Impact of Mixing process on the physical and Rheological Characteristics of Asphalt Binder Modified with Nano- Silica Powder

Hussein H Zghair¹, Hasan H Joni¹ and Maan S Hassan¹

¹Civil Engineering Department, University of Technology, 10066, Baghdad, Iraq

*E-mail: 11539@uotechnology.edu.iq (Hussein H Zghair)

Abstract. In Iraq pavements materials are exposed to various distresses due to the adverse environmental conditions and heavy traffic. The improvement of performance of flexible pavements is necessary for decreasing these distresses. The objective of this research is to improve the mixing efficiency of nano-silica particles with asphalt binder. Two mixer type (mechanical and high shear), rotational speeds (2000, 4000 and 6000 rpm), and mixing period (30 and 60 minutes) were assessed. Three nano-silica per cent 2%, 4%, and 6% by weight of asphalt were added and the asphalt binder mixtures were prepared by heating at mixing temperature 140 °C. Furthermore, comparisons were made to assess the modified asphalt binder characteristics such as penetration value, softening point, temperature sensitivity (penetration index), ductility and rotational viscosity. Results showed that the use of nano silica as an additive was useful in improving the asphalt binder characteristics. Generally, has been found that the penetration value decreased and softening point increased, as well as the viscosity, were increased and the temperature sensitivity was developed. Additionally, high shear mixing was more useful especially to obtain a good dispersion of nano silica powder in asphalt binder and got a homogenous composite material.

1. Introduction

Pavements of a highway are exposed to various distresses in Iraq, due to the adverse environmental conditions and heavy traffic. The improvement of performances of flexible pavements is necessary for decreasing these distresses. In order to improve the performances properties and the long-time behaviour, a new group of asphalt mixture has been developed through the combination of various types of modifiers [1–6]. During the last decades, there have been a variety of asphalt binder additives such as styrene-butadiene-styrene (SBS) [7–10], lime [11, 12], crumb rubber [13] and electronic waste powders [14–16] used to develop physical and rheological characteristics of asphalt binder.

In recent years, many researchers are interested in using the nanomaterial in improving asphalt binder properties and performance [17]. Nanomaterials are particles that have at least one dimension less than 100 nm [18]. There are several types of nano materials that are commonly used in asphalt modification such as nano silica, nano-clay, carbon nanotube, nano titanium oxide, and nano-hydrated lime(19-24).Many researchers concluded that nano silica powder has been used to reinforce polymers (25), Portland cement concrete mixtures (26, 27). Addition of nano silica with polymer modified asphalt to enhance the rheological properties and increased rutting resistance (28). Asphalt
binder was modified with nano-silica and seen that nano silica increased penetration value, reduced softening point and increased the rotational viscosity (29). Nano-silica has several characteristics such as high chemical purity, good dispersal ability, strong adsorption, large specific surface area, excellent and stability. Using nano silica powder by 2-4% of binder weight has been presented to decrease the rut depth by about half (30).

The major challenge in developing nano composite is the nanoparticles dispersion or chemical compatibility with composite materials. As a result, researchers are using proper procedures means to make uniform nano materials and stably disperse these in the asphalt binder. They found out that the issue of improving the nano silica (nS)/matrix interfacial adhesion and whole distribution must be resolved before completing the full potential of nano-reinforced composite materials. Dispersion of nanoparticles has been one of the main challenges due to the aggregation of the nanoparticles [31].

The object of study is to assess the mixing efficiency of a conventional mechanical mixer and the high shear mixer on a dispersion of nano silica into the asphalt binder by three contents 2%, 4% and 6% by weight. Also, evaluating the influence of mixing time on the physical and rheological properties of the modified asphalt binder.

