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# RUTTING PARAMETERS OF MODIFIED ASPHALT BINDER WITH MICRO AND NANO-SIZE OF METAKAOLIN POWDER

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**Abstract**

This research studies the feasibility of using micro and Nano-size of Metakaolin powder to develop the characteristics of the asphalt binder by three contents (1 %, 3 %, and 5 %) of asphalt weight with high shear mixer for a 60 min.set at 4000 rpm to prepare a homogeneous composite. Rheological properties were studied by the rotational viscosity value, penetration test, softening point temperature, penetration index, and ductility test. Rutting characteristics were determined also by the dynamic shear rheometer test on the control and the modified binder. Overall, the addition of Metakaolin has a helpful influence on the rheological properties of the modified asphalt binder. The greater percentages of improve the viscosity and softening point values at 6 % content of Nano (nMK) and micro (mMK) of Metakaolin were about (77, 40) % and (14, 9) %, respectively. Besides greater percentages of decrease in ductility and penetration values at 6 % content of Nano (nMK) and micro (mMK) of Metakaolin were approximately (18, 11) % and (30,16) %, respectively. Also, the complex shear modulus values increased and phase angle value reduced, attributable to the addition the micro (mMK) and Nano (nMK) of Metakaolin contents. The greater value of the complex shear modulus was detected at 5 % of the micro and Nano size of Metakaolin content. Furthermore, the results exhibited the greater values of complex shear modulus were at PG 76, PG 64 and PG 58 for types of Nano Metakaolin, micro Metakaolin, and control asphalt binder, respectively. It is an indication of improves the rutting performance as well as become appropriate for building the flexible pavements of the highways in hot weather circumstances.

Keywords: Micro and nano Metakaolin, Modification technique, Rheological properties, Rutting performance.

## 1. Introduction

In general, the asphalt binder can be categorized as a viscous-elastic material with temperature and time-dependent characteristics [1]. During the last durations, in Iraq country the pavements materials of roads are exposed to many failure cases, because of the greater loads of traffic and adversely environmental circumstances. The failures of pavements materials of roads are chief problems to sustain the performance of roads pavements. To overcome these problems, have prompted researchers to improve the properties of asphalt binders and mixes by investigated some alternative modifiers and additives such as fillers, fibres, plastics, waste material powders and polymers to be used in recent decades [2-7].

Recently, numerous studies orientate to nanotechnology science using nanomaterials presented promising results in enhancing the modified asphalt binder characteristics. Due to, its fine particle size compared with other modifiers materials which make it easy to be compatible with asphalt binder to produce nano-composite. The positive results that gained from modifying polymer with nanomaterials, which gave the researchers the motive to adopt the nanomaterials which enable them to study the asphalt binder properties at the nano size level especially rutting and fatigue performance [8-14].

One of the greatest commonly used layered silicates is montmorillonite material, which has a 2:1 layered structure with two silica tetrahedron sandwiching an alumina octahedron [15]. Nano modification process of asphalt binder has the potential to create up completely new procedures and types of modified asphalt binders, with extensive implications for the infrastructure of transportation. The ability to target material modification at the nano-size level promises to provide the optimization of material performance needed to significantly progress performance, durability, and sustainability [16].

Metakaolin (MK) is supplementary cementitious materials (SCM) that met the requirements of ASTM C618 standard [17]. Metakaolin ($Al_2O_3$:$2SiO_2$) powder is an unique type of filler material, due to it is not produced from waste and by-products materials of manufacturing processes such as silica fume, and fly ash powders. It is may be mined from the naturally occurring minerals, and it is used for especially industrial in the products of cement material. It is manufactured for a specific purpose under controlled conditions, in order to burnish it is colour strip rigid contaminations and decreased a particle size. Metakaolin is produced by thermal treatment (calcination process) of the kaolin clay at a temperature of approximately (600 to 800 $^{o}$C), and a calcining time of two hours resulted from de-hydroxylation and disorder shows pozzolanic properties, which can be used as a filler material [18, 19]. Many researchers studied the pozzolanic behaviour of MK powder and its effect on cement and concrete properties and shown that MK powder is an effective pozzolanic and results in improved of concrete strength [20, 21].

The Nano metakaolin (nMK) powder varies from the Micro metakaolin (MK) powder mainly in the physical properties such as particle size and specific surface area. Micro metakaolin powders are (99 %) less than (16 μm.) by the specific surface area approximately (12000 $m^2$/kg), and the average dimensions about (300×100×20 nm). Whereas the nMK powders are 88 % less than (2 μm.) by the specific surface area approximately 48000 $m^2$/kg and the average dimensions about 200×100×20 nm [20].

