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# Effects of asphalt modification by Nanosilica and Nanoclay on asphalt binder and hot mix asphalt properties

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**Abstract**. In the Hot mix asphalt (HMA) is widely used in asphalt paving in most of countries. The goal of this paper is to study the effect of adding nanomaterials on the properties of asphalt binder and HMA. An experimental program has been conducted to study how to evaluate the performance of the modified asphalt binder and the modified HMA. Nanosilica (NS) and Nanoclay (NC) were used as modifiers. NS and NC were added at 3%, 5% and 7% by bitumen weight. Tests were performed to evaluate enhancement occurred in Penetration, softening point (SP), rotational viscosity (RV). Marshall stability tests, loss of stability and indirect tensile strength were performed on modified asphalt mixtures. Results were compared with control mix. Results shows significant enhancement in most of properties of asphalt binder and HMA. Penetration decreased by 24.07% at 3% NC and 20.37% at 5% NS. 3% NC and 5% NS were chosen to be optimum nanomaterial content (ONMC). Modified bitumens by 3% NC and 5% NS were used to produce modified asphalt mixtures. The modified mixtures showed a significant enhancement in stability and indirect tensile strength (IDT), but they, in contrast, had a harmful effect on the loss of stability (LOS).

**Keywords:** Modified Asphalt; Asphalt; Hot Mix Asphalt; Nanomaterial; Nanosilica; Nanoclay; Modified Bitumen; Loss of stability

## 1. Introduction

Nowadays, there is a growing concern in highway community on improving quality of asphalt mixes and asphalt binders to enhance design life of flexible pavements. Bitumen is also known as the most popular asphalt binder. It is widely used in asphalt pavements. It mainly consists of different chemical compounds as carbon, hydrogen, and nitrogen compounds[1]. Virgin bitumen (not modified by additives or others) has multiple properties and characteristics that have major effects on asphalt pavement performance as consistency, adhesion, visco-elasticity and temperature susceptibility[2]. These properties could be called physical and rheological properties[3, 4, 5].

Due to the high traffic loads and weather effects, many problems and distresses occurs in the asphalt pavement, such as cracks and various distresses. So, Asphalt modification with different modifiers has become a trend for many researchers to improve the asphalt binders and asphalt pavement performance [6 , 7].  There are several requirements and conditions when choosing the appropriate modifier type as maintaining asphalt distinctive characteristics, physical and chemical stability and achieving  suitable  viscosity  at  normal  application  temperatures[2].  Through  research  and

development, researchers identified several good asphalt modifiers such as: crumbed rubber, styrene butadiene styrene (SBS), polyethylene, ethylene vinyl acetate, and nanomaterials [6, 8].

As is agreed upon, the term nanomaterial is the material which particles reach the nano size ($10^{-9}$) m [9]. Because of nanomaterials large surface area and small size, they represent better characteristics compared to other materials[10]. From research experience and history, it was observed that most of modified asphalt samples with nanomaterials had lower values of penetration and higher degrees of temperature of softening point than virgin asphalt. From this there is an indication of improvements of the flexibility and stiffness of modified asphalt binders[11].

Khattak et.al [12] studied the behaviour of HMA designed according to Superpave™ method. A comparison between properties of control mix and modified HMA was performed to evaluate the rate of enhancement in asphalt properties. Carbon Nano Fibers (CNF) was used as bitumen modifier. It was found that the complex shear modulus (E*) increased by 24% - 46% and phase angle ( ) decreased by 6% - 25%. That means higher resistance to permanent deformation for CNF modified asphalt mixtures.

Yao Zhang et.al[13] studied the effect of asphalt modification by SBS. Four types of SBS were used in HMA mixtures preparation. An empirical vertically loaded wheel test was created to measure rutting performance of modified HMA mixtures. From the analysis of results, they calculated the compressive dynamic modulus that indicates the rutting development. It was concluded that SBS enhanced the permanent rutting performance of asphalt mixtures.

Shu Wei Goh et.al [14] studied the effect of adding Nanoclay on superpave HMA mixtures. Asphalt binder used is a PG58-28 bitumen. Modified specimens were classified into two groups. The first group was exposed to water in freeze-thaw conditions. The second remained in dry condition. It was observed that the first group gave higher IDT with the increasing in NC content. On the contrast the second group gave opposite results.

A. E. A. Mostafa [7] studied the effect of bitumen modification by adding Nanosilica (NS) and Nano-Carbon (N-C). NS was added at percentages of 1%, 3%, 5%, 7% AND 9% by weight of bitumen. N-C was added at the percentages 0.01%, 0.1%, 0.5% and 1% of weight of bitumen. It was observed that with the increasing in NS and N-C penetration values decreased but values of softening point and viscosity increased.

## 2. Materials

The bitumen used in this research was produced by El-Nasr Petroleum Company. It was brought from one of highway projects in the New Administrative Capital established by Orascom Company for constructions.

Aggregate (course and fine) was completely produced by crushers, supplied from Attaqa crushers. Validation tests were performed to evaluate the quality of aggregate particles.

Nanomaterials used in this experimental work were NS and NC. They were manufactured locally by the laboratories of the Faculty of Science, Beni Suef University.

A large part of the experimental tests were conducted in the roads laboratory of the Faculty of Engineering, Ain Shams University.

## 3. Experimental work

An experimental program was established to test the behaviour of modified bitumen compared with virgin bitumen (bitumen without any additives). The program is mainly based on evaluation of physical and rheological properties of bitumen. Tests performed were Penetration, SP and RV tests.

Conventional tests of bitumen, aggregate and HMA were performed. Aggregate validation tests were conducted to ensure aggregate suitability for use in the production of HMA. Tests were LAA test, flat & elongated particles, plastic and liquid limits.

HMA control specimens and modified were tested as shown below, to evaluate the effects of asphalt modification.

