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A laboratory and field evaluation**

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DOI

[10.1016/j.cscm.2024.e03823](https://doi.org/10.1016/j.cscm.2024.e03823)

Publication date

2024

Document Version

Final published version

Published in

Case Studies in Construction Materials

Citation (APA)

Akinmade, D., Anupam, K., Kasbergen, C., Erkens, S., Samson, D., & Marco, P. (2024). Performance of natural asphalt as a paving material: A laboratory and field evaluation. *Case Studies in Construction Materials*, 21, Article e03823. <https://doi.org/10.1016/j.cscm.2024.e03823>

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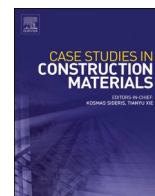
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Performance of natural asphalt as a paving material: A laboratory and field evaluation

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ARTICLE INFO

Keywords:

Natural bitumen
Petroleum bitumen
Marshall mixture design
Pavement construction

ABSTRACT

The ever-growing need to build roads to meet the necessary transportation demands is challenging, especially for developing countries. Low-volume roads (LVRs) are usually the backbone of catalyzing economic growth in these countries. With impediments surrounding Petroleum bitumen (price fluctuations) and environmental concerns, scientists are putting their effort into finding an alternative. The presented research is an attempt to check if Natural asphalt can be used as a full or partial replacement of the Petroleum bitumen. To the best of the authors' knowledge, only limited studies have focused on characterizing and understanding the engineering properties of Natural asphalt. The available techniques do not provide reliable information to the road authorities and hence they are discouraged from using it in practice. Particularly for countries, where the Natural asphalt source is available, the overall dependence on importing the Bitumen could be substantially reduced. Empirical and experience-based design criteria may not be sufficient as the standards were never developed for such materials, hence, a scientific approach is required before bringing it into practice. In this research, Natural asphalt sourced from different locations in Nigeria was assessed. Before performing the mixture level tests using Marshall and Cantabro design methods, the rheological and fatigue properties of the extracted Natural bitumen were examined in the laboratory. In the design of the experiment, various percentages of Natural asphalt were added between 0% and 20% by total mix weight; implying that the remaining required fraction of binder was fulfilled by the addition of petroleum bitumen. By using a ranking system (supported by statistics), an optimal design of mixture was obtained which was used in the field (exposed to normal traffic) at 30 different sections. Overall, results showed a good correlation between laboratory and field performance as compared to the petroleum-based mixture which was used as a reference mixture

1. Introduction

The construction processes involved in asphalt concrete pavement are known to have high energy consumption due to the need for elevated production temperatures, typically ranging from 170 °C to 180 °C [1,2]. When constructing a pavement, aggregates of

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<https://doi.org/10.1016/j.cscm.2024.e03823>

Received 12 April 2024; Received in revised form 15 September 2024; Accepted 6 October 2024

Available online 12 October 2024

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different sizes are typically combined using bitumen at high temperatures to create asphalt concrete mixes. In addition, energy is consumed during the sourcing of materials such as aggregates and bitumen. Bitumen, which serves as the binding material in pavement applications, has been utilised since ancient times around 6000 BC, making it one of the oldest and most widely accepted structural engineering materials [3]. As early as 3000 BC, the Persians employed bitumen for road construction; the Sumerians utilised it in ship building. Furthermore, in the Babylonian era bitumen was used in castle construction [4]. Bitumen plays a vital role in modern-day road pavement construction due to its economic engineering advantages and ability to stabilise the aggregate stone structure [5]. It offers resistance against water penetration and possesses the necessary strength to withstand traffic loads and adverse weather conditions. As a result, the global demand for bitumen exceeds 100 million metric tons annually [6–8]. However, the focus of this present study is on the application in the road industry and other potential applications are beyond the scope of this paper.

Prior to the industrialisation era, bitumen was obtained from the Natural bitumen deposits found at the bottom of the Dead Sea [9]. Nowadays, bitumen is mostly obtained as a by-product during the fractional distillation of crude oil at temperatures above 500 °C [4]. These processes may have a notable impact on the environment, contributing to global warming. In an attempt to address the environmental challenges linked to bitumen exploration, a majority of countries have committed to the COP 21 Paris Agreement. This is a collective agreement among nations to decrease CO₂ emissions significantly, aiming to limit the current average global temperature rise to well below 2 degrees Celsius compared to the pre-industrial era [10]. Hence, given the excessive reliance on Petroleum-derived products and the high-temperature production requirements, the current operations are most likely to become unsustainable in the near future.