Until nowadays, limited studies have been conducted to evaluate the rheological characteristics of a modified asphalt binder with Nano Metakaolin powder. This research aimed to assess the rheological properties of the modified asphalt binder with micro and Nano metakaolin powder by three contents (1 %, 3 %, and 5 %) of asphalt weight. It is prepared by the high shear mixer fixed at 4000 rpm for a 60 min. to obtain a homogeneous composite material. Rheological and physical characteristics were studied by the rotational viscosity values, penetration values, softening point values, penetration index values, and ductility values. Rutting performance were evaluated also by the dynamic shear rheometer test on the original binder and the modified binder, besides also short- term aged asphalts. The short-term aging is performed by the rolling thin film oven test.

## 2. Characteristics of Materials Used

### 2.1. Asphalt cement used

An original asphalt binder is penetration grade 60/70 was used in this research, supplied by the Dora refinery in Iraq. Physical properties of an original asphalt binder as a show in a Table 1.

**Table 1. Characteristics of the control asphalt binder (60/70).**

| Test | units | Standard ASTM | Results values | Limits of Specificati on, [22] |
|---|---|---|---|---|
| **Penetration** | 0.1 mm | [23] | 66 | 60-70 |
| **Ductility** | cm | [24] | 130 | >100 |
| **Flashpoint & Fire point** | oc | [25] | Flash 303 °C<br>Fire 311 °C | > 232 °C<br>--- |
| **Thin film oven** | 0.1mm<br>cm<br>gm | [26] | Pen. 89.9<br>Duc. 120<br>Mass loss 0.222 | >52<br>>50<br>< 1 |
| **Rotational Viscosity** | c.p | [27] | @ 135 °C 430<br>@ 165 °C 128 | ---- |
| **Softening point** | oc | [28] | 49.5 | ---- |
| **Penetration index (PI)** | --- | | - 0.665 | ---- |

### 2.2. The micro metakaolin (mMK) powder

The Micro Metakaolin (mMK) is an amorphous fine powder, calcined kaolin clay that has been usually used in the porcelain manufacture. The temperature degree and time of thermal treatment (calcination) are 750 ˚C, 2 hours respectively. The Iraqi kaolin clay obtained from the Dwekhle region at west zone of Iraq, which has been used as a modifier. The kaolin clay has been used after the thermal treatment process alters to the metakaolin clay, as presented in Fig. 1. The chemical analysis of mMK powder is exposed in Table 2, while the physical characteristics are exposed in Table 3.

## 2.3. Nano metakaolin (nMK) powder

The nano Metakaolin (nMK) is calcined Nano kaolin clay powder, was imported from the USA (Nanocor Inc,) company as nano clay and was used after the calcination process, and the temperature degree and time of thermal treatment at that temperature adopted in this research were 750 ˚C, two hours correspondingly [29]. The chemical analysis of nMK powder is displayed in Table 2, while the physical characteristics are presented in Table 3.


**Fig. 1. Nano & micro Metakaolin powder.**

**Table 2. Chemical analysis of mMK and nMK powder.**

| Chemical content | mMK | nMK |
|---|---|---|
| **$SiO_2$ (%)** | 95.30 | 74.92 |
| **$Al_2O_3$ (%)** | 3.95 | 1.41 |
| **$Fe_2O_3$ (%)** | 5.21 | 1.17 |
| **CaO (%)** | 8.80 | 0.18 |
| **L.S.F (%)** | 0.032 | 0.001 |
| **MgO (%)** | 4.20 | 0.34 |
| **$SO_3$ (%)** | 0.37 | 0.08 |
| **Loss on Ignition, (%)** | 3.96 | 0.72 |
| **Insoluble residue, (%)** | 86.64 | 57.63 |

**Table 3. Physical properties of mMK and nMK powder.**

| Property | Results | |
|---|---|---|
| **Color** | nMK<br>dark grey | mMK<br>light grey |
| **Specific surface area, $cm^2/g$.** | 48000 | 19000 |
| **Specific gravity,** | 2.69 | 2.62 |
| **Physical form** | powder | powder |

## 2.4. The high shear mixer (S.M.)

The high shear blender is used to combine the asphalt binder with micro and Nano powder. The high shear blender combines between the high rate of revolution (rpm.), flow level, and low pumping [30]. The high rotational speed of blender can be determined based on the used additives type; in this work the rate of revolution was 4000 rpm. The mixing method creates the modified asphalt binder with Nano and micro Metakaolin particles are a more homogeneous mixture and reducing the aggregation of the nano Metakaolin powder.

