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Effect of Organophilic Nano clay Modified Binder on High-Temperature Performance of Asphalt Mixture

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Abstract: Organophilic nano clay is the composition of mostly clay and organic molecules designed to improve various materials' physical and chemical properties. Permanent deformation in asphalt pavements is primarily the result of inadequate compaction, excessive loading, and high temperatures. Rutting can result in aquaplaning, leading to accidents and costly maintenance and rehabilitation works that drain the economy. Such behavior of asphalt pavements may be attributable to poor selection of aggregate, asphalt binder, or substandard asphalt binders. This study used different percentages of organophilic nano clay ranging from 3.0% - 5.0% with two penetration grade bitumen, i.e., N.R.L 60-70 & N.R.L 80-100. Adding organophilic nano clay to asphalt binder significantly reduced the rutting potential under cyclic loading at high temperatures (55 °C). Ten modified formulations and two virgin bitumen specimens were prepared. The modified binders' viscosity and rutting were tested using the Rotational Viscometer and Wheel Tracker Test (W.T.T.), respectively. The results were analyzed using software for Statistical Analysis. It was recorded that organophilic nano clay-modified bitumen significantly affected rutting to interpret the results. The results indicated that rutting decreased and viscosity increased in all nano clay-modified bitumen samples, with a 4.5% O.N.C modified binder showing the most significant improvement.

Keywords: binder modification; organophilic nano clay; rutting; viscosity; and wheel tracker test

1. Introduction

An asphalt mix is a composite material consisting of asphalt binder, i-e: bitumen, mineral aggregates, i-e coarse aggregate, fine aggregate, and mineral filler. While aggregates give an asphalt mix its load-bearing structure, the binder holds the particles together and subsequently gives the mix its tensile strength. A pavement is a multi-layered structure that rests on the soil's subgrade. In the last few decades, asphalt pavement has been widely used in constructing roads and highways [1]. As pavement is subjected to traffic loads, insufficient compaction of pavement layers, or temperature variations, permanent deformation may arise within the pavement or on its surface, such as rutting. Rutting usually occurs in both asphalt and the underlying unbound layers, but almost 85% - 95% of rutting accumulates in the asphaltic layers. Damage such as rutting would occur during the design life of asphalt pavement and at high temperatures. It would be dangerous to the users and produce a substantial amount of waste asphalt [2,3].

Rutting is the permanent deformation most dominant in the asphaltic course of a pavement or sometimes in the underlying layers, such as base or subgrade layers, caused by repeated action of traffic loads. Recent years have seen an increase in academic interest in the study of improving the resistance properties of raw virgin asphalt binders to pavement failures due to the growing emphasis on the sustainable development of resources, as well as the rising cost of asphalt binders and the

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scarcity of high-quality aggregates. [3–5]. After compaction, the coarse aggregate particles in an asphalt mix are tightly in contact with one another, thus forming a firmly bonded interlocked skeleton structure. This interlocked structure bears the loads applied by heavy traffic flow. The viscoelastic asphalt binder and mineral filler fill the voids between coarse aggregate. This results in a stable asphalt pavement structure [6].

Rutting deformation in an asphalt pavement can occur in 2 main modes: shear in one of the underlying layers or plastic flow in the Hot Mix Asphalt (H.M.A.) layer. According to (Button, Perdomo, and Lytton, 1990) [7], rutting is caused due to three different mechanisms; these are as follows: (1) consolidation or over-compaction of the pavement due to traffic volume or cyclic loads; (2) plastic deformation due to the instability of the mix; and finally, (3) instability, due to the stripping of binder underneath the surface course. Rutting typically occurs on pavement in three stages: (i) Primary rutting, also known as wear rutting. Traffic loads and environmental factors contribute to this; (ii) Secondary rutting, also known as structural rutting, results from the permanent deformation of underlying structural layers; and (iii) Tertiary rutting, also known as instability rutting, results from the lateral movement of materials within an asphalt pavement [8,9].

Bitumen modification is carried out to enhance its chemical, physical, and rheological properties to better resist the drastic effects of temperature variation, loading, and water damage on asphaltic concrete roads and to minimize road failures which may lead to rehabilitation and frequent maintenance, which would ultimately add towards spending extra capital. Researchers across the globe have been working tirelessly to innovate new materials capable of mating with bitumen to enhance their properties. Researchers and authors from Pakistan have also been involved in this field of bitumen modification, as Pakistan is amongst the most severely affected countries regarding road failures. In their study, Aman et al., 2020 [4] explore the effect of adding a phosphorous methyl compound (P.M.C.) to asphalt binder. The study involved preparing asphalt binder samples with varying concentrations of P.M.C. and conducting a range of laboratory tests to evaluate the physical and rheological properties of the samples. According to the findings, incorporating P.M.C. into the asphalt binder improved some of its essential attributes, including its point of softening, penetration, and ductility. The author proposes that adding polymer-modified bitumen, or P.M.C., to asphalt binder may have substantial consequences for designing and constructing asphalt pavements. It is because using P.M.C. as an additive in asphalt binder may improve the pavement's durability and resistance to many different types of distress, such as cracking and rutting.

In another research by Irfan et al., 2018 [10], the authors analyze how asphalt mixtures amended with crumb rubber were performed in a laboratory and actual at-site settings. As part of the study, laboratory testing on crumb rubber-modified asphalt mixes were carried out. These tests included evaluations of the mixture's physical qualities, as well as evaluations of its rutting and fatigue resistance. The authors also conducted field testing on portions of pavement containing crumb rubber-modified asphalt, employing several performance parameters to evaluate the results. Compared to unmodified mixes, the results showed that crumb rubber-modified asphalt mixtures enhanced physical qualities. When tested in the laboratory, these mixtures also exhibited improved rutting and fatigue resistance. In the field testing, the sections of the pavement that were changed with crumb rubber exhibited significantly less cracking and rutting than the sections that used a mathematical model that can reliably forecast the rut depth in asphalt pavements based on the data obtained from unmodified asphalt.

According to Authors Hussan et al., 2020 [11], authors have recently published research works that have effectively devised various laboratory tests. The researchers evaluated several asphalt mixtures in the laboratory using a variety of procedures, such as the Marshall Stability test, the Dynamic Modulus test, the Indirect Tensile Strength test, and the Wheel Tracking test. After collecting data on the rut depth observed in each test, they analyzed the correlation between the several test findings. The research showed that there is a connection between the depth of the ruts that were seen in the various performance tests. The researchers developed a mathematical model incorporating test parameters to predict the rut depth. The model provides a valuable tool for assessing the rutting potential of asphalt mixtures and can aid in designing and evaluating durable asphalt pavements. The study highlights the importance of considering multiple performance tests to evaluate the rutting resistance of asphalt mixtures. By showing correlations between different test results, engineers and researchers can gain valuable insights into the performance of asphalt pavements and make informed decisions regarding pavement design and material selection.

However, due to the asphalt mixture's viscoelastic plasticity under high temperatures combined with the pavement's vehicular load, the asphalt mortar and coarse aggregate flows out of the skeleton gap resulting in destabilization of the asphalt mixture's structure, which results in rutting to accumulate. Nanomaterials are progressively adopted to improve the resistance of modern asphalt pavement against rutting. At high temperatures, to enhance the performance of virgin asphalt binder, nanomaterials such as nano clay [12], carbon nanotubes [13–15], nano silica [16], carbon black [17], and graphene [18,19] are used.

With advancements and research in nanotechnology, a new material comprising layered silicate nanoparticles, called Nano clay, became an additive in asphalt [20]. Nano clay is an organic clay that is nanometer-sized. It comprises clay particles that are less than 100 nanometers in size. Nano clay is utilized in various products such as food, pharmaceuticals, and cosmetics. It can also be used as a filler in plastics and rubbers. Nano clay can improve these materials' strength, stiffness, and heat resistance. Nano clay is also studied for batteries, fuel, and solar cells. Nano clay is an agglomerated clay where, for 50% or more of the particles in the number size distribution, one or more external dimensions is in the size range of 1 nm – 100 nm [21,22].

Nano clay Modified bitumen composition has been successfully used to improve the physical and rheological properties as well as the performance of bitumen [23]. According to the findings of (Fu et al., 2004; Hanyu et al., 2005; Chen and Huang, 2007; Yildirim, 2007) [24–27], using nano clay as a modifier to improve the performance of S.B.S. (Styrene–Butadiene–Styrene) polymer-modified asphalt by adding Organophilic Montmorillonite (OMMT) nano clays, it was found that the viscosity of the SBS-modified asphalt as well as the stiffness (complex modulus) increased while the phase angle decreased. These outcomes confirmed that Organophilic Montmorillonite (OMMT) nano clays could reduce permanent deformation (rutting) in pavements, especially in the asphaltic layers.

Pakistan faces significant pavement failure due to excessive rutting because of high temperatures [28–31]. Many urban highways have excessive rutting due to high temperatures and excessive loading. Repair and maintenance work leads to traffic congestion and choke points at multiple work points. Traffic hazards due to loss of vehicle control and stability may lead to loss of life and damage to property. Constructing roads requiring minimal maintenance and rehabilitation work reduces rehabilitation costs, time, and resources. The purpose is to make a much safer and more reliable mode of transportation by reducing road accidents due to aquaplaning. By using modified binders and mixtures, the increase in resistance to such failures can help increase the life of pavements [32,33].

Researchers in this era are thriving in developing new materials which are not only environmentally friendly but are cost-effective to improve the properties of bitumen; such materials include Nano clay. Pakistan has abundant raw materials for road construction, but to optimize organophilic nano clay to be used as an effective modifier to enhance the binder properties, testing for its compatibility with locally abundant materials is required, which is targeted in this study. In previous research studies, authors have used different organic and inorganic compounds as additives to determine their impact on rutting in asphalt [14,16,18,32,33]. Still, organophilic nano clay was confined to measuring the rheological properties only [34–36] instead of its rut resistance capabilities. After thorough research, it was concluded that limited studies on organophilic nano clay in Pakistan were available to enhance the rut resistance properties at high temperatures [37].