Modifiers used in this research were NS and NC. Both Nanomaterials were characterized described and characterized using the SEM, XRD and TEM techniques.
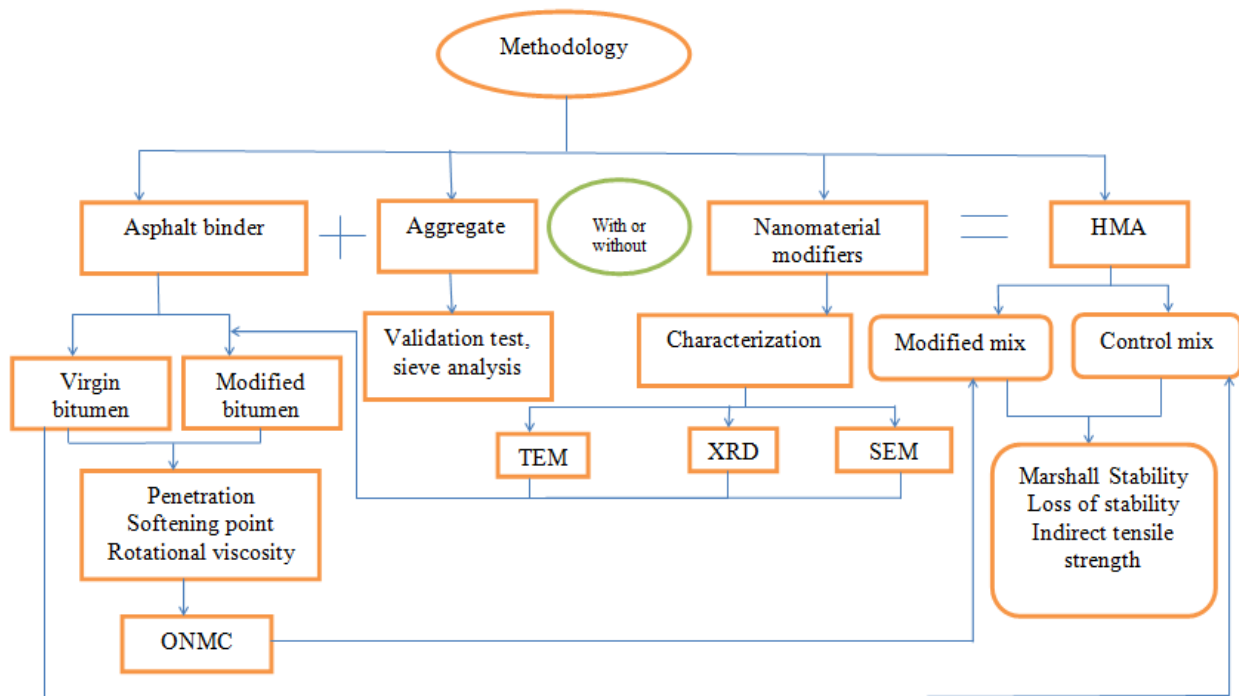
### 3.1 Bitumen

Bitumen is divided into small samples to be ready for testing. Tests were performed according to the procedures of the American Society of Testing Materials (ASTM) and American Association of State Highway and Transportation Officials (AASHTO). Results were compared with limits of Egyptian Code of Practice (ECP, 2008), [2,16,17, 13].

- Penetration test[15]: (ASTM D5/ 5M) Penetration testing measures standard needle penetration values for a solid and semi-solid bitumen surface. The needle is left to penetrate the surface of bitumen -in standard container- freely under its weight. The unit of penetration is 0.1 mm. the penetration value expresses the consistency of bitumen.

- SP test[16]: (ASTM D36/ 36M) This test measures softening point of bitumen by ring and ball apparatus. In this method two rings with standard dimensions are filled with bitumen. Two steel spheres with standard weights are put over the surface of bitumen in the rings. All of these components are put in a water bath. Under the influence of heat, the two spheres fall with the bitumen to touch a small horizontal plate under the rings with a standard distance. The temperature at which contact occurs is considered softening point. Average readings are taken. SP is an indication that expresses the tendency of bitumen to flow at high temperatures, encountered in service.

- RV test: (ASTM D4402/ 4402M) Brookfield viscometer is used to determine RV. A spindle of weight 27 gram is let to rotate at speed 20 rpm under various temperatures. It can measure viscosity at different units as Pa.s or CP.

*3.2 Aggregates*

Tests were performed on coarse and fine aggregate that were completely resulting from crusher millers. Aggregates used are:

- Aggregate A (Agg. A): The aggregates grain size ranges from 12 to 24 mm.
- Aggregate B (Agg. B): The aggregates grain size ranges from 9 to 12 mm.
- Aggregate C (Agg. C): The aggregates grain size ranges from 5 to 9 mm.
- Crushed sand (C.S): The aggregates grain size < 5mm.
- Mineral filler (M.F): Limestone powder.

Aggregates were tested according to ASTM and AASHTO specifications and compared with ECP, 2008 limits. Tests were[17]:

- Los Angeles Abrasion test (LAA)[18]: Test was performed according the procedures of ASTM C131.
- Flat and elongated particles: Test was conducted according the procedures of ASTM D 693.
- Water absorption: Test was conducted according the procedures of ASTM C 127.
- Liquid and plastic limits (LL, PL)[19]: Test was performed according the procedures ASTM D 4318.

*3.3 Modifiers*

In this research two different nanomaterials are used as asphalt modifiers. Nanomaterials were produced by the laboratories of the Faculty of Science, Beni-Suef University. The materials used in this research as modifiers are Nanosilica (NS) and nanoclay (NC). Various methods of scanning were used to show particle size distribution of the two nanomaterials. NS and NC were characterized using various techniques. They were characterized using X-ray diffraction (XRD), and Transmission Electronic Microscope (TEM). Scanning Electron Microscope (SEM).

For NS, The TEM images of the synthetic silica nanoparticles reflected their formation as agglomerated grain in the nano-scale. The grains appear in spherical form with size less than 10 nm (Fig.1). The XRD pattern shows broad peak around 2Theta $22.5^{\circ}$ which characterize silica particle of non-crystallize properties (amorphous). NS mainly consists of $SiO_2$, where $SiO_2$ represents about 99.8% of the chemical composition of NS.
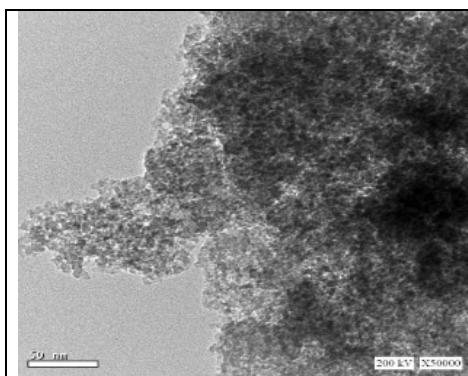