### 2.5. Preparation method of the modified asphalt samples

The 400 gm of 60/70 asphalt binder were heated to 140 °C, based on the rotational viscosity of used control asphalt. The high shear blender can be set at a constant rate of revolution 4000 rpm for mixing time an hour. Progressively added of micro and nano metakaolin by three contents (1 %, 3 %, and 5 %) of asphalt weight, mixed to the pre-heated asphalt binder in closed steel vessel applied on the electrical oven for an hour. To achieved satisfying distribution of micro and nanoparticles and gained a homogeneous mix of modified asphalt binder by ductility test. Then, tested to assess the rheological characteristics and rutting parameters of the modified asphalt binder.

## 3. Laboratory testing of modified asphalt binder

### 3.1. Physical tests of the control and modified asphalt binder

The rheological and physical properties of the control and modified asphalt binder were assessed by using the Brookfield rotational viscosity, penetration values, softening point temperature, penetration index, and ductility values. These tests were achieved according to the procedures of standard specification (ASTM).

### 3.2. Performance tests of modified asphalt binder

The permanent deformation parameters of the base and modified asphalt binder were assessed by the Dynamic Shear Rheometer (DSR) by exposed to the shear stress loading to determine the resistance of deformations. DSR test used to determine together viscous and elastic behaviours of the control and modified asphalt at intermediate and high-temperature degrees, based on the complex shear modulus($G^*$), which represents the stiffness of asphalt binder and phase angle ($\delta$), which characterizes the elasticity asphalt binder. Unaged and RTFO-aged asphalt binders specimens are examined by 1*25 plate. The DSR test was conducted at the frequency rate at 10 rad/s, the strain amplitude value of 0.1 % and initial temperature degree at 58 of with an increase of 6 temperature degrees on the original specimens. the preparation procedures of the control asphalt and the modified asphalt binder specimens were according to the standard specification [31], as shown in Fig. 2.


**Fig. 2. Testing method of the modified asphalt specimens by the DSR equipment.**

## 4. Results and discussion

Empirical assessments that are used to evaluate the rheological properties of the control and modified asphalt binder at moderate temperatures were penetration values and softening point values, which carried out on the control and modified asphalt binder with several contents of Metakaolin powder. Figures 3 and 4 presented that the consequences of penetration values and softening point values of the control and MK modified asphalt.

It can be detected that the penetration values of the MK modified asphalt binder are reduced with increasing the softening point values by addition the Metakaolin powder. This result may be an enhancement of the stiffness of the modified asphalt binder by dispersion and adsorption of Metakaolin particles in the asphalt binder, indications to decrease the light volatiles material in the maltene phase and alter to resin in the asphalting phase of the modified asphalt [32]. Besides, the stiffness of the Metakaolin powder is higher than the stiffness of the asphalt binder.

Furthermore, the decrease in the penetration values and higher increment in the softening point values noticed within nMK modified asphalt binder, indicates that the huge surface area of nMK particles addition creates the nMK modified asphalt to be stiffer than the other types of the asphalt binders. The 5 % of nMK powder showed a higher reduction in the penetration value and higher increment in softening point for the modified binder compared with the other types of modified asphalt binder; as there is insignificant alteration between 1 % and 3 % of MK content, as approved with El-Shafie et al. [33].