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The desired state for this study is to design an asphalt mix that can reduce failure in roads, primarily rutting. Also, there is only a tiny amount of information available; further research must be conducted before these alterations can be implemented in real-world settings because there is insufficient information on how these modifications improve rutting resistance in asphalt mixtures. Therefore, the research study was shaped to optimize the beneficial properties of organophilic nano clay with the locally available material in Pakistan to reduce rutting in asphaltic pavements. In this research, different percentages of nano clay ranging from 3.0%, 3.5%, 4.0%, 4.5%, and 5.0% of the total weight of bitumen in asphalt mix were used to make multiple molds or cakes of asphalt. The goal was to create an asphaltic mix comprising nano clay that would adhere to the binder at optimum nano clay content without compromising the rheological performance. The NC-modified asphalt mixtures were tested using a wheel tracker apparatus to measure permanent deformation/rutting and find the influence of nano clay on reducing rutting depth. After running the Cooper wheel tracker test, the target was determining the nano clay percentage yielding the least rutting in the asphalt cake/molds. Investigations were accompanied by multiple statistical tests to fortify and validate the authenticity of the results.

2. Materials and Methods

This section uses the materials, procedures, and methodologies discussed in various tests to achieve the targeted goals/ objectives. An organophilic nano clay modified binder is developed using raw bitumen and adding nano clay; different samples of modified asphalt were then made to conduct tests. Afterward, a detailed study, analysis of results, comparison of any change or improvement in performance properties, and rheology of the binder will be discussed. Different test protocols of ASTM were adopted, and test conditions were kept similar and constant across all samples. The behavior of raw virgin binder was evaluated by conducting tests mentioned below according to ASTM specifications to evaluate its nature. The binders that passed the tests were then used for modified asphalt sample preparation. Figure 1 illustrates that Phase – 1 consisted of a collection of materials. N.R.L 60/70 and N.R.L 80/100 Bitumen was used as an asphalt binder. The aggregate was sourced from Khanpur Crush in KPK province, Pakistan. Organophilic nano clay was used as a modifier. In Phase – II, specimens were prepared for conventional and mixture tests to check the properties of selected asphalt binder. Results of conventional tests on asphalt binder have been given in Table 1. In Phase – III, mixture tests were run on the specimens, including modified and virgin N.R.L 60/70 and N.R.L 80/100.

Table 1. Conventional Binder Test Results.

Test Name	Standard	Binder (N.R.L 60/70)	Binder (N.R.L 80/100)	Range
Softening point (°C)	ASTM D36	46	44	46-54
Penetration Test (0.1mm)	ASTM D5	63	89	60/100

2.1. Materials

To start the experimental stage of this research study, materials that were used were selected. The main ingredients used were Asphalt Binder, Aggregates, and Modifier Organophilic Nano clay, as illustrated in Figure 2 (a), (b), and (c), respectively. These materials are thoroughly discussed below.

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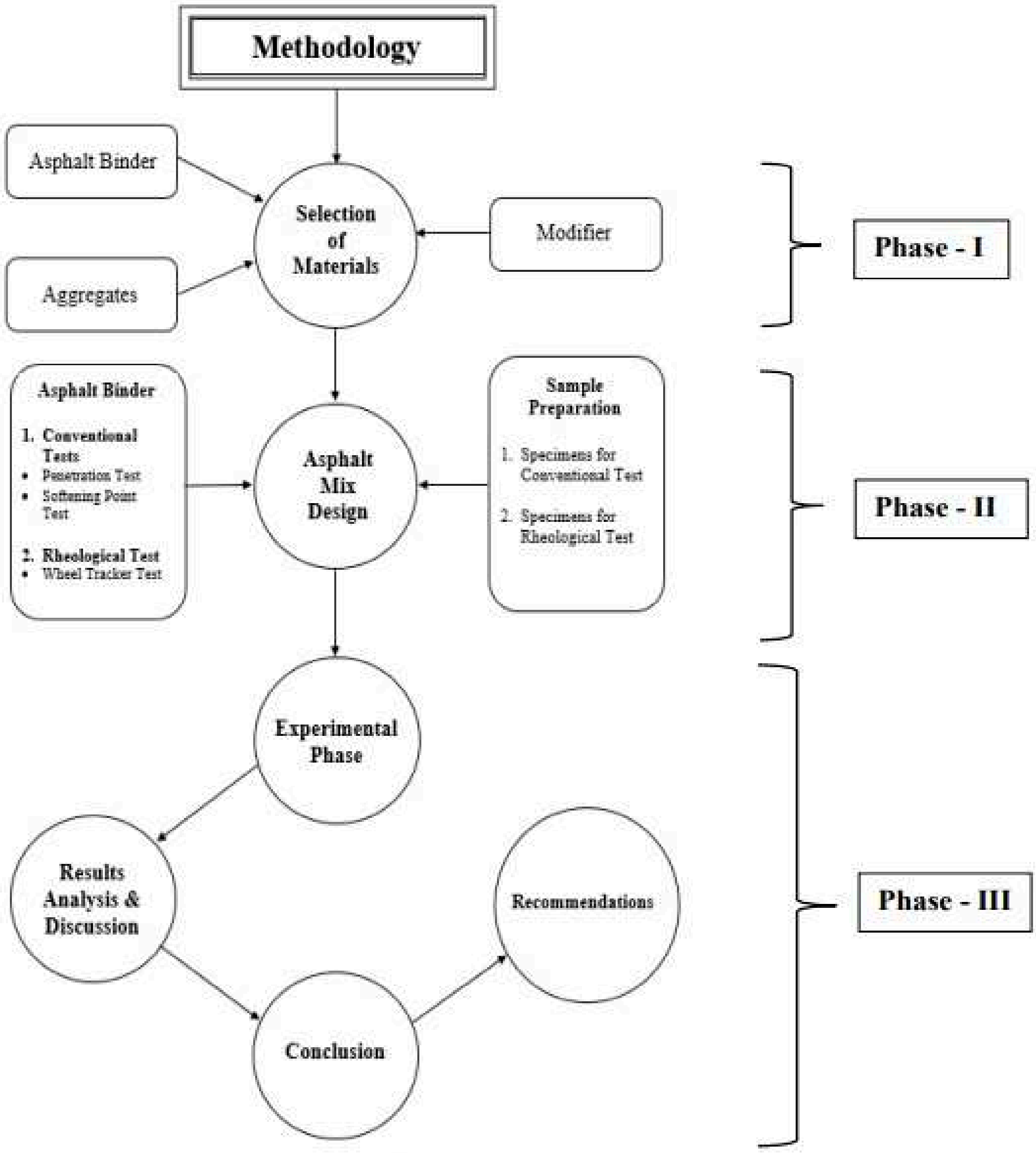

Figure 1. An experimental program was adopted for the research study.

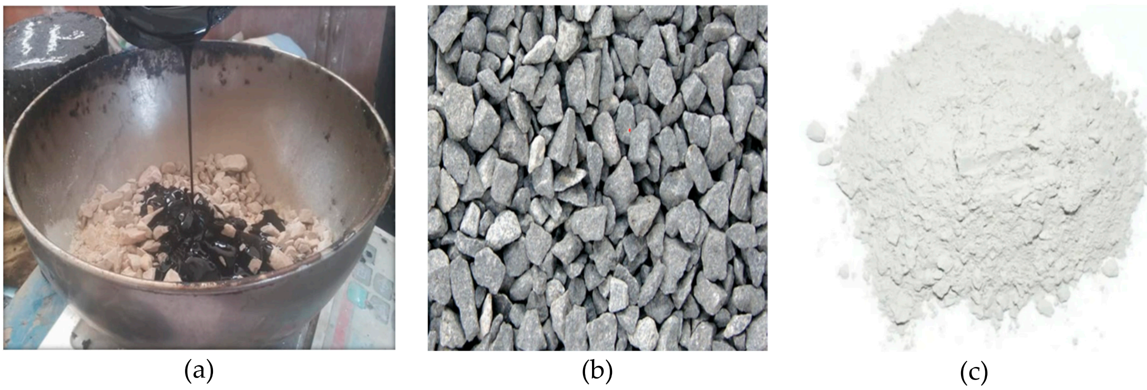

Figure 2. (a) Bitumen, (b)Khanpur crush, and (c) Organophilic Nano clay.

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2.1.1. Aggregates

An aggregate quarry was selected as per the petrography of the rock/ aggregate. The nature of an aggregate, whether acidic or basic, depends upon the amount of SiO_2 in its chemical composition. A source of rock with higher silica content, including biotite, feldspar, or quartz, tends to be acidic. Similarly, a source of rock with lower silica content, including pyroxene and olivine, tends to be basic. The nearest source of aggregate, i.e., Khanpur Crush, was selected. Khanpur crush tends to be basic due to the presence of Calcium Carbonate, $CaCO_3$. The location of the Khanpur crush quarry is pointed out in Figure 3(a).

(a) (b)

Figure 3. (a) Location of Khanpur Crush plant, (b) Location of source of Aggregates.

Khanpur is located in the Haripur district of the province of Khyber Pakhtunkhwa, coordinates of the site of the aggregate source are $33°50'15"N,\ 72°54'56"E$. Figure 3(a) and (b) were developed and edited using google earth. The aggregates were sieved, and the results are shown in Table 2.

Table 2. Aggregate gradation after sieve analysis.

Sieve Size	Passing %	Tolerance	N.H.A. Specifications
19 mm	100	± 7	100
12.50 mm	76.9	± 7	75-90
9.50 mm	62-76	± 7	60-80
#4	43-51	± 4	40-60
#8	31-39	± 4	20-40
#50	7-15	± 4	5-15
#200	4-6	± 1	3-8

Specimens for molds used in this research study were prepared using Bailey gradation, shown in Figure 4. It may be noted that the aggregate gradation used in this research study for specimen preparation is well-graded. The particle size distribution of aggregates is such that small or fine particles would fill gaps or voids amongst the larger particles/ aggregates.

