



The Effect of Pre-Heating Duration and Temperature Conditioning on Rheological and Rotational Viscosity of Asphalt Bitumen Modified with Nano Clay, Nano Hydrated Lime, and Nano Olive Husk Binders

Sandra Matarneh,^{1,*} Tariq Alkhrissat¹ and Mu'tasim Abdel-Jaber²

Abstract

Overheating asphalt bitumen is well recognized to result in oxidation and the hardening of the material. Heating bitumen is a crucial step in preparing samples, and it is vital to determine the specific time and temperature settings for the oven used in laboratory testing. The current AASHTO guidelines do not include specific instructions for the precise oven settings required for preparing bitumen samples before conducting laboratory tests. This research aims to assess the impact of varying oven heating time and pouring temperatures on the rheological characteristics of control and modified binders, namely nano clay (NC), nano hydrated lime (NHL), and nano olive husk (NOH), during sample preparation. Rheological qualities are assessed by using a rotational viscometer and a dynamic shear rheometer at temperatures relevant to the grade being tested. Three different temperatures (150, 165, and 180 °C) were used to test a control bitumen and two modified binders with percentages of 3% and 5%. The binders were evaluated in their original, un-aged conditions for a duration of 2 hours. The research suggests that a heating temperature of 150 °C and a duration of 2 hours is a suitable method for adequate preparation before conducting routine laboratory tests.

Keywords: Bitumen; Rheology; Complex modulus; Nano clay; Nano hydrated lime; Nano olive husk.

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1. Introduction

Nanotechnology comprises all advanced technologies that operate at the nanoscale. The primary distinction between nanotechnology and other technologies lies in the size of materials and structures employed in this field. The variation is attributed to the alteration in the intrinsic characteristics of the substance at this particular magnitude.^[1] As the particle size falls, a greater proportion of atoms and molecules become exposed on the surface.^[2] Consequently, the surface features of the materials become increasingly significant and influential, hence exerting a bigger influence on their physical and chemical properties.^[3] Consequently, the use of these materials

in several sectors of civil engineering has garnered significant interest. Currently, these compounds are also employed in the alteration of bitumen and asphalt. Iskender,^[4] Ghanoun *et al.*,^[5] Amini *et al.*,^[6] and Al-Mistarehi *et al.*^[7] discussed the effects of nano clay (NC) on bitumen and asphalt mixes. All tests consistently show the beneficial effects of both individual and combined use of NC. This enhancement also improved the rheological features of the bitumen while lowering its penetration grade. There were also some enhancements observed in the resistance to asphalt rutting and the depth of rutting. Ziari *et al.*^[8] conducted a recent study and saw favorable outcomes when incorporating NC with bitumen.

Nano olive husk (NOH) refers to a biomass waste that has a cellulose structure. It is composed of the seed, skin, and pulp that are left over after processing olive grains in the manufacturing of olive oil.^[9] Olives have an oil content ranging from 10% to 30%, a water content ranging from 40% to 60%, and a solid content of 30%. Once the olives are

¹ Civil Engineering Department, Faculty of Engineering, Al-Ahliyya Amman University, Amman, 19111, Jordan

² Civil Engineering Department, School of Engineering, The University of Jordan, Amman, 11942, Jordan

*Email: s.matarneh@ammanu.edu.jo (S. Matharneh)

pressed in olive oil factories, around 20-30% of the resulting substance is oil, while the remaining 70-80% is composed of NOH.^[10,11] Extensive research has been conducted on the application of NOH in various fields. It has been studied as a fertilizer in soil improvement research in agriculture to mitigate its negative impact on the environment.^[12-14] Additionally, it has been explored as an additive in animal feed for livestock,^[15] as a bio-charred fuel for energy production,^[16] and as an additive for clay bricks in construction,^[17,18] and as an ingredient in skin care products in the cosmetic industry.^[19] Zhang *et al.*^[20] conducted research on the usage of NOH in asphalt modification in road engineering and found that asphalt mixtures with NOH additives exhibited enhanced antioxidant and cracking performance. Geckil *et al.*^[21] also noted that NOH additives can improve asphalt performance by modifying the bituminous binder. Additionally, Khedaywi *et al.*^[22] reported that olive waste ash improved the performance of asphalt binders in their study, where they tested different proportions of olive waste ash. Further study is required to determine whether specific NOH wastes may be recycled because of the substantial volume of NOH waste, despite the existing body of studies on its utilization in many fields.

