Hot Asphalt Mixture Characteristics with Nano-Metakaolin Materials

Mustafa A Kareem¹, Abbas Al-Hdabi², Nibras A Al-Sahaf³ and Laith F Hasan⁴

¹ Post graduate student, Department of Civil Engineering, Faculty of Engineering, University of Kufa, Iraq. Email: kareemmustafa41@gmail.com
² Asst. Prof., Department of Civil Engineering, Faculty of Engineering, University of Kufa, Iraq. Email: abbas.alhadabi@uokufa.edu.iq
³ Lecturer, Department of Civil Engineering, Faculty of Engineering, University of Kufa, Iraq. Email: nibrasa.alsahhaf@uokufa.edu.iq
⁴ B.Sc. Civil Engineering, University of Kufa, Iraq. Email: laithf.hasan@uokufa.edu.iq

Abstract. In this research, Nano-metakaolin was used to investigate the achievable prospects to enhance asphalt mixture properties. The aim of this study was to evaluate the effect of adding Nano-Metakaolin on the properties of asphalt mix and comparing it with the local and international requirements, besides identifying the optimum percent of NMK to be incorporated in the hot mix asphalt. As a modifier to the bitumen several percentages of Nano-Metakaolin was used i.e. 1, 2, 4, 8, 16 and 20% by weight of bitumen. Modified asphalt binders were subjected to physical and conventional tests which were penetration, kinematic viscosity, softening point, and ductility. From these tests, optimum Nano-Metakaolin was 16%. After obtaining the optimum Nano-Metakaolin, two sets of asphalt concrete mixtures were prepared, the first was with the pure asphalt (control mixture) and the second was with the optimum Nano-Metakaolin i.e. 16% (modified mixes). The control and modified mixtures have been evaluated based on experimental tests, such as Marshall Stability (MS), Marshall flow, Marshall stiffness, Indirect Tensile Strength (ITS) and Index of Retained Strength (IRS). The results for these tests indicated an improvement in the mechanical proprieties i.e. increased Marshall Stability, Indirect Tensile Strength (ITS), and index of retained strength. As a final result, the use of (16%) Nano-metakaolin by weight of asphalt enhanced asphalt concrete properties and produce durable mixes for highway construction.

Keywords: Nanotechnology; Asphalt mixtures; Pure asphalt; Nano-metakaolin; mechanical properties.

1. Introduction
Asphalt concrete is one of the most common types of pavement surface materials used in the world. Asphalt pavements must undergo heavy loads and unfavorable environmental conditions and their resulting distresses such as high-temperature rutting and low-temperature cracking for an acceptable period of time. Therefore, modification of asphalt layers is necessary.

It is a composite material consisting of mineral aggregates, asphalt binder, and air voids. The load-carrying behavior and resulting failure of such material depend on many mechanisms that are strongly related to the local load transfer between aggregate particles [1].
Due to the rapid urbanization and industrialization of the world over the last century, the construction and maintenance of transportation roadways is a constant demand in both urban and rural areas. Furthermore, due to excessive traffic loads and environmental factors, many existing pavements have already reached the end of their service life and other pavements will soon require maintenance [2].

Fatigue cracking and rutting (permanent deformation) distresses are the most significant challenges that can affect the functional performance of asphalt highways. The reducing of the problems would be more economical for recovery while reconstructing defects require more costs. One approach to enhance asphalt characteristics is by using a variety of modifiers including rubbers and polymers. Lately, several effective solutions have been proposed such as the adoption of nanomaterials to modify asphalt and/or asphalt mixtures characteristics performance [3].

Nanomaterials in present days are being utilized widely in a spectrum of fields. Nanotechnologies work on modifying base materials on the molecular level to produce new materials with better properties. For example, modifying the properties of Portland cement using Nanomaterials have been examined by several studies. Lately, Nanomaterials have been used in road paving. Many studies have been carried out to investigate the potential advantage of adding different Nanomaterials to asphalt binders such as “Nano clay, Nano silica, Nano-hydrated lime, Nano-sized plastic powders, or polymerized powders, Nanofibers, and Nanotubes” [4].

