica and nano-silica materials on the characteristics of bitumen binder and mix. Various ratios of micro and nano-silica have been added to neat asphalt binder. Moreover, an experimental study has been implemented to distinguish mechanical bitumen mix performance which has been modified with the MS and NS based on Marshall Properties, moisture damage evaluation, and permanent deformation. The following findings can be summarized:

- The addition of the nano-materials may improve the asphalt binders' performance. In particular, properties like softening point and kinematics viscosity may be improved (increased), whereas the penetration grade (decreased). Furthermore, the rheological characterization revealed that the viscosity increases mainly for both MS and NS modified binder comparing with the control, For NS additive gives higher viscosity values than MS.
- The micro- and nanomaterials in the bitumen mixtures leads to a reduction in the rut-depth of modified mixes according to the WTT rutting test data that has been compatible with factor $G^*/\sin\boldsymbol{\delta}$ results at a specified loading condition.
- The modified asphalt has a higher level of the stiffness at high traffic conditions and high surface temperature creep stiffness. M-value has been improved for all of the modified asphalt binders, especially at 6% NS adding. Hence, an improvement in stiffness and m-value exists in nanosilica modified specimen is higher compared to those in microsilica modified specimens, which demonstrates better performance of the modified samples at the low frequency values as well as high temperature degrees.
- The tensile strength of modified bitumen is improved in comparison with the non-modified one. It has been found that the addition of 8% NS to asphalt mixture show the maximum increase in ITS. The same applies for the Marshall Stability which is considerably better than the standard one.
- The results indicate that NS is considered the best modifier that achieved maximum stability, minimum flow, higher tensile strength; and lower rutting depth. Finally, 6 and 8% of Micro and Nano-silica were assigned as the optimal content for asphalt modification.

In summary, the use of micro- MS and nanosilica NS in the asphalt mixture improves the mechanical properties of these modified asphalt mixtures. Based on the test results of micro- and nanomaterials modified asphalt binders in this study, there is an excellent agreement with the results of previous studies [14, 15, 21, 25]. In addition, future work may focus on additional tests for evaluating the low-temperature performance of asphalt mixtures.

Conflict of Interest

The authors of this study confirm that there is no conflict of interest or any financial interest with any agency or person.

References

1. Al-Taher, M.G.; Hassanin, H.D.; Ibrahim, M.F.; and Sawan, A.M. (2018), Comparative study of performance of modified asphalt mixtures using

different traditional and nano additives. *International Journal of Scientific and Engineering Research*, 9(3), 1243-1250.

2. Leiva-Villacorta, F.; and Vargas-Nordcbeck, A. (2019). Optimum content of nano-silica to ensure proper performance of an asphalt binder. *Road Materials and Pavement Design*, 20(2), 414-425.
3. Adlinge, S.S.; and Gupta, A.K. (2013). Pavement deterioration and its causes. *International Journal of Innovative Research and Development*, 2(4), 437-450.
4. Fang, C.; Yu, R.; Liu, S.; and Li, Y. (2013). Nanomaterials applied in asphalt modification: A review. *Journal of Materials Science and Technology*, 29(7), 589-594.
5. Mostafa, A.A. (2016). Examining the performance of hot mix asphalt using nano-materials. *International Organization of Scientific Research(IOSRJEN)*, 6(02), 25-34.
6. You, Z.; Mills-Beale, J.; Foley, J.M.; Roy, S.; Odegard, G.M.; Dai, Q.; and Goh, S.W. (2011). Nanoclay-modified asphalt materials: Preparation and characterization. *Construction and Building Materials*, 25(2), 1072-1078.
7. Ameri, M.; Kouchaki, S.; and Roshani, H. (2013). Laboratory evaluation of the effect of nano-organosilane anti-stripping additive on the moisture susceptibility of HMA mixtures under freeze–thaw cycles. *Construction and Building Materials*, 48, 1009-1016.
8. Yang, J.; and Tighe, S. (2013). A review of advances of nanotechnology in asphalt mixtures. *Procedia-Social and Behavioral Sciences*, 96, 1269-1276.
9. Li, R.; Xiao, F.; Amirkhanian, S.; You, Z.; and Huang, J. (2017). Developments of nano materials and technologies on asphalt materials–A review. *Construction and Building Materials*, 143, 633-648.
10. Jamshidi, A.; Hasan, M.R.; Yao, H.; You, Z.; and Hamzah, M. (2015). Characterization of the rate of change of rheological properties of nano-modified asphalt. *Construction and Building Materials*, 98, 437-446.
11. Zhang, MH.; and Islam, J. (2012). Use of nano-silica to reduce setting time and increase early strength of concretes with high volumes of fly ash or slag. *Construction and Building Materials*, 29, 573-580.
12. Singh, L.P.; Karade, S.R.; Bhattacharyya, S.K.; Yousuf, M.M.; and Ahalawat, S. (2013). Beneficial role of nanosilica in cement based materials–A review. *Construction and Building Materials*, 47, 1069-1077.
13. Kong, D.; Du, X.; Wei, S.; Zhang, H.; Yang, Y.; and Shah, S.P. (2012). Influence of nano-silica agglomeration on microstructure and properties of the hardened cement-based materials. *Construction and Building Materials*, 37, 707-715.
14. Yao, H.; You, Z.; Li, L.; Lee, C.H.; Wingard, D.; Yap, Y.K.; and Goh, S.W. (2013). Rheological properties and chemical bonding of asphalt modified with nanosilica. *Journal of Materials in Civil Engineering*, 25(11), 1619-1630.
15. Lazzara, G.; and Milioto, S. (2010). Dispersions of nanosilica in biocompatible copolymers. *Polymer Degradation and Stability*, 95(4), 610-617.
16. Karthikeyan, M.; and Bindhu, B.K. (2010). Comparative study on bituminous concrete modified by nano silica and micro silica. *IJSTE International Journal of Science Technology and Engineering*, 4(10).