Nano-hydrated lime (NHL) is a type of additive that may be used in bitumen to enhance the characteristics and performance of asphalt mixes. The use of hydrated lime in hot mix asphalt (HMA) yields several advantages. There is a substantial body of research that discusses the efficacy of NHL in managing water sensitivity and its well-recognized role as an anti-stripping agent in preventing moisture damage. Nevertheless, new research indicates that NHL produces additional effects in asphalt mixes. NHL functions as an active filler with antioxidant properties.^[23] These characteristics yield several advantages for pavements. For a significant duration, NHL has been employed as an ingredient in asphalt mixes. Nevertheless, its effects are not completely acknowledged. NHL, because of its distinct characteristics, has the potential to significantly influence the rheology and damage mechanics of asphalt cement when used as an addition.^[24]

It is widely recognized that subjecting bitumen to excessive heat can result in oxidation and cause it to stiffen. Heating is a crucial step in preparing laboratory samples. Thus, it is necessary to determine the amount of time the oven has to be set and the temperature at which the samples should be poured before conducting tests. The current AASHTO guidelines for bitumen sample preparation advise against heating unaged asphalt to temperatures above 135 °C. However, for some modified asphalts or extensively aged bitumen, pouring temperatures beyond 135 °C may be

necessary. According to AASHTO,^[25] “It is important not to exceed a heating temperature of 163 °C for bitumen” and “Reduce the heating temperature and duration to prevent the sample from becoming stiffening properties”.

There is currently no established process for determining the precise heating temperature and heating duration for unaged, NC, NOH, and NHL bitumen-modified samples. This research aims to provide appropriate heating parameters for bitumen sample preparation and to examine the impact of aging on the rheological characteristics of the bitumen. The complicated viscosity reduction and temperature aging indices were utilized to determine optimal heating parameters. In contrast, the RTFO aging index, dynamic shear rheometer (DSR) function map, and critical stiffness temperature were employed to analyze the impact of aging.

The study objectives were achieved by performing rheological analysis of the unmodified and modified bitumen samples utilizing the rotational viscometer (RV) and DSR analysis. The process of aging is carried out by employing the rolling thin film oven (RTFO) for the purpose of short-term aging. The temperature and RTFO aging indices are calculated by comparing the rheological characteristics of aged and unaged bitumen. The DSR function map is utilized to monitor the evolution of the rheological characteristics of the bitumen over time. This is achieved by examining the correlation between the storage modulus and the ratio of dynamic viscosity to loss modulus. The dynamic viscosity is calculated by dividing the loss modulus by the angular frequency.

2. Materials and methods

The experimental program involved heating the control (base) bitumen and the three modified bitumen samples, which were modified with two percentages (3 and 5% by weight of binder) of NC, NOH, and NHL at three different temperatures, 150, 165 and 180 °C. The maximum mixing temperature was limited to 180 °C, since at higher temperatures the bitumen might be oxidized leading to brittleness of the bitumen at low temperatures. For all mixes, to neutralize the effect of heating period, heating period for all mixes was fixed at 2 hours. Then, a preset amount of NC, NHL and NOH was slowly added and mixed for a duration of 2 hours at a rotational speed of 2500 rpm in shear mixer. The modified bitumen samples were tested immediately after the completion of the mixing process without allowing them to mature. The testing techniques involved conducting a series of rheological experiments, specifically the RV and DSR tests at different testing temperatures and frequencies. The study utilized three replicates for each test group, and the reported test results are the mean values for each test. The testing program is summarized in Fig. 1.