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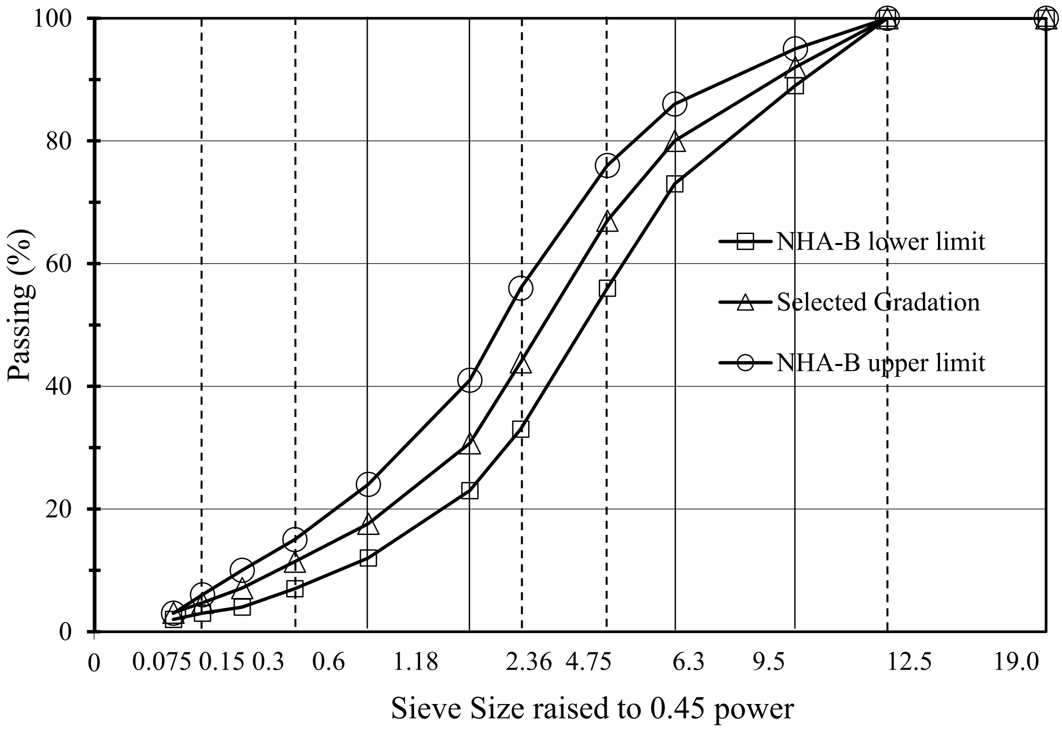

Figure 4. Aggregate Gradation Curve using Bailey method of gradation.

The elemental composition of the Khanpur aggregate quarry is presented in Table 3. The data was obtained by Geoservices engineering consultants located in Rawalpindi, Punjab, Pakistan. The ASTM C295 standard guide for the petrographic examination of aggregates was followed To perform the petrographic analysis of the sample.

Table 3. Elemental Composition of Khanpur Aggregate.

Name of element	Percentage %
Dark Grey Limestone	31
Light Grey Limestone	13
Dark Grey to Grey Veined Limestone	8
Cremish Brown limestone	3
Quartzwacke	2
Grey Limestone	43

Physical tests were conducted according to ASTM and B.S. Standards to conclude the properties of the aggregate to access the physical and mechanical properties of aggregates. The results obtained from these tests are provided in Table 3. It may be noted that the results of aggregates' physical and mechanical properties lie within the bounds of British Standards (B.S.) and ASTM. It shows that the source of aggregates is good and can be relied upon for further use in testing as per ASTM Standards.

Table 4. Physical properties of Aggregates.

Sr. #	Properties of aggregate	BS/ ASTM Standards	Results	Test Limits
1	Flakiness Index (%)	BS 812.108	5.25	10 (max)
2	Elongation index (%)	BS 812.109	5.8	10 (max)
3	Water Absorption (%)	ASTM C127	1.32	3 (max)
4	Specific Gravity	ASTM C128	2.83	3.0 (max)
5	Bulk Density (kg/m3)	ASTM D1895	1508.0	1750 (max)
6	Voids (%)	ASTM D3203	44.56	45 (max)
7	Impact Value (%)	ASTM C125	16.50	20 (max)
8	Crushing Value (%)	ASTM D5821	29.80	30 (max)

2.1.2. Asphalt Binder

Bitumen was used as a binder in this study. National Refinery Limited bitumen was chosen for this research study as N.R.L. bitumen is one of Pakistan's most widely used bitumen. N.R.L grade 60/70 and N.R.L grade 80/100 bitumen were used to make specimens and conduct testing. Both the grades of bitumen, N.R.L 60/70 and N.R.L 80/100 were sourced locally. Penetration tests and softening point tests were conducted on the bitumen sample to assess the physical properties of this bitumen. Both the asphalt binders were modified for analyses of mixture tests.

The first test conducted was to determine the penetration value of the bitumen. For this, a penetration test was executed using a penetrometer. The uniformity of a bituminous material is expressed as the distance in tenths of a millimeter that a standard needle vertically penetrates a sample of the bitumen under known loading, time, and temperature conditions. For each grade of bitumen, the test was repeated on the same sample at different locations using the same lab conditions. The results presented in Table 5 were very accurate for both the bitumen grades, N.R.L grade 60/70 and N.R.L grade 80/100. These results showed that both grades had a uniform consistency and that both samples could be used in this study for consistent and accurate results during further testing.

Table 5. Penetration Test and Softening Point Results for Virgin and Modified N.R.L 60/70 and N.R.L 80/100 Binder.

Properties	Unit	Virgin		Modified		Specification Limit (minimum)
		N.R.L 60/70	N.R.L 80/100	N.R.L 60/70	N.R.L 80/100	
Penetration 0.1 mm @ 25 °C	$\frac{1}{10}$mm	61	82	69	89	60/80
Softening point (°C)	°C	46	49	41	44	43

The second test was carried out to assess the softening point of both the bitumen grades using ring and ball apparatus. In Softening Point Test, the mean temperature at which disks of bitumen soften and sags downward for a distance of 25 mm under the weight of a steel ball is noted. This test method covers the determination of the softening point of bitumen in the range from 30 °C to 157 °C using the ring-and-ball apparatus immersed in distilled water from 30 °C to 80 °C. The results of Table 5 were analyzed after conducting tests on both the bitumen grades. It was determined that for both the penetration grades of bitumen, the results were very consistent and fell in the pre-specified ranges of ASTM, referring that the bitumen grades were free of contaminants and usage of it in this study would provide valid results rather than inaccurate results.

2.1.3. Asphalt Modifier

Organophilic nano clay was used as a modifier in this study. The nano clay was sourced by importing it from China. In its physical form, organophilic nano clay is a white powder-like clay. Figure 5 shows a sample of Organophilic Nano clay in a laboratory before being added to bitumen.

Figure 5. Organophilic Nano clay.

Nano clay comprises nanoparticles of layered mineral silicates [38]. Organophilic nano clays are clay minerals chemically modified with organic cations to make them more compatible with organic materials, such as polymers, resins, and oils [39]. Some fundamental properties of organophilic nano clays include Hydrophobicity and Thixotrophy [40]. Organophilic nano clays are hydrophobic, repelling water and other polar substances. Increased compatibility: Adding organic cations to the clay structure increases its compatibility with organic materials, allowing for improved dispersion and intercalation. Improved mechanical properties: Organophilic nano clays can improve the mechanical properties of polymers and other materials, such as tensile strength, modulus, and impact resistance [41]. Increased barrier properties: Adding organophilic nano clays can improve the barrier properties of polymers and other materials, such as gas permeability and water vapor transmission [42].

Organophilic nano clays can exhibit thixotropic behavior, meaning their viscosity can change with time and applied stress. High surface area: The high surface area of organophilic nano clays allows for increased interaction with other materials and can facilitate chemical reactions and adsorption. Compatibility with different organic systems: Organophilic nano clays are compatible with various organic systems, including thermoplastics, thermosets, coatings, and adhesives [43].

2.2. Asphalt mix design and specimen preparation

Materials required to prepare the test specimens were sourced locally and from China. The aggregates were sourced from the Khanpur quarry, and physical tests were conducted. Asphalt binders of grades PG 60/70 and P.G. 80/100 were sourced from National Refinery Limited. Organophilic nano clay was sourced from China. After heating the raw bitumen to a temperature of 120 °C, it was added to the mixing can to mix it thoroughly by high shear mixer so that all the bitumen could adhere to the nano clay completely at 2800 rpm for 60 minutes [44]. The coarse aggregate was heated before adding modified bitumen, enabling the aggregate to mix and bind to the bitumen completely. Twelve molds were made; out of these twelve molds, six were made from N.R.L 60/70 grade bitumen. Amongst these six samples, five were made up of modified bitumen, and modification was done by adding 3.0%, 3.5%, 4.0%, 4.5%, and 5.0% organophilic nano clay by total weight of bitumen.

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The sixth sample was made as a test sample of raw virgin N.R.L 60/70 grade bitumen, and the results of all the modified samples were compared to the virgin sample, and analysis was done. Similarly, the same procedure was repeated for N.R.L 80/100-grade bitumen. A total of six samples were made; five of these six samples were made up of modified bitumen, and modification was done by adding 3.0%, 3.5%, 4.0%, 4.5%, and 5.0% organophilic nano clay by total weight of bitumen. The sixth sample was made as a test sample of raw virgin N.R.L 80/100-grade bitumen, and the results of all the modified samples were compared to the virgin sample, and analysis was done. After making all the samples, each was assigned an I.D. tag for easier identification, shown below in Table 6.

Table 6. Identification Numbers of different bitumen samples.

Sampling Type	Respective ID
Organophilic Nano clay Modified binder	ONMB
Penetration Grade 60/70 Bitumen	N.R.L 60/70
Penetration Grade 80/100 Bitumen	N.R.L 80/100
Asphalt Binder containing 3.0% nano clay	3.0% N.C
Asphalt Binder containing 3.5% nano clay	3.5% N.C
Asphalt Binder containing 4.0% nano clay	4.0% N.C
Asphalt Binder containing 4.5% nano clay	4.5% N.C
Asphalt Binder containing 5.0% nano clay	5.0% N.C

Optimum binder content graphs of all modified asphalt samples have been given in Annexure. The volumetric properties of asphalt mixtures have been given in Table 7. It may be noted from Table 7 that the percentage of air voids (VA%) and voids in minerals aggregates (VMA%) are within the limitation values. Only voids filled with asphalt (VFA%) of the N.R.L 60/70 modified bitumen and the N.R.L 80/100 modified bitumen are less than 65%. Marshall method was used to mix modified bitumen with aggregate. The optimum bitumen content for neat bitumen 60/70 and 80/100 was 4.5%, and for comparison purposes, the same OBC % was selected for each NC-modified mixture. An experimental program was established to track the research and achieve the target goal at each step.