The most common distresses that could face unmodified paving asphalt is the rutting deformation (due to high temperature) and cracking (due to low temperature). Modifying and strengthening asphalt hence should be considered in the asphalt pavement industry [5].

As the world continues to urbanize and construct more transportation roadways, the need for high-quality and sustainable pavement is a constant need. Due to these demands, transportation experts and engineers focus on improving the performance and service-life of pavements. Such modifications aim to improve the mechanical properties and durability of asphalt mixtures by adding nano-metacaolin to the asphalt cement.

2. Methodology

2.1 Selected Materials

In this research, the materials provided are commonly used in preparation of hot asphalt mixtures especially in the middle and south regions of Iraq such as aggregate, cement, asphalt binder.

2.1.1 Aggregates

The coarse aggregate has been brought from a hot-mix asphalt plant in Al-Najaf city (Iraq) and it was originally brought from Al-Nibaee quarry (North of Baghdad). Tables 1 and 2 list the main chemical and physical characteristics of the used aggregate which were tested in accordance to ASTM, (2004) [6]. While Table 3 shows the selected gradation for type IIIA surface course mixture in accordance to Iraqi specification for roads and bridges (SCRB, 2003) [7].
### Table 1. Chemical composition of Nibaee aggregate.

| Chemical compound | Results, % |
|-------------------|------------|
| L.O.I.            | 6.55       |
| MgO               | 0.78       |
| SiO₂              | 82.52      |
| CaO               | 5.37       |
| SO₃               | 2.7        |
| Fe₂O₃             | 0.69       |
| Al₂O₃             | 0.48       |

Mineral composition

| Mineral   | Results, % |
|-----------|------------|
| Calcite   | 10.92      |
| Quartz    | 80.03      |

### Table 2. Physical properties of aggregates

| Property                                      | ASTM designation | Test Results | SCRB specifications |
|-----------------------------------------------|-------------------|--------------|---------------------|
| Bulk specific gravity                         | C127              | 2.610        |                     |
| Apparent specific gravity                     | C127              | 2.641        |                     |
| Percent wear by Los Angeles abrasion, %       | C131              | 22.7         | 30 Max              |
| Soundness loss by sodium sulfate Solution, %  | C88               | 3.4          | 12 Max              |
| Flat and elongated particles, %               | C4791             | 5            | 10 Max              |
| The degree of crushing, %                     | D5821             | 96           | 90 Max              |

**Fine aggregate**

| Property                                      | ASTM designation | Test Results | SCRB specifications |
|-----------------------------------------------|-------------------|--------------|---------------------|
| Apparent specific gravity                     | C128              | 2.68         |                     |
| Sand equivalent, %                            | D2419             | 57           | 45 Min              |
| Bulk specific gravity                         | C128              | 2.631        |                     |
| Clay lumps and friable particles, %           | C142              | 1.85         | 3 Max               |
| Angularity, %                                 | C1252             | 54           |                     |

### Table 3. Aggregate size separation by using sieve analysis.

| Sieve size, mm | Specification limit | % passing by total weight of aggregate |
|----------------|---------------------|----------------------------------------|
| 19             | 100                 | 100                                    |
| 12.5           | 90-100              | 95                                     |
| 9.5            | 76-90               | 83                                     |
| 4.75           | 44-74               | 59                                     |
| 2.36           | 28-58               | 43                                     |
| 0.300          | 5-21                | 13                                     |
| 0.075          | 4-10                | 7                                      |
2.1.2 Asphalt Cement

One type of asphalt cement is used with (40-50) penetration grade brought from AL Nasiriya refinery. Table (4) shows the physical properties of asphalt cement which were evaluated according to ASTM standards ASTM, (2004) [6] and compared with Iraqi Specification known as a general specification for roads and bridges [7].