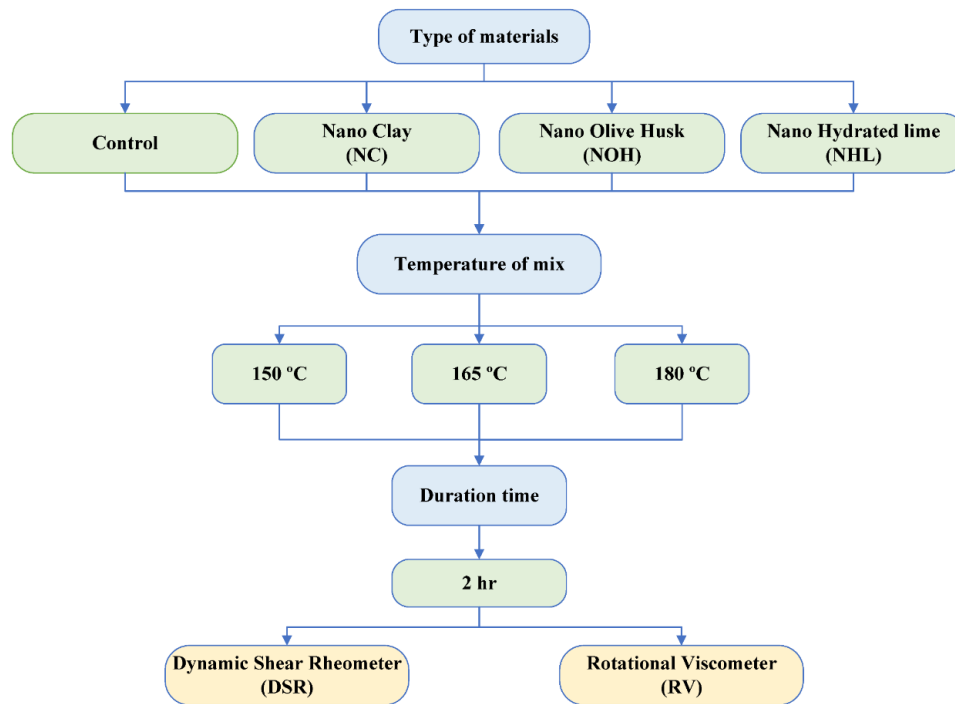


Fig. 1: Summary of the testing approach.

2.1 Rotational viscometer

The viscosity was determined by measuring the rotational speed of a spindle rotating at a rate of 20 rpm, following 30 minutes of thermal stabilization at a temperature of 135, 165, and 195 °C, in accordance with the AASHTO T316.^[26]

2.2 Dynamic shear rheometer

The DSR testing was conducted according to the AASHTO T315,^[27] utilizing a parallel plate geometry with a diameter of 25 mm and a spacing of 1 mm. Frequency sweep experiments were performed across a range of 0.1 rad/s to 100 rad/s at temperatures of 58, 64, 70, 76, and 82 °C. The values for the complex shear modulus (G*) and phase angle (δ) were acquired.

3. Results and discussion

3.1 Rotational viscosity

The viscosity of an asphalt binder was used to assess the flow properties of the binder, ensuring its suitability for pumping and handling at the hot mixing facility. Additionally, it was employed to identify the appropriate temperatures for mixing and compacting asphalt mixes. In order to examine the impact of different temperatures on modified binders NC, NHL, and NOH, viscosity-temperature curves were created, and regression analysis was performed. These results are shown in Fig. 2 and Table 1.

Typically, as anticipated, the viscosity of the modified binders reduces with increasing test temperature. The viscosity-temperature curves in this research adhere to a power function, and all coefficients of determination have values of 0.99 or more. The connection may be

mathematically represented as $y = Ax^B$. The equation represents the relationship between rotational viscosity (y), temperature (x), and regression coefficients (A and B). The viscosity value rises in direct proportion to the concentration (A) increase. The regression coefficient B quantifies the degree to which the binder is affected by changes in temperature. The temperature susceptibility increased as the absolute value of B rose. The values of A and absolute B in Table 1 indicate that binders modified with 3% NC and NHL exhibit a reduction in viscosity compared to the control binder at different mixing temperatures. However, binders modified with 5% NC and NHL show an increase in viscosity compared to the control binder at different mixing temperatures. The addition of NOH to the binder, at concentrations of 3% and 5%, results in a decrease in viscosity compared to the control binder at different mixing temperatures. Simply said, adding an extra 3% of NC and NHL, as well as 3% and 5% of NOH, may marginally lower the viscosity of the modified binders. Consequently, this leads to a drop in the mixing and compaction temperatures of the HMA mixture.

The research investigated the impact of different nanoparticles on the characteristics of asphalt binders. Nano-clay and nano-silica enhanced the high-temperature performance and viscosity of bio-modified asphalt, with nano-clay demonstrating more pronounced improvements in aging and rutting resistance.^[28] The amalgamation of nano clay and nano lime-modified bitumen enhanced viscosity and optimized rutting characteristics, with nano-lime exhibiting a more pronounced effect.^[29] The alteration of organophilic nanoclay enhanced the softening point and viscosity while reducing penetration values, hence influencing mixing and

