Hot Mix Asphalt Characteristics Improved With Nano Materials

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Abstract. Common failures in road asphalt pavement includes fatigue, rutting and cracking. It is also common that these failures are usually resulted from either temperature sensitivity or moisture susceptibility of asphalt mix or one of its component – asphalt or aggregate. Many studies have recently dealt with these problems in asphalt roads and several solutions were proposed. Using nano-materials is one of these advanced solutions. Investigating the feasibility of using nano-materials as a mechanism for improving asphalt mix characteristics is the aim of this project. Specifically, the potential effects of nano-carbon in improving bituminous mixtures used in road paving have been experimentally studied. Several tests on bitumen and bituminous mixture with and without nano-carbon have been conducted. Penetration, softening point and ductility were conducted on the base and modified bitumen, while Marshall Stability, retained strength and Indirect Tensile Strength (ITS) tests have been carried out on asphalt mixture. The results proved that using nano carbon can largely improve Marshall Stability, ITS and index of retained strength. On the other hand, it is found that the penetration at high temperatures decreased significantly despite there was a slight decrease in it at low temperatures; therefore, it is recommended that modified mixtures - i.e. those with nano carbon - can greatly withstand permanent deformation in comparison with control mixtures.

Keywords. Nano-Carbon, hot asphalt mixtures, Ordinary Portland Cement, durability, mechanical properties.

1. Introduction

Fatigue, rutting and cracking are the most significant challenges that can affect the functional performance of asphalt (bitumen) highways. Many studies have recently dealt with these problems in asphalt roads and several solutions were proposed. Using nano-materials to modify asphalt and/or asphalt mixtures characteristics is one of these state-of-the-art solutions. [1].

The current research studies the effect of using nano-material such as nano-carbon tube and nano-silica on the physical properties of bitumen such as penetration, softening point, and viscosity.

Worldwide, the main role and function of asphalt when using it in road paving asphalt mixtures is to bind aggregate particles in order to compose pavement that is as strong as possible to sustain external...
stresses. Such stresses could be due to the loads of passing traffic (vehicles) or due to severe environmental boundary conditions (moisture and temperature) for long time. 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 is hence should be considered in asphalt pavement industry [2 and 3]. Such modifications aim to improve the key characteristics of unmodified asphalt such as temperature sensitivity, durability, adhesion, aging resistance, oxidation resistance, and friction.

According to Shen (2011), asphalt characteristics can be modified using a variety of modifiers including rubbers, polymers, resins, metals, chemical agents, fibres, and sulfur. Nano-materials in present days are being utilized widely in highway pavement engineering industry and in research as a new generation of bitumen modifier [4].

The distinct feature of nano-materials is that they 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 nano-materials have been examined by several studies. Lately, many studies have been carried out to investigate the potential advantage of adding different nano-materials to bituminous binders. Such nano-scale modifiers include but not limited to nano-silica, nano-clay, nano-tubes, nano-hydrated lime and other nano-scale polymer and plastic powders [5].

It is worthwhile mentioning that materials with nano-sized particles are with physico-chemical characteristics that are significantly different from their original materials (with natural size particles). The main reason is that the generated new nanomaterial have relatively enormously larger surface area as shown in Figure (1). Nano-materials can be technically defined as any material or chemical substance with particles of at least one of its dimension is (1-100) nanometers.

While nano-materials can be useful on many commercial and technical aspects, their environmental and safety potential hazards should be carefully appraised and determined. In Europe, there are nowadays many products in the market that contain nano-materials; coating, anti-bacterial clothing, food products, batteries and cosmetics, just to name a few.

Road asphalt pavement is typically manufactured using optimum amount of asphalt binder with aggregate of specific proportions, gradation and characteristics. The ultimate purpose of asphalt mix design is to produce road paving mixture that are capable of withstanding heavy traffic loads and environmental impacts [7]. For instance, exposure to environmental challenge such as water (moisture) is one of the most recognized challenges that could reduce the asphalt strength and increase the possibility of rutting and fatigue problems. The characteristics of asphalt and asphalt mixture materials can largely affects their strength. Adding modifiers such as nano-materials is expected to enhance these characteristics.

