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RESEARCH ARTICLE Open Access

The Role Of Nano-Alumina Particles In Enhancing Asphalt Binder Rheological Properties

Baran Ramadhan Omer [1]*, and Ganjeena J. Khoshnaw [2]

[1] Department of Civil Engineering, College of Engineering, University of Duhok, Duhok, Kurdistan Region, Iraq.
[2] Civil Engineering Department, Faculty of Engineering, International Tishk University, Erbil, Kurdistan Region, Iraq.

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*Email address: Baran.omer@uod.ac
Corresponding Author

Abstract: Traditional asphalt binders face significant challenges such as aging, temperature susceptibility, and fatigue cracking, which adversely impact highway maintenance and rehabilitation. Modern road networks also face emerging challenges such as increased traffic loads, climate change-induced temperature extremes, and in return, the need for more sustainable and eco-friendly materials to reduce environmental impact is essential. Aging reduces the binder's flexibility, increasing the likelihood of pavement cracking under traffic loads. Temperature susceptibility leads to rutting at high temperatures and thermal cracking in cold conditions, while fatigue cracking compromises the structural integrity of roads under repeated loading. These issues result in higher maintenance costs, reduced infrastructure durability, and potential safety risks for road users. Addressing these challenges is critical to developing sustainable, cost-effective, and safe road networks. Researchers have explored innovative solutions, including incorporating nanomaterials into modified asphalt binders alongside additives like rubbers and polymers. This study investigates the direct integration of nano-alumina into a 60/70 penetration grade asphalt binder without additional additives. Using a high-shear mixer, nano-alumina was incorporated at varying concentrations (2%, 4%, 6%, and 8% by weight). Conventional and rheological tests revealed that the nano-modified binder exhibited enhanced temperature susceptibility, improved overall viscoelastic properties, increased stiffness, better ageing resistance, and improved performance at low temperatures. For example, penetration improved up to 30% for 6% nano content and temperature susceptibility, reaching up to 35% in some contents
Keywords: *Asphalt Binder; Nanomaterial, Nanotechnology, Nano-Alumina, Rheological Properties.*

1. Introduction

The main composition of asphalt pavement is Asphalt binder, which is the first material used, and pavement binder acts as a semi-fluid or molten material above 25°C, while brittle at lower temperatures [1-3]. As stated by others, the softness or temperature-related properties, as well as age-related properties of asphalt binder, are one of the primary leveraging factors in asphalt pavement performance deterioration, while an increase in traffic volume, an increase in the mass of vehicles and higher tire pressure worsen the rutting, fatigue cracking and stripping phenomena. The latest research made some attempts to enhance asphalt benders' properties using different additives such as rubbers, polymers and bonding agents, but still issues remain with high temperature self-ignition during storage and low level of the binder's tolerance to these materials [4, 5]. Therefore, scientists had to use advanced nanotechnology, which presents great enhancers to materials used, such as nano titanium, nano silica, nano alumina and nano clays. As a result of the abovementioned, binders had appreciable alterations in their physicochemical properties and brought about a paradigm shift in material science [6].

In this regard, nanomaterials of various forms were used in numerous investigations aimed at improving the physicochemical properties of bituminous materials. Studies considering nano-silica as a modifier for base asphalt had reduced penetration and softening points in addition to increasing the viscosity. Furthermore, on the other hand, rheological behaviour, as determined by dynamic shear rheometer (DSR) testing, of the modified binders indicated that the materials had a greater complex modulus and lower phase angle [7, 8]. Also, the Nano-CuO modifications were obtained that increased the specific heat capacity of asphalt binders and decreased the temperature and moisture susceptibility, as well as improved the ageing resistance of the binders [9]. Moreover, Kadhim et al. reported that the use of nano-alumina significantly improved the bitumen's rheological properties at high and intermediate temperatures when added to bituminous mixtures with low concentrations of SBS, as a consequence of having the modifications done. The other pleasing outcome of this modification has been the provision of a stable system when stored with remarkable anti-ageing properties [10].