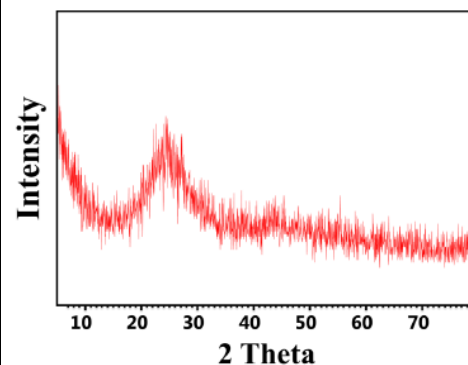| Fig. 1.TEM image of silica nanoparticles | Fig. 2. XRD pattern of silica nanoparticles |

For NC, The chemical analysis of the sample reflected its composition from MgO (40.2%), $SiO_2$ (46.3%) and $Al_2O_3$ (5.5%) in addition to other traces for other elements that related to the impurities of the using natural raw materials. The SEM image reflected the formation of the clay sample as aggregates of nano nudes agglomerated to each other in some samples.

Table 1: Major Oxides of NC

| Major oxides of NC | Percentage % |
| --- | --- |

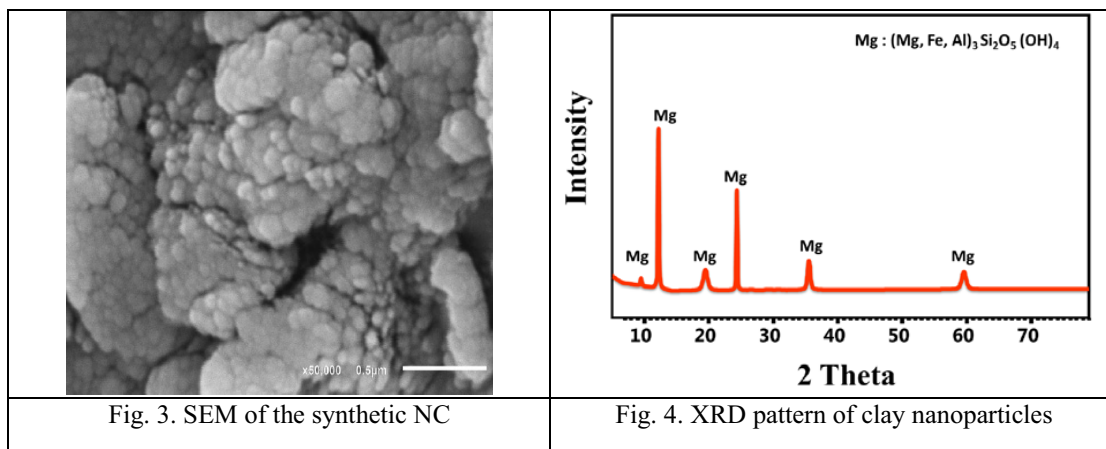| | |
|---|---|
| MgO | 40.2 |
| $Al_2O_3$ | 5.48 |
| $SiO_2$ | 46.3 |
| $TiO_2$ | 0.41 |
| $K_2O$ | 0.34 |
| $Na_2O$ | 0.46 |
| $Cr_2O_3$ | 1.5 |
| $Fe_2O_3$ | 4.2 |
| CaO | 1.13 |

| | |
|---|---|
|  |  |
| Fig. 3. SEM of the synthetic NC | Fig. 4. XRD pattern of clay nanoparticles |

### 3.4 Control mix design

Sieve analysis tests [21 , 20] were conducted Aggregate 1, Aggregate 2, Aggregate3, crushed sand, and limestone powder. The gradations were as showed in table 5. Gradation chosen is gradation 4C. Bitumen was added with percentages 4%, 4.5%, 5%, 5.5% and 6% of the weight of aggregates.

Table 2 job mix formula and ECP, 2008 limits

| sieve | JMF | Specifications | |
|---|---|---|---|
| | | L.L | U.L |
| 1" | 100 | 100 | 100 |
| ¾ " | 98.7 | 80 | 100 |
| 3/8" | 74.6 | 60 | 80 |
| #4 | 52.0 | 48 | 65 |
| #8 | 41.6 | 35 | 50 |
| #30 | 22.6 | 19 | 30 |
| #50 | 16.5 | 13 | 23 |
| #100 | 9.7 | 7 | 15 |
| #200 | 5.2 | 3 | 8 |

### 3.5 modified mixtures

NS and NC was added to virgin bitumen at percentages 3%, 5% and 7%. Modified mixes were prepared by using modified bitumen by NS and NC. They were tested and evaluated by Marshall Stability, loss of stability and IDT

## 4. Results and discussion

*4.1 Bitumen:*

The results of tests performed on bitumen were as shown in table 3. The table shows a numerical comparison between the results of tests performed. The comparison was used to evaluate the effects of bitumen modification.

Table 3 Properties of asphalt cement (Bitumen)

| Test | specifications | Virgin bitumen | 3% NS | 5% NS | 7% NS | 3% NC | 5% NC | 7% NC |
|---|---|---|---|---|---|---|---|---|
| **Penetration(0.1mm)** | ASTM D5 | 54 | 52 | 43 | 55 | 41 | 57 | 56 |
| | | Changes (%) | -3.7 | -20.37 | +1.85 | -24.07 | +5.56 | +3.7 |
| **Softening point(ºC)** | ASTM D36 | 42.5 | 44.25 | 46 | 45.75 | 47.5 | 45 | 40 |
| | | Changes (%) | +4.12 | +8.24 | +7.65 | +11.76 | +5.88 | -5.88 |
| **Viscosity at 135ºC(CP)** | ASTM D4402 | 345.67 | 429 | 447 | 496 | 495.67 | 404 | 433 |
| | | Changes (%) | +24.11 | +29.31 | +43.5 | +43.2 | +16.87 | +25.26 |
| **Viscosity at 165ºC(CP)** | ASTM D4402 | 75 | 91 | 95.67 | 125.2 | 120.67 | 87 | 91.33 |
| | | Changes (%) | +21.33 | +27.56 | +66.67 | +60.89 | +16 | +21.77 |

The results were recorded in the previous table of tests conducted on virgin bitumen and modified bitumen (Table 3). The following charts represent comparisons between the penetration values, the softening point, and the rotational viscosity of the different NS and NC percentages.