This research studies the potential effects of Nano carbon on the physical properties of bitumen such as penetration, softening, and ductility used in hot-mix asphalt. Also, the mechanical properties and water damage have been studied to indicate the main advantages from producing the new binder.

2. Materials and Type Testing

2.1 Materials

Two sets of asphalt mixes have been prepared for laboratory testing for the current study. The first set includes the base (unmodified) mixture whereas the second set includes the modified mixture (with nano-carbon). The working materials which include aggregate, filler and asphalt cement are sampled, prepared and tested according to the State Commission for Roads and Bridges specifications in Iraq (SCRB/R9) 2003 [8].

2.1.1 Aggregate
Concrete asphalt usually contains coarse aggregate, fine aggregate and mineral fillers. Crushed quartz is the aggregate type utilized in the lab tests of the current study. It is usually used for highway paving. It has been brought from a hot-mix asphalt plant in Al-Najaf city (Iraq) and it was original brought from Al-Nibaee quarry (North Baghdad). Tables 1 and 2 lists the main chemical and physical characteristics of the used aggregate. Figure 2 shows aggregate gradation in accordance to the sieve analysis for the selected gradation which was 12.5mm maximum size for surface course type III, SCRB/R9 (2003) [8].

2.1.2 Asphalt cement
Marshall test samples required to evaluate the hot mix asphalt stability and flow have been prepared using 60-70 penetration grade asphalt cement. The physical properties of this binder is shown in Table 3. Then Nano carbon was added to this type of bitumen with different percentages i.e. 0.5, 1, and 2% by weight of asphalt.

2.1.3 Mineral filler
Ordinary Portland cement was used as the mineral filler in HMA sample. Table 4 shows the key properties of this cement.

2.1.4 Nano-Carbon
Carbon nanotubes (CNTs) are allotropes of carbon with a cylindrical nanostructure. These cylindrical carbon molecules have unusual properties, which are valuable for nanotechnology, electronics, optics and other fields of materials science and technology. Owing to the material's exceptional strength and stiffness, nanotubes have been constructed with length-to-diameter ratio of up to 132,000,000:1, significantly larger than for any other material. Carbon nanotubes are the strongest and stiffest materials yet discovered in terms of tensile strength and elastic modulus respectively. Nano-Carbon have a tensile strength of 63 gigapascals (9,100,000 psi). In addition, Nano-Carbon shells have strengths of up to ≈100 gigapascals (15,000,000 psi), and a low density for a solid of 1.3 to 1.4 g/cm³, its specific strength of up to 48,000 kN·m·kg⁻1 is the best of known materials, compared to high-carbon steel's 154 kN·m·kg⁻1.

2.2 Type testing
As stated previously, both unmodified asphalt cement and modified samples have been prepared. The modified samples are comprised by adding three different ratios of nano-carbons – 0.5%, 1% and 2%. The effects of nanocarbon added on bitumen characteristics were evaluated by the aid of penetration and softening point tests results.
ASTM specification D1559-89 [9], was used as a guide to prepare the HMA mixture. At first and before mixing with AC, all the aggregate groups (coarse, fine and filler) previously separated by particle size (gradation) were heated up to 150°C by an electric oven. The used 60-70 AC is also heated up to 150°C to assure the adequate mixing temperature with the aggregate. After mixing, the mixture is compacted according to the Marshall mix design method standards which states that Marshall compactor should be used to apply 75 blows per each sample side. Automatic compactor was used. Three important tests were carried out on the prepared asphalt mix; these are indirect tensile test, Marshall stability test, and Index of Retained Strength (IRS). The experimental program in this project includes conducting a number of standard tests that can reflect key properties of the original and modified HMA mixtures. The properties include durability, volumetric and mechanical properties. IRS test is used to evaluate AC mix durability. Marshall test results (stability and flow) and Indirect Tensile Strength test are used to quantify the mechanical properties of the AC mixture. In addition to the control sample, three modified samples were prepared using 0.5%, 1% and 2% nano-carbon ratios (% of bitumen weight). Three test sample of each of these main samples have been prepared for testing. The average values were finally computed.