Even though there are numerous studies on the improvement of asphalt binder, not many have focused on the use of nanomaterials like Nano-Alumina on its own or in conjunction with other substances. This gap is important since when it comes to the sole effect of the nanomaterials on the performance of the asphalt binder, it also has the potential to provide great economical and efficient alternatives.

With that, can Nano-Alumina alone enhance the physico-chemical and durability properties of unmodified asphalt binder 60/70? Taking colloidal aims and objectives into account, the addition of Nano-Alumina to a certain asphalt binder that is unmodified can improve its physicochemical and durability properties due to the inherent hybrid characteristics of the nanomaterial. This assists in improving oxidative resistance and long-term durability as well by reducing the effects caused by thermal cycling. Accordingly, it is hypothesized to interact with asphalt binder at the material's nanoscale, creating a more compounded and resilient framework that is more effective under several forces and exposures. In this study, in order to validate this theory, a number of tests were performed.

2. Materials and Methodology

2.1 Materials

The materials used in this study were a local base asphalt binder 60/70 penetration grade provided by the Directorate of Roads and Bridges – Duhok, and Nano-Alumina, as shown in Fig.1, requested from Hebei Suoyi New Material Technology Co., Ltd. – China, with the specifications listed in Table 1. Conventional tests following ASTM guidelines were performed to examine the overall characteristics of the base asphalt binder, as detailed in Table 2. which met the requirements specified by ASTM D946M-15.

Table 1: Properties of Nano-Alumina

Properties	Nano-Alumina
Chemical Formula	Al2O3
Appearance	White powder
Purity	99.99%
Particle size	45nm
Particle Morphology	Spherical
Specific surface area	185m2/g
Ph	7-8
Molecular weight	101.96g/mol
Melting point	2054°C

Table 2: Base asphalt binder characteristics 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

Figure 1: Nano-Alumina appearance

2.2 Sample Preparation Progress

The modified asphalt binder sample is made by heating the base asphalt binder in an oven just above the softening point. This creates a liquid consistency that makes it simple to pour into the mixing pan. The amount of nano-alumina (2%, 4%, 6%, and 8% by binder weight) was carefully measured and added to the liquid asphalt binder gradually. To guarantee even dispersion, avoid clumping, and get rid of bubbles, mixing was done for five minutes using a high-shear mixer set to 4500 rpm. After that, for 15 minutes, the speed was raised to 15,000 rpm while maintaining a 157 ± 1°C regulated mixing temperature.

2.3 Aging Progress

In compliance with ASTM D2872 guidelines, the rolling thin film oven (RTFOT) test was used to replicate controlled short-term aging that takes place during paving, mixing, storing, and transportation operations. The main causes of bitumen's aging are oxidation and volatilization brought on by operational and environmental variables [11, 12]. However, the pressure aging vessel (PAV) test was carried out in accordance with ASTM D 6521 for long-term aging, which represents the performance of asphalt binder in service.

2.4 Asphalt Binder Rheological Properties

The binder was subjected to standard laboratory tests both prior to and following the aging process. These tests comprised evaluations of ductility in compliance with ASTM D113 guidelines, penetration in compliance with ASTM D5/D5M-13 criteria, the softening point in compliance with ASTM D36/D36M-14 standards, and temperature susceptibility as measured by the penetration index (PI). The PI indicates how sensitive the binder is to temperature changes and is calculated from the softening point and penetration test data. A higher PI means that binder samples are less sensitive to temperature changes, which is better from a temperature sensitivity perspective. Equation (1) yields the PI value, which gives information on the binder's resistance to temperature changes. It ranges from -3 (high susceptibility) to +7 (low susceptibility) [13-15].