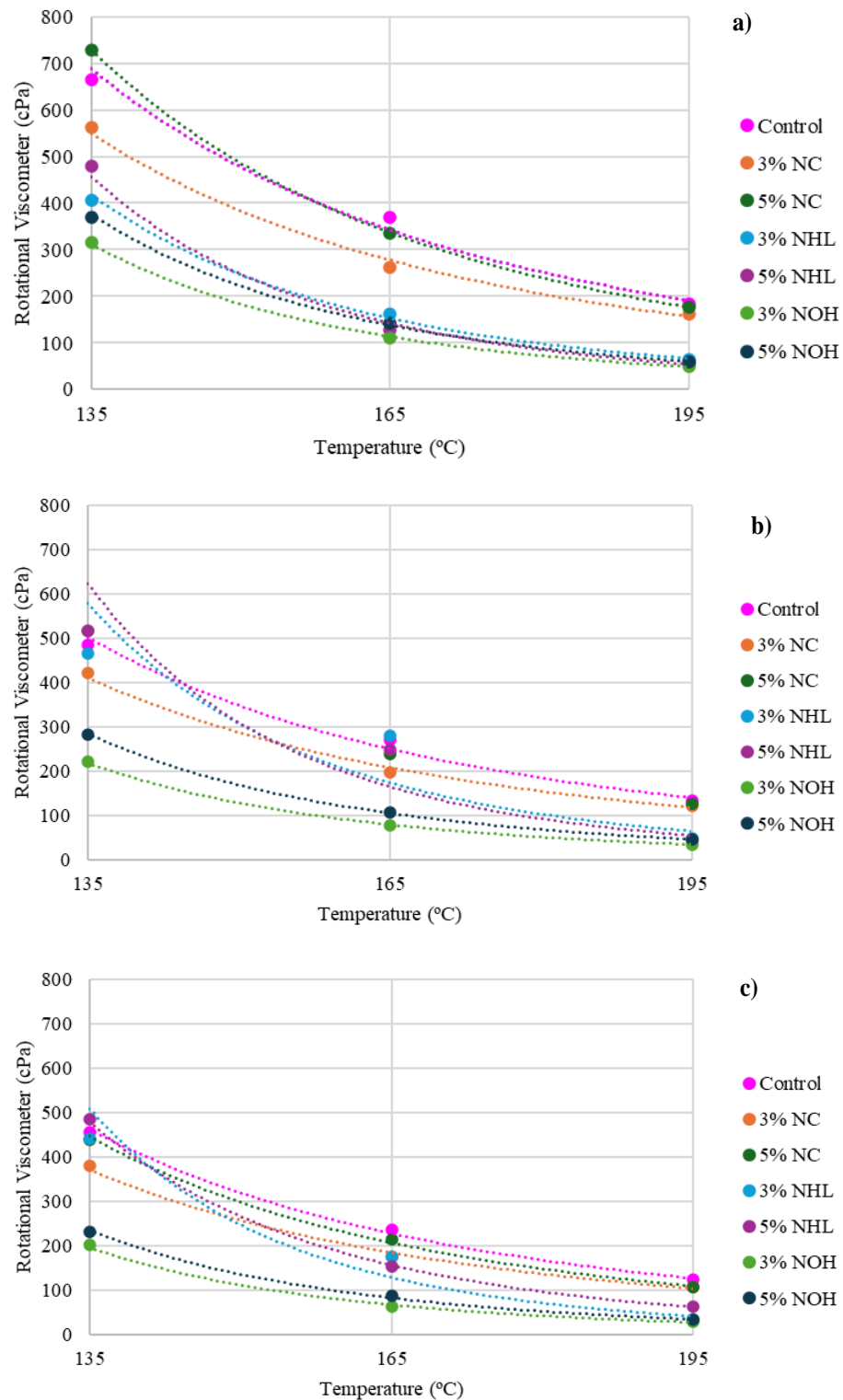


Fig. 2: Regression analysis of rational viscosity and temperature of modified binders NC, NHL, and NOH at a) 150 °C, b) 165 °C and c) 180 °C.

compaction temperatures.^[30] Nano-sized hydrated lime enhanced rutting resistance in foamed warm mix asphalt relative to conventional-sized hydrated lime without significantly influencing viscosity.^[31] These findings underscore the capacity of nanoparticles to improve many characteristics of asphalt binders, especially regarding high-temperature performance and resistance to rutting.

3.2 Dynamic shear rheometer

3.2.1 Phase angle

The phase angle is the measure of the time delay between shear strain and shear stress, indicating the viscosity of the asphalt. In general, asphalt with a lower phase angle has superior elastic recovery ability. Fig. 3 shows the effects of the quantities of NC, NHL, and NOH on the phase angle with

Table 1: The rotational viscosity regression analysis during heating at temperatures of 150, 165, and 180 °C for durations of 4 hours.

Temperature of mixing	Type of materials	Percentage (%)	Rotational viscosity (cPa)			Equation of regression	Coefficient of determination	
			135 °C	165 °C	195 °C			
150 °C	Control	0	664.5	369.1	183.8	$2E+10x^{-3.476}$	0.9917	
		3	562.5	261.9	162.1	$9E+9x^{-3.398}$	0.9961	
	NC	5	728.1	336.3	177	$1E+11x^{-3.846}$	1.0000	
		3	405.2	163.1	64	$2E+13x^{-5.003}$	0.9978	
	NHL	5	480	127.7	57.3	$1E+15x^{-5.807}$	0.9970	
		3	315.4	110.2	49.5	$2E+13x^{-5.043}$	0.9997	
	NOH	5	370.5	141.8	59.3	$1E+13x^{-4.976}$	0.9997	
		Control	0	465.1	249.4	124.2	$2E+10x^{-3.575}$	0.9944
	165 °C	NC	3	421.9	196.4	121.6	$2E+10x^{-3.565}$	0.9969
			5	517	238.8	125.7	$2E+11x^{-4.017}$	1.0000
NHL		3	504.1	229.6	99.3	$1E+12x^{-4.402}$	0.9966	
		5	516.8	200.1	59.6	$2E+15x^{-5.838}$	0.9910	
NOH		3	220.8	77.1	34.7	$7E+12x^{-4.936}$	0.9955	
		5	281.6	107.8	45.1	$2E+14x^{5.551}$	0.9984	
180 °C		Control	0	455.7	235.2	123.9	$3E+10x^{-3.700}$	0.9980
			3	380.6	173.9	106.8	$2E+10x^{-3.665}$	0.9969
		NC	5	438.9	214.1	106.6	$2E+11x^{-4.043}$	0.9979
			3	442.1	175.1	34.5	$2E+17x^{-6.805}$	0.9778
	NHL	5	484.5	152.4	62.3	$3E+15x^{-5.997}$	1.0000	
		3	201.2	62.9	28.1	$3E+13x^{-5.257}$	0.9922	
	NOH	5	230.2	86.3	33.3	$2E+13x^{-5.143}$	0.9998	