Figure 5 represents the relationship between softening point and the amount of modifiers. In case of NS, the chart represents a direct relationship between increasing the amount of NS and increasing the value of the softening point. The relationship remains increased until it reaches 5% NS, after which the softening point value when increasing the ratio decreased to 7%. The softening point has decreased in value very slightly. In the case of NC, the value of SP increased sharply at the amount of 3% NC, and then decreased, decreasing thereafter to be an inverse relationship with the ratio of the modifier.

Figure 6 represents the relationship between penetration in unit of 0.1 mm, and the amount of modifiers. In the case of the NC, the curve decreased dramatically with adding NC by ratio 3%. After that ratio, the penetration value increases to the highest value at 5% then returns to decrease slightly.

In the case of the NS, the value of penetration began to decrease gradually to the value of 3% NS, and then the curve took a greater rate of decline to reach the lowest value at 5% NS, Then the relationship turns to be a direct relationship after the 5% ratio.

| Fig. 5 softening point values versus  Nanomaterial content (NMC) | Fig. 6 Penetration value versus NMC |
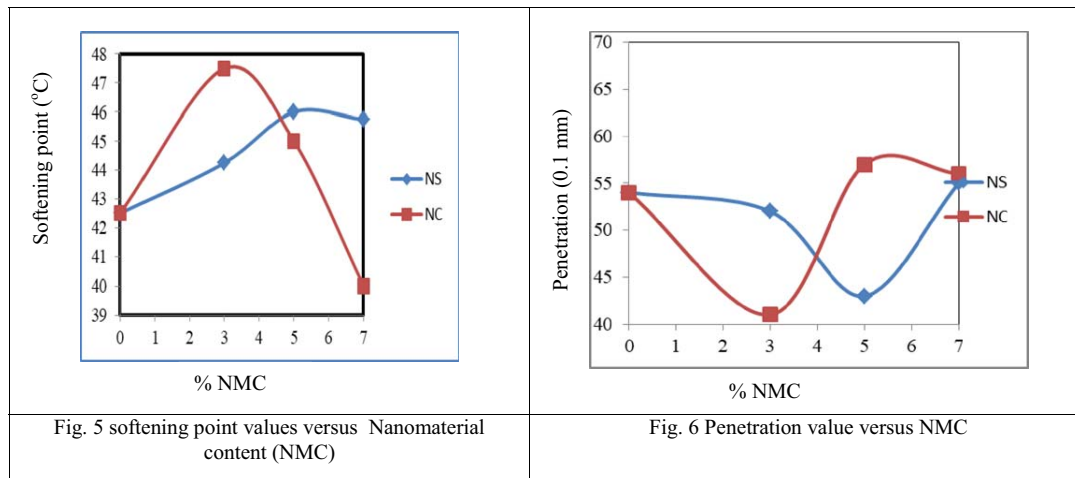
The rotational viscosity of the bitumen was measured at two different temperatures. The temperatures at which viscosity is measured are 135°C and 165°C. At temperature of 135°C, from figure 7, and in the case of NC, it was found that the relationship between the amount of modifier and RV behaved as follows, RV increased in accelerated rate to reach 495.67 CP increasing by 43.2%.  After reaching the peak value at 3% NC, the manner of the RV reacts diversely. RV decreases from the highest point to reach 404 CP at 5% NC. In the case of NS, In the case of the NS, it was found that the relationship was constantly increasing viscosity with increasing quantity of NS. Figure 8 shows the relationship between the quantity of modifier and RV. It was found that the proportion between the quantity of the modifier and the RV takes almost the same behavior as their behavior at 135 ° C with different values of the RV.



| Fig. 7 Rotational viscosity at 135°C | Fig. 8 Rotational viscosity at 165°C versus NMC |
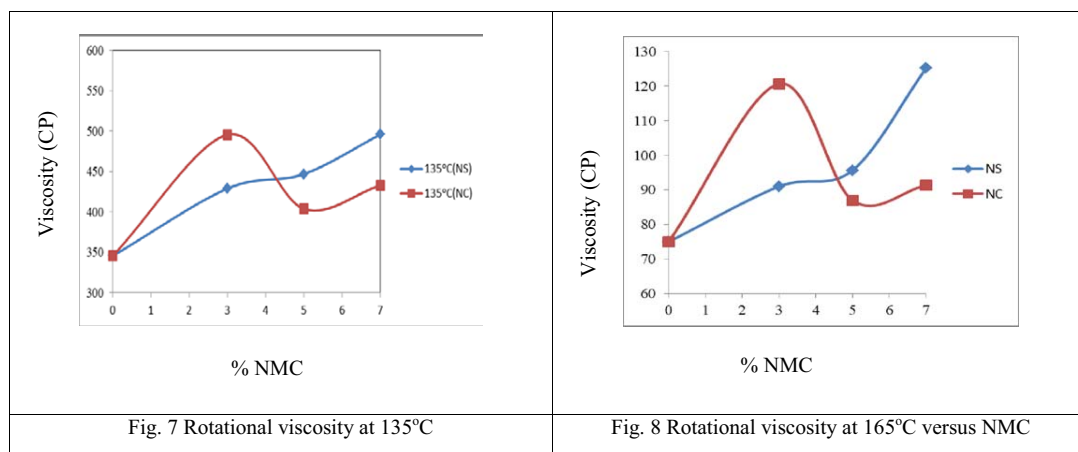
To determine the optimum quantities of modifiers (ONMC), those are the ratios that achieve the best performance of the asphalt bond for different properties. The results of the previous tests for the bitumen reached were analyzed. The amount of 3% of the NC has the highest softening point values, the lowest penetration values and the viscosity achieves its highest values, either at 135 or 165

7

temperatures. The quantity of 5% of the NS has the highest softening point values, the lowest penetration values and the viscosity achieves suitable values of viscosity at 135°C and 165°C

