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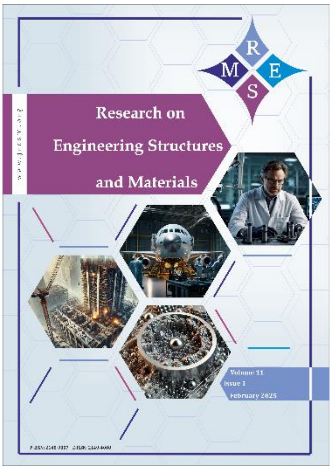

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Research Article

Enriching asphalt binders' rheology by joining nano-copper oxide as a modifier

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Abstract

The durability of road surfaces faces persistent challenges due to increasing traffic loads and the vulnerability of traditional asphalt binders to aging, temperature fluctuations, and fatigue cracking. To address these issues, researchers have explored the integration of nanomaterials into asphalt binders to enhance their durability and rheological performance. Bitumen 60/70 Penetration Grade, commonly produced in Iraq, is rarely used locally due to its sensitivity to temperature variations. In this study, Nano-Copper Oxide was incorporated as a modifier into the asphalt binder at concentrations of 2%, 4%, 6%, and 8% by weight. The modified binders underwent conventional tests such as Penetration, Softening Point, and Ductility, alongside rheological tests including the Dynamic Shear Rheometer and Bending Beam Rheometer. The results revealed significant improvements in penetration reaching about 25% and 37% at 6% content for both unaged and aged samples, respectively. While the lowest temperature susceptibility found at 4% content. Additionally, the viscoelastic behavior was improved, as indicated by higher G* values and lower δ values compared to the original binder with enhanced stiffness and reduced aging effects as confirmed by the Rolling Thin Film Oven Test. Furthermore, the nano-modified binders met SuperPave™ specifications and exhibited resistance to low-temperature cracking only at -6 °C and failed at -12 °C. These findings suggest the potential for extended pavement service life and increased durability through the use of nano-modified asphalt binders.

1. Introduction

The success of any modifier for asphalt binder can be assessed built on two main criteria: (a) its behavior while performing, which includes features such as resistance to rutting, moisture damage, fatigue, and low-temperature thermal cracking once applied on sites; and (b) its practical requirements before site usage, including modifier dispersal within the asphalt binder medium, stability at high storage temperatures, resistance to aging throughout the construction phase, and workability [1-4]. Although currently applied modifiers, such as crumb rubber, polymers, and additives have demonstrated significant improvements in in-service performance, they still face challenges related to phase separation at high temperatures and poor compatibility before field application. Additionally, unmodified asphalt binders tend to become stiffer before application due to a higher degree of oxidation, presenting another functional challenge [5, 6].

Researchers are therefore working to address these challenges in modified binders while also seeking to improve the performance of unmodified asphalt binders through the use of innovative materials. Recently, nanotechnology has raised as a potential field, using elements such as nano-clay, nano-silica (SiO_2), nano-alumina (Al_2O_3), nano-copper oxide (CuO), carbon nanotubes (CNT),

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nano-titanium oxide (TiO_2) and other nanomaterials to improve the characteristics of bitumen. This technique provides unique options for sustainable and durable asphalt infrastructures, with several nano-sized elements demonstrating potential in enhancing the physicochemical characteristics of bitumen [7-9]. This advanced technology is regarded as the most recent innovation for creating safe and sustainable pavement infrastructure. Despite many years of research and exploration by scientists, material manufacturers, and engineers, the application of nanotechnology has remained restricted. Thus, there has been renewed effort and investigation into developing nanomaterials for pavement applications, focusing on enhancing the nanoscale rheological properties and the durability of the mixtures [10-15].

Numerous studies have demonstrated the potential of nanomaterials in enhancing bitumen properties. Yao et al. found that nano silica significantly improved rheological properties, stiffness, and resistance to rutting and aging, with optimal results at 4–6% concentrations [16]. Cheraghian et al. highlighted the benefits of fumed silica nanoparticles in enhancing binder stability and aging resistance [17], while Masri et al. identified 2% nano silica as optimal for maintaining viscoelastic behavior and minimizing strain under stress. Similarly [18], Alhamali et al. and Zghair et al. observed improved performance at higher nano silica concentrations, though challenges like reduced ductility and storage stability were noted [19, 20]. Also, recent studies by Ashish and Singh and Amini et al. demonstrated the use of nano-CuO with multi-wall carbon nanotubes to improve temperature susceptibility, viscosity, and resistance to aging and rutting, showcasing the potential for hybrid nanocomposites [12, 21]. Also, nanomaterials' role in improving moisture damage resistance, aging resistance, and high-temperature performance has been emphasized by reviewer [22, 23]. Other study by Yang and Tighe, explored the effectiveness of various nanoparticles like nano clay, nanotubes, and nano-titanium dioxide, with improvements in crack resistance, fatigue resistance, and environmental sustainability [24].

Despite extensive research on enhancing asphalt binders, limited studies have focused exclusively on the use of nanomaterials, such as nano-CuO, either independently or without combining them with other compounds. This gap is critical, as it pertains to understanding the unique impact of nanoparticles on the performance of asphalt binders, offering the potential for more cost-effective and efficient alternatives. The primary objective of this study is to address the shortcomings of traditional asphalt binders, particularly in terms of aging resistance, temperature susceptibility, and fatigue cracking, which collectively contribute to the premature deterioration of asphalt pavements. This study aims to enhance the durability and performance of asphalt binders through the direct incorporation of nano-CuO, without the inclusion of other additives such as polymers, elastomers, rubbers, or chemical additives.