2. Material and Methodology

2.1. Asphalt cement
An original asphalt 60/70 penetration grade obtained from the Dora refinery at the middle region of Iraq was used in this study. The basic characteristics of the asphalt binder were tested in the laboratory and showed in Table 1.

| Test               | Test Conditions | Standard                  | Results | Standard Limits of Iraqi Specification (SCRB/ R9, 2003)[37] |
|--------------------|----------------|---------------------------|---------|-------------------------------------------------------------|
| Penetration        | (0.1mm) cm     | ASTM D5[32]               | 66      | 60-70                                                       |
| Ductility          |                | ASTM D113 [33]            | 130     | >100                                                        |
| Flash point        |                | ASTM D92 [34]             | 302°C   | >232 °C                                                     |
| Fire point         |                | Fire 310°C Pen.           |         |                                                             |
| Thin – film oven test | 163 °C, 50gm, 5 hr. | 0.222<1 Mass loss        | 89.9    | >52                                                         |
| Rotational Viscosity | Pa. sec       | ASTM D4402 [35]           | 430     | @ 135 °C                                                   |
| Softening point    |                | ASTM D36 [36]             | 49.5    | @ 165 °C 128                                               |
| Penetration index  |                | ASTM D36 [36]             | -0.665  |                                                             |

2.2. Nano size of SiO2 powder (nS )
Nano silica is a white colour and hydrophobic powdered, a synthetic amorphous silica purchased from Aerosol Company in North America and shown in plate1, was used as the modifier of the binder. The main characteristics of nano silica, according to the manufacturer’s sheets are listed in Tables 2& 3.
Plate 1. Nano Size of SiO2 Powder

Table 2. Properties of Nano SiO2 Powder According to the Manufacturer Characteristics.

| Property         | Result       |
|------------------|--------------|
| Physical form    | Powder       |
| Color            | White        |
| Size             | 11-12 nm     |
| Bulk Density     | 0.1<         |
| (gr/cm³)         |              |
| True Density     | 2.4          |
| (gr/cm³)         |              |
| Specific surface | 200          |
| area, m²/g       |              |

Table 3. Chemical Properties of Nano SiO2 Powder According to the Manufacturer Characteristics.

| Property               | Result |
|------------------------|--------|
| SiO₂ content based on  | 99.8   |
| ignited material, wt.% |        |
| PH                     | 3.7-4.5|

2.3. Mixer Type

Conventional mechanical mixer and high shear mixer are apparatuses of the laboratory used for mixing modifiers such as polymers and nanomaterials with asphalt binder. Therefore, each mixer has its special qualifications. For example, due to the constant rotational speed of a mechanical mixer, its mixing time is supposed as a variable. And the rotational speed of the mixer is considered as variables for a high shear mixer.

2.3.1 Mechanical Mixer (M.M) is a mixing technique as shown in the plate 2, the high-speed rotary motion of the mixer produces a high centrifugal force that helps to mix asphalt binder with additives. The rotational speed of the motor is constant through the time of mixing. The rotational speed based on the additive type and the operator’s opinion. The mixer is used by assuming its mixing time parameter variable, while the rotational speed of the motor is constant in each minute, the composite material must be mixed as extended to obtain a homogenous material.
2.3.2 Shear Mixer (S.M.) is a special design of the tip that makes the high shear speed most proper mixers the previously mention mixers are used to mix asphalt binder with nanomaterial as shown in the plate 2. High shear mixer joins between the high shear speed degree and low pumping and circulations level [38]. High revolution rate pulls the materials from the bottom of the container to the top then a centrifugal force drives materials quickly out through the stator screen into lower speed area of the mix. Speed difference creates a hydraulic shearing force which produces homogenous composite materials [39].

Plate 2. Mixers Used to Mix Nano SiO₂ Powder with Asphalt Binder, Mechanical Mixer at Left Side and High Shear Mixer at Right Side.

2.4 Mixing Nano SiO₂ Powder with Asphalt Binder
Two mixers were used to mix nano SiO₂ powder with asphalt binder. The mechanical mixer can be set at a constant rotational speed (2000) rpm and high shear mixer can be set at angular velocities (2000, 4000 and 6000) rpm. Mixing time parameter is considered as a variable for mixers ranges (30 to 60) minutes to produce a homogeneous composite material. The asphalt binder was preheated to 140°C, based on the viscosity of base asphalt binder. Then, nano SiO₂ powder was gradually added in three percent (2%, 4% and 6 %) to the heated asphalt binder as shown the plate 3; and then tested to assess the physical and rheological properties of the modified asphalt binder.