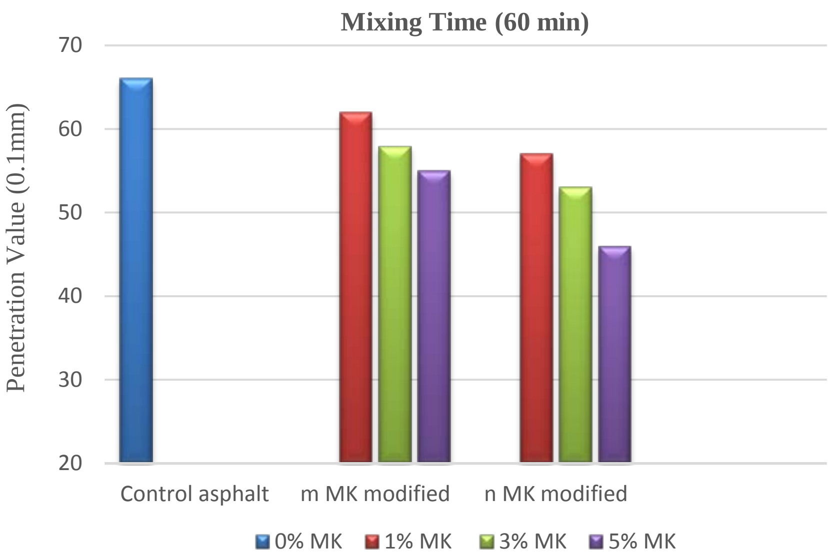


**Fig. 3. Penetration grades of the control asphalt and modified asphalt with micro and nano MK powder.**

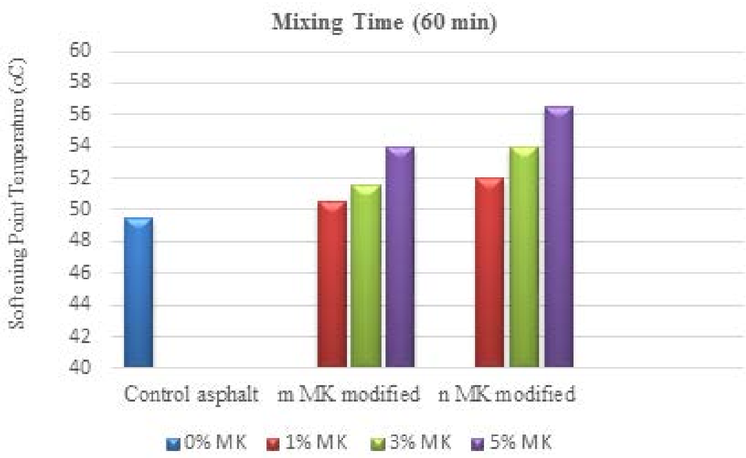


**Fig. 4. Softening point values of the control asphalt and modified asphalt with micro and nano MK powder.**

Figure 5 presented that the penetration index (PI) values contrasted with the micro and Nano Metakaolin percent. It can be noted that the PI values of the MK modified asphalt are increased by the addition of the MK percent. Though, the adding of MK powder contents have been a positive influence on the susceptibility of temperature. The 5 % of nMK modified asphalt showed the greater of the PI value compared with the other types of the asphalt binders. Whereas the PI values of the MK modified asphalt binder were within the range of PI values as (-2 to +2), which can be used in the construction of the highway flexible pavements [34, 35].

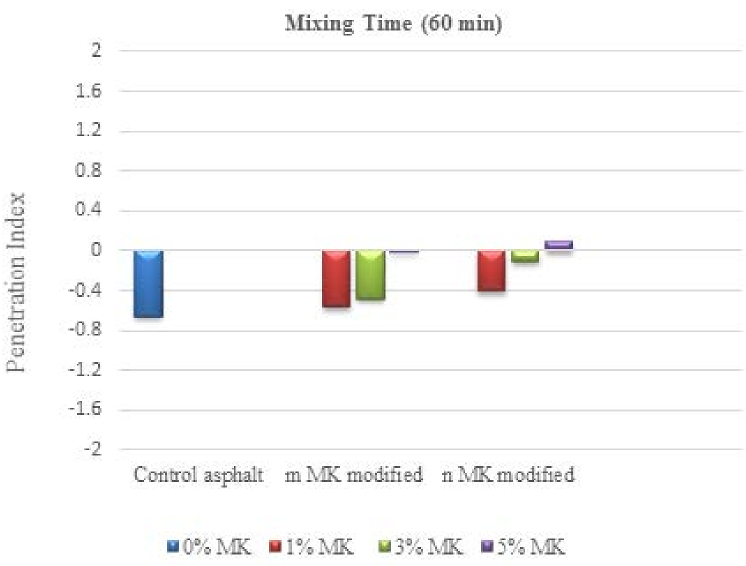


**Fig. 5. Penetration index values of the control asphalt and modified asphalt with micro and nano MK powder.**

The ductility test is used to show cohesion of the asphalt material. Further, the test is reflected the elasticity and homogeneity of the MK modified asphalt, also a result, indicates the resistance of low temperature cracks. Figure 6 displayed that the ductility values contrary to MK contents. It can be detected that the ductility value reduced by the addition of the MK powder content. These results might be endorsed to the absorption of the light volatiles in the maltene phase and change to resin in the asphaltene phase of the modified asphalt. Furthermore, lower value in the ductility was observed with 5 % of nMK content, due to the that the large surface area of nMK particles creates the nMK modified asphalt binder to be stiffer than the other types of the asphalt binders. This performance may be the result of chemical reaction and adjustment in chemical structure, as mentioned by Ghile [36].