2. Materials and Methods

2.1. Materials

2.1.1. Asphalt Binder

In this study, a 60/70 penetration grade asphalt binder, supplied by the Directorate of Roads and Bridges – Duhok, was used in this study. Conventional tests according to American Society for Testing and Materials (ASTM) standards were conducted to analyze the rheological properties of the original asphalt binder, as detailed in Table 1. The results of the tests indicate that the asphalt binder generally meets the requirements specified by ASTM D946M-15, which is related to the standard specification for penetration-graded asphalt binder for use in pavement construction.

Table 1. Asphalt binder properties per ASTM D946M-15

Tests (Unit)	Requirements	Results
Penetration at 25°C (0.1mm)	60-70	68.8
Ductility at 25°C (cm)	Min. 100	106
Softening Point (°C)	Min. 46	47
Ductility after RTFOT (cm)	Min. 50	93
Retained Penetration after RTFOT (%)	50 +	78
Rotational Viscosity at 135°C, 165°C (cp)	-	365, 112.5

The penetration value of 68.8 (0.1 mm) indicates adequate stiffness, while the ductility of 106 cm reflects excellent resistance to cracking under tensile stress. The softening point of 47°C exceeds the required minimum, ensuring thermal stability during temperature fluctuations. The retained penetration value of 78% and ductility of 93 cm after rolling thin film oven test (RTFOT) confirm the binder's strong resistance to short-term aging, maintaining flexibility and durability. Additionally, rotational viscosity values at 135°C (365 cP) and 165°C (112.5 cP) ensure good workability during mixing and paving

2.1.2. Nano-Material

Nano-Copper Oxide, appears as a black to brown powder as shown in Fig. 1, was used to modify the asphalt binder, which were ordered from Hebei Suoyi New Material Technology Co., Ltd. – China. The nanomaterial properties are presented in Table 2.

Table 2. Nanomaterial’s properties

Properties	Nano-Copper
Chemical Formula	CuO
Appearance	Black to Brown powder
Purity	99%
Particle size	45nm
Particle Morphology	Spherical
Specific surface area	100-200 m^2/g
pH	5.5-6.5
Molecular weight	79.55g/mol
Melting point	1201°C

Fig. 1. Nano-copper oxide powder

2.2. Asphalt Binder Sample Preparation

2.2.1. Sample

An oven is used to heat the original asphalt binder to achieve liquidity, slightly above its softening point, to allow easy pouring into the mixing pan. Precise amounts of nano-copper oxide, comprising 2%, 4%, 6%, and 8% by weight of the asphalt binder, are then carefully measured and gradually added to the liquefied binder. Then a high shear mixer was used for mixing. Initially, the speed of the mixer was 4500 rpm for 5 minutes to ensure uniform dispersion of the nanomaterials, avoid clumping, and manage floating particles and bubbles [20]. After this, the mixer's speed is increased to 15000 rpm for 15 minutes, with the mixing temperature kept at 157 ± 1°C, which is determined in accordance with ASTM D2493 guidelines. The mixing criteria were selected based on findings from previous studies, which indicate a relationship between mixing time and speed. Specifically, when the mixing speed is high, the required mixing time decreases, and vice versa, to ensure optimal dispersion of materials [25-29]. Once the desired dispersion is achieved, the modified bitumen is cooled and stored in sealed containers for subsequent testing and application. Fig. 2 illustrates the procedure for sample preparation.

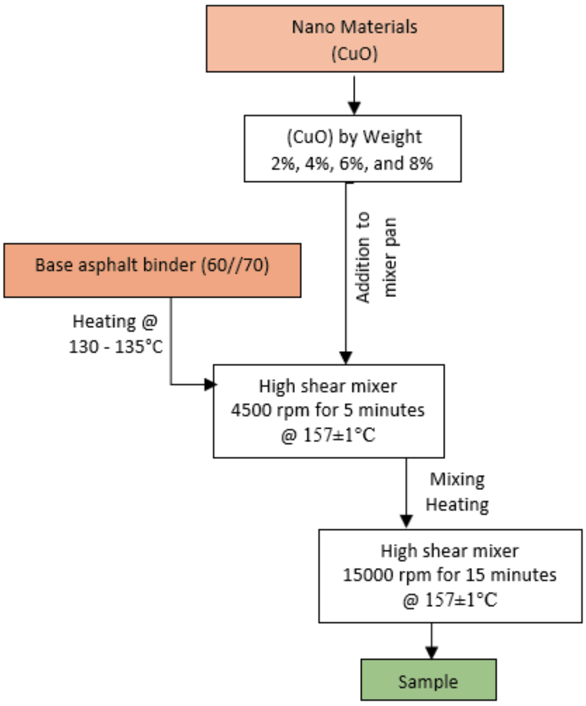


Fig. 1. Asphalt binder sample preparation procedure

2.2.2. Aging Process

Bitumen gradually ages due to exposure to various environmental and operational factors, resulting in oxidation and volatilization [30, 31]. Volatilization and oxidation are the primary processes responsible for the formation of carbonyl and sulfoxide groups, which contribute to the aging of asphalt binders. These processes increase the viscosity of the binder, resulting in stiffer mixes and subsequently leading to various pavement distresses, such as raveling, fatigue cracking, and thermal cracking [32]. Short-term aging takes place throughout storage, mixing, transportation, and paving activities. To simulate this aging process in a controlled environment, the RTFOT test was conducted following ASTM D2872 standards [33]. In RTFOT test, a thin layer of asphalt binder is exposed to heat and air in a rotating oven to simulate oxidative aging. The test is carried out at 163°C for 85 minutes to ensure uniform exposure, and the results, which include changes in penetration, softening point, and viscosity, offer information about the binder's resistance to aging and suitability for practical uses.