Table 7. Volumetric Properties of modified asphalt mixtures.

Type of Mix	Aggregate Source	VA (%)	VMA (%)	VFA (%)	Modifiers quantity by the weight of asphalt binder	Mixing Method
---	---	Limits 04 - 07	14 (Min.)	Limits 65 - 75	---	---
O.N.C. modified Mix N.R.L 60/70	Khanpur Crush	5.22	14.76	64.63	3% - 5%	Marshal method
O.N.C. modified Mix N.R.L 80/100	Khanpur Crush	6.43	16.57	61.2	3% - 5%	Marshal method

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2.3. Test Methods

Test Methods are divided into two categories, i-e., Conventional Testing, which includes Standard Penetration Test and Softening Point Test, and the second testing category, Mixture testing, which includes Cooper Wheel Tracker Test.

2.3.1. Rotational Viscometer Test

The rheology of the virgin bitumen pen grade 60/70 and 80/100 and modified bitumen with nano clay was determined using Rotational Viscometer, as illustrated in Figure 6. The sample is sheared at a constant strain rate and temperature. Compared to other technologies, the 135 °C (275 °F) temperature used by rotational viscometers is more accurate and representative of asphalt laying field temperatures. A binder's mixing and compaction viscosity can be determined using R.V. testing per the AASHTO TP48 standard to calculate the bitumen spinning viscometer values. Using a Brookfield rotating rheometer, bitumen's viscosity is tested. During the experiment, the temperature was maintained at 135 °C.

Figure 6. Rotational Viscometer (R.V.) apparatus.

The original/modified binder was poured into the sample chamber and placed in a thermoset. Then the spindle moves down into the sample chamber and ensures that the tip of the spindle does not touch the bottom of the chamber. Now set out the required temperature and set the torque. After 30 minutes, the thermoset achieved the critical temperature. The sample must be maintained for 10 minutes at the test temperature for equilibrium. The spindle is then rotated for 10 minutes. After that, bypassing every minute, three consecutive readings are taken, then take their average.

2.3.2. Cooper Wheel Tracker Test (W.T.T.)

The Wheel tracker test assesses the resistance to permanent deformations such as rutting of asphalt mixtures following BS EN 12697-22: 2003 [45], as illustrated in Figure 7. Superpave gyratory compacted samples (150 mm diameter, 50 mm height) were prepared in the laboratory. The W.T.T. device is an electrically powered machine that can move a rubber wheel over test specimens. A wheel with a 705.0 ± 4.5 N load passes at 50 - 54 passes per/minute. The specimens were tested at a constant temperature of 55 °C, and 10,000 wheel passes at 53 passes/ minute or 26.5 cycles/ minute were made on both the virgin and modified asphalt mixtures. The mold height was 50 mm after compacting each mold with a Superpave gyratory compactor. The low rut depth obtained from the W.T.T. apparatus indicates the mixture has high resistance against rutting.

Figure 7. Wheel Tracker Test Apparatus.

3. Results

In this chapter, results obtained from all the tests, conventional and mixture tests, conducted in the laboratory on both the bitumen grades, N.R.L 60/70 and N.R.L 80/100, as well as the molds/ cakes of modified binder, will be thoroughly analyzed and interpreted and discussed in extensive detail. Firstly, the physical property of bitumen is analyzed using the results obtained from conventional tests, such as softening point and penetration tests. These tests will confirm the consistency and purity of the bitumen samples and ensure consistent results during further use in this study. Secondly, the mechanical behavior of both virgin and modified mixtures is analyzed through the results obtained from mixture tests such as the Cooper wheel tracker test. The results of each research phase are discussed in detail after analyzing the graphical representation of data.

3.1. Effect of Nano clay on Binder

The discussion is broken down into the following categories to analyze different parameters of nano clay and its effect on bitumen.

3.2. Effect of Nano clay on Binder Rheology

The two binder grades incorporate Different percentages (3.0%, 3.5%, 4%, 4.5%, and 5%) of organophilic Nano Clay. The nano clay is thoroughly mixed with bitumen at the specified temperature, and modified asphalt is prepared. Continuous stirring is carried out to ensure uniform dispersion of Nano Clay. Samples from virgin bitumen are also prepared to compare the rheological results with the modified mix, as presented in Table 8. The absolute viscosity of modified and virgin bitumen is determined. By analyzing Figure 8, it can be presumed that with an increase in nano clay, from 3.0% to 4.5% in 60/70 pen grade bitumen, the modified bitumen's viscosity increases, and the average torque decreases consistently.

Table 8. Impact of Nano clay on viscosity and torque of Pen 60/70 and 80/100-grade bitumen.

Pen 60/70				Pen 80/100		
Sr. No	**Nano Clay %**	**Average Viscosity**	**Average torque (%)**	**Nano Clay %**	**Average Viscosity**	**Average torque (%)**
1	0	353.3	4.7	0	342.3	3.5
2	3	391.6	4.5	3	394.6	2.7
3	3.5	417.9	4.2	3.5	417.5	2.1
4	4	456.6	3.7	4	434.8	1.5
5	4.5	495.2	2.9	4.5	463.9	1.2
6	5	488.7	3.2	5	456.2	1.3

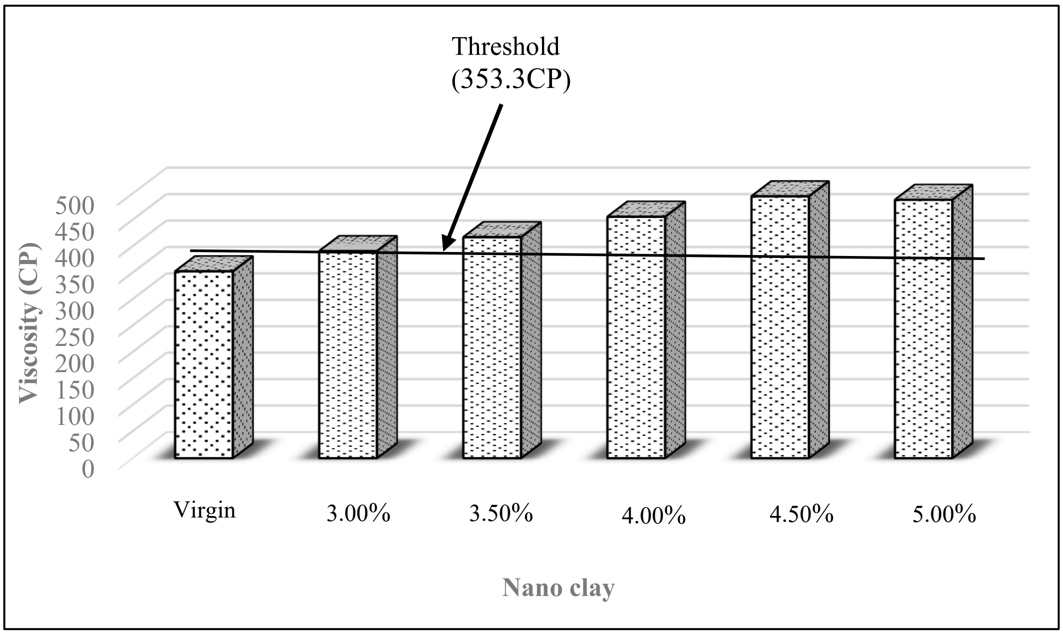

Figure 8. Impact of Nano clay on the viscosity of virgin and modified PG 60/70 bitumen.

Figure 8 illustrates a decreasing trend at 5.0% nano clay content. The threshold value denotes the performance of the virgin binder without any modification. Comparing the results of the modified binders to that of virgin binder by analyzing the threshold value, it may be concluded that the viscosity of modified binder samples (From 3.00% To 5.00%) had improved significantly, with the maximum increase seen on 4.50% N.C sample. Organophilic Nano clay particles, because of their nanoscale size, have a very high surface area to volume ratio. These particles have the potential to interact with the molecules of the bitumen when they are disseminated in a liquid medium like bitumen, which could increase the number of entanglements and interactions. Because of this, a three-dimensional network structure is created, contributing to the material's increased viscosity. Organophilic Nano clay particles, because of their nanoscale size, have a very high surface area to volume ratio. These particles have the potential to interact with the molecules of the bitumen when they are disseminated in a liquid medium like bitumen, which could increase the number of entanglements and interactions. Because of this, a three-dimensional network structure is created, contributing to the material's increased viscosity [39,46].

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Similar results can be concluded for P.G. 80/100 bitumen by analyzing Figure 9 compared to PG 60/70 bitumen. Virgin binder shows the most negligible viscosity; by addition of Nano clay, viscosity increases, and the average torque decreases. These similar results show the compatibility of nano clay with bitumen and its positive effects. Similar results trends are reported to the previous findings [34–36].