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

The two main parameters that researchers look at to assess short-term aging resistance are retained penetration (RP) and the increase in softening point (ISP). While RP is the proportion of penetration retained in aged samples relative to their initial unaged state, ISP calculates the difference in softening points of aged and unaged samples. Equations 2 and 3 are used to calculate these parameters [16]. Where; PI is Penetration Index, SP is Softening point (°C), and Pen25 is Penetration value at 25°C.

(2) $$ISP\ (°C) = SPaged – SPunaged$$

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

A higher ISP and lower RP value help to reverse further aging of the asphalt binder samples because as the asphalt binder ages, its penetration value decreases while its softening point and viscosity increase. In general, higher ISP and lower RP values are indicative of better aging resistance of asphalt [16, 17].

2.5 Dynamic Rheologic Parameters for Nano-Modified Asphalt Binders

The dynamic rheological parameters of nano-modified asphalt binders explore how the material reacts to stress or strain changes with different temperatures and frequencies, using DSR and BBR tests.

2.5.1 Dynamic Shear Rheometer Test

For examining the rheological properties of the binder at medium to high temperatures, a dynamic shear rheometer (Anton Paar RheoCompass™), shown in Fig. 2, was used according to AASHTO T315 [18]. The DSR test provides two main parameters: the complex shear modulus (G*) and the phase angle (δ) [19]. These parameters aim to evaluate both the elastic and viscous properties of the modified asphalt binders. The binder sample undergoes regular shearing, causing it to deform, and the resistance of the sample to this deformation is termed G*. Meanwhile, the difference between the resulting shear strain and the applied shear stress is called the δ, which reflects the relationship between the viscous and elastic properties [20-22]. Fatigue cracking and rutting are two outcomes derived from the G* and δ results. A higher δ indicates a more viscous asphalt binder, with values generally between 0° and 90°.

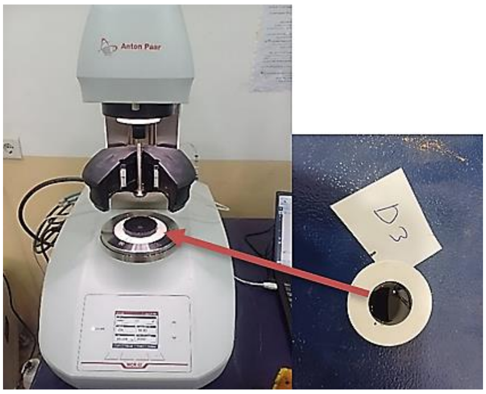

Figure 2: DSR Tester

2.5.2 Bending Beam Rheometer Test

Thermal cracking susceptibility of the binder samples was determined using the Bending Beam Rheometer test, following AASHTO T313 standards, for the base and nano-modified asphalt binders as illustrated in Fig. 3. The goal of this test is to assess the low temperature stiffness and relaxation properties of asphalt binders, which is an important characteristic for prediction of the places where and how the binder will perform in cold climates [9, 23-26].

Figure 3: BBR Tester

2.6 Dispersion of nanomaterials in binder

The surface morphology of the samples and the dispersion of nanomaterials within the asphalt binder matrix can be studied by scanning electron microscope (SEM). SEM imaging, conducted at a magnification level of 1000x, offered comprehensive details of the morphological and surface properties, allowing the determination of any agglomerations or voids.[27-30].

3. Results

3.1 Conventional Properties

Regarding the Penetration, as shown in Fig. 4, adding nano-alumina into the asphalt binder has caused a drop in penetration values for both aged and unaged models. This states that the stiffness of the asphalt binder has been enhanced, particularly with contents with high nano-alumina. The reduced penetration proposes that nano-alumina augments the binder's capability to resist deformation under load, which is valuable for high-temperature performance.