2.3. Rheological Properties of Asphalt Binder

Conventional laboratory experiments were performed on the binder both before and after the aging process. These tests included assessments of ductility in accordance with ASTM D113-2007 guidelines, the softening point following ASTM D36/D36M-14 standards, penetration according to ASTM D5/D5M-13 specifications, and thermal susceptibility evaluated by the penetration index (PI). The Penetration Index (PI), derived from the softening point and penetration test results, measures the binder's susceptibility to temperature variations. A higher PI indicates a lower susceptibility of binder samples to temperature variations, which is preferable in terms of temperature sensitivity. The PI value, determined by equation 1, ranges from -3 (high susceptibility) to +7 (low susceptibility), providing insight into the binder's resilience to temperature fluctuations. [34-36].

$$PI = \frac{1952 - 500\,log\,(Pen_{25}) - 20SP}{50\,log(Pen_{25}) - SP - 120} \tag{1}$$

To evaluate short-term aging resistance, researchers focus on two primary factors: the increase in softening point (ISP) and retained penetration (RP). ISP measures the change between the softening points of aged and unaged samples, while RP represents the percentage of penetration retained in aged samples compared to their original unaged state. These parameters are calculated using Equations 2 and 3 [15].

$$ISP\ (°C) = SP_{aged} - SP_{unaged} \tag{2}$$

$$RP\ (\%) = \frac{Penetration\ before\ aging}{Penetration\ after\ aging} * 100 \tag{3}$$

Where; PI is Penetration Index, SP is Softening point (°C), and Pen25 is Penetration value at 25°C. As the asphalt binder ages, its penetration value decreases, and simultaneously, its softening point and viscosity increase. Generally, for aging resistance of bitumen, higher ISP and lower RP values are indicative of better aging resistance. Therefore, a higher ISP and lower RP value help to reverse further aging of the asphalt binder samples [15, 25].

Typically, a rise in viscosity after aging is detected, representing higher stiffness. Although, at elevated temperatures, raised viscosity can advance rutting resistance, extreme stiffness may lead to cracking, particularly under low temperatures or frequent loads. Conversely, a reduction in ductility post-aging suggests less flexibility, causing the binder more prone to thermal and fatigue cracking.

2.4. Dynamic Rheologic Parameters for Nano-Modified Asphalt Binders

The dynamic rheological parameters of nano-modified asphalt binders assess the material's response to applied stress or strain under varying temperatures and frequencies using both DSR and BBR tests.

2.4.1. Dynamic Shear Rheometer Test

Concerning the rheological characteristics of the binder at intermediate to high temperatures, a dynamic shear rheometer tester (Anton Paar RheoCompass™), as shown in Fig. 3, was utilized in accordance with AASHTO T315 [16]. The test was performed at a mean temperature of 70.0°C using a frequency of 10 rad/s (1.59 Hz) and a strain of 12%. The sample geometry included a parallel plate setup with 25mm diameter and 1mm thick. Each test included 10 data points, with significant values determined by averaging the results. For instance, the mean complex shear modulus (|G*|) was 0.968 kPa, and the |G*|/sin(δ) value was 0.9685 kPa, with a standard deviation of 0.0003 kPa and a 95% confidence interval of 0.9683–0.9687 kPa. The dynamic shear rheometer DSR test results yield two key parameters: the complex shear modulus (G*) and the phase angle (δ) [37]. These parameters aim to assess both the elastic and viscous behaviors of the modified asphalt binders. The asphalt binder specimen is subjected to frequent shearing which deforms the specimen and the overall specimen resistance to this deformation is known as complex modulus (G*), while the shift between the resulting shear strain and the practiced shear stress is called the phase angle (δ) which describes the relationship between components of the viscous and elastic behaviors [38-40]. Fatigue cracking and rutting are two indicators obtained from the (G*) and (δ) results [41]. A higher G* value is associated with greater stiffness, which enhances rutting resistance at high temperatures by limiting permanent deformation under repetitive loads. Conversely, the phase angle (δ) quantifies the lag between applied stress and resultant strain, with lower δ values indicating a more elastic behavior. This elastic behavior contributes to improved load recovery, further mitigating rutting potential. At low temperatures, cracking resistance is influenced by the binder's ability to relax thermal stresses, which is related to its viscous properties. While higher G* values enhance stiffness, they may reduce flexibility, increasing susceptibility to thermal cracking if not balanced by a sufficiently high δ value. The ratio G*/sin(δ), commonly referred to as the rutting factor, is used to evaluate high-temperature performance, with higher values indicating superior resistance to rutting deformation [14, 23]. After RTOFT aging, a greater (G*) value suggests improved stiffness and resistance to rutting, but if it is not balanced with an appropriate phase angle, it may also indicate a risk of brittleness.