| Property                                                | ASTM Designation | Test Results | Requirements for penetration graded asphalt cement |
|---------------------------------------------------------|------------------|--------------|---------------------------------------------------|
| Penetration at 25°C, 100 gm, 5 sec (1/10 mm)           | D5               | 40           | 40-50                                             |
| Softening point, Ring and ball °C                       | D36              | 50           | ………………                                        |
| Ductility at 25 °C, cm                                  | D113             | 105          | ………………                                        |
| Flash point, °C                                         | D92              | 273          | >232                                             |
| Specific gravity at 25 °C                              | D70              | 1.03         | ………………                                        |
| Solubility in trichloroethylene, % wt                   | D2042            | 99.31        | >99                                              |
| Residue from thin-film oven test                        | D1754            |              | ………………                                        |
| -Ductility at 25 °C, cm                                 | D113             | 33           | >25                                              |
| Retained penetration, % of original                     | D5               | 59           | >55                                              |

2.1.3 Filler

Filler materials represent mineral particles that pass Sieve No.200 (0.075mm). The mineral filler used in this research was ordinary Portland cement (OPC) obtained from Al Kufa factory, at 7 percent content, which represents the average value of SCRB specifications [7]. Table (5) shows the Physical properties of this cement.

2.1.4 Nano-metakaolin (NMK)

The nano-metakaolin used in this study is manufactured by Iranian Nanomaterials Pioneers Company in Iran [8]. The properties of nano metakaolin were with surface area of 480000 cm²/g and of average dimensions of 200*100*20 nm. Table (6) gives the chemical composition of NMK. Practical steps of the preparation process of nano-metakaolin (NMK) is shown in Figure (1).

| Property                              | Portland cement |
|---------------------------------------|-----------------|
| Passing sieve No. 200, (%)            | 94.76           |
| Specific surface area (m²/kg)         | 418             |
| Specific gravity (ASTMC188-95)        | 3.1             |

| Chemical content | %    |
|------------------|------|
| SiO₂             | 45.5 |
| Al₂O₃            | 37   |
| Fe₂O₃            | 0.2  |
| TiO₂             | 1.5  |
| CaO              | 0.01 |
| MgO              | 0.02 |
| Na₂O             | 0.03 |
| K₂O              | 0.07 |
| L.O.I            | 12.5 |
3. Experimental works

3.1 Testing on Asphalt Binder
Asphalt binder (40-50) was used in this research. In order to evaluate asphalt cement properties, several tests were conducted on both unmodified asphalt binder and modified asphalt specimens that have been prepared. The modified specimens are prepared by adding various percent of Nano-metakaolin 1, 2, 4, 8, 16, and 20%. Different laboratory tests have been performed such as the penetration, softening point, kinematic viscosity, and ductility.

3.2 Testing Program
All materials and mixture were prepared under ASTM specification D1559-89. The mixture was compacted according to the Marshall mix design method standards. Three important tests were carried out on the prepared asphalt mixture; these are Marshall stability, indirect tensile and index of retained strength tests.

3.2.1 Marshall Test
Marshall Method for designing hot asphalt mixtures was used to determine the optimum asphalt content. According to standard 75-blows on each face. Marshall design method designated as ASTM D1559-89, (AASHTO T 245-13) [9] a number of specimens each of 1200 gm in weight was prepared using five different asphalt contents (from 4 - 6% with 0.5 % incremental). Three specimens were used to prepare asphalt mixture with one- asphalt content to have an average value of Marshall Flow, bulk density, and stability.

3.2.2 Indirect Tensile Strength (ITS) Test
A mechanical displacement control testing frame was used to conduct the indirect tensile tests in accordance with ASTM D4123 (ASTM, 2004) in order to evaluate the tensile strength of asphalt concrete mixtures.

3.2.3 Index of Retained Strength (IRS) Test
Index of retained strength (IRS) test is used to evaluate moisture damage of asphalt pavement in accordance with method ASTM D1075 (ASTM, 2004). It is one of the tests required by (SCRB, R/9, 2003) [8] specifications to be performed on asphalt mixes used in a surface course in addition to Marshall Tests [10].