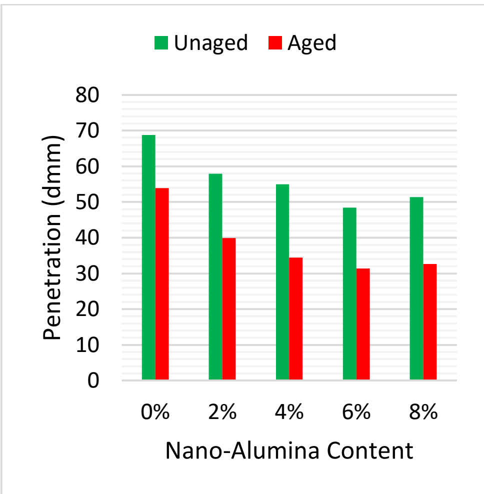

Figure 4: The influence of nano-alumina content on penetration

Relating to temperature Susceptibility, which is the measure of the PI value, boosted with rising nano-alumina content, as shown in Fig. 5. This improvement shows a decline in the binder's sensitivity to temperature variations. Greater PI values represent that nano-alumina supports to thermal stability, making the binder more suitable for areas with significant temperature differences. Similar trends were found in a study by [31], which noted that 2% nano-alumina reduced penetration, thereby enhancing binder stiffness and aging resistance.

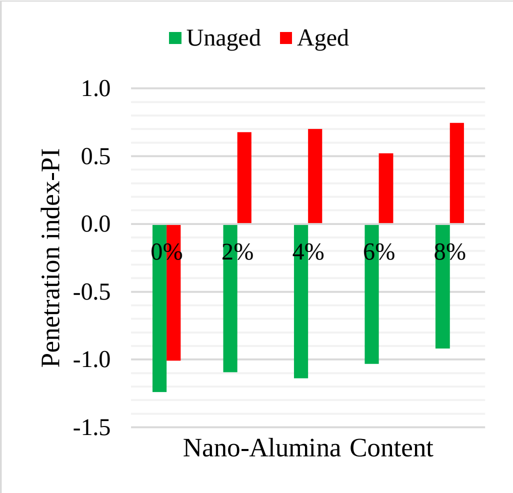

Figure 5: The influence of nano-alumina content on temperature susceptibility

Also, for short-term aging resistance, as illustrated in Fig. 6. The RP% is reducing, while the ISP is enhancing with increasing nano-alumina content. These results suggest that during mixing and paving operations, nano-alumina can improve the oxidative stability of the binder and reduce the undesirable effects of short-term aging processes such as volatilization and oxidation. The same impact of nano-alumina contribution was observed in studies conducted by [32, 33]

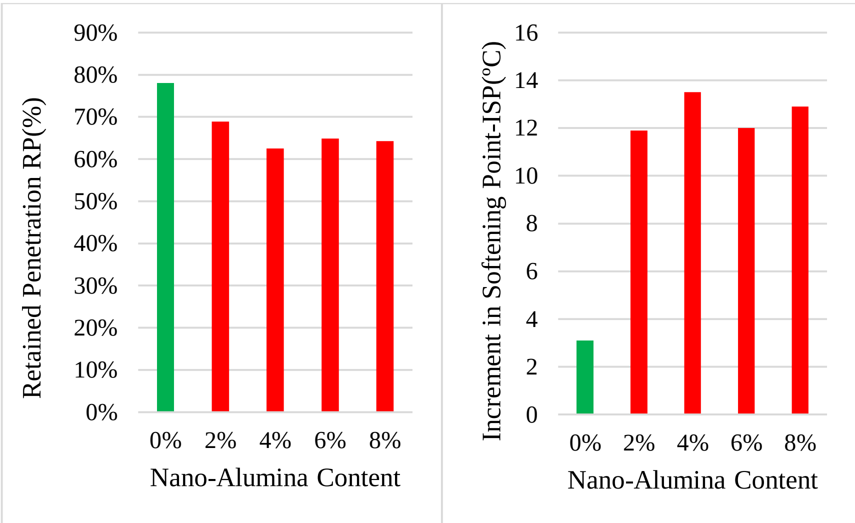

Figure 6: the influence of nano-alumina content on short-term aging resistance represented by (a) RP% (b) ISP (°C)

In contrary, the ductility of the binder, as depicted in Fig. 7, reduced with increasing nano-alumina content for both aged and unaged samples. This decrease can be attributed to the stiffening effect of nano-alumina. While a decrease in ductility might affect flexibility, it enhances stiffness, which is critical for load-bearing capacity under heavy traffics.