Fig. 3. DSR tester

2.4.2. Bending Beam Rheometer Test

The Bending Beam Rheometer test was conducted on both original and modified asphalt binders, in accordance with AASHTO T313 standards, to assess the thermal cracking susceptibility of the binder samples, as shown in Fig. 4. This test was performed at temperatures of -6°C and -12°C and its increment, with a constant load applied for 240 seconds to measure the binder's ability to resist thermal cracking. The specimen geometry consisted of beams measuring 125 mm in length, 12.5 mm in width, and 6.25 mm in thickness. The equipment used included a calibrated bending beam rheometer capable of maintaining precise temperature control. Each test involved multiple repetitions to ensure reliability, with significant values determined by averaging the results. This test aids in evaluating the stiffness and relaxation properties of asphalt binders at low temperatures, which is essential for predicting their performance in cold climates [7, 17, 42-44].

Fig. 4. BBR tester

2.5. Morphology of the Nano-Modified Binder

The energy-dispersive X-ray spectrometer and scanning electron microscope were used in tandem to provide information on the elemental composition of samples, identifying the elements present along with their concentration and dispersal. The EDX was operated at a magnification of 1000x and a voltage acceleration of 15kV to generate an elemental map for evaluating the dispersion of nanomaterials within the sample. Utilizing an X-ray spectrum, the EDX compresses compositional and topographical information of the sample into a single view. The elemental dispersion is then visualized with a colored map, where each color represents an element present in the sample [45-48]

3. Results and Discussions

3.1. Conventional Tests

Throughout the aforementioned process, the integration of Nano-copper oxide into the asphalt binder has yielded notable enhancements in its physical characteristics. Fig. 5, Fig. 6, and Fig. 7 illustrate its impact dosages on both aged and unaged samples concerning penetration, resistance to short-term aging, and susceptibility to thermal changes.

Regarding the penetration, adding nano-copper oxide led to improvements in the penetration of unaged and aged modified asphalt binders. As illustrated in Fig. 5, the penetration value decreased with the increase in (CuO) ratio up to 6%, then started to increase with (CuO) 8%. This increase might be attributed to the excessive concentration of nanoparticles within the asphalt binder matrix, which can lead to particle agglomeration and subsequently increase the stiffness.

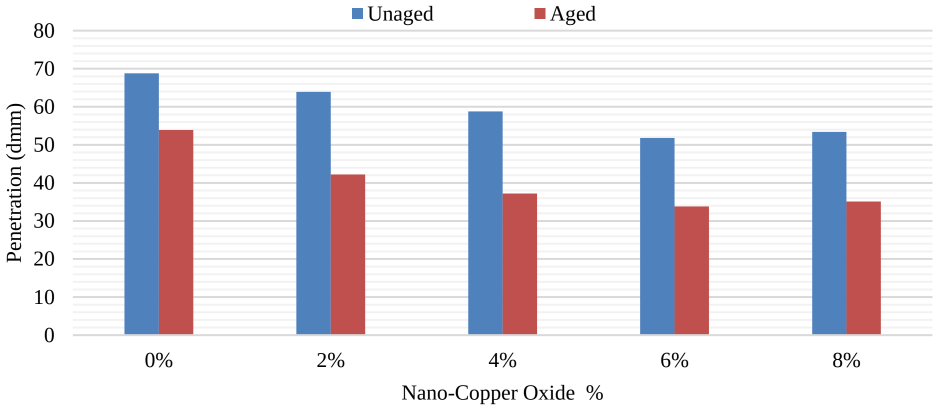
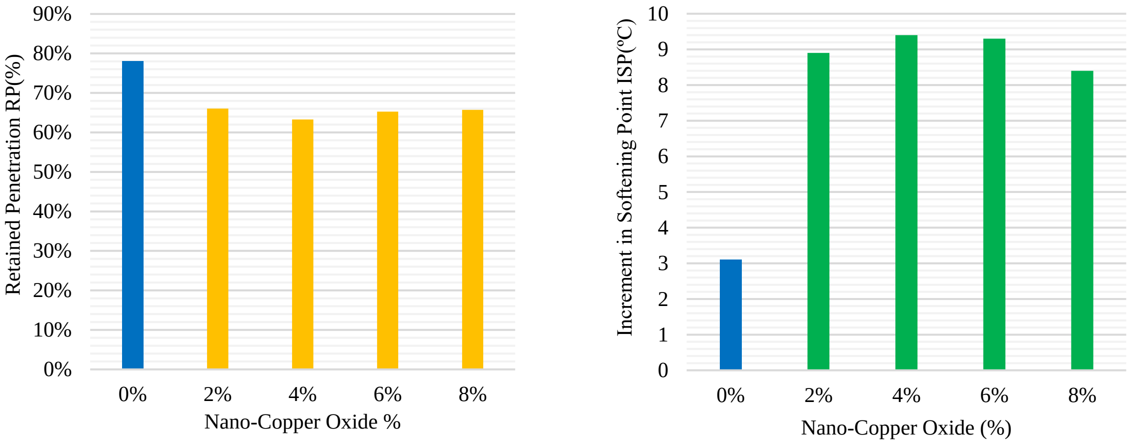


Fig. 5. Penetration test results

The data presented in Fig. 6 (a) and Fig. 6 (b) indicate that the lowest RP and maximum ISP value occurs at a concentration of 4%, respectively. Therefore, the 4% nano-copper oxide concentration exhibits the lowest RP and highest ISP values, demonstrating superior short-term aging resistance. Furthermore, 4% gives the lowest PI which demonstrate the lowest temperature susceptibility as shown in Fig. 7.