The results indicate noticeable improvements in the properties of the modified bitumen. From analyzing the results and identifying the improvement rates for the different bitumen properties, the 3% NC and 5% NS ratios were found to be the ONMC.
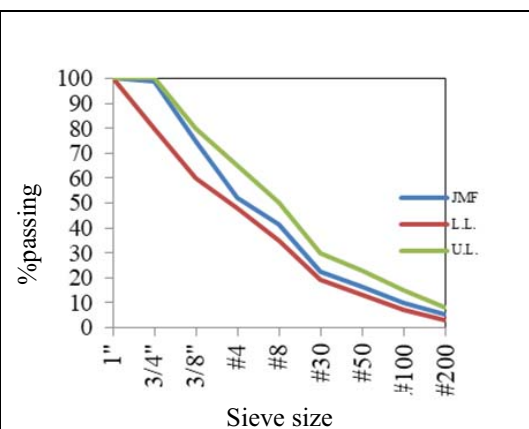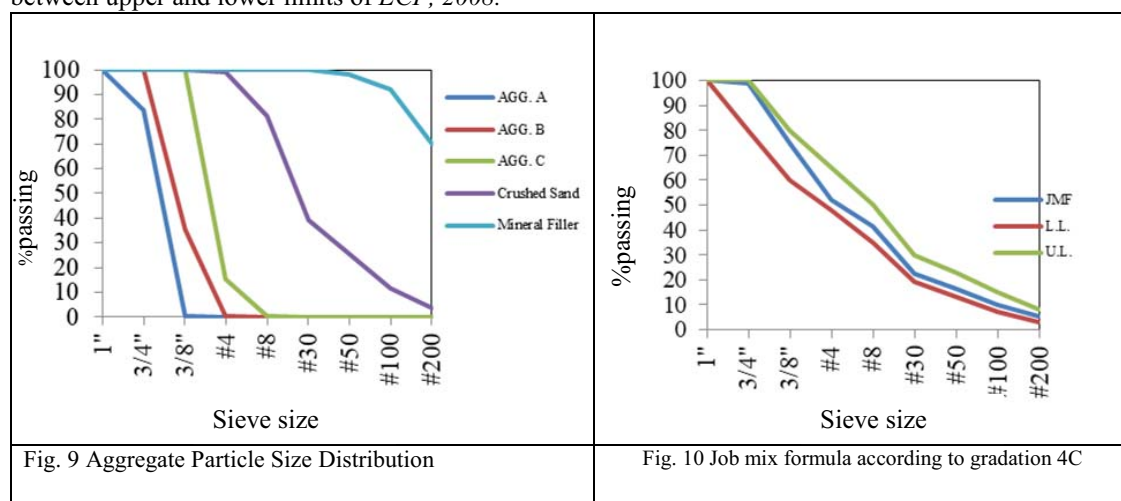
*4.2 Aggregates:*

The aggregate was tested according to the following specifications [20,21]. LAA test is the resistance of coarse aggregate to degradation under the conditions of Los Angeles machine. Flat and elongated particles represent a weak point for the aggregate if it exceeds the permissible limits in the *ECP, 2008*, where inertia is weak in one or more directions, which may expose the particles to breakage and fragmentation during handling, use or operation of the pavement. Table 2 shows results of the tested aggregates.

Table 4 Aggregates properties and specifications

| Test | Test spec. | Agg. A | Agg. B | Agg. C | Crushed Sand |
|------|-----------|--------|--------|--------|--------------|
| LA abrasion | ASTM C131 | 23.8 | 24.1 | 25.4 | -- |
| Flat particles | ASTM D 693 | 3 | 3 | 2 | -- |
| Elongated particles | ASTM D693 | 3 | 4 | 3 | -- |
| Water absorption | ASTM C 127 | 2 | 3 | 2 | 1.1 |
| LL, PL | ASTM D 4318 | -- | -- | NP | NP |

*4.3 Asphalt mixture:*

A sieve analysis was performed for all types of aggregate involved in the production of asphalt mixtures. Asphalt mixture for surface layer is designed according to gradation 4C. A blend of aggregates was collected in varying proportions of each type of aggregate for the design of Job Mix Formula (JMF). Figure 9 represents the gradation of aggregate types Agg. A, Agg. B, Agg. C, Crushed sand and Mineral filler (limestone powder). Figure 10 shows that he JMF gradation is located between upper and lower limits of *ECP, 2008*.



| Fig. 9 Aggregate Particle Size Distribution | Fig. 10 Job mix formula according to gradation 4C |
|---|---|

The control mix was designed by Marshall Mix design method and results were as follows:

Aggregates are graded by sieve analysis test. Gradation 4C was selected to be the gradation of HMA. Bitumen was added with 4, 4.5, 5, 5.5 and 6% of the weight of aggregates. Mixes were prepared according to procedures of Marshall Mix design method.

Figure 11 shows the relationship between stability of specimens prepared and asphalt content. The highest stability was at asphalt content 5%. The limits of air voids in asphalt mix used in surface layer should be not less than 3% and not more than 5%. At 4% air voids, the asphalt content was 5.5% as shown in figure 12



| Fig. 11 Asphalt content versus stability | Fig. 12 Asphalt content versus air voids |
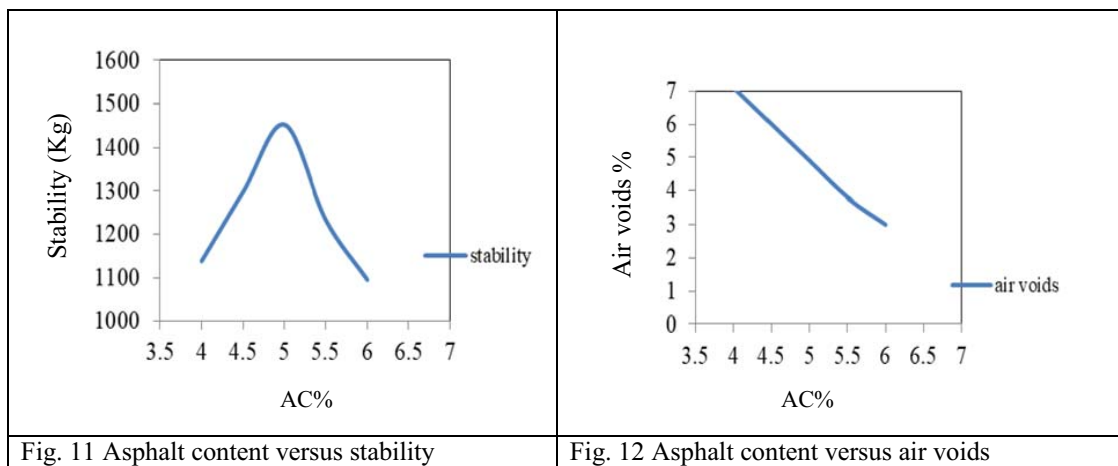
In figure 13 the highest value of unit weight was at 5.1% asphalt content. The optimum asphalt content (OAC) is the average of the previous three values of asphalt content. OAC is 5.2%. The percentage 5.2% achieved the limits of ECP, 2008 in the case of flow as shown in figure 14 and VMA as shown in figure 15.
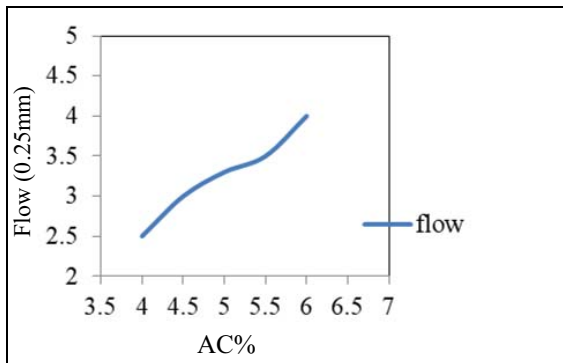