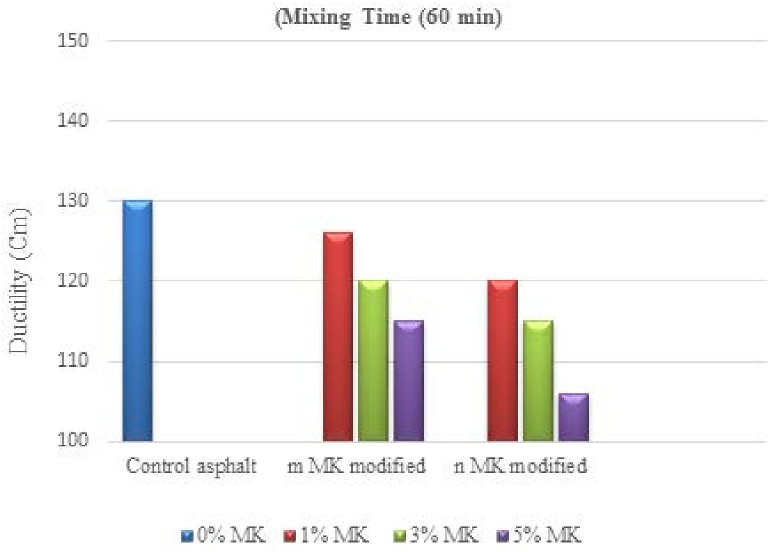


**Fig. 6. Ductility values of the control asphalt and modified asphalt binders with micro and nano MK powder.**

Rotational viscosity test that measures the suitable temperatures of mixing and compaction processes for the asphalt binder. Figures 7 and 8 presented that the relation of rotational viscosity values against the MK powder contents for the control and modified asphalt binder at temperatures 135 to 165 ºC. It can be detected that the viscosity value increasing because of the addition of the MK content. This enhancement of the stiffness of the MK modified asphalt might be caused by the adsorption of the MK powders into the asphalt binder. Consequently, the loss of the light oily material in the maltene phase and alter to resin in the asphaltene phase of the modified asphalt binder. Besides, the stiffness of the Metakaolin particles is higher than the stiffness of the asphalt. Furthermore, the higher viscosity value was observed with 5 % of nMK content, due to the great surface area of nMK particles creates the exfoliated structure which resulted from a stiffer thicker film of the nMK modified asphalt binder. Another reason is it impacts be supposed that if the nMK particles are more commonly discrete they ought to add extra strength to increase the resistance to the shear flow of molecular [37].