(a) (b)

Fig. 6. Retained penetration-RP (%) and Increment in softening point-ISP results (°C) (a) RP % (b) ISP (°C)

The ductility test results are utilized to assess the anti-deformation and anti-cracking characteristics of modified asphalt binders. As depicted in Fig. 8, for unaged and aged modified binder asphalts, improvements in ductility cannot be observed except for unaged 2% content sample which exhibits improvements about 4% compared to the original asphalt binders. This is attributed to the elasticity characteristics of the nano-CuO, which is not viscoelastic.

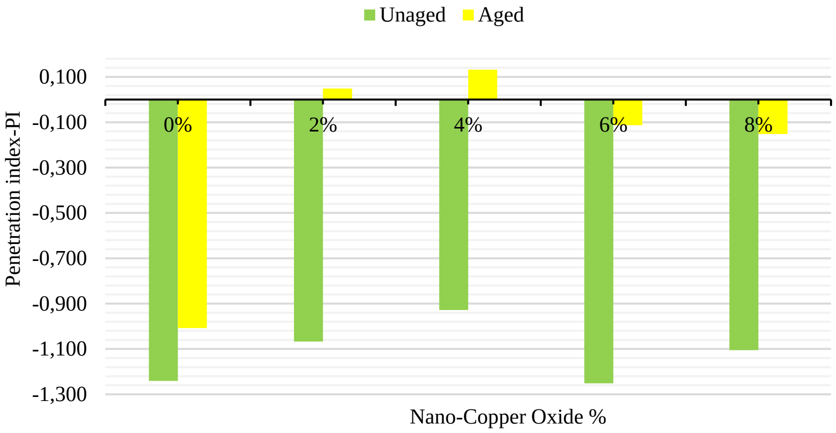


Fig. 7. Penetration Index (PI) results

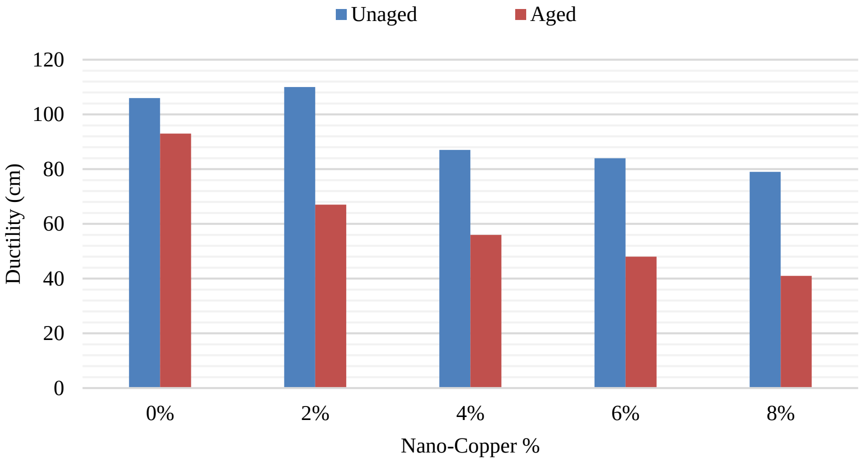


Fig. 8. Ductility test results

3.2. Rheological Tests

3.2.1. Dynamic Shear Rheometer Test (DSR)

This study evaluated modified asphalt binders in both aged and unaged states. Fig. 9 and Fig. 10 display the complex shear modulus (G*) and phase angle (δ) of modified asphalt binder mixes across a range of temperatures. An increase in temperature resulted in a reduction of the complex shear modulus and a concurrent rise in the phase angle. Thus, higher temperatures led to increased viscosity in the asphalt binder. Notably, all modified asphalt binder samples demonstrated higher G* values and lower δ values compared to the original binder at the same temperatures, indicating enhanced stiffness and elastic properties in both aged and unaged samples. The effect of aging on the complex shear modulus (G*) and phase angle (δ) provides critical insights into the performance of asphalt binders. Aging typically increases G* due to stiffening of the binder, improving rutting resistance at high temperatures but potentially reducing flexibility. The phase angle (δ) generally decreases with aging, indicating a shift toward more elastic behavior. This effect is temperature and frequency-dependent, as higher temperatures and lower frequencies tend to amplify the viscoelastic changes caused by aging, which could influence the binder's susceptibility to thermal and fatigue cracking under varying field conditions.