Plate 3. Different Mixing Process of Nano SiO₂ Powder with Asphalt Binder.
2.5 Laboratory Testing
Nano SiO2 powder was mixed with a 60-70 penetration grade asphalt binder at 140 °C using two various mixers. The characteristics of the control and modified asphalts were assessed by using penetration grade, softening point temperature, temperature sensitivity, Brookfield rotational viscosity and ductility property.

3. Results and Discussions
Figures 1 and 2 illustrated the relationship of penetration grade and softening point temperature for two mixing processes of non-modified and modified asphalt binder. It can be observed that the penetration grade is declined and the softening point going up by adding the nano-silica concentration for two mixing processes. This development in the stiffness of modified asphalt binder may consequence from adsorption and diffusion of nano-silica in asphalt binder. Therefore, indications to loss and absorption oily materials in the maltene phase by nano-silica and alter to the resin in the asphalting phase of modified asphalt. As well, the stiffness of nano silica particles is harder than the stiffness of base asphalt binder [40]. Furthermore, the great decrease in penetration value and growth of softening point seen within the mechanical mixing process, due to the fact that the non-homogenous dispersion of nano silica powder and leads to the agglomeration.

![Figure 1. Penetration Value with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 30 Minute.](image1)

![Figure 2. Softening Point with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 30 Minute.](image2)
Figures 3 and 4, display the prolonged mixing time improving the physical and rheological characteristics up to 60 minutes for the high shear mixing process. Despite the fact, the mechanical mixing process leads to produce the hard asphalt binder attributed to increasing nano silica agglomeration.

![Mixing Time (60 min.)](image)

**Figure 3.** Penetration Value with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 60 Minute.

![Mixing Time (60 min.)](image)

**Figure 4.** Softening Point with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 60 Minute.

Figure 5 showed penetration index (PI) values against nano silica content. It can be observed that the PI value is reduced by adding the nano-silica for two mixing processes, which was rotational speed set at 2000 rpm. While the PI values for the high shear mixing process, which was rotational speed set at (4000 to 6000) rpm are increased by adding the nano-silica content. The incompatible influence by adding the nano-silica concentration for PI values, due to that the non-homogenous dispersion of nano silica powder and leads to the agglomeration [41].
Figure 5. Penetration Index with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 30 Minute.

Figure 6, exhibited the extended mixing time leads to great growth in PI value within high shear mixing process indicates that the dispersion of nano silica was more homogenous than the mechanical mixing process [42]. Representing that the rutting resistance increases with an addition of nano silica concentration attributed to form an exfoliated structure [43]. On the other hand the PI values still within the limits of specifications (+2.0 to -2.0), that can be used in pavement materials in reference [44].

Figure 6. Penetration Index with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 60 Minute.

Figure 7. Presented the ductility values against nano silica content. It can be observed that the ductility is declined by adding the nano-silica. An indication of loss and absorption oily materials in the maltene phase by nano silica particles. Furthermore, a great decline in ductility value seen within the mechanical mixing process, due to the fact that the non-homogenous dispersion of nano silica powder and leads to the agglomeration, as shown in plate 4.
Figure 7. Ductility value with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 30 Minute.

Plate 4. Dispersion of Nano-Silica particles in Asphalt Binder for Different Mixer (Mechanical Mixer at Left Side and High Shear Mixer at Right Side).