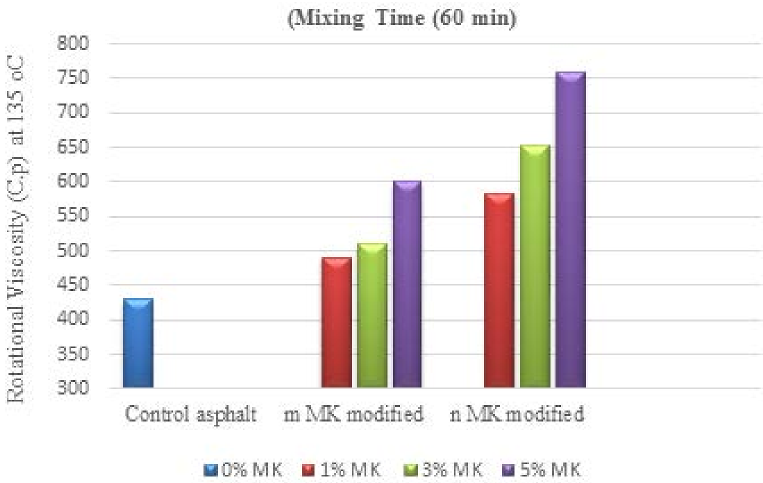


**Fig. 7. Viscosity values of the control asphalt and modified asphalt binders with micro and nano MK powder.**

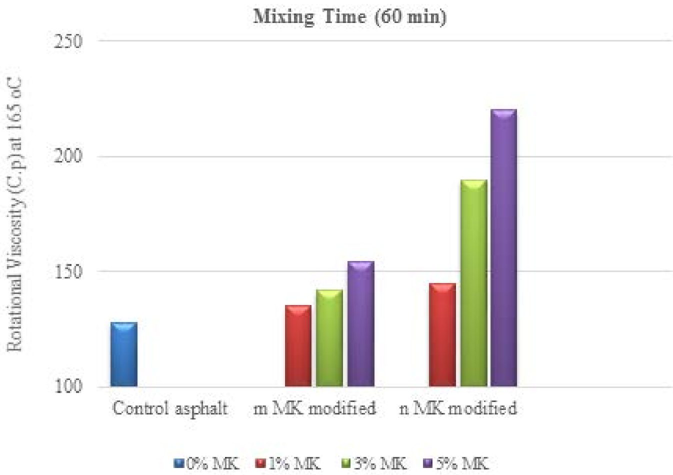


**Fig. 8. Viscosity values of the control asphalt and modified asphalt binders with micro and nano MK powder.**

Figures 9-12 showed that the relation of the complex shear modulus ($G^*/\sin\delta$) values against the MK powder contents for the control asphalt and modified asphalt. It can be noticed that the ($G^*/\sin\delta$) value increased and phase angle ($\delta$) reduced, attributable to the adding of the MK powder contents. It discovered that the higher value of complex shear modulus ($G^*/\sin\delta$) was at 5 % of micro and Nano size of MK content.

The major reason for these variations in the rheological properties of the MK modified asphalt because of the MK particle absorbers of the light volatiles of the asphalt. Which effects the enhancement of the stiffness of the MK modified asphalt binder. Furthermore, it may be caused in the formation of the exfoliated structure of

MK powder and indicates to greater dissipated energy per loading cycle which implies the adding of MK particles, as mentioned in [11, 38]. On the other, the high degrees of temperature have important influences on the asphalt properties.

The variance in the stiffness of materials changes with different temperatures susceptibility. In general, the control asphalt binder has a high susceptibility of temperature. Furthermore, the greater values of ($G^*$/sin $\delta$) of the MK modified asphalt binder were at (PG 76), (PG 70) and (PG 64) for types of nMK, mMK, and control asphalt, respectively. These results attributed that the reactivity and large surface area of Nano size of MK powder adding creates the stiffer and thicker film of nMK modifier asphalt compared with the other types of the asphalt. Furthermore, the greater enhancement in the nMK modified asphalt displayed superior rutting resistance compared with mMK modified asphalt.