Figure 1. Practical steps of the preparation process of Nano-metakaolin
4. Results and discussion
The results of laboratory work have been obtained and analyzed in order to identify the optimum percent of Nano-metakaolin to be added to the asphalt cement; in addition to study the effect of adding the optimum percent of Nano-metakaolin on mechanical properties of asphalt concrete mixtures. Laboratory work results are presented in this chapter in two stages. First, several sorts of testing have been conducted to investigate the impact of adding Nano-metakaolin material to the base asphalt (40-50 penetration grade). The tests include kinematic viscosity, penetration, softening point and ductility to find the optimum Nano-metakaolin content. The second stage, Marshall Test was carried out with different percentages of asphalt which are (4.0, 4.5, 5.0, 5.5, and 6.0%) and the results are analyzed in order to obtain the optimum asphalt content. After obtaining OAC and optimum NMK, two sets of asphalt concrete specimens with and without NMK were prepared. The results are analyzed on several experimental tests including Marshall Test, Marshall Stiffness, indirect tensile strength, Index of retained strength and Volumetric Properties. Finally, the results were compared with the Iraqi SCRB specifications [8].

4.1 Effect of Nano-metakaolin on Penetration Test Results
Four tests were carried out to study the effect. Penetration tests were carried out for the base and modified asphalt with different percentages of Nano-metakaolin i.e. 1, 2, 4, 8, 16, and 20% by weight of asphalt at different temperatures which were (15, 25 and 35°C). Figure (2) shows the penetration test results for different percentages of Nano-metakaolin. According to the results, with the addition of different percentages of Nano-metakaolin, the penetration value at high temperature decreased dramatically while there is a little decrease at low temperature in comparison with unmodified asphalt. The best results for the asphalt binder used in this study obtained in the asphalt modified with 16% Nano-metakaolin.

![Figure 2. Variation of penetration values versus Nano NMK content](image)

4.2 Effect of Nano-metakaolin on Viscosity Test Results
The Kinematic viscosity test has been carried out at percentage of 4, 8, and 16% by weight of asphalt at different temperatures which were 90, 110, and 130°C. Figure (3) shows the effect of Nano-metakaolin content on the Kinematic viscosity of asphalt with change temperature. By adding Nano-metakaolin to the base asphalt binder, the viscosity increased. The maximum viscosity was related to the case of adding 16% (NMK) which occurred at 130 °C.
4.3 Effect of Nano-metakaolin on Softening Point Test Results
The results of softening point test for base asphalt binder and asphalt binder modified with different percentages of Nano-metakaolin are shown in Figure (4). The figure demonstrates a change in softening point from adding 1% NMK to 20% NMK. There is a significant increase in softening point after adding 4, 8, and 16% Nano-metakaolin by weight of asphalt.

4.4 Effect of Nano-metakaolin on Ductility Test Results
Figure (5) demonstrates the results of ductility test for both unmodified asphalt and modified asphalt with different percentages of Nano-metakaolin.

In accordance with the above testing, the results showed that the optimal percent for Nano-metakaolin is 16%.

![Figure 3. Variation of kinematic viscosity versus Nano NMK content](image1)

![Figure 4. Variation of softening point versus Nano NMK content](image2)
4.5 Optimum Asphalt Content
1200 gm in weight were prepared using five different asphalt contents (4 - 6% with 0.5 % incremental) in order to find optimum asphalt content (OAC). Figure (6) shows a summary of Marshall test outcomes.

4.5.1 Determination of optimum asphalt content (OAC)
Figure 6 is utilized to obtain three AC values as follows:
The asphalt content at the highest MS = 4.6%
Asphalt content at the highest (% GMB) bulk density = 5.5%
Asphalt content at the median of allowed percentages of air voids (% AV) i.e. 4% for surface course mixtures = (5.35) %
Optimum asphalt content (OAC) = \( \frac{4.6\% + 5.5\% + 5.35\%}{3} \) = 5.15%
The result of OAC satisfies with Iraqi specifications (SCRB, R/9, 2003) [7] and (ASTM, 2004) [6].

4.6 Effect of NMK on Mechanical Properties of HMA
Two sets of the specimens were prepared; asphalt (40-50) penetration grade and the second mixture is prepared as a modified binder with Nano-metakaolin of 16% metakaolin by weight. They have been evaluated based on experimental tests which are Marshall Stability MS, Marshall Flow, Marshall Stiffness, ITS, and IRS. Figure (7) illustrates the influence of the addition of Nano-metakaolin on Marshall Stability test. The figure implies that Marshall Stability increased significantly when 16% of Nano-metakaolin has been added to the base asphalt binder. Figure (8) represents the influence of the addition of Nano-metakaolin on Marshall Flow test. There is a slight decrease in Marshall Flow when 16 percent of NMK has been added to the base asphalt binder.