Fig. 13 Asphalt content versus unit weight
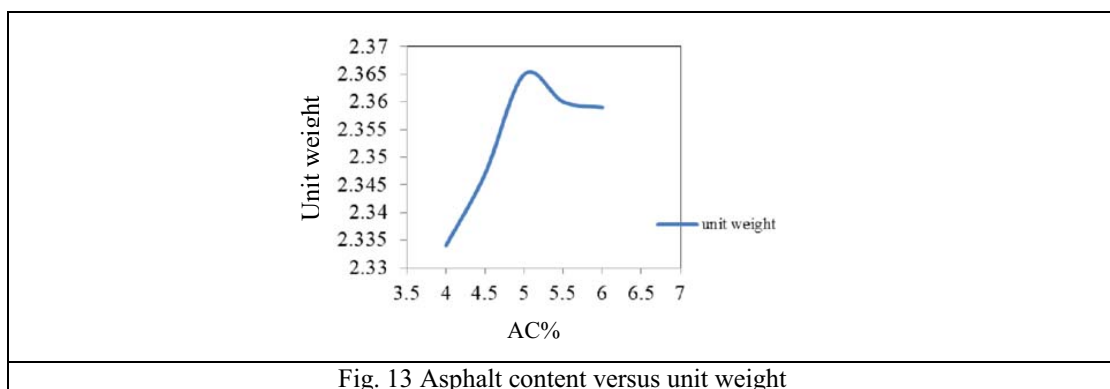
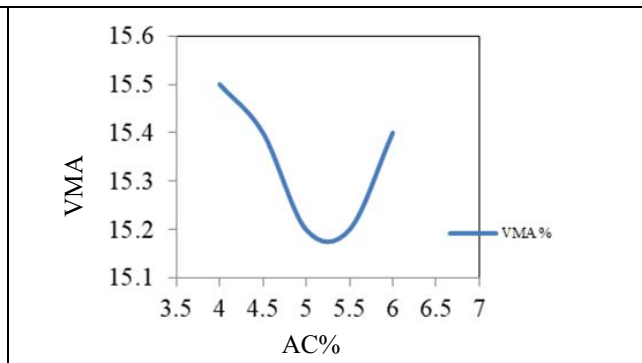| | |
|---|---|
|  |  |
| Fig. 14  Asphalt content versus flow. | Fig. 15 Asphalt content versus voids in mineral aggregates (VMA) |

Modified bitumens with 3% NC and 5% NS were used to produce modified HMA mixtures. Modified specimens were tested by Marshall Stability, flow, and Indirect Tensile Strength (IDT).  The results of the mentioned tested specimens were compared with control mix results.

Table 5 results of testing HMA mixtures

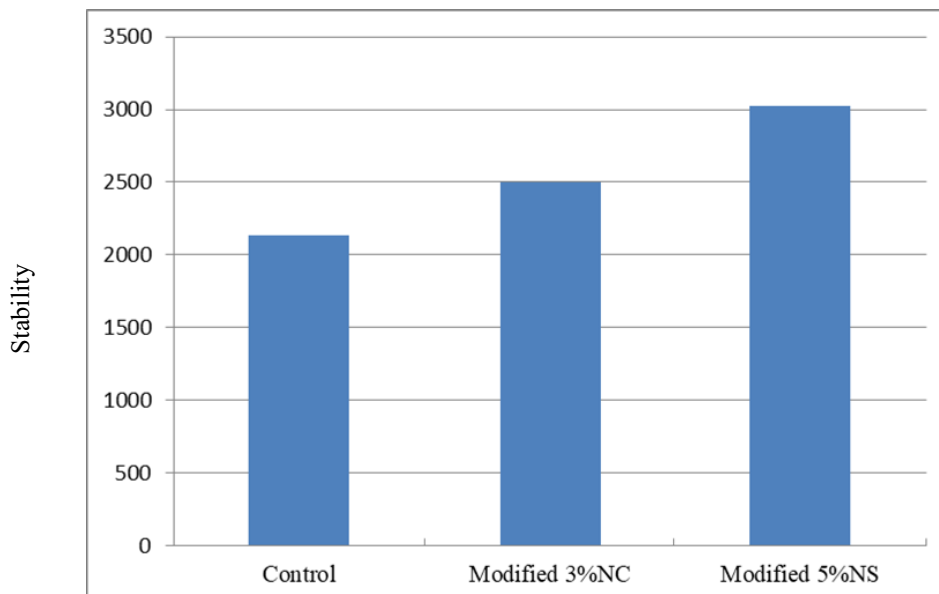| Specimen | Stability | %change in stability | Flow (0.1 in) | Density (gm/cm³) | Marshall Quotient | %change in MQ | Stability after 24 hrs in waterbath | (LOS)% | Change in LOS% | IDT | %change in IDT |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | 2134 | -- | 13.65 | 2.29 | 615.54 | -- | 1879 | 11.95 | -- | 2.36 | -- |
| Modified 3%NC | 2499 | +17.1% | 15.49 | 2.35 | 635.34 | 3.22 | 2046 | 18.13 | +9.06 | 4.17 | +76.77 |
| Modified 5%NS | 3027 | +41.85% | 13.91 | 2.35 | 856.69 | 39.18 | 2109 | 30.33 | +12.42 | 6.63 | +92.28 |