different mixing temperatures. The phase angle was found to be highly influenced by the quantities of NC, NHL, and NOH, as well as the test temperatures. Typically, the phase angle of the binders rises as the test temperatures increase and when NC, NHL, and NOH are added. The modified binder containing NOH exhibits smaller phase angles compared to the ones containing NC and NHL under the same conditions. Therefore, the asphalt with NOH exhibits superior elastic recovery compared to asphalt with NC and NHL. Furthermore, the phase angle of the modified binder, which includes 3% of NC, NHL, and NOH, is marginally lower compared to the binders containing 5% of NC, NHL, and NOH. The phase angle (δ) of asphalt binders is affected by nano additives such as NC, NHL and NOH which alter the viscoelastic properties. NC decreases the phase angle by augmenting stiffness and elasticity via its stratified architecture, hence improving rutting resistance.^[32] Likewise, NHL diminishes the phase angle by enhancing stiffness and elastic rebound, while concurrently decreasing moisture susceptibility.^[33] NOH, used as a bio-additive, may decrease the phase angle at ideal levels by enhancing the rigidity of the binder; nevertheless, excessive quantities may disturb the equilibrium between elasticity and viscosity. The effects are contingent upon the kind and amount of the addition, enhancing binder performance for certain situations.^[34]

3.2.2 Complex modulus

The dynamic shear complex modulus (G^*) is defined as the

inverse of the dynamic shear complex compliances. G^* is the quotient of the greatest stress experienced during shear loading divided by the maximum strain. It quantifies the overall resistance to deformation during shear loading. Asphalt with a high complex modulus has enhanced resistance to flow deformation. Fig. 4 illustrates the effects of the NC, NHL, and NOH composition on the complex modulus. In general, the complex modulus of the binders reduces as the test temperatures rise and when NC, NHL, and NOH are added. The study found that the complex modulus is highly influenced by the concentration of NC, NHL, and NOH, as well as the temperature of the test. The regenerated asphalt comprising NC and NHL exhibits a reduced complex modulus compared to those containing NOH under the same conditions. Therefore, the asphalt containing NOH exhibited superior resistance to flow deformation compared to the asphalt containing NC and NHL.

Fig. 4 illustrates the impact of the three altered binders on the complex modulus. For instance, like the rutting resistance factor and phase angle, the modified binder NOH exhibits the strongest resistance to flow deformation. Furthermore, the specific kind of altered binder has a significant impact on the G^* value in this research. NC enhances G^* due to its elevated surface area and interfacial bonding, hence augmenting elasticity and diminishing viscosity.^[35] NHL chemically engages with the binder, enhancing stiffness, elasticity, and moisture damage resistance.^[36] NOH used as a bio-additive enhances the rigidity of the binder and fortifies its structure;