The indirect tensile strength test results are shown in Figure (9). The value has been increased by adding Nano-metakaolin to the base asphalt binder. High tensile strength at failure is a desirable property for stiff mixtures. ITS increased almost by 8% for modified mixtures in comparison with the control mixtures. While, Figure (10) shows the influence of the addition of NKM on the index of retained strength test for modified and unmodified mixtures.

Due to the results above, the Using of Nano Metakaolin enhanced the Characteristics Hot Asphalt Mixture with the optimum ration of 16% by weight of asphalt material.
Figure 6. The relationships between asphalt cement and the properties of control mixtures

Figure 7. Effect of Nano metakaolin on Marshall Stability
Figure 8. Effect of Nano metakaolin on Marshall Flow

Figure 9. Effect of Nano metakaolin on indirect tensile strength

Figure 10. Effect of Nano metakaolin on the index of retained strength
5. Conclusions

The aim of this research was to evaluate the effects of Nano-metakaolin as an asphalt binder modifier on the asphalt mixtures. Several laboratory tests were conducted to evaluate the characteristics of HMA by different contents of Nano-metakaolin added to the base asphalt binder. Based on the limited test results obtained in this research, the following conclusions can be drawn.

1. Regarding kinematic viscosity, penetration, softening point, and ductility test. The optimum amount of Nano-metakaolin to be added as a modifier of asphalt mix was obtained to be 16% by the total weight of asphalt mix.

2. The results of the penetration test and ductility test represent decreased penetration degree and the ductility also declined after adding Nano-metakaolin to the base asphalt binder. This indicates that modified asphalt with NMK is more stiffer in comparison with base asphalt binder.

3. Marshall stability is increased by adding 16% of NMK to the base mixture. Also, Marshall stability is increased by almost 17% for the modified mixes in comparison with the control mixes.

4. By means of Indirect Tensile Strength. ITS increased by almost 7% for modified mixtures in comparison with the unmodified asphalt concrete mixtures.

5. In terms of the Index of Retained Strength test. IRS is increased from 85 to 92%. IRS reduced performance and increased maintenance for asphalt pavements. Thus, enhance IRS rise performance and reduce maintenance.

References

[1] Sadd M H, Dai Q, Parameswaran Vand Shukla A 2004 Microstructural Simulation of Asphalt Materials: Modeling and Experimental Studies Journal of Materials in Civil Engineering, 16(2), 107-115.

[2] Siriwardane H, Gondle R and Kutuk B 2010 Analysis of Flexible Pavements Reinforced with Geogrids Geotechnical and Geological Engineering, 28(3), 287-297.

[3] Shafabakhsh G H, Mirabdolazimi S M, and Sadeghnejad M 2014 Evaluation the effect of nano-TiO2 on the rutting and fatigue behavior of asphalt mixtures Construction and Building Materials, 54, 566-571.

[4] Sadeghnejad M and Shafabakhsh G 2017 Experimental Study on the Physical and Rheological Properties of Bitumen Modified with Different Nano Materials (Nano SiO2 & Nano TiO2) International Journal of Nanoscience and Nanotechnology, 13(3), 253-263.

[5] Lewandowski L H 1994 Polymer modification of paving asphalt binders Rubber Chemistry and Technology, 67(3), 447-480.

[6] ASTM Standards 2004 Roads and Paving Materials Annual Book of the American Society for Testing and Materials Standards, Section 4, Vol. 04-03.

[7] State Commission of Roads and Bridges (SCRB/R9) 2003 General Specification for Roads and Bridges Republic of Iraq, Ministry of Housing and Construction, Department of Planning and Studies, Baghdad, Revised Edition, Addendum No.3.

[8] Iranian Nanomaterials Pioneers Company http://www.irananotech.com

[9] AASHTO 2007 Standard Specifications for Transportation Materials and Methods of Sampling and Testing. 5th edition, American Association of State Highway and Transportation Officials, Washington, D.C., USA.

[10] Al-Jumaily M A H 2008 Evaluation of Hydrated Lime Filler in Asphalt.