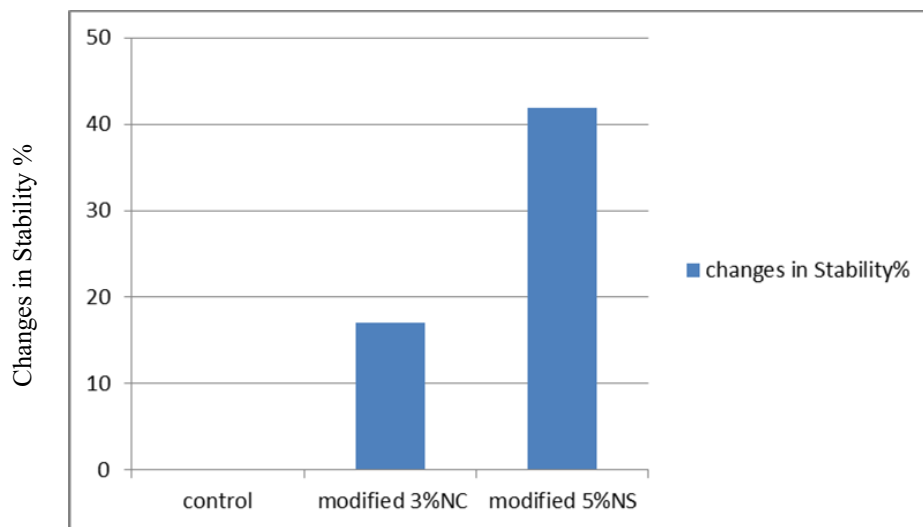Fig. 16 stability values for control mix and modified mixtures
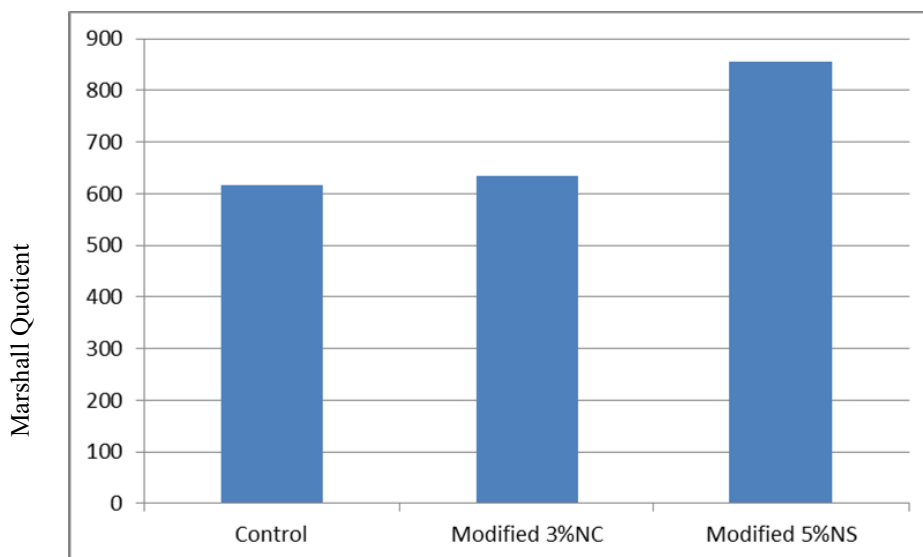
Fig. 17 Changes in Stability %



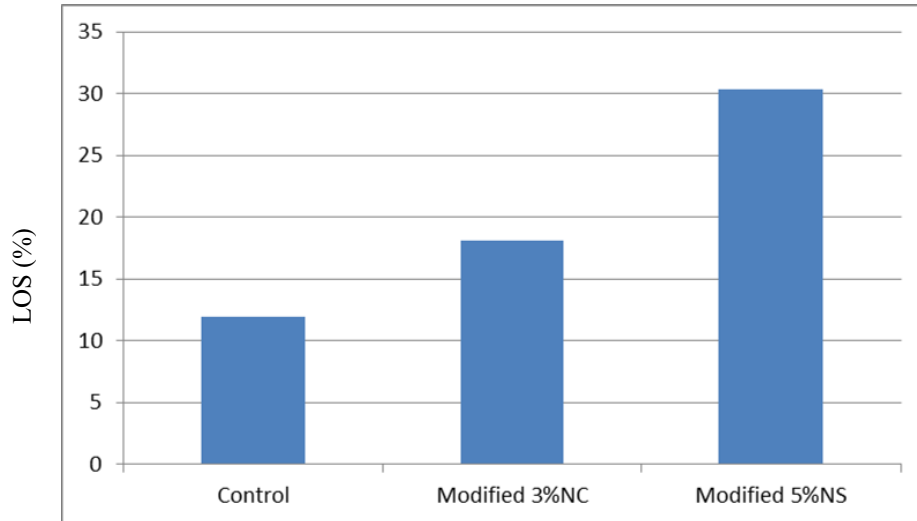Fig. 18 MQ values for control mix and modified mixtures