Low-volume roads (LVRs), defined by FHWA 2009 as road networks with an annual average daily traffic (AADT) of less than 400 vehicles per day [5], are essential for the economic development and progress of emerging and developing economies, particularly in developing nations [11]. The construction of low-volume roads needs quality bitumen and given the challenges associated with Petroleum bitumen and other economic factors, it is crucial to explore alternatives to replace Petroleum-sourced bitumen partially or completely. It is noted that this is occurring alongside the growing demand for high-quality bitumen in the road sector. Road agencies in various countries are actively looking for alternative binder sources such as crude oil shale, biomass, microalgae, and Natural bitumen [12–15]. Researchers have also investigated the feasibility of utilising bio-binders from animal waste [16], agricultural waste [17], wood waste [18], lignin [19], cedar wood [20] and Natural bitumen [21] for paving operations. Hence, this study focuses on using Natural bitumen as an alternative for paving operations in modern-day paving. The volume of Natural bitumen deposits is currently considered to be 1,856,853 billion barrels, as demonstrated and 864,841 billion barrels inferred, as shown in Table 1 [22]. Natural bitumen can be found in rock enriched with bitumen, often known as Rock asphalt [23] and Gilsonite [24]. The lake binder is commonly found in Alberta in Canada, east Venezuela, Malagasy, Utah, Kentucky in the United States, Buton Island in Indonesia; Albania; Romania; Kazakhstan; France (Gard &Tham); Switzerland(TraverValley);Italy(Ragusa)[25].

Natural bitumen occurs as bitumen enriched with fine-grain sands known as Natural sand bitumen or oil sands [26]. Oil sands are loose sands or mainly consolidated sandstones saturated with highly viscous bitumen, mostly mined for crude oil production found in various countries throughout the world, but vast quantities are found in Canada, Nigeria, and Venezuela [27]. Canada has approximately 900 billion barrels of oil sands, and its bitumen content is about 12–13 %. In contrast, Nigeria has a reserve of about 42 billion tonnes of Natural bitumen, estimated to be the second-largest in the world [26,28]. The Natural occurring plain bitumen is found to be soft with low viscosity [23] as the type found in Ondo State, Nigeria [23,29]. Farmers within this region have used it to repair their mud houses, applying it directly to paint their homes [23].

For several decades, these Natural sand bitumen mixtures have been used as an alternative material in constructing flexible pavements, especially for low-volume roads in rural areas directly without much scientific investigation [30]. The consequence is that roads built with these materials without scientific inputs in the past and future will result in early failures, a waste of resources and time. However, most of these Natural bitumen sources have not been fully characterized or understood to provide reliable information to predict their long-term performance adequately. In addition, the importation of Petroleum bitumen may not be economically viable for road paving operations in countries that have abundant natural bitumen. Empirical and experience-based design criteria may not

Table 1
World Natural bitumen resources in 1990 (in a million barrels)Source:[22].

Area	Demonstrated	Inferred
North America		
Total	1,708,548	864,809
South America		
Total	50,460	
Europe		
Total	20,493	
Asia		
Total	76,263	
Africa		
Total	1065	31
Middle East		
Total	13	
Southeast Asia		
Total	11	1
World Total	1,856,853	864,841

be sufficient as the standards were never developed for such materials, hence, a scientific approach is required. To the best of the authors' knowledge, limited studies have focused on understanding the engineering properties of Natural bitumen, particularly utilizing advanced testing methods and optimizing their use in asphalt mixtures. Hence, this study examines the performance of Natural bitumen modified petroleum bitumen mixtures with reference to the mixtures prepared using Petroleum bitumen in laboratory and field [31].

1.1. Terminologies used in this study

Natural bitumen is consistently referred to as the binding agent extracted from the Natural asphalt otherwise mentioned in this article. Similarly, in the paper, Natural asphalt will consistently be used for asphalt mixtures of bitumen and mineral matter present naturally on earth. Petroleum bitumen is bitumen derived from the fractional distillation of crude oil from the abandoned industrial process. Furthermore, the term "Natural occurring Plain binder (NOPB)" is used to describe bitumen without minerals occurring naturally.