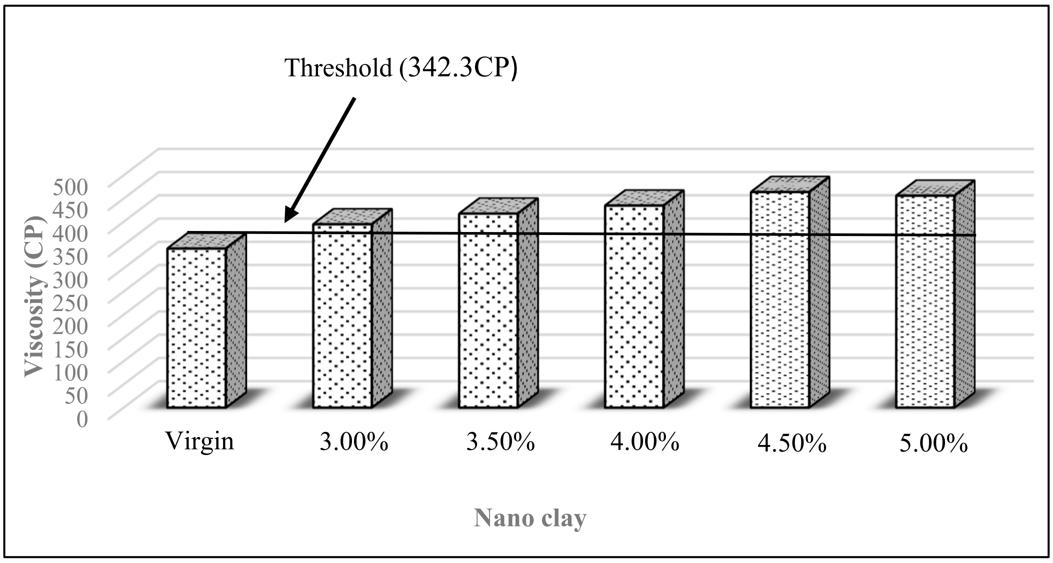

Figure 9. Impact of Nano clay on the viscosity of virgin and modified P.G. 80/100 bitumen.

3.3. *Effect of Nano clay on Rut Resistance*

A total of ten (10) tests were conducted on Organophilic nano clay-modified Bitumen with Nano clay percentages ranging from 3.0% - 5.0%, and two tests were conducted on raw virgin bitumen PG 60/70 and P.G. 80/100, as illustrated in Figures 10 and 11. The tests were conducted at 55°C, and the total number of passes of W.T.T. was 10,000. Each mold was compacted using a Superpave gyratory compactor and trimmed to a height of 50 mm. The test was initiated, and the results showed that Organophilic nano clay modified bitumen had much more excellent rut resistance, especially at the advanced stages of the test, as compared to raw virgin bitumen. For both the binder grades, rutting was noted to decrease exceptionally.

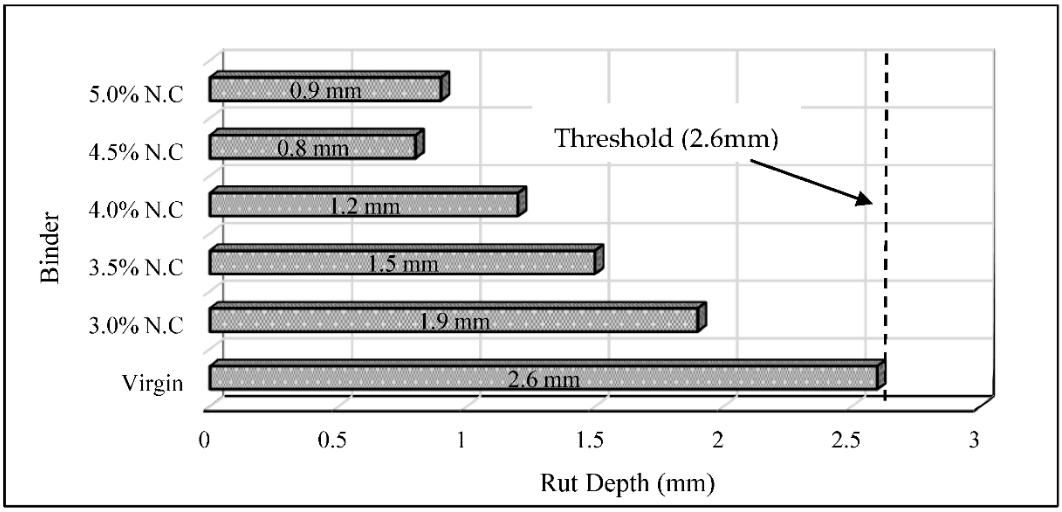

Figure 10. Impact of PG 60/70 grade O.N.C modified binder on Rut Depth.

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For PG 60/70 Grade bitumen, the result showed a maximum decrease in rut resistance at 4.5% nano clay content, which was 0.8 mm compared to the virgin's 2.6 mm, a decrease of 69.23% in rutting, as illustrated in Figure 10. The results for P.G. 80/100 were almost similar, with a maximum decrease in rutting of 67.64%. The maximum decrease in rut resistance was again noted at 4.5% nano clay, which was 1.1 mm compared to the virgin's 3.4 mm, as shown in Figure 11. It may be noted from Figures 10 and 11 the threshold values for both the binder grades. The threshold value for N.R.L 60/70 is 2.6 mm. It shows the rutting accumulated in virgin asphalt mix, compared to the modified mixes with organophilic nano clay. The threshold value for N.R.L 80/100 is 3.4 mm. Asphalt mixtures that contain organophilic micro clay are less susceptible to temperature variations. It indicates that the mixtures are less affected by high swings in temperature, which might assist in alleviating rutting concerns brought on by temperature-induced changes in the stiffness of the asphalt [36,37,47]. It shows the rutting accumulated in virgin asphalt mix, compared to the modified mixes with organophilic nano clay.

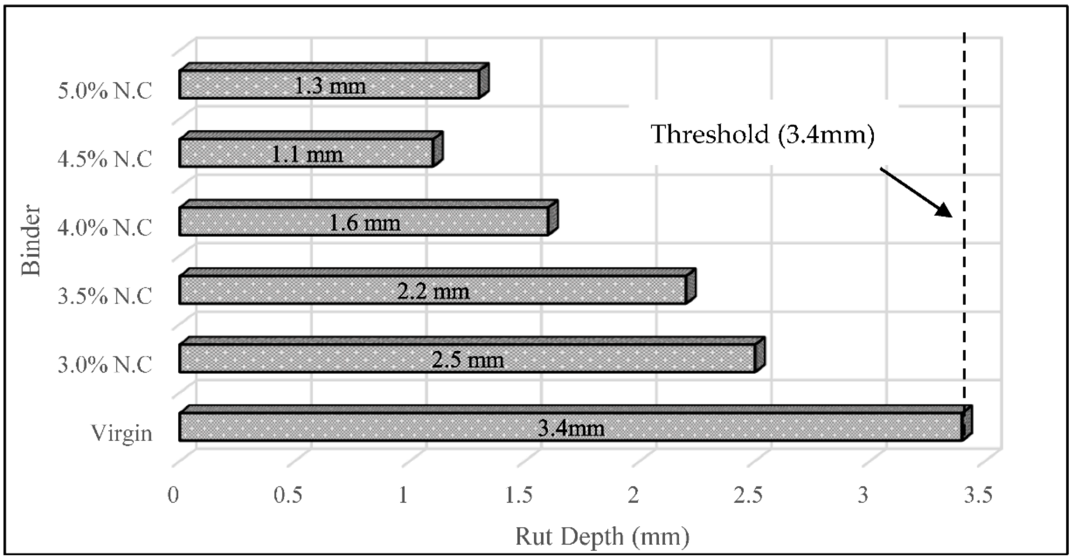

Figure 11. Impact of P.G. 80/100-grade O.N.C modified binder on Rut Depth.

3.4. Effect of Wheel Tracker Test Passes on asphalt mixes

The tests were run under similar conditions, keeping the temperature constant at 55 °C, the mold height at 50 mm, and the number of passes at 10,000. A relationship between the number of passes of wheel tracker apparatus on the molds, both modified and unmodified for the two binder grades, and the amount of rutting accumulated in the molds were developed to identify the optimum nano clay percentage that produced the best results when added to virgin bitumen. A scatter diagram for each binder grade was developed, showing the effects of passes on virgin and modified bitumen to achieve this. The results are shown in Figure 12 for N.R.L 60/70 grade bitumen. Test data of N.R.L 60/70 and 80/100 modified asphalt mixtures are given in Table 9.

Table 9. Test data obtained after completion of 10,000 passes for N.R.L 60/70 and 80/100.

	N.R.L 60/70 with N.C						N.R.L 80/100 with N.C					
No. of Passes	3	3.5	4	4.5	5	Virgin	3	3.5	4	4.5	5	Virgin
0	0	0	0	0	0	0	0	0	0	0	0	0
500	0.31	0.28	0.27	0.2	0.11	0.5	0.39	0.35	0.27	0.2	0.11	0.49
1000	0.59	0.52	0.49	0.36	0.22	0.91	0.71	0.64	0.49	0.36	0.22	0.89
1500	0.8	0.7	0.6	0.41	0.29	1.13	0.88	0.78	0.6	0.41	0.29	1.09
2000	0.97	0.82	0.68	0.43	0.32	1.26	0.97	0.84	0.68	0.43	0.3	1.18
2500	1.09	0.91	0.75	0.47	0.38	1.37	1.09	0.91	0.75	0.47	0.31	1.29
3000	1.18	0.99	0.79	0.49	0.39	1.45	1.18	0.99	0.79	0.49	0.35	1.35

3500	1.27	1.05	0.81	0.51	0.41	1.52	1.28	1.05	0.81	0.5	0.38	1.4
4000	1.33	1.11	0.85	0.55	0.43	1.59	1.33	1.11	0.85	0.52	0.4	1.47
4500	1.39	1.15	0.89	0.59	0.45	1.68	1.4	1.14	0.89	0.55	0.44	1.55
5000	1.45	1.17	0.91	0.6	0.47	1.74	1.46	1.17	0.91	0.6	0.49	1.66
5500	2.02	1.21	0.95	0.64	0.51	1.81	1.51	1.21	0.95	0.62	0.51	1.72
6000	1.59	1.26	0.98	0.67	0.56	1.89	1.59	1.26	0.98	0.67	0.57	1.81
6500	1.65	1.29	1.1	0.7	0.6	1.97	1.62	1.29	1.1	0.7	0.61	1.9
7000	1.69	1.32	1.05	0.71	0.62	2.05	1.69	1.32	1.05	0.71	0.68	1.99
7500	1.71	1.35	1.09	0.74	0.65	2.15	1.71	1.35	1.09	0.74	0.78	2.1
8000	1.77	1.39	1.11	0.76	0.69	2.25	1.77	1.39	1.11	0.76	0.86	2.21
8500	1.8	1.42	1.15	0.79	0.7	2.35	1.85	1.48	1.12	0.79	0.92	2.36
9000	1.84	1.46	1.17	0.8	0.74	2.43	1.99	1.6	1.17	0.81	1.01	2.59
9500	1.89	1.49	1.19	0.8	0.82	2.51	2.21	1.89	1.38	0.94	1.14	2.95
10000	1.93	1.5	1.2	0.81	0.93	2.6	2.5	2.2	1.6	1.1	1.3	3.4