2.2.1 Marshall Stability and flow
The ASTM D6927 specification [9], is used to prepare, compact and test the samples for computing Marshall Stability MS and Marshall Flow MF. The test quantifies the AC sample resistance to plastic flow. The asphalt mix sample is a 2.5in. height by 4.0in diameter cylinder. The load applied using the Marshall apparatus is applied on the AC specimen’s lateral surface at 60°C. The applying load continues with 50.8 mm/min pressing rate until reaching the failure load. Marshall Stability is computed based on the maximum load (failure load) whereas the corresponding strain recorded by the gage is the Marshall flow.

2.2.2 The Volumetric properties
ASTM D2726 specification [9], is utilized to compute dry bulk density whereas ASTM D3203 is used to specify and compute Voids in Mineral Aggregate (VMA), Air Voids (AV) and Voids Filled with Asphalt (VFA). Following are the corresponding mathematical models:

\[
\text{Dry density} = \frac{\text{dry weight}}{\text{SSDweight} - \text{weight in water}}
\]

(1)

where SSD is saturated-surface dry weight, gm.

\[
AV = \left(1 - \frac{\text{dry density}}{\text{SGmax}}\right) \times 100\%
\]

(2)

where AV is air voids, SG\text{max} is the theoretical specific gravity for the mixture computed as per ASTM D2041 specification.

\[
\text{VMA} = 100\% - \frac{Gmb \times Ps}{Gs} \times 100\%
\]

(3)

Where Ps is aggregate percentage in the mix and Gs is the bulk specific gravity of aggregate.

\[
\text{VFA} = \left(\frac{VMA - AV}{VMA}\right) \times 100\%
\]

(4)

2.2.3 Indirect tensile strength test
ASTM D4123 specification [9], standard was followed to perform the Indirect Tensile Strength and to compute the tensile or splitting strength. The test includes applying compressive load on the AC cylindrical specimen along its diameter and with 50.8 mm/min rate. The applied compression load will develop an indirect tensile stress along the specimen diameter which can be computed as follows:

\[
\sigma_t = \frac{2 \times P_{max}}{\pi HD}
\]

(5)

where \(\sigma_t\) is the indirect tensile strength in KPa, \(P_{max}\) is the maximum applied load, KN, D is the specimen diameter, m and H is the specimen height, m.

2.2.4 Moisture damage
The ASTM D6927 standard specification [9], entitled “Effect of water on Cohesion of Compacted Bitumen Mixtures” is utilized to prepare the AC specimen and to conduct the test procedure. The IRS is very important in measuring the moisture susceptibility and hence damage of asphalt mixtures. The test was carried out on both control and nano-carbon modified samples. IRS test attempts to determine to what extent the water can reduce the AC cohesion. In the Iraqi highway specification (SCRB), this test is required for surface courses; the minimum IRS value is set to be 70%. Marshall stability values are employed to calculate the retained strength. Hence, Marshall test procedure including sample preparation, compacting and loading details are adopted here. Accordingly, the Index of retained strength is computed mathematically using Eq. (6) below.

\[
\text{IRS} = \frac{S2}{S1} \times 100\%
\]

(6)

Where IRS is the percentage of Index of Retained Strength, S1 is the Marshall stability of the dry condition specimens, kN, and S2 is the Marshall stability of the wet condition specimens, kN.

The first set of specimens are prepared by immersing the specimen for only 30 to 40 minutes (considered as dry conditions); while the second set is prepared under wet conditions by immersion in water for 24 hours. Both immersing conditions should be at 60°C. Marshall test is then applied for both dry and wet conditions specimens – both original and modified.
**Long-term aging**

Long Term Ageing (LTA) simulates age hardening through road use. The LTA method is approved by the Strategy Highway Research Program (SHRP) A-003A which is recommended that the compacted samples are cured in an oven at 85°C for 2 or 5 days to simulate 5 or 10 years’ age hardening in the field, respectively KLIEWER et al. 1995 [10].