1.2. Objective and scope of this study

The objective of this research is to study Natural asphalt for pavement construction as an alternative material for low-volume roads by performing a laboratory investigation. Furthermore, the results of the field trial using Natural bitumen-modified asphalt mixtures with Petroleum asphalt on low-volume roads are compared. The trial was closely monitored over a two-year period(August 2021-July 2023) to evaluate the long-term performances of the Natural bitumen-modified asphalt mixtures in comparison with the Petroleum asphalt mixtures. The scope of the proposed study is as follows:

- To study the mineralogy of the extracted minerals of Natural asphalt;
 - Comparison of the performance of Natural bitumen from four different sources of Natural asphalt with Petroleum bitumen(60/100)
 - Comparison of the laboratory and field performance of mixture prepared using Natural asphalt and mixtures prepared using Petroleum bitumen(60/70).
- To make it easier for the reader, the research framework followed in the research is shown in Fig. 1.

2. Research methodology

The following section outlines the methodology and experimental approach used in this research, from the binder to the mixture scale level. This includes the material collection process, characterization process, and various test protocols as presented in the following subsections.

2.1. Material collection

The Natural bitumen was collected from three locations(Illubirin, Isowa, Akindolu Emaye) identified through geological studies as

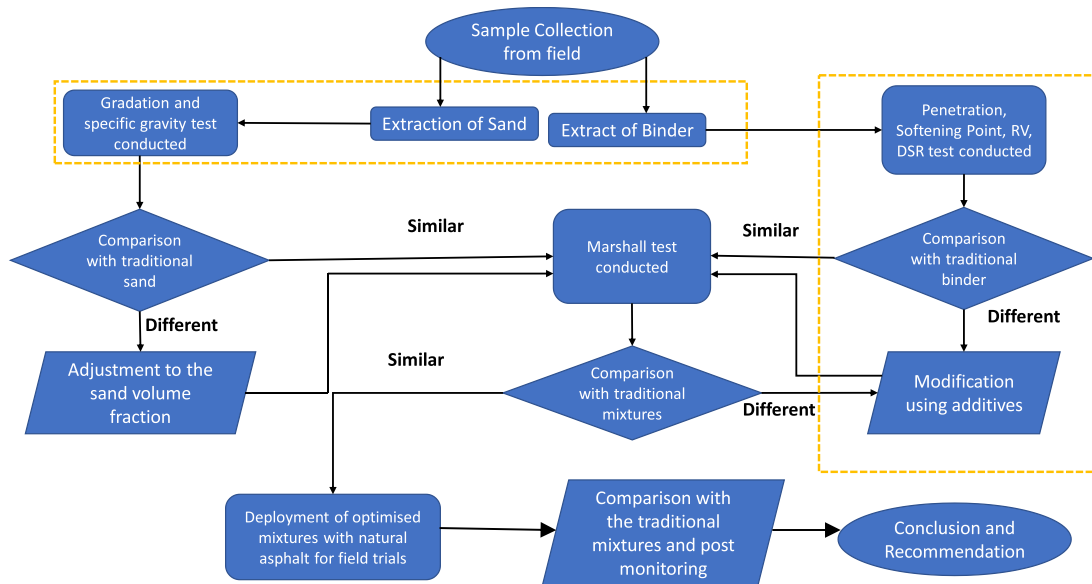


Fig. 1. Optimization of Natural bitumen for road construction Note: Traditional mixtures: Typical dense asphalt mixtures of different aggregate sizes and 60/70 bitumen. Traditional Binder: The typical 60/70 penetration grade bitumen used in Nigeria for road construction.

potential sources for high quality and abundant paving materials [32] as shown in Table 2. Samples of NOPB were obtained from wells in the vicinity of Illubirin [33].

2.2. Bitumen extraction process

The Natural bitumen from Illubirin, Isowa and Akindolu emaye samples were placed in an oven between $100\text{ }^{\circ}\text{C} \pm 5$ to soften the material and enhance miscibility with the solvent. Following the reflux method (ASTM D 2172 Method B) [34], the extraction process was carried out to separate the mineral aggregates from the mixtures. Following the extraction processes, aggregates and solvent-bitumen mixtures were obtained. The solvent-bitumen solution was then subjected to the recovery process to separate the solvent from the bitumen following the ASTM standard [35].

2.3. Experimental procedure

This section outlines the various test methods used in this study to evaluate Natural bitumen in paving operations. The obtained Natural bitumen (see 2.1 & 2.2) was tested qualitatively compared with the conventional Petroleum bitumen. Subsequently, the mineral sands were further graded to check if they adhered to the specified gradation of fine sand according to the given specification.

Following the two aforementioned tests, a preliminary mixture scale test was conducted utilising the Marshall approach and Cantabro test to assess the performance of asphalt mixtures at varying percentages. In this study, the amount of sand present in the Natural bitumen was considered during the laboratory optimisation of the overall mixtures. An experimental trial utilising the finalised optimised mixture modified with Natural bitumen was carried out for rural road maintenance. The schematic approach adopted in this research is presented in Fig. 1. The Following sub-sections present some standardised test procedures used in Nigeria.