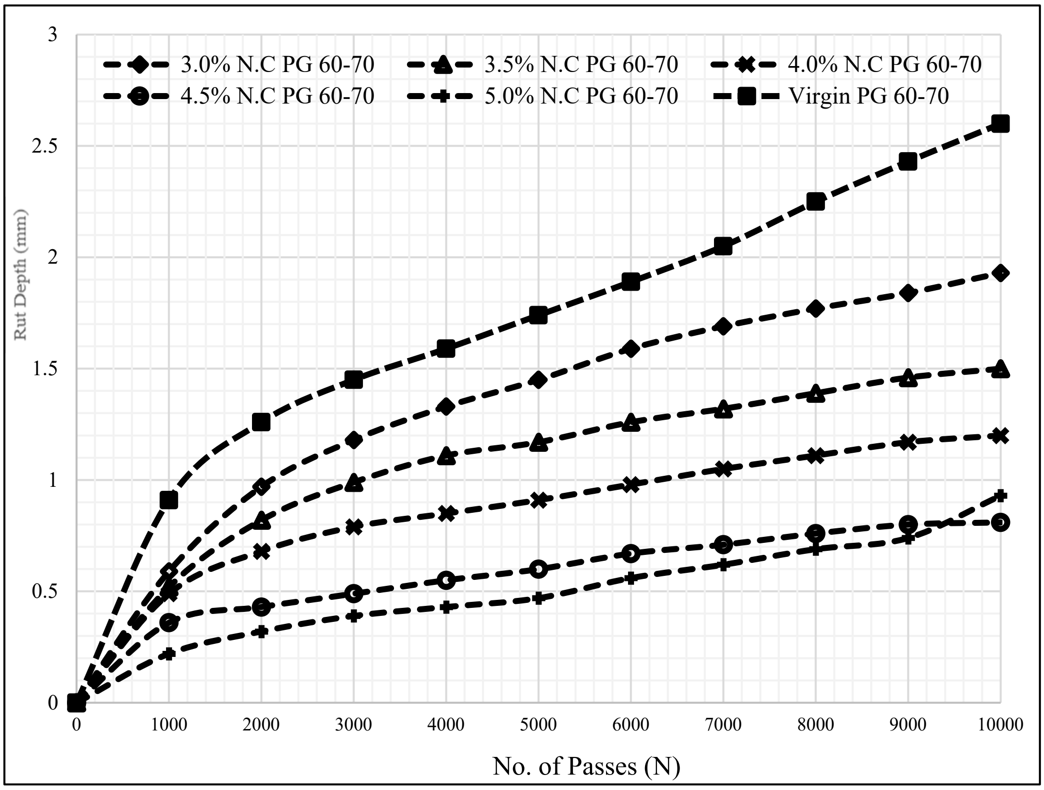

Figure 12. Effect of load cycles on Rut Depth of PG 60/70 bitumen.

It may be noted from Figure 12 that as the test progresses, induced rutting also increases with the number of passes increasing. Rutting decreases with an increase in the percentage of nano clay compared to virgin binder. The trend of rutting decreases concerning the increase in organophilic nano clay until the addition of 4.5% nano clay, and the minimum amount of rutting may also be noted to be in the same mold containing 4.5% organophilic nano clay, and comparison is illustrated in Figure 13. It is a decrease of 69.23% in rutting compared to virgin binder. In the case of adding 5.0% nano clay, the rutting seems to increase. It may be because the bitumen is compatible with its maximum strength of 4.5% nano clay. The further increase might disrupt the optimum bitumen content (O.B.C.), hence disrupting the J.M.F. of the mixture. The decrease in rutting depth is because nano clay has a high aspect ratio and an enormous surface area; organophilic nano clay can function as a reinforcing filler within the asphalt matrix. The nanoparticles can organize themselves into a three-dimensional network within the asphalt, which improves the material's mechanical properties and makes it more resistant to deformation caused by traffic stresses [46,48,49]. It may be concluded that with the addition of nano clay, the rut depth decreases with increasing the number of passes compared to virgin binder; hence it is evident that nano clay substantially affects rut resistance [50].

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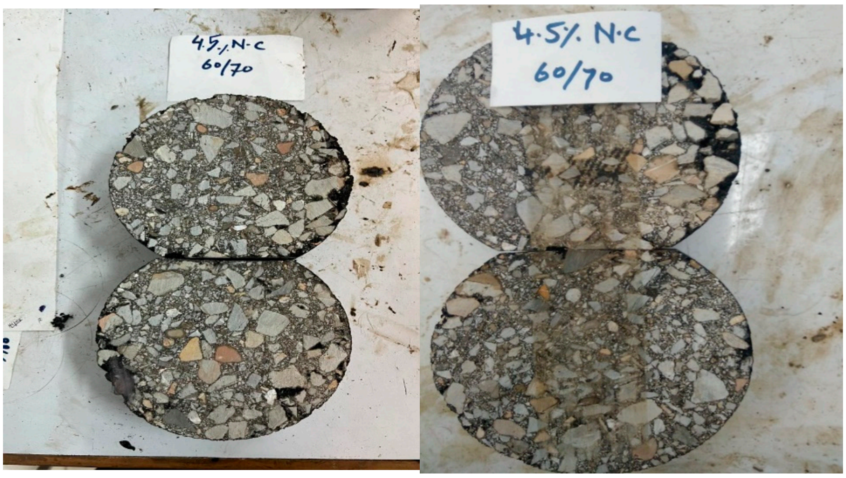
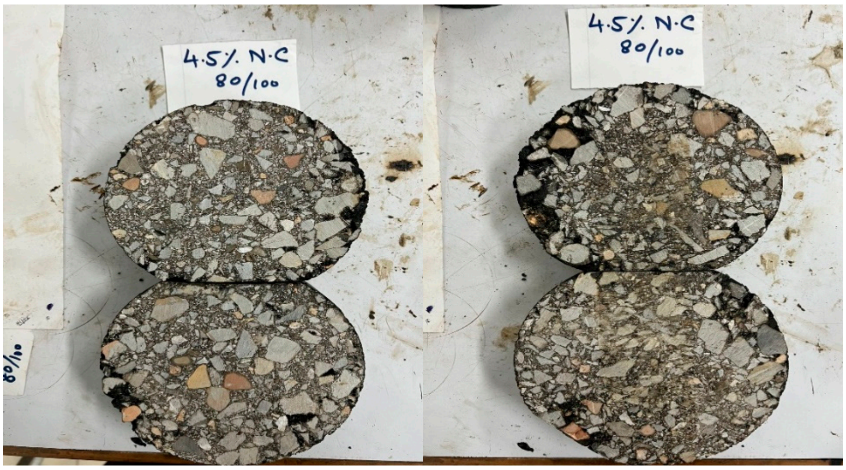


(a) (b)

Figure 13. P.G. 60/70 with 4.5% N.C. asphalt mixtures before and after Wheel Tracker Test.

Figure 14 illustrates that the permanent deformation of Pen 80/100 with 4.5% nano clay asphalt mixture reduced compared to virgin binder. Figure 15 shows the scatter diagram for P.G. 80/100 bitumen, and it may be noted that the result is similar to PG 60/70. The trend of rutting increases as the number of passes propagates, but comparing the rut accumulation to a virgin binder, nano clay modified bitumen shows decreased rutting in advanced stages of W.T.T. passes. The viscosity test results confirmed that modified nano clay asphalt binder increases significantly after adding organophilic nano clay. A greater viscosity can contribute to enhanced resistance to deformation at high temperatures, reducing the chance of rutting in the presence of heavy traffic loads and extreme temperatures [36,37]. Another reason is that nano clay has an organophilic character that can diffuse more uniformly inside the asphalt binder. A more uniform mixture is produced due to the improved dispersion, which also contributes to the increased resistance to rutting [46,48,49]. The minimum rutting could again be noted at 4.5% nano clay, decreasing from virgin's 3.4 mm to 1.1 mm; this shows a decrease of 67.64% in overall rutting as compared to virgin binder.



(a) (b)

Figure 14. P.G. 80/100 with 4.5% N.C. asphalt mixtures before and after Wheel Tracker Test.

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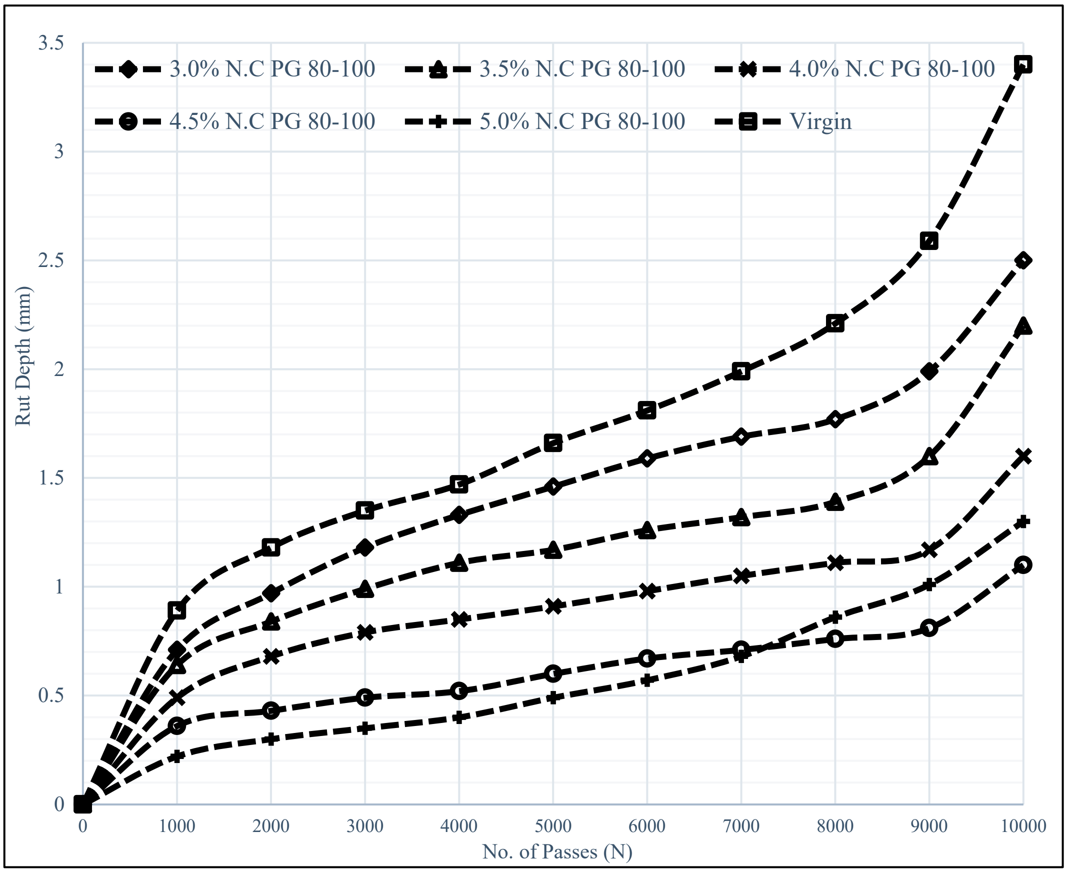