Figure 8, shows that the extended mixing time enhancing the ductility property up to 60 minutes for the high shear mixing process. Although the huge decline of ductility value is seen within the mechanical mixing process of modified asphalt up to 60 minutes, indicating that the agglomeration of nano silica particles [42].
Figures 9 - 12, illustrate the relationship of rotational viscosity values and various nano silica content at a temperature of 135 and 165°C. As can be observed that the viscosity is improved by the addition of nano silica content and it can consequence from adsorption and diffusion of nano-silica in asphalt binder and growth the stiffness of binder. Also, the great increase of viscosity value is seen within mechanical mixing process up to 60 minutes, indicating that the non-homogenous dispersion of nano silica powder and leads to the agglomeration, as shown in plate 4. While the slight a reduction of viscosity value is seen within high shear mixing of modified asphalt binder up to 60 minutes as shown in figures, as agreed with references [40&41].
Figure 10. Viscosity value with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 60 Minute.

Figure 11. Viscosity value with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 30 Minute.

Figure 12. Viscosity value with Different Contents of Nano-Silica for Two Types of Mixer at Mixing Period 60 Minute.
4. Conclusions
With a view to enhance the mixing efficiency of the mechanical mixer and the high shear mixer on a dispersion of nano silica into the asphalt binder. Moreover, the following conclusions were made according to the results obtained:

1. Using nano silica powder as an asphalt modifier reduces the penetration value and increases the softening point temperature.

2. The great decrease in penetration value and growth of softening point is seen within the mechanical mixing process, due to that the non-homogenous dispersion of nano silica powder and leads to the agglomeration.

3. A sensitivity of temperature (PI) is reduced by adding the nano-silica for two mixing processes, which was rotational speed set at 2000 rpm. While the PI values for the high shear mixing process, which was rotational speed set at (4000 to 6000) rpm are increased by adding the nano-silica content.

4. The ductility value declined by adding the nano-silica per cent. Also, a great decline in ductility value is seen within the mechanical mixing process, due to the non-homogenous dispersion of nano silica powder and leads to the agglomeration.

5. Long mixing time is enhancing the ductility property up to 60 minutes for the high shear mixing process. Although the huge declination of ductility value is seen within the mechanical mixing process of modified asphalt up to 60 minutes, indicating that the agglomeration of nano silica particles.

6. The viscosity is improved by the addition of nano silica content. It can consequence growth the stiffness of binder. The great increase of viscosity value is seen within mechanical mixing process up to 60 minutes. Although the slight declination of viscosity value is seen within high shear mixing of modified asphalt up to 60 minutes. This effect depends on the dispersion of nano silica powder and the agglomeration process.

7. High shear mixing set at (4000 to 6000) rpm for mixing time reached (30) minutes at 140°C mixing temperature was more useful especially to obtain a good dispersion of nano silica powder in asphalt binder and got a homogenous composite material. The prolonged mixing time enhances the physical and rheological characteristics of asphalt binder up to 60 minutes, but it is considered more consumption of energy and more costly.

8. Based on the characteristics of modified asphalt binder, the 4% of nano silica content was satisfactory to enhance the physical characteristics. Additionally, it become more appropriate for construction the pavements of highway in hot climate conditions.