2.3.1. Bitumen: penetration test

The penetration test was done to determine the consistency of Natural bitumen from Isowa, Illubirin, and NOPB in comparison with the Petroleum binder. The penetration test was conducted by measuring the depth at a standard needle penetrating the conditioned test specimen under a 100 g load at $25\text{ }^{\circ}\text{C}$ temperature for 5 s as outlined in EN specification [36]. Afterwards, the bitumen were all graded according to the depth of penetration observed during the test.

2.3.2. Bitumen: softening point

The softening point test was carried out to compare the consistency of the Natural bitumen and NOPB against Petroleum binders. The softening point test was conducted following the EN specification [37]. It is noted that a higher value of softening point would indicate hard bitumen while a lower value of softening point would indicate soft bitumen.

2.3.3. Bitumen: viscosity test

The rotational viscosity test was conducted on the Natural bitumen, NOPB and the Petroleum bitumen to determine their viscous nature and resistance to flow as described in the European standards [38]. The behaviour of Natural bitumen towards the mixing and compaction temperature was measured using Brookfield rotational viscometer. The thermo-chamber spindle speed and temperature were set at 20 rpm and $135\text{ }^{\circ}\text{C}$ respectively.

2.3.4. Bitumen: rheological properties

The rheological properties were investigated to study the visco-elastic properties of bitumen time-temperature dependent behaviour when subjected to thermal and mechanical conditions [39]. The rheological properties of the Natural bitumen and the NOPB were characterised following NEN-EN 14770 [40] specifications for determining the complex shear modulus (G^*) and phase angle (δ). In addition, the amplitude sweep, frequency sweep, and fatigue tests were conducted on all the extracted Natural bitumen compared to the Petroleum binder. The Natural bitumen, NOPB and the Petroleum bitumen were tested based on strain-controlled using parallel plates geometry of 8 mm plate diameter and 2 mm gap configuration. The frequency used varied from 10 to 0.1 Hz in a logarithmic manner with a strain load of 0.1 % at temperatures of 0,10,20,30 and $40\text{ }^{\circ}\text{C}$.

2.3.5. Bitumen: fatigue properties

Fatigue tests on the Natural bitumen and Petroleum binders was conducted using the Linear amplitude sweep test(LAS) procedure according to AASHTO TP 101-14 [41]. The test was conducted in the application of cyclic torsional load in increasing amplitude to accelerate the damage on the binders. The LAS test was conducted in 2-phase using the 8 mm parallel plate with a 2 mm gap. The first

Table 2
Sample collection location.

Sample Location	Geographic Coordinate
Illubirin	$6^{\circ}38'33''\text{N}, 4^{\circ}49'44''\text{E}$
Isowa	$6^{\circ}42'25''\text{N}, 4^{\circ}52'12''\text{E}$
Akindolu Emaye	$6^{\circ}39'3''\text{N}, 4^{\circ}53'29''\text{E}$
Akindolu Emaye	$6^{\circ}39'19''\text{N}, 4^{\circ}51'48''\text{E}$

phase examines the rheological properties using frequency sweep test applying oscillatory shear at constant amplitude within a specified loading frequency at 20 °C constant temperature.

The applied load of 0.1 % strain over a range of frequency 0.1–10 Hz was used following AASHTO TP 101–14 [41]. The second phase was conducted using continuous oscillatory

strain-controlled cycles with linearly increasing strain amplitudes from 0 % to 30 % on the material at a constant frequency of 10 Hz and temperature of 20 °C. The frequency sweep and

the amplitude sweep data were then used to compute the binders resistance to fatigue based on Eqs. (1)–(4). The damage accumulation, $D(t)$, of the Natural bitumen, NOPB and Petroleum bitumen is expressed using Eq. (1) with respect to the testing time, t :

$$D(t) \cong \sum_{i=1}^N [\pi\gamma_0^2 [C_{i-1} - C_i]]^{\frac{x}{1+\alpha}} (t_i - t_{i-1})^{\frac{1}{1+\alpha}} \tag{1}$$

$C(t) = \frac{G^*(t)}{G_{initial}^*}$, which is G^* at time t divided by the initial value of G^* (undamaged), γ_0 is the applied strain for a given data point, percent, and t is the testing time in seconds, $\alpha = \frac{1}{m}$

where m is the slope plot of the logarithmic scale relationship between storage modulus and frequency, the values of $C(t)$ and $D(t)$ can be obtained by fitting the following relationship at any given time.