All the mixtures are conditioned in an oven at 85°C for 5 days to simulate 10 years age-hardening. Then the specimens were tested in accordance with ASTM D1559, ASTM Standards 2004, to indicate MS values after ageing at 60°C. In addition, Mean Marshall Stability Ratio (MMSR) was determined, which represents the ratio between MS after and before ageing.

3. **Results and discussion**

Several types of testing have been conducted to investigate the effect of adding Nano carbon material to the pure (unmodified) bitumen (60-70 penetration grade). The tests include penetration, softening point and ductility. From the results of the tests on the binder, the optimum Nano carbon percentage was indicated. Two sets of samples were considered; the first one is with 60-70 penetration grade which called control mixtures whereas the second is prepared with modified binder (base bitumen plus optimum Nano carbon) which named as Nano mix within this study.

3.1 **Effect of Nano-carbon on penetration test results**

Penetration tests was carried out for the base bitumen and modified bitumen with different percentages of Nano carbon i.e. 0.5%, 1% and 2% by weight of asphalt. This test was performed also at different temperatures which were 15, 25 and 35°C. Figure 3 shows the penetration results at these temperatures for the base and modified binder. It can be stated that, the penetration has been decreased when Nano carbon added at all percentages. Also, for all types of binder it is increased with different slopes when the temperature increased. The most interesting point is that the slope of the binder with 1% Nano carbon is the lowest one and the penetration at high temperature decreased highly in comparison with the base bitumen while there was no very little decrease at low temperature. From this point it can be observed that the modified binder will resist the permanent deformation more than the control mixtures.

3.2 **Effect of Nano-Carbon on softening point test results**

Table 5 shows the results of the softening point for all types of binder i.e. base and modified binders with different percentages of Nano carbon. It is clearly shown that softening point increased largely when the percentage of Nano carbon increased.

3.2.1 **Effect of Nano-Carbon on Ductility**

According to the Iraqi highways standard, the value of ductility should be higher than 100 cm, all the results have achieved this value with the observation that the addition of nano carbon achieved a slight increase in the ductility as shown in Table 6.

In accordance to the above testing, the results showed that the optimal percent for Nano carbon is 1%; the bitumen used in this study was with graded 60-70. This can attributed to that the stiffness increased significantly at high temperature with little effect at low temperature. Therefore, the produced mixtures i.e. with modified binder will perform adequately at high and low temperatures.

3.3 **Effect of Nano-carbon on Marshall Stability and Flow**

The results of Marshall Stability were shown in Figure 4. According to the Iraqi standard specifications, Marshall stability should be larger or equal to 8 KN for surface courses. So, it is not comply with the specification for the mixtures with 60-70 asphalt. But Marshall stability increased significantly when 1% of nano carbon has been added to the base bitumen. Also, Marshall stability increased by more than 30% for the modified mixtures in comparison with untreated mixtures. Figures 5 and 6 present Marshall flow and Marshall stiffness, respectively.
3.3.1 Effect of Nano-Carbon on the volumetric properties

Figures 7, 8 and 9 show the results of specific gravity, air voids and voids filled with asphalt, respectively. It can be seen that together control and modified mixtures are in consistency with the requirements.

3.4 Effect of Nano-Carbon on Indirect Tensile Strength

ITS results are shown in Figure 10. From the result of testing, ITS increased by almost 24% for modified mixtures in comparison with the control mixtures.

3.5 Effect of Nano-Carbon on Index of Retained Strength (IRS)

The results of index of retained tensile strength are shown in Figure 11. From the results IRS increased from 62 to 72%. According to the Iraqi standard specifications, IRS must exceed 70%, therefore the modified mixtures comply with this specification.

| Chemical Composition of Nibae aggregate | Chemical compound | Results, % |
|----------------------------------------|-------------------|------------|
|                                       | L.O.I.            | 6.55       |
|                                       | SiO₂              | 82.52      |
|                                       | CaO               | 5.37       |
|                                       | MgO               | 0.78       |
|                                       | SO₃               | 2.7        |
|                                       | Fe₂O₃             | 0.69       |
|                                       | Al₂O₃             | 0.48       |
| Mineral composition                    | Quartz            | 80.03      |
|                                       | Calcite           | 10.92      |