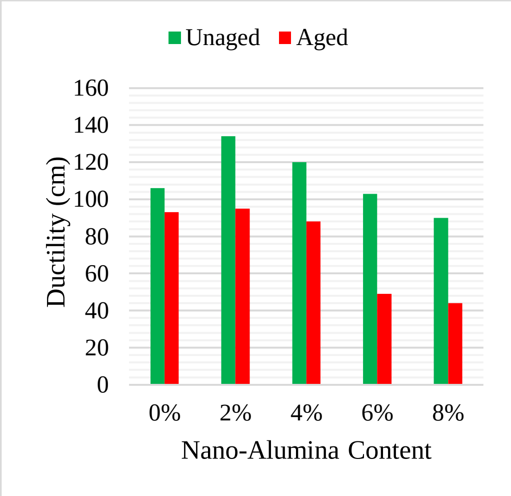

Figure 7: The influence of nanomaterial content on ductility

3.2 Rheological Properties

The results of the DSR test, shown in Fig. 8 and Fig. 9, specify that the integration of nano-alumina increases the complex shear modulus (G*) and decreases the phase angle (δ) for both unaged and RTFOT-aged samples. A higher G* implies improved resistance to permanent deformation (rutting) under repeated loading, while a lower δ indicates enhanced elastic behaviour. These findings confirm that nano-alumina significantly improves the binder's rheological performance, particularly at medium to high temperatures.