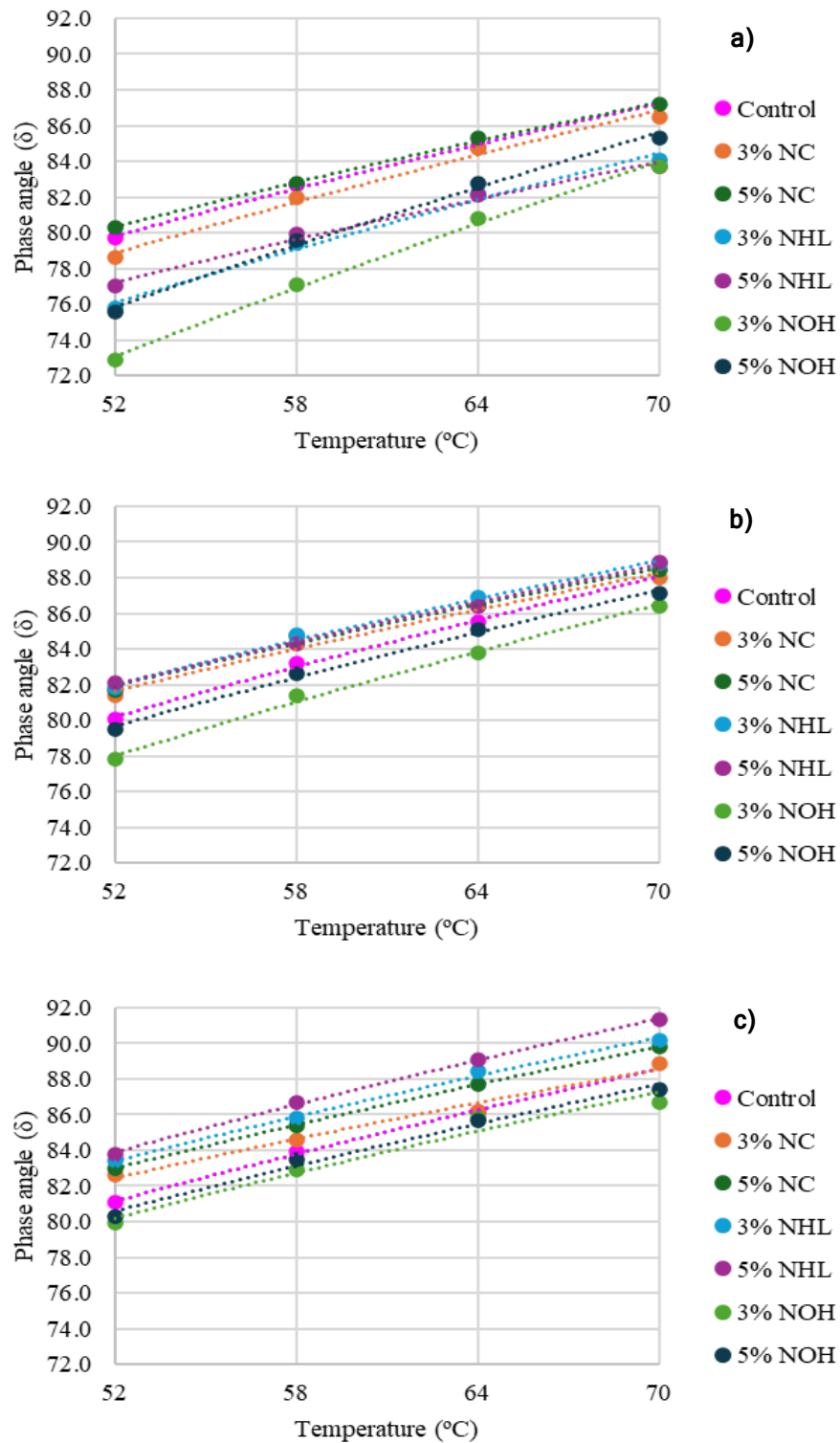


Fig. 3: Phase angle and temperature of modified binders NC, NHL, and NOH at a) 150 °C, b) 165 °C and c) 180 °C.

nevertheless, excessive quantities may result in agglomeration, hence diminishing efficacy.^[37]

3.2.3 TrueGrade temperature

The failure temperature of the time-temperature equivalence principle is the crucial temperature at which the concept no longer applies to mixtures above or below this temperature.

Higher failure temperatures make it more challenging to cause a persistent deformation in asphalt concrete. Put simply, if the failure temperature of asphalt rises, it will enhance the resistance of the mixes to rutting. Typically, it is comparable to the product of G and the reciprocal of the sine of δ . Fig. 5 demonstrates the impact of the quantities of NC, NHL, and NOH on the temperature at which modified binders fail. The