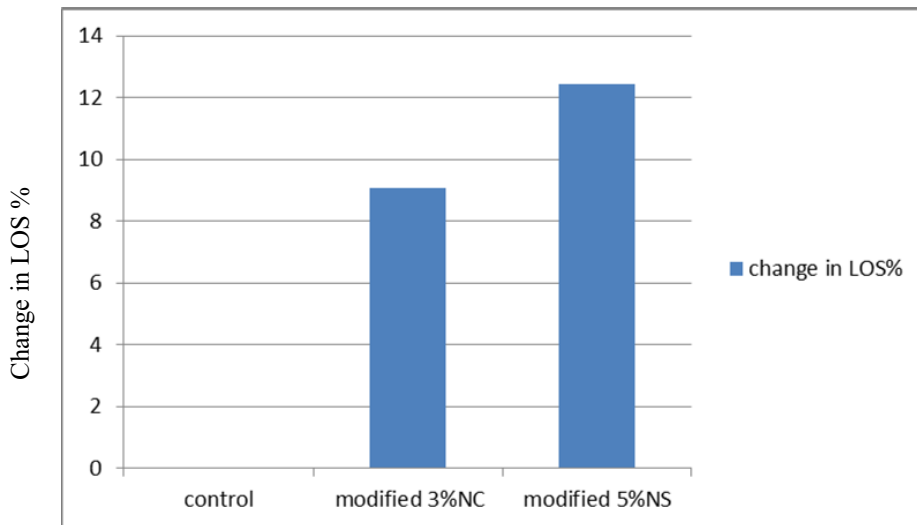Fig. 19 LOS values for control mix and modified mixtures
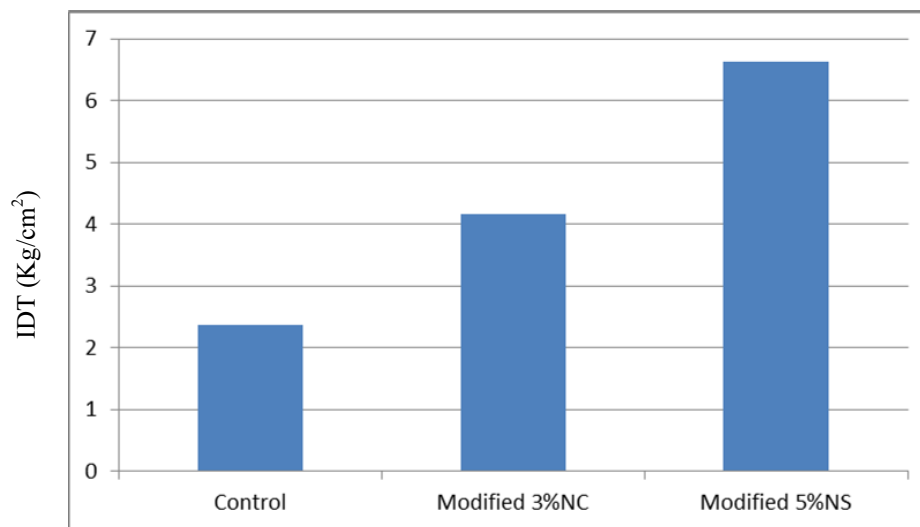


Fig. 20 change in LOS %

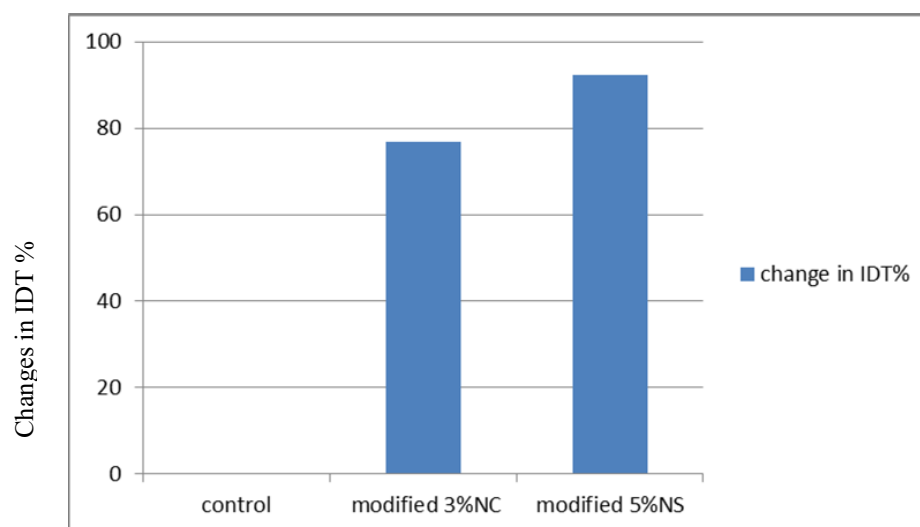Fig. 21  IDT values for control mix and modified mixtures



Fig. 22 Changes in IDT %

As shown in table 5 and figures 16 to 22, there is a clear trend to increase different values of HMA properties. Figure 16 and figure 17 are comparisons between the values of stability of control specimen and modified specimens. The two types of modified specimens show significant improvement in stability. Modified specimens with 3% NC showed an increase in the stability value of the modified samples by 17.1%. Modified specimens with 5% NS showed an increase in the stability value of the modified samples by 41.85%.

Figure 18 shows a comparison between the values of MQ for control and modified specimens. In the case of modified specimens with 3% NC, MQ increased by 3.22%. In the case of 5% NS modified specimens, the MQ dramatically increased to reach an increase by 39.18%.

Figure 19 and figure 20 show also a comparison between loss of stability for control and modified specimens. Control specimen lost 11.95% of stability after one cycle of submerging in a water bath. 3% NC modified specimens lost 18.78% of stability under the same previous conditions. 5% NS

modified specimens lost about 30.33% after the same period under the same previous conditions. The change occurred on modified specimens compared with control specimens that LOS increased by 9.06% in case of modified specimens with 3% NC and LOS increased by 12.42% in the case of modified specimens with 5% NS.

Figure 21 and figure 22 show the effect of adding the two nanomaterials on IDT. The two modifiers caused very large increase in the IDT. In the case of 3% NC, IDT increased very significantly by 76.77%. The same behavior occurred in the case of 5% NS. IDT increased dramatically to reach 92.28%.

## 5. Summary and conclusion

In this study, an experimental investigation was conducted to evaluate the effect of asphalt modification by using two different nanomaterials NC and NS. The study included the behaviours of bitumen and HMA mixtures. The conclusion of the study is as follows:

o   NS and NC have great effects on enhancing the properties of asphalt binders and HMA.
o   Penetration values, which are an effective indication of the consistency of asphalt binder, were strongly influenced by the modification of asphalt with nanomaterials. Lower values of penetration mean more viscous consistency and harder binder. Penetration decreased with increasing NS content in bitumen until reaching 5%, after that penetration value returned to increase.
o   NS increased rotational viscosity of bitumen at different temperatures as $135^{\circ}$C and $165^{\circ}$C significantly.
o   For HMA mixtures, the results of tests performed on control mix and modified mixtures points to noticeable enhancement in stability, MQ and IDT values for the modified mixtures. But in contrast, loss of stability in modified mixtures is higher than of control mixture. That means faster aging in asphalt mixtures.
o   It is recommended to study the performance of bitumen when modified with both nanomaterials. It is also recommended to evaluate the resistance to permanent deformation by wheel tracker in case of modified mixtures with NS and NC. Skid resistance is also recommended to be evaluated to know the effect of NS and NC on surface roughness of asphalt mixtures.

## 6. References

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