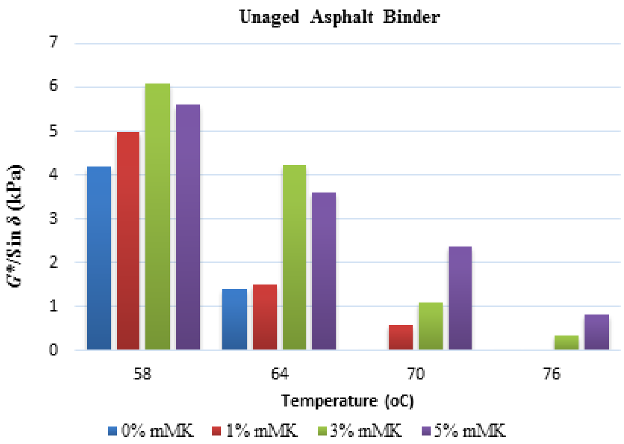


**Fig. 9. The $G^*$/sin $\delta$ values of the control asphalt and modified asphalt binders with micro MK powder.**

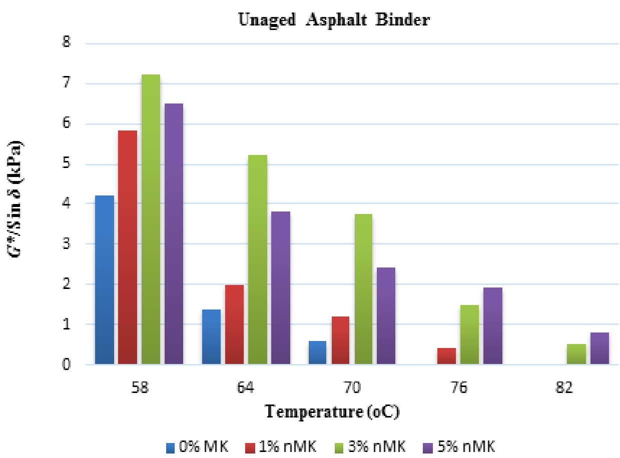


**Fig. 10. The $G^*$/sin $\delta$ values of the control asphalt and modified asphalt binders with nano MK powder.**

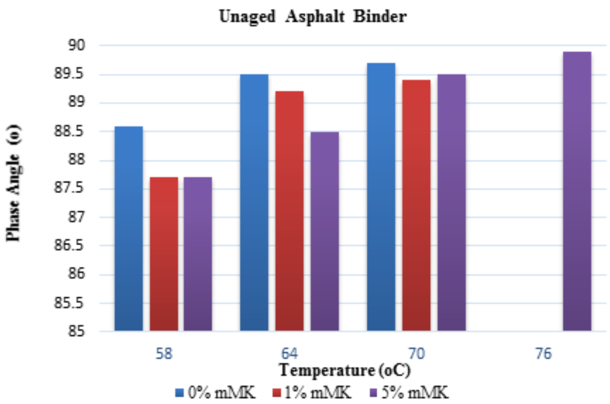


**Fig. 11. The phase angle values of the control asphalt and modified asphalt binders with micro MK powder.**

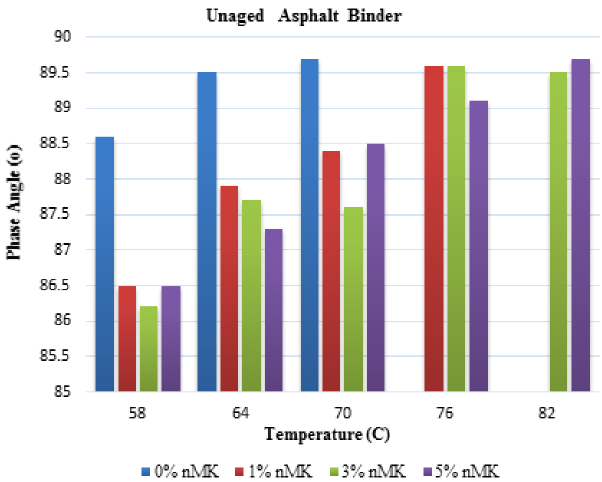


**Fig. 12. The phase angle values of the control asphalt and modified asphalt binders with nano MK powder.**

The aging process is the principal influence for the deterioration of asphalt binder. It occurs during the mixing procedures of the hot asphalt mixture. From results in Figs. 13-16, detected the nMK modified asphalt exposed that the higher ($G^*/\sin\ \delta$) values and lower phase angle. It is well known that adding nMK powder has the probable to progress the complex shear modulus ($G^*/\sin\ \delta$) and decrease the phase angle value. Consequently, it would be show an improvement of viscous-elastic behaviours of the modified asphalt binder after the aging test. Additionally, the greater values of ($G^*/\sin\ \delta$) were at PG 76, PG 64 and PG 58 for MK modified asphalt, and control asphalt binder, respectively. The 5 % of nMK modified asphalt exhibited greater dissipated energy per loading cycle which implied the addition of nMK develop the resistance of rutting, as agreed with Ezzat et al. [38, 39].