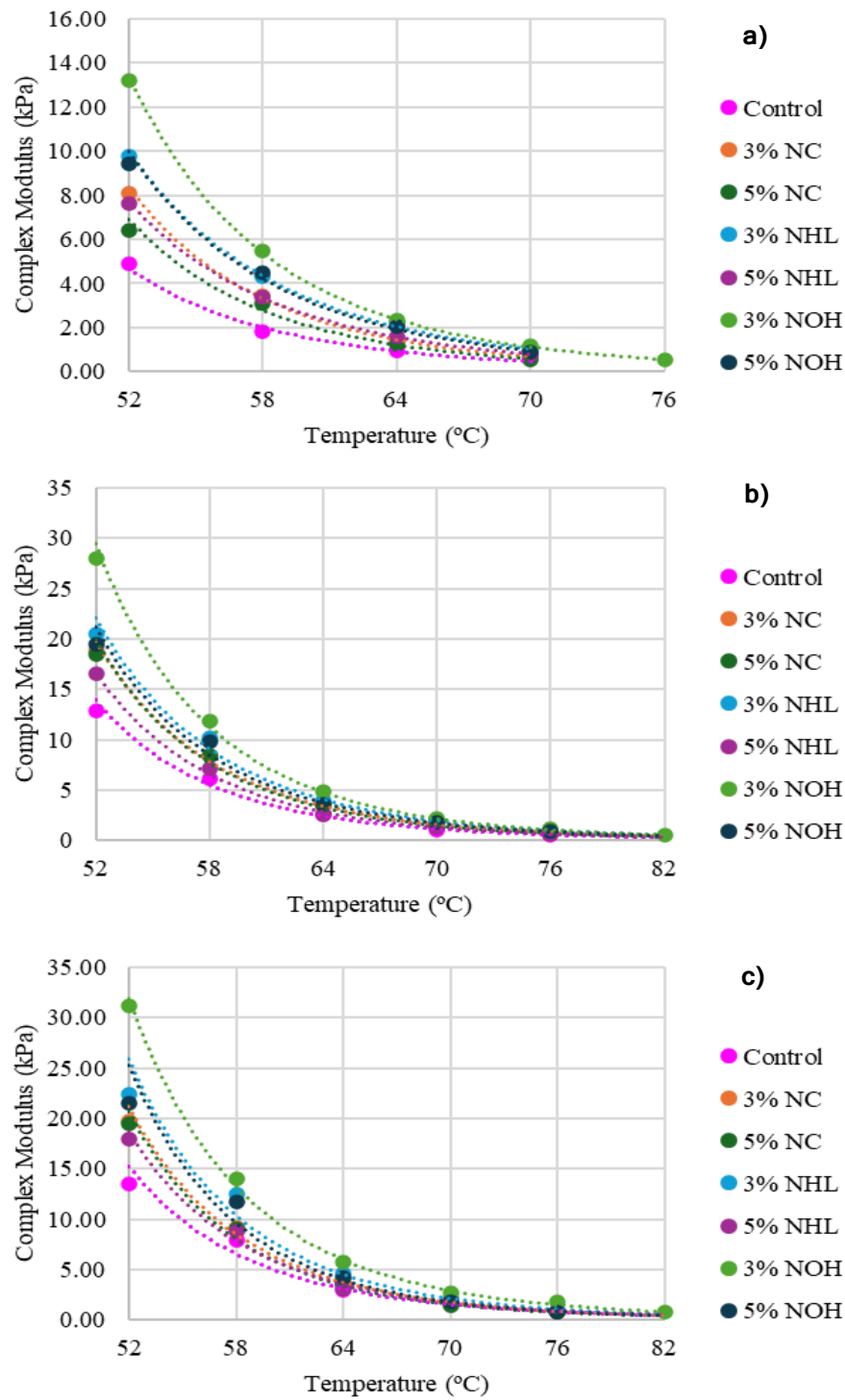


Fig. 4: Complex modulus and temperature of modified binders NC, NHL, and NOH at a) 150 °C, a) 165 °C and c) 180 °C.

failure temperature of the modified binders was shown to be highly dependent on the concentrations of NC, NHL, and NOH. Typically, the failure temperature rises with the addition of NC, NHL, and NOH. The binders that have been changed with NC and NHL exhibit reduced failure temperature values compared to the asphalts that have the same NOH content. Therefore, while comparing under same conditions, it can be seen that the asphalt having NOH exhibits a greater temperature at which failure occurs compared to the modified

binders including NC and NHL. Furthermore, the binders that have been changed to include 5% NHL have failure temperature values similar to the binders that have been treated with 5% NC.

NC elevates failure temperature because of its stratified architecture and robust interfacial adhesion, hence improving elasticity and resistance to deformation.^[38] NHL enhances stiffness and moisture resistance, hence indirectly boosting high-temperature performance.^[39] NOH elevates the failure

temperature by enhancing the rigidity and fortifying the binder matrix; however, excessive quantities may compromise thermal stability owing to agglomeration.^[40]

impacts of using these modified asphalt binders in comparison to conventional materials.^[28,30,32,41,42] In conclusion, carbon nanoparticles (CNPs) and nano-aluminum oxide (Nano-Al₂O₃) excel in high-temperature applications, but nano-titanium .