References
[1] Collins J H, Boudin M G, Gelles R and Berker A. 1991 Improved Performance Of Paving Asphalts By Polymer Modification Proc Assoc Asphalt PavTechnol 43–79
[2] Saeed Ghaffarpour Jahromi and Ali Khodaii 2009 Effects of Nano clay on Rheological Properties of Bitumen Binder Construction and Building Materials 23, 2894–2904
[3] Zhanping You and Julian Mills-Beale 2011 Nano clay-modified asphalt materials: Preparation and characterization Construction and Building Materials 25 1072–1078
[4] Saeed Sadeghpour Galouyak and Bahram Dabir 2010 Rheological Properties and Storage Stability of Bitumen/SBS/Montmorillonite Composites Construction and Building Materials 24 300–307
[5] Sengoz B and Isikyakar G 2007 Evaluation of the Properties and Microstructure of SBS and EVA Polymer Modified Bitumen Construction and Building Materials 22 1897–905
[6] Lu X and Isacsson U 2001 Modification of Road Bitumen with Thermoplastic Polymers PolymTest 20 77–86
[7] Ouyang C, Wang S, Zhang Y and Zhang Y 2006 Improving the Ageing Resistance of Styrene-Butadiene-Styrene Tri-Block Copolymer Modified Asphalt by Addition of Antioxidants Polymer Degradation and Stability 91 795–804
[8] Kok B V and Yilmaz M 2009 The Effects of Using Lime and Styrene Butadiene-Styrene on Moisture Sensitivity Resistance of Hot Mix Asphalt Construction and Building Materials 23 1999–2006
[9] Khattak MJ, Baladi GY and Drzal LT 2007 Low-Temperature Binder-Aggregate Adhesion and Mechanistic Characteristics of Polymer Modified Asphalt Mixtures Journal of Materials in Civil Engineering 19 5 411–422
[10] Hasan H Joni and Ethar K Shaker 2017 Determination of the Acceptable Range of Mixing and Compaction Temperatures for Modified Asphalt Mixture with Styrene Butadiene Styrene (SBS) International Journal of Current Engineering and Technology
[11] Huang SC, Robertson RE, Branthaver JJF and Petersen JC 2005 Impact of Lime Modification of Asphalt and Freeze-Thaw Cycling on the Asphalt-Aggregate Interaction and Moisture Resistance to Moisture Damage Journal of Materials in Civil Engineering 17 6 711–718
[12] Lesueur D, Petit J and Ritter HJ 2013 The Mechanisms of Hydrated Lime Modification of Asphalt Mixtures: A State-of-The-Art Review Road Materials and Pavement Design 14 1 1–16
[13] Lo Presti D 2013 Recycled Tire Rubber Modified Bitumen’s for Road Asphalt Mixtures: A Literature Review Construction and Building Materials 49 863–881
[14] Colbert BW and You Z 2012 Properties of Modified Asphalt Binders Blended with Electronic Waste Powders Journal of Materials in Civil Engineering 24 10 1261–1267
[15] Joni H H and Zghair H H 2016 Effect of Adding Used-Foundry Sand on Hot Asphalt Mixtures Performance Eng. &Tech. Journal 34 (A) 6
[16] Zghair HH, Joni HH and Hassan MS 2018 Influence of Micro-Size Silica Powder on Physical and Rheological Characteristics of Asphalt binder International Journal of Engineering & Technology 7 4.20 180-184
[17] Hasan Z, Kamran R, Mohammad F, Ahmad G, and Hosein F 2001 Evaluation of Different Conditions on the Mixing Bitumen and Carbon Nano-tubes International Journal of Civil & Environmental Engineering 12 6 12-53
[18] Faramarzi M, Arabani M, Haghi A and Mottaghitalab V 2015 Carbon Nanotubes-Modified Asphalt Binder: Preparation and Characterization International Journal of Pavement Research and Technology 8 1 29-37
[19] Fang C, Yu R, Liu S and Li Y 2013 Nanomaterials Applied in Asphalt Modification: A Review Journal of Materials Science & Technology 29 7 589-594
[20] Shafabakhsh G H, Mirabdolazimi S M and Sadeghinejad M 2015 Evaluation the Effect of Nano-Tio2 on the Rutting and Fatigue Behavior of Asphalt Mixtures Journal of Construction and Building Materials 54 1 566-572
[21] Shafabahsh G H and Jafari Ani O 2015 Experimental Investigation of Effect of Nano TiO2/SiO2 Modified Bitumen on The Rutting and Fatigue Performance of Asphalt Mixtures Containing Steel Slag Aggregates Journal of Construction and Building Materials 85 1 136-143