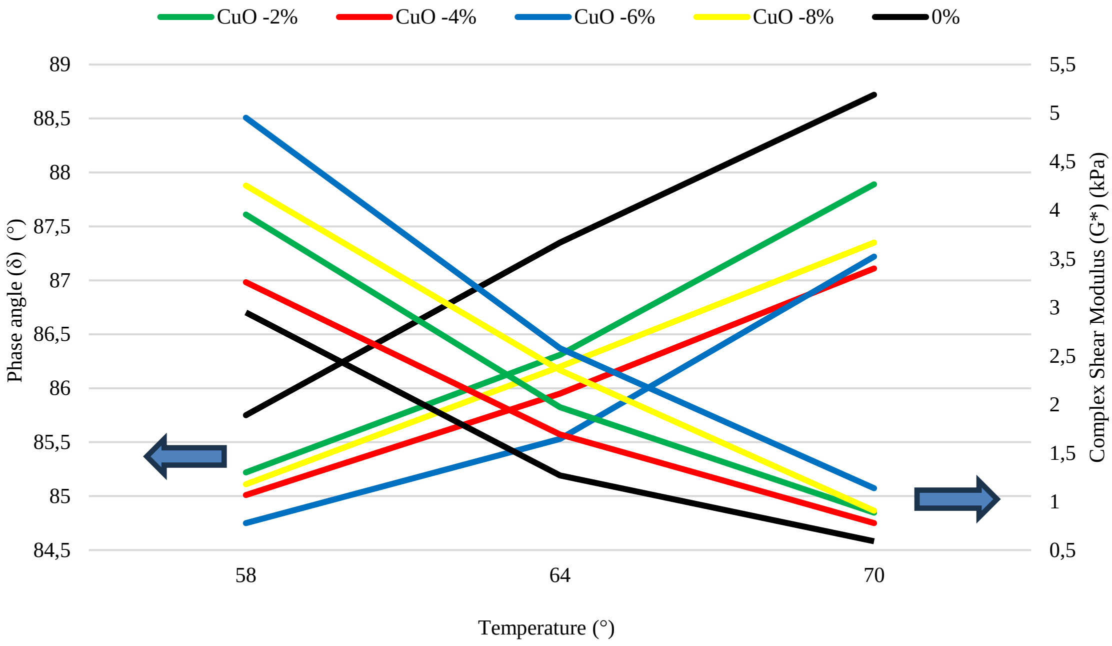


Fig. 9. Unaged DSR results - complex shear modulus (G*) and phase angle (δ)

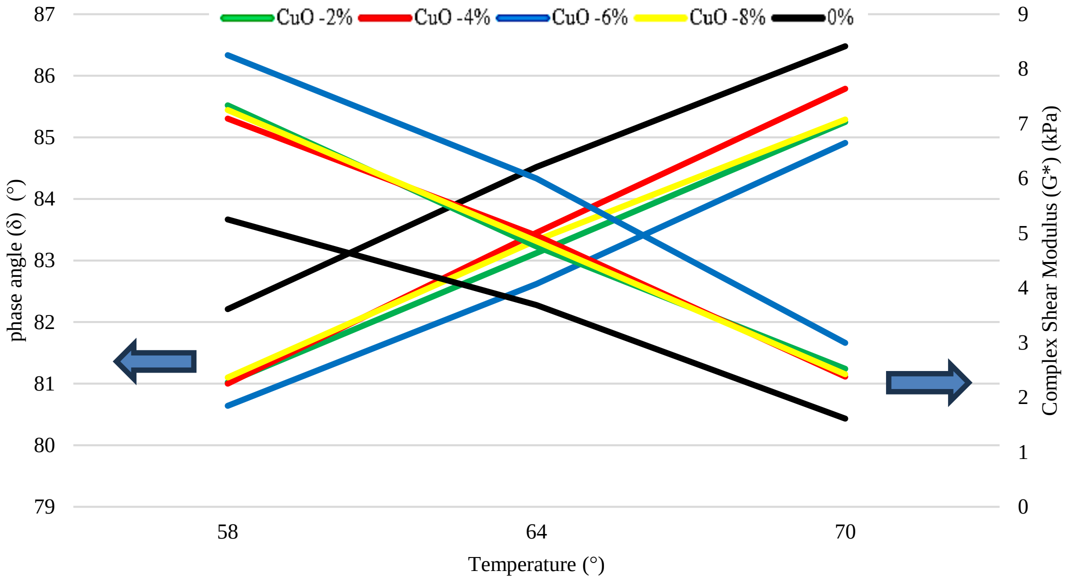


Fig. 10. RTOFT aged DSR results – complex shear modulus (G*) and phase angle (δ)

3.2.2. Bending Beam Rheometer Test

To evaluate the impact of nanomaterial modifications on low-temperature performance, the creep stiffness and m-value parameters were assessed. The results, as illustrated in Fig. 11 and Fig. 12, demonstrate the effects of integrating nano-copper oxide into the asphalt binder. Notably, there were improvements in certain mix contents, particularly with 6% Content, where the m-value increased by approximately 3%, which reflect better stress relaxation capabilities, allowing the binder to dissipate thermal stresses more effectively, and creep stiffness decreased by about 10% indicating improved flexibility, reducing the binder's brittleness and enhancing its ability to resist thermal cracking at low temperatures. However, some contents showed a decline in performance. Despite these variations, all samples met the SuperPave™ requirements at a low temperature of -6ºC, with an m-value of ≥ 0.3 and a creep stiffness of ≤ 300 MPa. Nevertheless, all samples failed to meet these requirements at -12ºC. This indicates that differences in the chemical composition of the nanoparticles and the asphalt binder, as well as their interaction, may be the primary cause of the failure at lower temperatures.