Table 2 delineated the economic and environmental

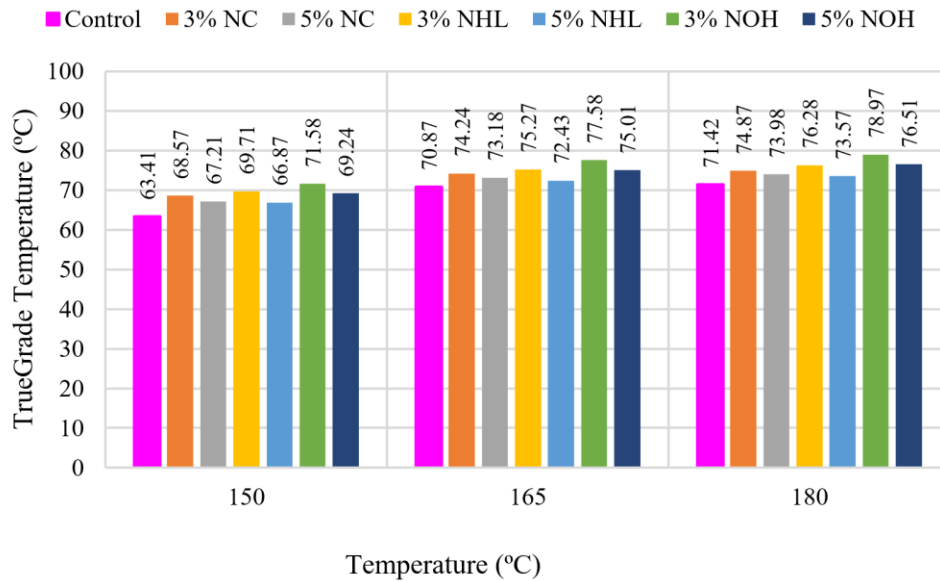


Fig. 5: Failure temperature of modified binders NC, NHL, and NOH at 150, 165, and 180 °C.

Table 2: The economic and environmental impact of using these modified asphalt binders versus traditional materials.

Factor	CNPs ^[41]	Nano-Al ₂ O ₃ ^[42]	Nano-TiO ₂ ^[42]	NC ^[28]	NHL ^[32]	NOH ^[30]
Initial cost	High due to complex production processes	Moderate to high cost due to production complexity	High, due to complex production and dispersion	Moderate cost, though, varies with type and supplier	Relatively moderate, cheaper than many other nano-modifiers.	Relatively low, particularly as a bio-based modifier
Environmental impact	Production can have a significant environmental impact; however, improved binder longevity may offset this.	Moderate environmental impact due to energy-intensive production	High carbon footprint from production but offers environmental benefits through UV degradation reduction	Improved sustainability compared to traditional additives, particularly when produced sustainably	Moderate environmental impact, though, provides long-term performance benefits and moisture resistance.	Environmentally friendly due to its bio-based nature, contributing to sustainability
Recyclability	Potential for improving recyclability of asphalt due to enhanced stability and longevity	Slight improvement in recyclability by enhancing the binder's resistance to degradation	It may improve recyclability by enhancing resistance to degradation and oxidative damage	Enhances recyclability by improving binder resistance to degradation	Improves recyclability by enhancing the binder's resistance to aging and degradation	Enhances recyclability due to improved binder stability and resistance to degradation
Cost-effectiveness	High initial cost but potentially cost-effective in the long term due to reduced maintenance and enhanced durability	Moderate cost with good long-term performance benefits, making it cost-effective in the right applications	High initial cost but durable and UV-resistant properties may provide long-term savings	More cost-effective compared to other nano-modifiers, especially for hot climates	Offers a good balance between cost and performance, with long-term benefits	Cost-effective due to its natural and renewable nature, providing long-term benefits

dioxide (Nano-TiO₂) offers unique environmental benefits. NC, NHL, and NOH provide more cost-effective and ecologically sustainable options, with NOH being especially advantageous due to its bio-based formulation. The choice of a modifier depends on specific performance goals, environmental conditions, and financial constraints.

4. Conclusion

The test findings of three different modified binders, NC, NHL, and NOH, were analyzed using a range of conventional and particular tests. Based on this analysis, the following conclusions may be made:

1. The inclusion of NC, NHL, and NOH resulted in a reduction in the phase angles of binders. Under the same conditions, the modified binder with NOH exhibited superior elastic recovery performance compared to the modified binders with NC and NHL. The use of modified binders comprising 5% NC significantly enhances the phase angle of aged asphalt in comparison to NHL and NOH.
2. The inclusion of NC, NHL, and NOH resulted in a reduction in the complex modulus of binders. Furthermore, the asphalt containing NOH exhibited superior resistance to flow deformation compared to the asphalt containing NC and NHL. The inclusion of 3% NC and NHL in the modified binders significantly reduces the complex modulus compared to modified binders containing 3% NOH.
3. Typically, the failure temperature rises with the inclusion of NC, NHL, and NOH. The modified binders, which include 3% NOH, may be considered the most effective dose compared to the Control, NC, and NHL binders.
4. As anticipated, the inclusion of NC, NHL, and NOH resulted in a decrease in the viscosity measurements of the modified binders in this investigation. However, this reduction was more pronounced when using the modified binder NOH.

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

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