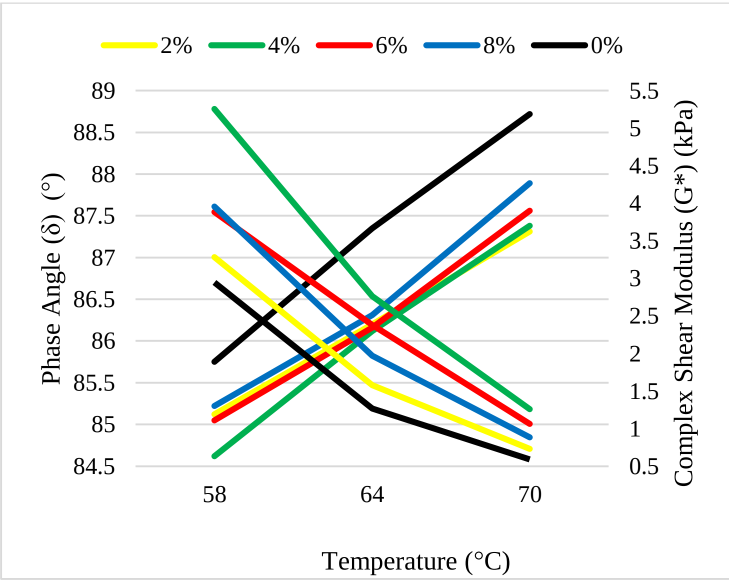

Figure 8: Unaged sample response to DSR test parameters

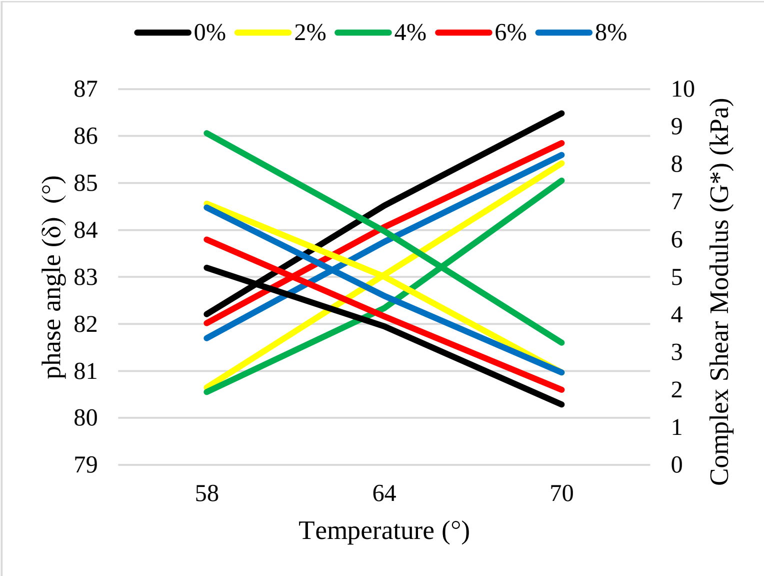

Figure 9: RTOF-aged sample response to DSR test parameters

The low-temperature performance of nano-modified binders was evaluated using the Bending Beam Rheometer test, as shown in Fig. 10. The creep stiffness decreased slightly with the addition of nano-alumina, while the m-value increased. These changes suggest improved flexibility and stress relaxation at low temperatures, reducing the likelihood of thermal cracking. Nano-alumina thus enhances the binder's overall performance in colder climates. Similar trends in improved low-temperature performance due to nanomaterial modification have also been reported by [34, 35], confirming the role of nano-alumina in enhancing binder properties at low temperatures.

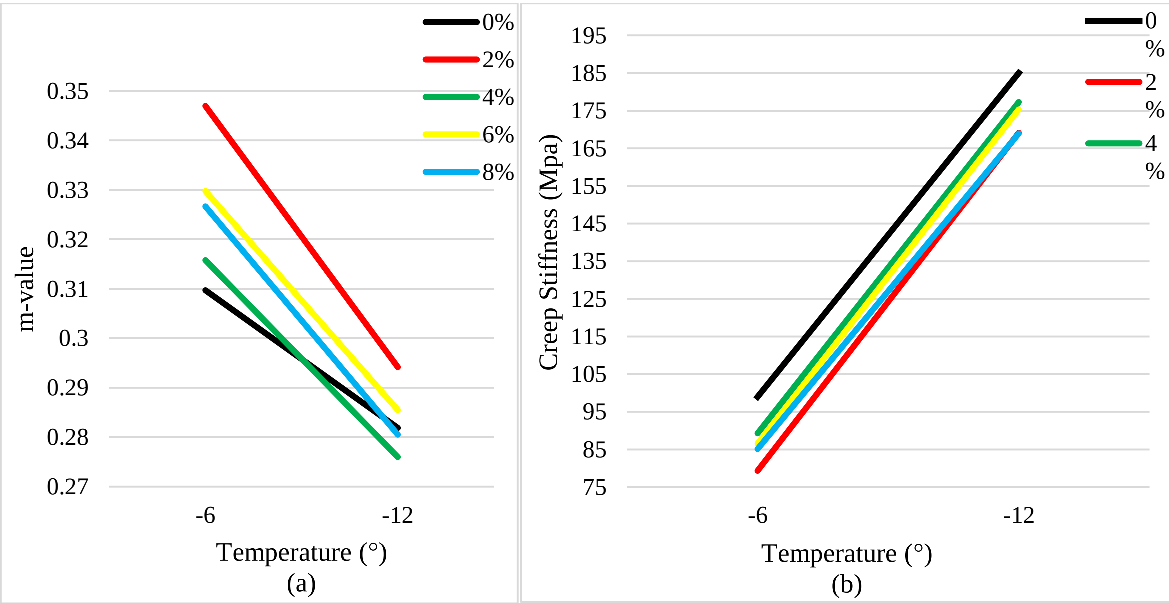

Figure 10: Low temperature susceptibility represented by (a) m-value, (b) Creep Stiffness (Mpa)