17. Hallock, M.F.; Greenley, P.; Diberardinis, L.; and Kallin, D. Potential risks of nano materials and how to safe handle materials of uncertain toxicity. *Journal of Chemical Health and Safety*, 16 (1), 16-23.
18. Abed, A.H.; Qasim, Z.I.; Al-Mosawe, H.; and Norri, H.H. (2019). The effect of hybrid anti-stripping agent with polymer on the moisture resistance of hot-mix asphalt mixtures. *Cogent Engineering*, 6(1), 1659125.
19. Fattah, M.Y.; Ibrahim Al Helo, K.H.; and Qasim, Z.I. (2016). Prediction models for fatigue resistance of local hot mix asphalt. *Road Materials and Pavement Design*, 17(4), 793-809.
20. Pacheco-Torgal, F.; and Jalali, S. (2011). Nanotechnology: Advantages and drawbacks in the field of construction and building materials. *Construction and Building Materials*, 25(2), 582-590.
21. Abutalib, N.; Fini, E.H.; Aflaki, S.; and Abu-Lebdeh, T.M. (2015). Investigating effects of application of silica fume to reduce asphalt oxidative aging. *American Journal of Engineering and Applied Sciences*, 8(1), 176-184.
22. Al-Taher, M.G.; Hassanin, H.D.; Elgendy, M.F.; and Sawan, A.M. (2017). Improving the performance of asphalt mixtures using nano silica. *World Applied Sciences Journal*, 35(12), 2614-2621.
23. Al-Taher, M.; Hassanin, H.; Ibrahim, M.; and Sawan, A. (2018). Investigation of the effect of adding silica fume on asphalt concrete properties. *International Journal of Engineering Research*, 7(4), 48-55.
24. SCRB SCRB/R9,(2003), General specification for roads and bridges. Section R/9, Hot Mix Asphalt Concrete Pavement, Revised Edition. State Corporation of Roads and Bridges, Ministry of Housing and construction, Republic of Iraq.
25. Santagata, E.; Baglieri, O.; Tsantilis, L.; Chiappinelli, G.; and Dalmazzo, D. (2016). Bituminous-based nanocomposites with improved high-temperature properties. *Composites Part B: Engineering*, 99, 9-16.
26. Ezzat, H.; El-Badawy, S.; Gabr, A.; Zaki, E.S.I.; and Breakah, T. (2016). Evaluation of asphalt binders modified with nanoclay and nanosilica. *Procedia Engineering*, 143, 1260-1267.
27. Yusoff, N.I.M.; Breem, A.A.S.; Alattug, H.N.; Hamim, A.; and Ahmad, J. (2014). The effects of moisture susceptibility and ageing conditions on nano-silica/polymer-modified asphalt mixtures. *Construction and Building Materials*, 72, 139-147.
28. Qasim, Z.I.; Abed, A.H.; and Almomen, K.A. (2019). Evaluation of mixing and compaction temperatures (MCT) for modified asphalt binders using zero shear viscosity and Cross-Williamson model. *Case Studies in Construction Materials*, 11, e00302.
29. EN 12697-22:2003+A1:2007 - Bituminous mixtures - Test methods for hot mix asphalt - Part 22: Wheel tracking.