[22] Yusoff N I M, Breem A A S, Alattug H N M, Hamim A and Ahmad J 2014 The Effects of Moisture Susceptibility and Ageing Conditions on Nano-Silica/Polymer-Modified Asphalt Mixtures Journal of Construction and Building Materials 72 1 139-147
[23] You Z, Mills-Beale J, Foley J M, Roy S, Odegard G M, Dai Q and Goh S W 2011 Nano Clay Modified Asphalt Materials: Preparation and Characterization Construction and Building Materials 25 2 1072-1078
[24] Khattak M J, Khattab A, Rizvi HR and Zhang P 2012 The Impact of Carbon Nano-Fiber Modification on Asphalt Binder Rheology Construction and Building Materials 30 5 257-264
[25] Chrissafis K, Paraskevopoulos KM, Papageorgiou GZ, Bikaris DN 2008 Thermal and Dynamic Mechanical Behavior of Bio-Nanocomposites; Fumed Silica Nanoparticles Dispersed in Poly (Vinyl Pyrrolidone), Chitosan, and Poly (vinyl alcohol) J. Appl. Polym. Sci. 110 1739-1749
[26] Jaber A, Gorgis I and Hassan M 2017 Relationship between Splitting Tensile and Compressive Strengths for Self-Compacting Concrete Containing Nano- and Micro Silica MATEC Web of Conferences 162 02013
[27] Quercia G and Brouwers HJJ 2010 Application of Nano-Silica (Ns) in Concrete Mixtures In 8th Fib International PhD Symposium in Civil Engineering Lyngby 20 431-436
[28] Alhamali DL, Wu J, Liu Q, Hassan NA, Yusoff NIM and Ali SIA 2015 Physical and Rheological Characteristics of Polymer Modified Bitumen with Nano silica Particles Arab Journal of Sci. Eng. 15
[29] Ezzat H, El Badawy S, Gabr A, Zaki EI and Breakah J 2016 Evaluation of Asphalt Binders Modified with Nano clay and Nanosilica. Advances in Transportation engineering: The Third International Conference on Transportation Geotechnology 143 1260-1267
[30] Yao H, You Z, Li L, Lee CH, Wingard D, Yap YK, Shi X, Goh SW 2012 Rheological Properties and Chemical Bonding of Asphalt Modified with Nano Silica J. Mater. Civ. Eng. 25 11 1619-1630
[31] Hussain F et al. 2006 Review Article: Polymer–Matrix Nanocomposites, Processing, Manufacturing, and Application: An Overview J Compos Mater 40 17 1511–75
[32] ASTM D5 2015 Standard Test Method for Penetration of Bituminous Materials Annual Book of Standards American Society for Testing and materials 04.03
[33] ASTM-D113 2015 Standard Test Method for Ductility of Bituminous Materials. Annual Book of Standards American Society for Testing and materials 04.03.
[34] ASTM D92 2015 Standard Test Method for Flash and Fire Points by Cleveland Open Cup Tester Annual Book of Standards American Society for Testing and materials 04.03
[35] ASTM D4402 2015 Standard Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscometer Annual Book of Standards American Society for Testing and materials 04.03
[36] ASTM D36 2015 Standard Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus) Annual Book of Standards American Society for Testing and materials 04.03
[37] State Organization for Roads and Bridges 2003 Hot Asphaltic Concrete Pavement, Iraqi Standard Specification Ministry of Housing and Construction, Department of Design and Study Section R9
[38] Ames G 2014 Mixing, Blending and Size Reduction Handbook. Available from: www.admix.com/pdfs/resource/library-tech-mixhandbook.pdf
[39] Silverson 2016 Mixing with a High Shear Laboratory Mixer Available from: http://www.silverstown.com/us/products/labatory-mixers/how-it-works
[40] Taherkhani H and Afrozoo S 2016 The Properties of Nano Silica-Modified Asphalt Cement. Petroleum Science and Technology 1381-1386
[41] Mahmoud Enieb and Aboelkasim Diabi 2017 Characteristics of Asphalt Binder and Mixture Containing Nano silica International Journal of Pavement Research and Technology 10 148-157
[42] Taherkhani H and Afrozoo S 2017 Investigating the Performance Characteristics of Asphaltic Concrete Containing Nano-Silica. Civil Engineering Infrastructures Journal 50 1 75 – 93
[43] Nura Bala, Ibrahim Kamaruddin, Madzlan Napiah and Nasiru Danlami 2017 Rheological and Rutting Evaluation of Composite Nano Silica/ Polyene Modified Bitumen Materials Science and Engineering 201
[44] White Oak D and Read J 2015 Shell Bitumen Handbook Shell Bitumen London UK