3.3 Dispersion of Nanomaterials with SEM Images

The SEM image shown in Fig. 11 shows how the nano-alumina particles disperse within the asphalt binder matrix. The uniform dispersion of the nanoparticles is crucial for reliable improvement of the binder's assets, such as stiffness, thermal stability, and the ability to remain undeformed. Though the dispersion is mostly regular, slight groups of nano-alumina particles are detected in specific zones, probable due to the high surface energy of the nanoparticles that endorses accumulation. Nevertheless, the relatively minor extent of these masses recommends that the mixing procedure, operating a high-shear mixer, was active in attaining a satisfactory level of consistency. Furthermore, the image underlines the smooth incorporation of nano-alumina into the binder matrix, with negligible voids, demonstrating durable physical and chemical interactions between the nanoparticles and the binder. These interactions contribute to the boosted mechanical and rheological properties detected in this study.

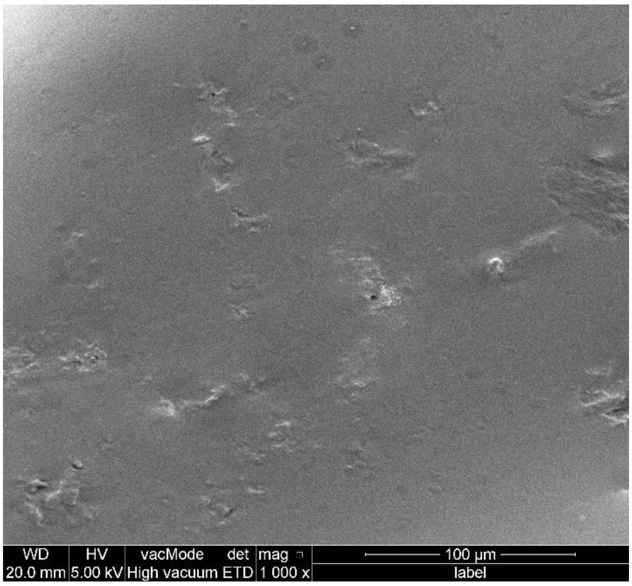

Figure 11: SEM image of nano-modified asphalt binder

4. Conclusion

This study examined the consequences of nano-alumina as a modifier for asphalt binder and its impact on conventional and rheological properties. The outcomes show that the addition of the nano-alumina into asphalt binder advances its performance in the subsequent ways:

- SEM images showed well-dispersion of nano-alumina particles within the asphalt binder matrix indicating suitable mixing conditions.
- The reduction in penetration values and greater Penetration Index with increasing nano-alumina content support greater stiffness and enhanced temperature susceptibility, making the modified binder appropriate for areas with fluctuating climatic situations.
- The enhanced retained penetration and increased softening point values highlight the enhanced oxidative stability of nano-modified binders, mitigating the adverse effects of short-term aging caused by mixing and paving operations.
- The increased complex shear modulus and decreased phase angle observed in the DSR tests indicate enhanced resistance to rutting and improved elasticity, crucial for medium to high-temperature performance under traffic loads.
- The improved m-value and reduced creep stiffness, as revealed by BBR tests, demonstrate better stress relaxation and reduced susceptibility to thermal cracking, ensuring improved performance in cold climates.

In summary, the study shows that nano-alumina meaningfully boosts the physicochemical and rheological properties of asphalt binders, contributing to better durability and flexibility in various functional and environmental situations. These results support the possible of nano-alumina as a cost-effective and effective nanomaterial for improving asphalt binder performance, paving the way for its wider application in flexible pavements.

Author's Contribution: We confirm that all named authors have read and approved the manuscript. We also confirm that each author has the same contribution to the paper. We further confirm that all authors have approved the order of authors listed in the manuscript.

Conflict of Interest: There is no conflict of interest for this paper.

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