Table 1. Physical properties of aggregates

| Property                                              | ASTM Designation | Test results | SCRB specifications |
|-------------------------------------------------------|------------------|--------------|---------------------|
| Coarse aggregate                                      |                  |              |                     |
| Bulk specific gravity                                  | C 127            | 2.64         | ....                |
| Apparent specific gravity                             | C 127            | 2.695        | ....                |
| Percent wear by Los Angeles abrasion, %                | C131             | 22.7         | 30 Max.             |
| Soundness loss by sodium sulphate solution, %          | C88              | 3.4          | 12 Max.             |
| Flat and elongated particles, %                       | C 4791           | 5            | 10 Max.             |
| Degree of crushing, %                                  | D5821            | 96           | 90 Min.             |
| Fine aggregate                                         |                  |              |                     |
| Bulk specific gravity                                  | C127             | 2.67         | ....                |
| Apparent specific gravity                             | C127             | 2.701        | ....                |
| Sand equivalent, %                                     | D2419            | 57           | 45 Min.             |
| Angularity, %                                          | C1252            | 54           | ....                |
| Clay lumps and friable particles, %                    | C142             | 1.85         | 3 Max.              |
Table 3. Properties of 60‒70 asphalt cement

| Property                                      | ASTM Designation | Test Results | Requirements Penetration Graded Asphalt Cement |
|-----------------------------------------------|------------------|--------------|-----------------------------------------------|
| Penetration at 25 °C, 0.10 mm                 | D5               | 62           | 60-70                                         |
| Ductility at 25 °C, cm                        | D113             | 105          | >100                                          |
| Specific gravity at 25 °C                    | D70              | 1.03         | -----                                         |
| Flash point, °C                               | D92              | 273          | >232                                          |
| Solubility in trichloroethylene, % wt         | D2042            | 99.31        | >99                                           |
| Residue from thin film oven test              | D1754            |              |                                               |
| - Retained penetration, % of original         | D5               | 69           | >55                                           |
| - Ductility at 25 °C, cm                      | D113             | 55           | >25                                           |

Table 4. Physical properties of fillers

| Property                                      | Portland Cement  |
|-----------------------------------------------|-------------------|
| Passing sieve No. 200, (%)                    | 94.76             |
| Specific gravity (ASTM C188-95)               | 3.05              |

Table (5) the results of softening point

| Ratio% | Softening point |
|--------|-----------------|
| 0      | 42              |
| 0.5    | 53              |
| 1      | 72              |
| 2      | 83              |

Table (6) the results of ductility test

| Ratio % | Ductility (cm) |
|---------|----------------|
| 0       | >100           |
| 0.5     | >100           |
| 1       | >100           |
| 2       | >100           |

Figure (1): Example of Surface Area of Nanomaterial for a Cubic [6]
Figure 2: Type IIIA surface course hot asphalt gradation

Figure 3: Effect of Nano-Carbon on Penetration
Figure 4: Effect of Nano carbon on Marshall Stability

Figure 5: Effect of Nano carbon on Marshall flow
Figure 6: Effect of Nano carbon on Marshall Stiffness

Figure 7: Effect of Nano carbon on specific gravity
Figure 8: Effect of Nano carbon on air voids

Figure 9: Effect of Nano carbon on voids filled asphalt
Several key conclusions can be reached based on the experimental results. Regarding penetration, softening point and ductility test, the optimal percent for Nano carbon is 1%; because the stiffness increased significantly at high temperature with little effect at low temperature. On the other hand, Marshall stability increased significantly when 1% of nano carbon has been added to the base bitumen. Also, Marshall stability increased by more than 30% for the modified mixtures in comparison with zero-nanocarbon AC mixtures. Also, ITS increased by almost 24% for modified mixtures in comparison with the unmodified asphalt concrete mixtures. Furthermore, IRS increased from 62 to 72%. According to the Iraqi standard specifications, IRS must exceed 70%, therefore the modified mixtures satisfy this specification.
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