$$C(t) = C_0 - C_1 D(t)^{C_2} \tag{2}$$

$C_0 = 1$, it is the initial value of C , C_1 and C_2 are the curve-fit coefficients derived through linearisation of the power law. The value of damage accumulation ($D_{(t)}$) at failure, D_f is the value of $D_{(t)}$ that corresponds to the initial complex shear modulus reduction at peak shear stress, which can be computed using Eq. (3). The binder fatigue performance is evaluated computed using(4).

$$D_f = \left[\frac{CatPeak}{c_1} \right]^{1/c_2} \tag{3}$$

$$N_f = A[\gamma_{max}]^{-B} \tag{4}$$

where N_{fis} the fatigue life, $A = \frac{f(D_f)^k}{k[\pi C_1 c_2]^{\frac{1}{\alpha}}}$, $k = 1 + (1 - C_2)\alpha$, $B = 2\alpha$, γ_{max} is the expected maximum strain (%), f is the loading frequency at 10 Hz.

The fatigue results obtained from the different types of Natural bitumen and NOPB in this study played a vital role in making informed decisions regarding the selection for trial asphalt mixture design. The subsequent section describes the standard test procedure used for mix design in Nigeria, which was adopted for a trial optimisation of the partial replacement of Petroleum bitumen using Natural bitumen directly.

2.3.6. Asphalt mixtures design: marshall stability and flow properties

In this research the asphalt mixture design including varying percentages of Natural bitumen into asphalt mixtures was done using the Marshall method according to the specification used in Nigeria[31]. The composition of the asphalt mixtures for the wearing course adopted is presented in Table 3.

The laboratory tests were done in three stages. In the first stage, the optimal binder content of the control mixtures was obtained by varying the percentages of Petroleum bitumen(see details in Table 10). The second stage was to substitute the optimal binder content

Table 3
Composition of dense mixture asphalt concrete (DAC)[42].

Sieve size(mm)	Specification		
	Maximum	Minimum	Design
12.5	100	85	100
9.5	92	75	92
6.35	82	65	82
2.36	65	46	65
1.18	51	36	51
0.6	40	26	40
0.3	30	18	30
0.15	24	14	24
0.075	14	7	14
60/70 bitumen content (%) Θ	5	8	6
Average voids content	3 %	5 %	
VMA		>14	14.5
VFA	82	75	81
Maximum density (Kg/m ³)	-	-	-

Bitumen content on 100 % aggregates by mass.

with Natural bitumen in the total mixtures. The Natural bitumen was used to replace the Petroleum bitumen by the percentage of the total mixtures at varying percentages of 5 %, 10 %, 15 %, and 20 % as replacements of the optimum Petroleum bitumen (see details in Table 12). Subsequently, the aggregate was combined with the filler and then heated in an oven at a temperature of 160 °C. The bitumen was pre-heated to 160 °C and thoroughly mixed with the heated aggregate and filler until a complete coating of the aggregate and filler was achieved.

In the third stage, the samples are according to the Marshall Mixture design, which involves the extraction process to re-evaluate the gradation limits. After every addition of the Natural bitumen in the mixtures, the sample was further separated and tested for gradation to assess if they fell within the gradation limits. The gradation limits check was done according to the standard [42]. This can be considered as the preliminary stage of evaluating the possibility of partial replacement of total binder in the mixtures using Natural bitumen. A total of 15 Marshall Samples were produced to determine the optimum binder, and another set of 15 samples was produced to determine the optimum percentage for Natural bitumen.

2.3.7. Cantabro loss properties

To assess the ravelling resistance of hot mix asphalt modified using Natural bitumen the Cantabro loss test was conducted following the AASHTO TP 108–14 standard [43]. In this test, cylindrical asphalt mixture specimens with a diameter of 102 mm and a height of 63.5 mm were prepared using Marshall Compactor at a heavy compaction level (75 blows). Two testing conditions namely dry and wet, were carried out during the sample preparation. Under the dry condition, samples were left at room temperature for 24 h prior to undergoing the test. Subsequently, for the wet condition, the originally dry-conditioned samples were placed in a temperature-controlled water bath at 60 ± 1 °C for an additional 24 hours before leaving room temperature before testing [44]. The samples were weighed to get the weight before abrasion (M_1) then placed into the Los Angeles machine drum without the charge of steel balls subjected to 300 revolutions at a speed of 30 rpm, at a constant temperature of 25 °C. The weight of the samples after the test was measured as weight after abrasion (M_2). The percentage of mass loss was calculated to indicate the ravelling of the asphalt mixture in the two conditions. Six replicates were conducted for each type of mixture (3 for dry and 3 for wet).