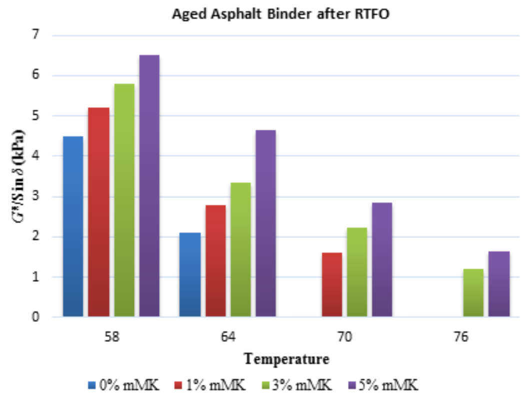


**Fig. 13. The $G^*/\sin\delta$ values of the control asphalt and mMK modified binders after RTFOT.**

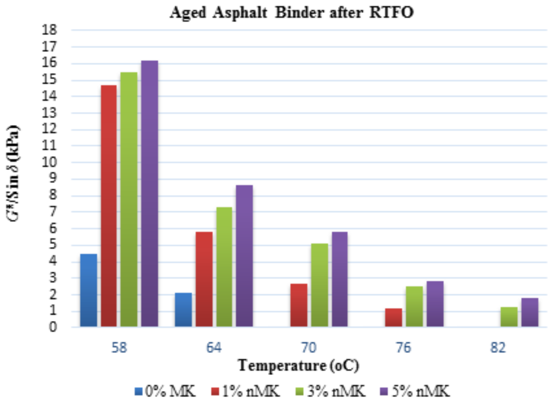


**Fig. 14. The $G^*/\sin\delta$ values of the control asphalt and nMK modified binders after RTFOT.**

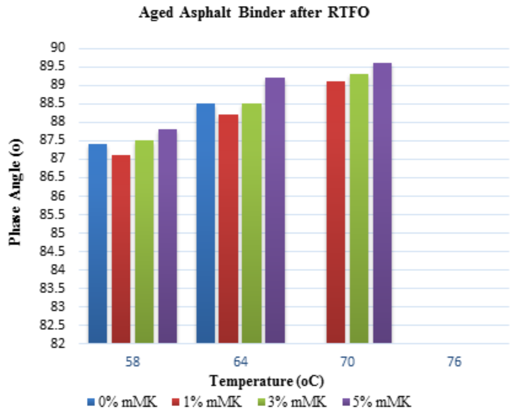


**Fig. 15. The phase angle values of the control asphalt and mMK modified binders after RTFOT.**

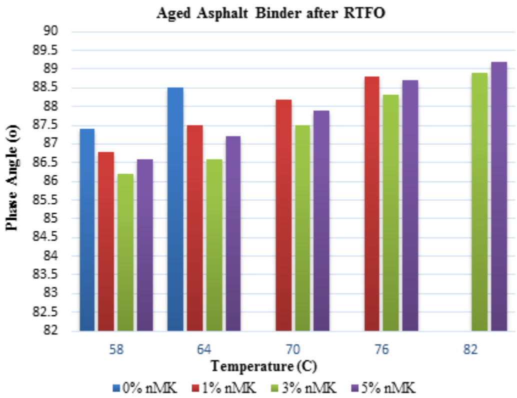


**Fig. 16. The phase angle values of the control asphalt and nMK modified binders after RTFOT.**

## 6. Conclusions

Based on the results of laboratory work, following conclusions have been drawn:

- MK modified asphalt binder penetration value reduced while increasing the softening point temperature value by the addition of the MK powder content. The greater enhancement in the penetration value and softening point temperature value was observed with 5 % of nMK powder content for all types of the modified asphalt compared with the control asphalt.
- The susceptibility of temperature PI values for the MK modified asphalt binder is improved by addition to the MK particles content. Though, the modified asphalt binder with 5 % of nMK powder content exhibited the higher value of PI compared with the other types of the asphalt binder. Consequently, it has a positive effect on the temperature susceptibility, it is an indication of develops the rutting resistance.
- The use of the micro and Nano size of MK powder significantly improved the rotational viscosity when compared with the control asphalt binder.
- Ductility value of the MK modified asphalt reduction with addition the mMK and nMK content indicated that its stiffness progresses with adding the MK powder content.
- The high shear mixer fixed at 4000 rpm for a 60 min. of mixing time, at a mixing temperature of 140°C was satisfactory to attain a good diffusion of micro and Nano MK powder into the asphalt binder and gained a homogeneous mix. Besides, it can be detected that the long mixing time may be a result of a slight enhancement of the dispersion of Nano MK particles, but it is greater energy consumption and more costly.
- The complex shear modulus ($G^*/\sin \delta$) increased and phase angle ($\delta$) reduced, attributable to the adding of the micro and Nano of MK powder contents. The higher value of complex shear modulus ($G^*/\sin \delta$) was detected at 5% of micro and Nano size of MK content.
- Based on the RTFOT results of the control asphalt and MK modified asphalt noted that the greater values of complex shear modulus ($G^*/\sin \delta$) were at PG 76, PG 64 and PG 58 for types of nMK, mMK, and control asphalt binder, respectively. It is

an indication of develops the resistance of permanent deformations for nMK modified asphalt binder compared with control and mMK modified asphalt binder.

- From the results of the rheological properties and the Dynamic Shear Rheometer test of the modified asphalt binder, 5 % of MK content was reasonable to develop the rheological characteristics, because of the MK particle absorbers of the light volatiles of the asphalt. Which effects the enhancement of the stiffness of the MK modified asphalt binder. Furthermore, it may be caused in the formation of the exfoliated structure of MK powder and indicates to greater dissipated energy per loading cycle which implies the adding of MK particles. Besides the rutting performance as well as become appropriate for building the flexible pavements of the highways in hot weather circumstances.

**Abbreviations**

| | |
|---|---|
| DSR | Dynamic Shear Rheometer |
| mMK | Micro Metakaolin |
| nMK | Nano Metakaolin |
| PI | Penetration Index |
| S.H | Shear Mixer |
| SCM | Supplementary Cementitious Materials |

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