Figure 15. Effect of load cycles on Rut Depth of P.G. 80/100 bitumen.

4. Statistical Analysis

Statistical analysis was performed to determine whether the results obtained were statistically determinant/ significant and if they could be trusted with reliability. IBM SPSS software was used to carry out statistical analysis. For some other computations, Microsoft Excel was also used. The analysis of the output results obtained from statistical analysis is based on the criteria that the confidence level of the test was kept at 95%. It means that values obtained from SPSS with results < 0.05 would be significant, and the additive/ modifier would positively or significantly improve virgin binder properties. The percentages from 1-6 portray the percentage of organophilic nano clay added to the 60/70 and 80/100 binders, where 1 represents 0% O.N.C addition (Virgin Binder), 2 represents 3.0% O.N.C addition, and so on up to 5.0% O.N.C.

4.1. One-Way ANOVA Analysis

The ANOVA Analysis [49] aims to validate that the effect of O.N.C in raw virgin bitumen would significantly reduce rutting. Variables were defined (dependent and independent), and ANOVA Analysis was conducted to execute this analysis. The results of this analysis are given in Table 10.

Table 10. ANOVA Analysis for Rutting concerning O.N.C.

ANOVA					
Rutting					
	Sum of Squares	**df**	**Mean Square**	**F**	**Sig.**
Between Groups	5.920	5	1.184	7.478	.015
Within Groups	.950	6	.158		
Total	6.870	11			

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It may be noted from Table 10 that after the addition of nano clay significance of the data has changed. As all the tests were run with a 95% confidence interval, the significance observed is 0.015, less than 0.05 (5%). Hence the data is statistically significant. Figure 16 shows that adding 3.0% to 4.5% of nano clay at 135 °C increases the viscosity for NRL 60/70 and NRL 80/100, respectively, compared to the unmodified binder. The maximum increment in viscosity is found at 4.5% nano clay content compared to unmodified binders.

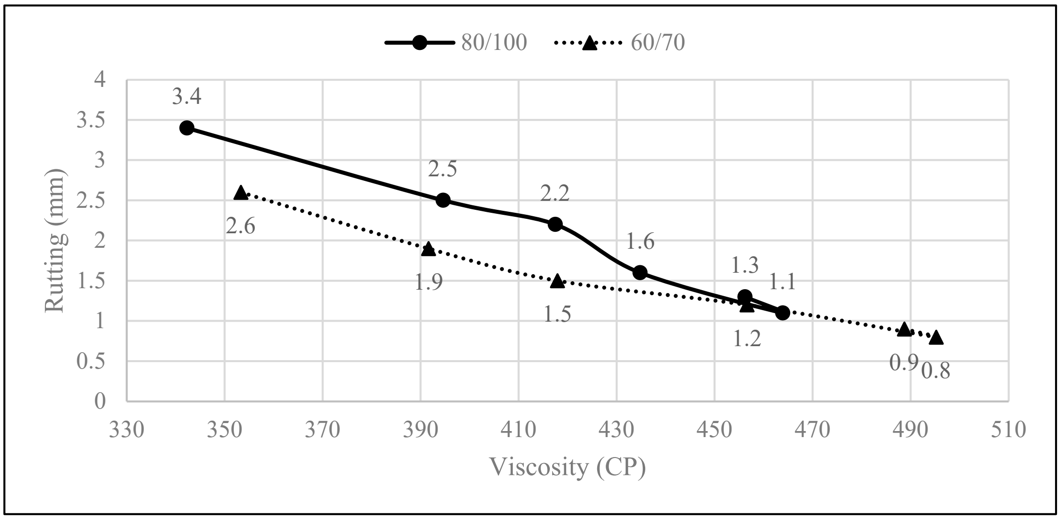

Figure 16. Relationship between Rutting and Viscosity for N.R.L 60/70 and N.R.L 80/100 bitumen.

Consequently, the rutting resistance is decreased. At 4.5% nano clay content, the maximum increase in viscosity and the maximum decrease in rutting resistance are observed. Increasing the viscosity value at high temperatures is a suitable property of rutting resistance. Nano clay was successfully incorporated into the base binder to investigate the flow properties of the bitumen and rut resistance of the asphalt mix. The significance of this is to improve the flow characteristics of bitumen in the pavement while decreasing the induced rutting in the asphalt mix.

4.2. Regression

Five equations were developed, including Linear, Logarithmic, Cubic, S, and Exponential between the nano dosages and the rutting depth, as illustrated in Figures 17 and 18. It was developed using SPSS to show each equation's impact on the modified binder's nature. Curve fitting equations for the 60/70 and 80/100-grade binders are developed and plotted. It may be noted that the impact of different equations on the trend of rutting across the modified binder. A decreasing trend in rutting may be noted across all the developed equations. These are further discussed below.

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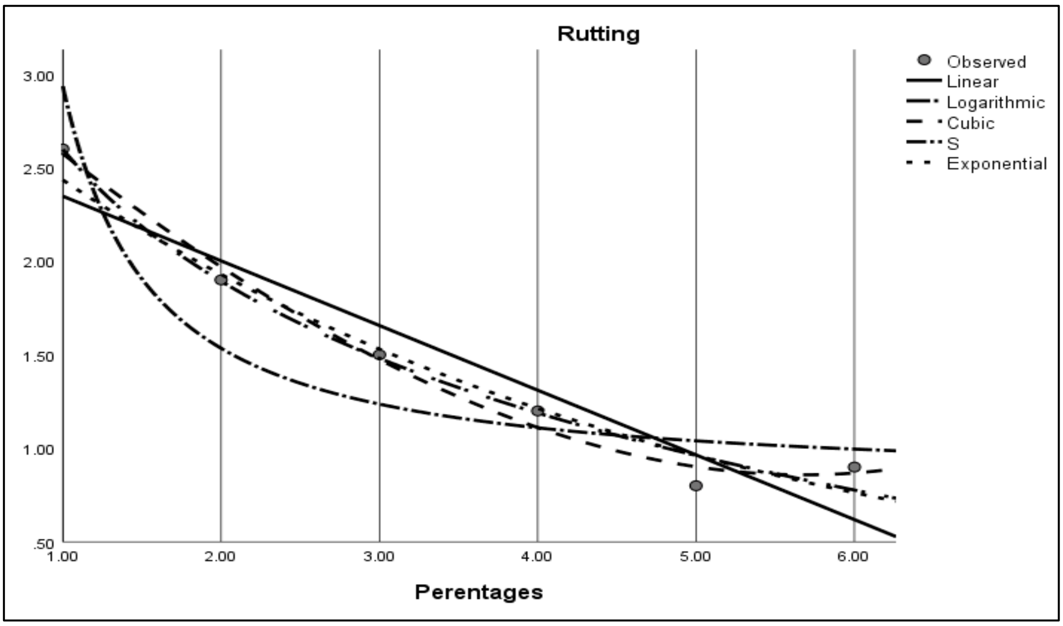

Figure 17. Graphical representation of rutting concerning different equations for 60/70 binder.

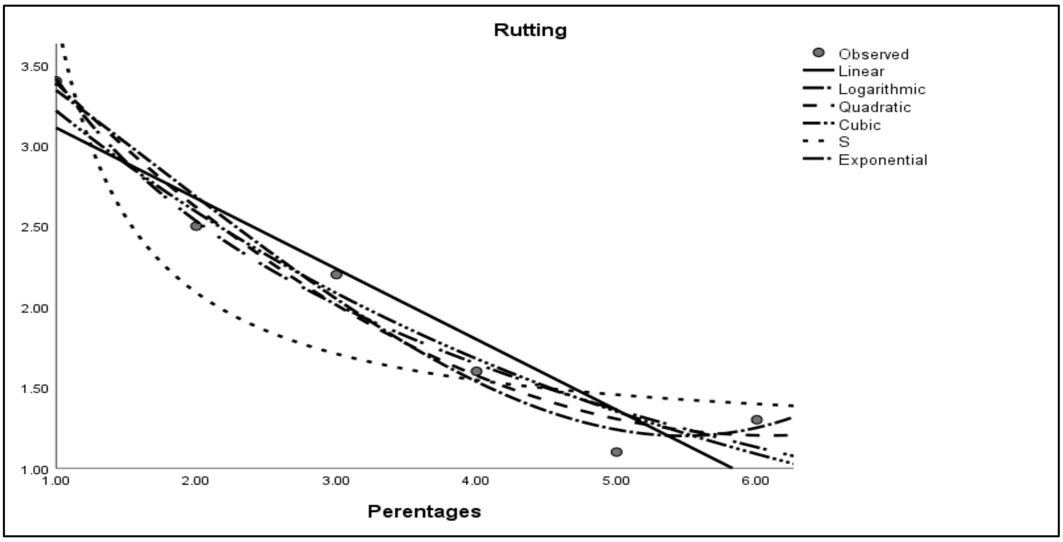

Figure 18. Graphical representation of rutting concerning different equations for 80/100 binder.