The cantabro mass loss was computed using Eq. (5). The degradation of both Natural bitumen and Petroleum bitumen were similar as shown in Fig. 2.

$$\text{Cantabro loss} = \frac{M_1 - M_2}{M_1} \times 100\% \quad (5)$$

M_1 = Mass of asphalt before testing, g; The specimen is removed, dusted, and weighed again after abrasion M_2 = Mass of asphalt before testing, g. Cantabro Loss index was computed using Eq. (6).

$$\frac{CL_{\text{wet}}}{CL_{\text{dry}}} \times 100\% \quad (6)$$

CL_{wet} = Cantabro loss% from moisture – conditioned samples

CL_{dry} = Cantabro loss% from dry conditioned samples.

The optimum Natural bitumen content was determined through testing was subsequently employed in the field maintenance project, with its performance closely monitored. (refer to 4 for more detailed information).

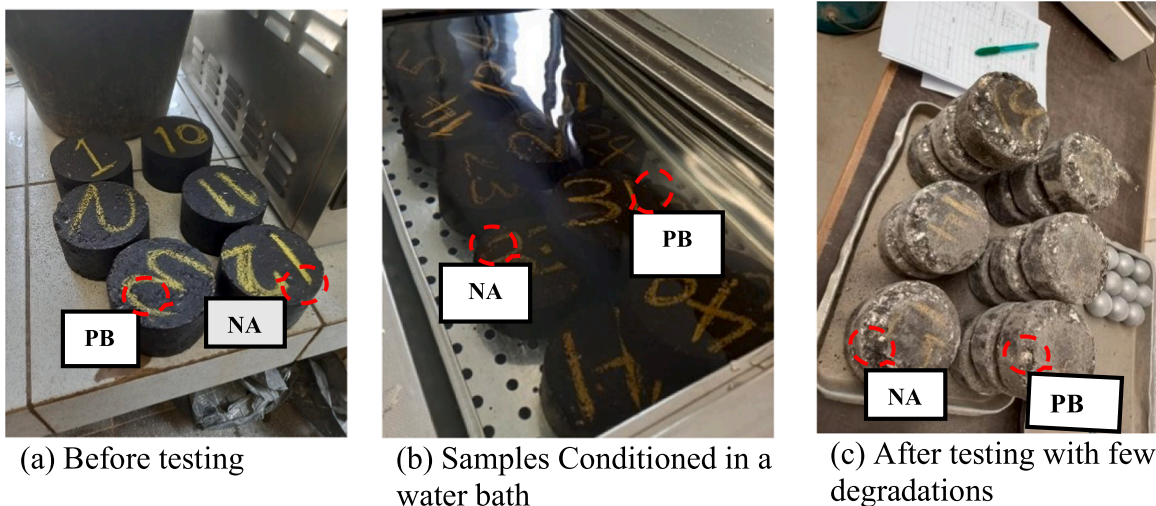


Fig. 2. Asphalt concrete before testing and after testing (NB: Natural bitumen, PB Petroleum bitumen).

3. Results and discussion

The following subsections gives the discussion of the performance of natural bitumen in conventional asphalt and their laboratory and field performance.

3.1. Extraction of binder from Natural bitumen and determination of binder content

The bitumen content obtained from the three locations ranged between 10 % and 12 % (see Table 4), which is consistent with findings reported in previous studies [30]. As shown in Table 4 the percentage of bitumen obtained from the Natural bitumen yields promising results, warranting further studies to explore its potential for paving operations.

3.2. Physical properties of Natural bitumen

The penetration value of the NOPB sample and the Natural bitumen from Isowa and Illubirin were considered for further analysis due to the available quantity. As presented in Table 5, the penetration values of Natural bitumen samples extracted from Isowa, Illubirin sites, and the Petroleum bitumen falls within the classification of a 60/100 grade binder. The softening point results for the Natural bitumen obtained from the Isowa and Illubirin site locations were determined to be 49 and 52, respectively. These values meet the specified requirements and criteria for use in paving operations, indicating that the Natural bitumen from these locations possesses favourable characteristics for application in road construction.

As shown in Table 5, NOPB can be classified as a softer binder with a grade of 160/220 with a softening point value of 39.8, falling within the specified range of 35–43 [37]. This shows that NOPB could be suitable for application in cold regions, where it could possibly be used as an emulsion and rejuvenator in paving operations. However, more research is needed to investigate this aspect.