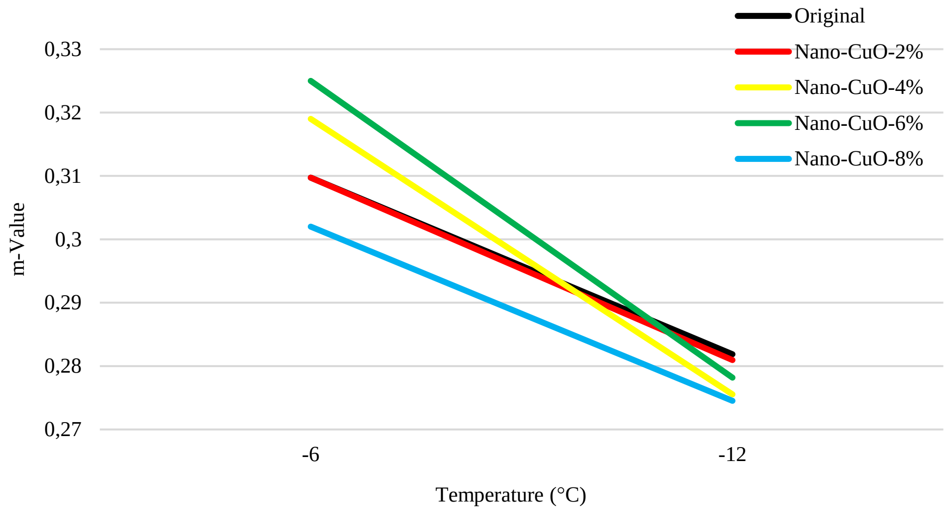


Fig. 11. m-Value

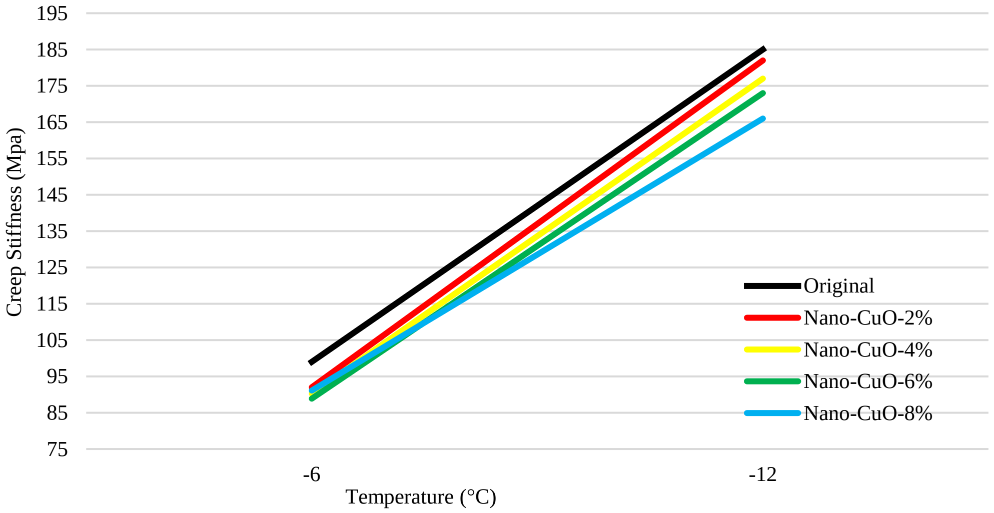
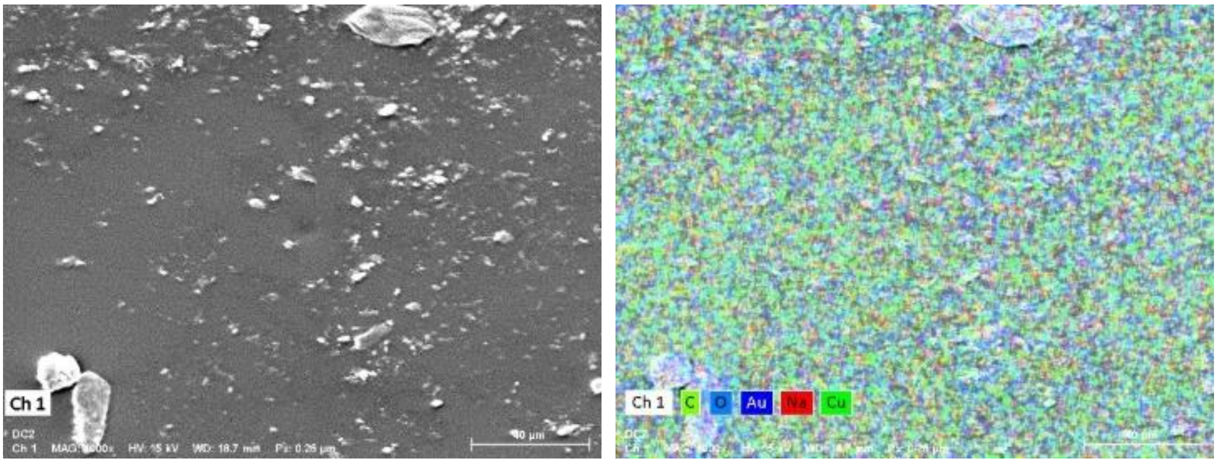