Figure 19 represents regression analysis for N.R.L 60/70 and N.R.L 80/100 bitumen by developing a curve-fitting equation using a least square method for Polynomial Regression. For N.R.L 60/70, the significance may be noted to be 0.005, which is < 0.05, and for N.R.L 80/100, the significance may be noted to be 0.016, which is < 0.05, this denotes that the modifier had a significant impact on both the binder grades. For N.R.L 60/70, the R^2 may be noted as 0.977, which is < 1. For N.R.L 80/100, the R^2 may be noted as 0.989, which is < 1. It signifies goodness of fit for both the binder grades. Further analysis may contribute towards the nature of the curve, which denotes a decreasing trend in rutting in both the binder grades.

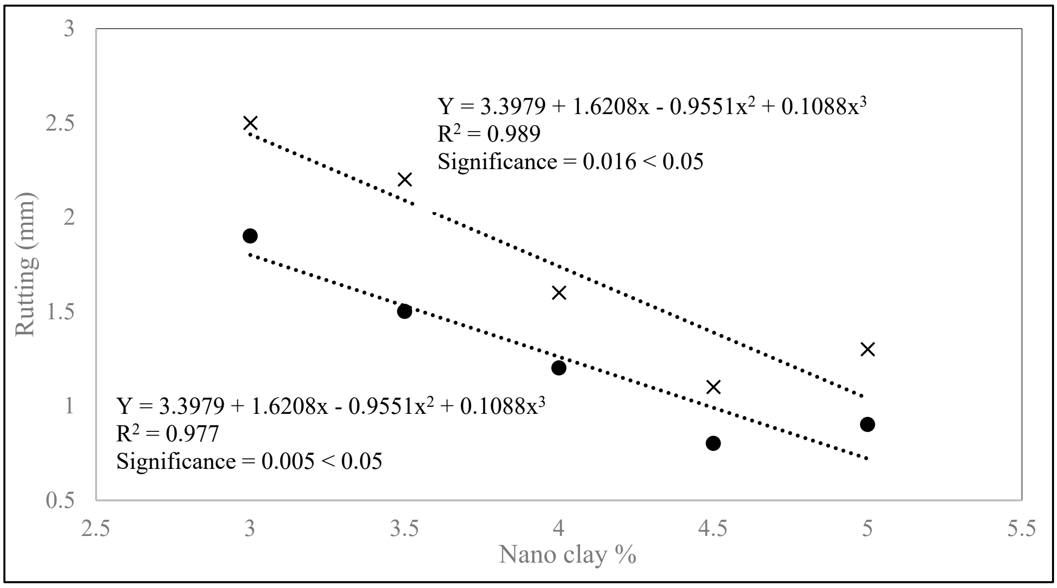

Figure 19. Polynomial Regression – Curve fitting for N.R.L 60/70 and N.R.L 80/100 bitumen.

Figure 20 shows the regression analysis for N.R.L 60/70 and N.R.L 80/100 bitumen by developing a curve-fitting equation using the exponential curve method for the exponential equation. For N.R.L 60/70, the significance may be < 0.003 or < 0.05, which denotes that the modifier significantly impacted the binder. The significance of N.R.L 80/100 bitumen may be noted as 0.002, which is < 0.05. It denotes that the modifier had a significant impact on the binder. For N.R.L 60/70 bitumen, the R^2 may be noted to be 0.977, which is < 1, which signifies goodness of fit. For N.R.L 80/100 bitumen, the R^2 may be noted to be 0.989, which is < 1, which signifies goodness of fit. Further analysis may contribute to the nature of the curves, which denotes a decreasing trend in rutting for both binder grades. After conducting statistical analysis on the data acquired from testing and interpreting it, it was found that considering Descriptive Statistics, Post Hoc Test, Homogeneous Subsets, Correlations, Regression, etc., the data was found to be statistically significant and that it used to make concluding statements regarding this research study would be substantive and reliable.

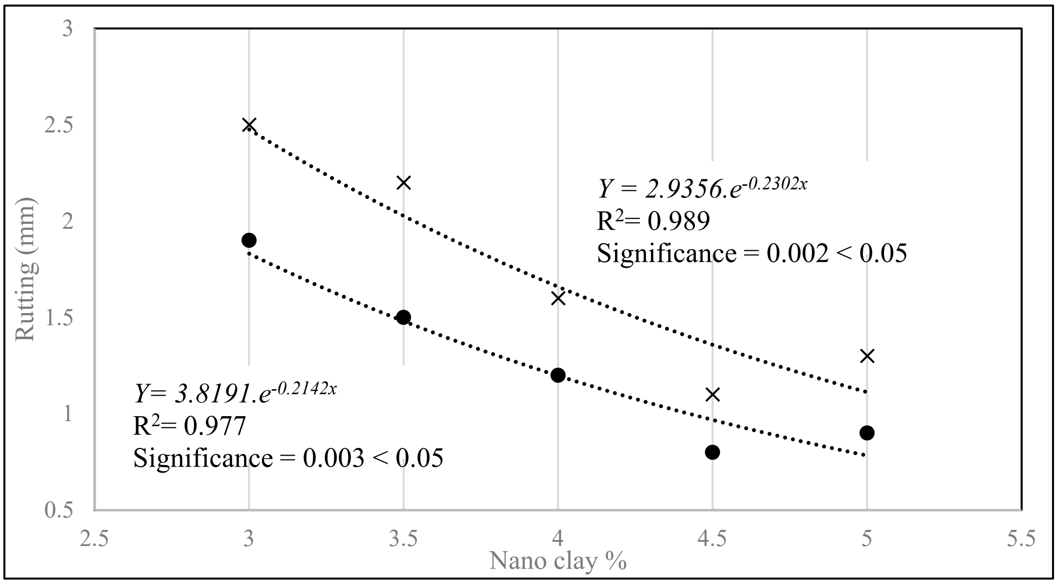

Figure 20. Exponential Curve – Curve fitting for N.R.L 60/70 and N.R.L 80/100 bitumen.

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5. Conclusions

The results of these tests were compared, and the modified binder was compared with a raw virgin binder to evaluate any changes or improvements in the mix properties. Based on the tests mentioned above, the following conclusions were made.

- For N.R.L 60/70 bitumen, the penetration value had increased from 61 to 69, which is an increase of 11.59%. Similarly, for N.R.L 80/100 bitumen, the penetration value had increased from 82 to 89, an increase of 7.0%. For N.R.L 60/70 bitumen, the Softening point decreased from 46°C to 41°C, which is a decrease of 5.0%. Similarly, for N.R.L 80/100 bitumen, the Softening point decreased from 49°C to 44°C, which is a decrease of 10.20%. The viscosity for N.R.L 60/70 bitumen had increased from 353.3 CP to 495.2 CP. It is an increase of 28.65%. Similarly, the viscosity of N.R.L 80/100 bitumen increased from 342.3CP to 463.9CP. It is an increase of 26.21%.
- The asphalt mixes used in this study were designed to assess the impact of high temperatures on the rut resistance of asphalt mixes. Laboratory tests were run according to high temperatures to replicate the mostly warm climates in Pakistan. The rut resistance properties of the P.G. binders were compared to virgin P.G. binders. In N.R.L 60/70 grade bitumen using 4.50% Nano clay, rutting was reduced to 0.8mm, a decrease of 69.23% compared to the virgin's 2.6 mm. Similarly, for N.R.L 80/100, the maximum decrease in rutting was again noted at 4.50% Nano clay content, decreasing from virgin's 3.4 mm to 1.1 mm, which decreased 67.64% in overall rutting. It can be concluded that organophilic nano clay positively affects raw bitumen and improves the rut resistance property of binders. The results in SPSS and running statistical analysis on them concluded that adding organophilic nano clay significantly impacted raw virgin bitumen, improving the conventional and rheological properties of bitumen.
- This study used the N.H.A. class B asphaltic wearing course of 50 mm thickness, and adding organophilic nano clay per bitumen weight modified the binder grades' mix. For N.R.L 60/70, after conducting tests and analyzing the results, the optimum nano clay percentage was 4.50%. The mix had a bitumen content of 4.25% with Bulk Specific Gravity, Gsb = 2.651, Specific Gravity of bitumen, Gb = 1.029, and Maximum Theoretical Specific Gravity of Loose Mix, Gmm = 2.490. The optimum nano clay percentage for N.R.L 80/100 was 4.50%. The mix had a bitumen content of 4.29% with Bulk Specific Gravity, Gsb = 2.673, Specific Gravity of bitumen, Gb = 1.029, and Maximum Theoretical Specific Gravity of Loose Mix, Gmm = 2.490.

6. Recommendations

Based upon the study conducted above, the following recommendation is given,

- As this research study was conducted using high temperatures, portraying the mostly warm climate of Pakistan, using organophilic nano clay modified binder is recommended as a viable alternative to using asphalt of raw virgin bitumen, considering its positive effect against rut resistance and increased viscosity.
- Based upon the future trend of the world shifting towards an environmentally friendly solution to the extraction of excessive crude oil and ultimately refining more bitumen, the use of organophilic nano clay in bitumen may be admired as not only does it decrease the overall bitumen content in an asphalt mix. Still, it would ultimately cut overall carbon emissions, contributing to a decrease in greenhouse gas emissions and global warming. Furthermore, more research needs to be done to explore the properties of nano clay at high and low temperatures with an increased percentage of nano clay in the asphaltic concrete mix.

Author Contributions: The concept was advised by A.A.G., H.J. performed the design and performance of experimental activities operated, and the implementation of practical activities was analyzed, and W.H. inscribed a paper draft by A.A.G., H.A. The W.H. evaluated the article. All authors have read and agreed to the published version of the manuscript.

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Informed Consent Statement: It is not required to provide any statement of informed consent to proceed with this experimental investigation.

Data Availability Statement: This experimental investigation does not call for any remark regarding data availability.

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