3.3. Viscosity test

The viscosity values of the NOPB sample and the Natural bitumen from Isowa and Illubirin in this study were found to be less than 3 Pa s at 135 °C and 160 °C as shown in Fig. 3.

According to binder specifications, these values are within the range stipulated for mixing and compaction [38]. The mixing and compaction temperatures for the Natural bitumen and Petroleum bitumen (70/100) were obtained by identifying the points where the viscosity-temperature line intersects within the specified limits of 0.17 ± 0.02 Pa-s for the mixing temperature range (MTR) and 0.28 ± 0.03 Pa-s for the compaction temperature range (CTR) as shown Fig. 3 [45]. The viscosity values corresponding to the mixing and compaction temperatures are set at 70 ± 20 mPa s and 280 ± 30 mPa s, respectively [46].

The obtained mixing and compaction temperatures of all values obtained are presented in Table 6. In summary, it can be concluded that using Natural bitumen in road construction is expected to not pose construction-related issues related to mixability at the plant and compaction at the construction site.

3.4. Rheological properties

Natural bitumen also exhibits rheological properties like Petroleum-sourced bitumen, which are crucial for various applications, particularly in the construction and road industries. Subsequently, we would focus more on the viscoelastic properties to describe their behavior.

3.4.1. Linear Viscoelastic complex modulus and phase angle master curves

The viscoelastic properties of the Natural bitumen and Petroleum bitumen were obtained using a dynamic shear rheometer (DSR) in an undamaged state using frequency sweep tests. The time-temperature superposition principle (TTSP) and sigmoidal functions [47, 48] were used to construct the master curves at a reference temperature of 20 °C. The plots of Fig. 4(a) and (b) shows that as the frequency increases, the complex modulus also increases while the phase angle decreases for both Natural and Petroleum bitumen. The 60/70-grade Petroleum bitumen generally exhibits a higher complex shear modulus than the 70/100-grade Petroleum bitumen and all Natural bitumen in low and high frequency. Nonetheless, within the low-frequency range, Illubirin-sourced Natural bitumen shows slightly higher complex shear modulus values than the 70/100 Petroleum bitumen and other Natural bitumen.

As shown Fig. 4(a) and (b) there is no significant disparity in the values of complex shear modulus at high frequencies. This shows that Natural bitumen sourced from Illubirin exhibits a more elastic behaviour than 70/100 bitumen and other Natural bitumen at high temperatures. However, at lower temperatures, the behaviour of the Natural bitumen and Petroleum bitumen are similar. a clearly illustrates that Natural bitumen from Isowa exhibits the lowest complex shear modulus at a frequency of approximately 10 Hz, with

Table 4
Weight of the extracted minerals.

Sample ID	Isowa	Illubirin	Akindolumaye
% Bitumen	11.4	10.7	11.9

Table 5
Penetration and softening point test values for fresh samples.

Source	Penetration (0.1 mm)	Softening point (°C)
Isowa	100	49
Illubirin	64	52
NOPB	210	40
70/100	90	48
60/70	65	48

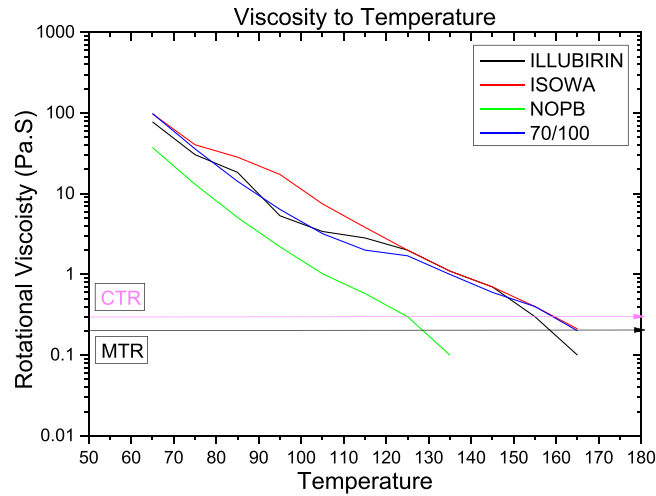
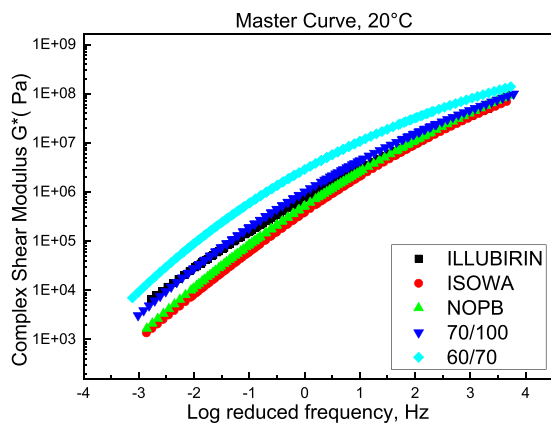