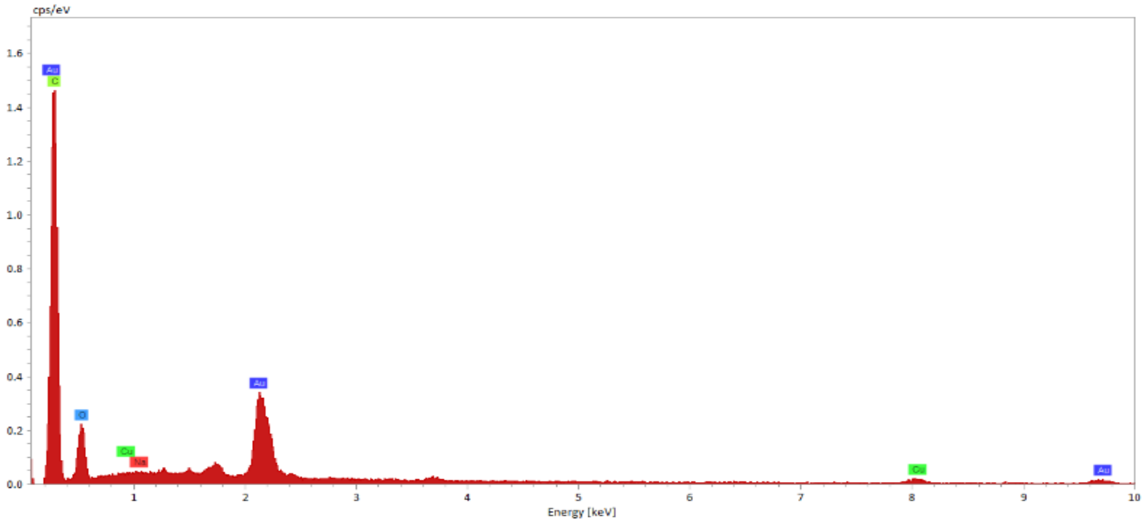
Fig.12. Creep Stiffness

3.3. Morphology of the Sample

Employing the Energy-dispersive X-ray (EDX) analysis method alongside scanning electron microscopy (SEM) enables the evaluation of sample chemical compositions and the distribution of nanomaterials within asphalt binder mixes. This technique assigns a unique color to each element, allowing for the construction of an elemental map by overlaying all elements present, thus illustrating the sample's elemental composition. SEM images, along with elemental EDX maps and EDX spectra of nano-copper modified asphalt binders, is presented in Fig. 13. The SEM images indicate a high-quality dispersion achieved through the mixing technique utilized across all samples. Moreover, the EDX spectrum provides insight into X-ray intensities and their relative concentrations emitted from the sample in relation to their energy levels. By identifying the peak of each element, the EDX spectrum aids in determining the presence of these elements in each modified asphalt binder sample.



(a) SEM images (b) Overly EDX map



(c) EDX spectrum

Fig.13. SEM/EDX elemental scan, Spectrum and map for nano-copper oxide modified binder

4. Conclusion

This investigation explored into the durability and physical assets of asphalt binder when subjected to modification with nano-copper oxide. The subsequent findings are as follows:

- The conditions used for mixing, including time, speed, and temperature, have resulted in a good dispersion of nanomaterials throughout the mixes which was established by utilizing EDX analysis and SEM images;
- The integration of Nano-Copper oxide has vital impact on improving the penetration for both aged and unaged modified asphalt binders. The best improvement has found at 6% in which reached about 25% for unaged samples and 37% for aged ones at the same concentration level;
- Significant improvements in short-term aging resistance were discerned in the modified asphalt binders with 4% ratio, characterized by increased increment in softening point ISP with 67% and reduced retained penetration RP with 19.2%, compared to the original asphalt binder samples;
- The susceptibility of temperature, evaluated by the penetration index (PI), experienced a significant boost with 4% samples for each of aged and unaged modified asphalt binder reaching 25% and 113% for unaged and aged samples, respectively;

- Due to the brittleness of the Nano-Copper oxide, the addition of high ratios of (more than 2%) led to decrease in ductility for unaged modified asphalt binders. However, post short-term aging (RTFOT), the ability to endure low-temperature performance and resist cracking did not commonly improve across all mix contents; instead, some samples experienced brittle; and
- Dynamic Shear Rheometer (DSR) testing revealed that the integration of nanomaterials notably bolstered resistance against rutting deformation and minimized bitumen's resistance to cracking and deformation under high temperatures. Furthermore, RTFOT-aged samples exhibited heightened stiffness, improved elasticity, and a greater rutting factor $G^*/\sin(\delta)$ upon incorporation of Nano-copper oxide, indicative of their exceptional capacity to withstand aging during construction, thereby ensuring prolonged durability for practical applications.
- Although all the asphalt binder samples successfully met the SuperPave™ criteria for low-temperature performance at -6°C, they failed to meet the required specifications at the more stringent temperature of -12°C, indicating a potential limitation in their low-temperature cracking resistance at colder climates or extreme condition.

Although the initial cost of nano-CuO may be higher, its long-term benefits can offset these expenses. By improving the durability and performance of asphalt binders, nano-CuO can reduce maintenance and operational costs over time. Furthermore, its cost-effectiveness during the service life of the asphalt binder makes it a favorable option for environmental sustainability, as it can lead to fewer repairs and longer-lasting road infrastructure. Therefore, In this context, further investigation may be conducted in future studies, including the incorporation of additional nanomaterials to create composite nanomaterials and evaluating their cost-effectiveness in road applications.

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