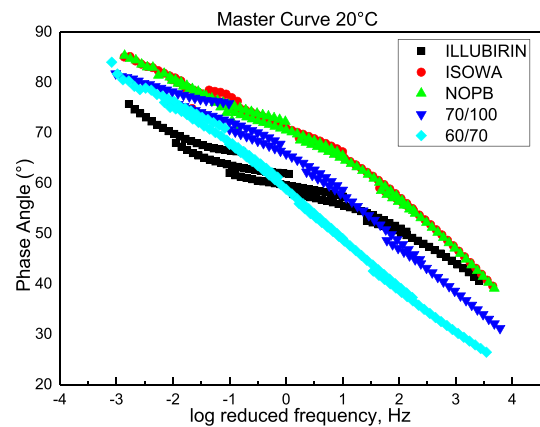
Fig. 3. Viscosity-temperature graph. Note: CTR: Compaction temperature range, MTR: Mixing temperature range.

Table 6
Mixing and compaction temperatures.

Source	Mixing Temperatures(°C)	Compaction temperatures (°C)
Isowa	165	160
Illubirin	158	155
NOPB	129	125
70/100	165	160
60/70	NR	NR



(a) Complex shear modulus



(b) Phase angle

Fig. 4. (a) Complex shear modulus and Phase angle for Natural and petroleum bitumen.

NOPB being

next, while Illubirin and the 70/100 bitumen show similar values. The same tendency was observed in the phase angle master curves shown in Fig. 4(b). and a summary of their rheological data is presented in Table 7.

3.4.2. Cole-Cole diagrams

The Cole-Cole diagram shown in Fig. 5 was used to understand the behaviour of Natural bitumen compared to Petroleum bitumen. The Cole-Cole diagram plots the loss modulus (G'') against the storage modulus (G') on linear and log-log scales, as shown in a and respectively. A straight line drawn on the graphs defines the data points zones, as shown by the plots of Fig. 5(a) and (b).

The Natural bitumen mostly shifted to the left which indicates a more elastic behaviour. The Natural bitumen and petroleum bitumen are similar at higher temperatures (the left side of the cole-cole diagram before reaching its peak), and there is no significant transition between curves. However, by decreasing the test temperature and moving towards the right side of the diagram, it can be seen that the highest peak belongs to the petroleum binders (60/70 and 70/100) and the lower ones are for Natural bitumen. It shows that adding Natural bitumen to petroleum bitumen would make it slightly stiffer and may cause a reduction of loss modulus.

3.4.3. Black diagrams

The black diagrams illustrate the rheological behavior of both Natural bitumen and Petroleum bitumen. These diagrams depict the complex shear modulus against the phase angle, with the frequency and temperature parameters eliminated, without applying the Temperature-Temperature Superposition Principle [49]. As presented, the black diagram curves of Natural bitumen from NOPB and Isowa shifted towards higher values of phase angles, indicating a reduction in stiffness. The Natural bitumen from Illubirin exhibits a curve that tends to shift to the left. These changes suggest a tendency towards a stiffer and less viscous material, potentially leading to failures such as brittle fractures, especially at lower temperatures.

The Petroleum bitumen 60/70 and 70/100 displayed a similar curvature to the NOPB and the Isowa sourced Natural bitumen as shown in Fig. 6. The following sub-section will highlight some preliminary findings from fatigue analysis of all the Natural bitumen source and comparing them with Petroleum bitumen using the Linear Amplitude Sweep test.

3.5. Fatigue Analysis

Fatigue analysis was conducted on the Natural bitumen in comparison to the Petroleum bitumen to assess their durability and performance. The Linear amplitude sweep (LAS) method as described in the subsequent sections was used in this study for evaluation.

Performance Predictions

The LAS method [50] is based on the visco-elastic continuum damage (VECD) approach to predict binder fatigue life as a function of strain level of asphalt materials. The LAS test was performed on Natural bitumen and petroleum bitumen to evaluate the fatigue performance of the binders [51]. The developed Fatigue models for the Natural bitumen and Petroleum bitumen based on (VECD) analysis are shown in Table 8. The Natural bitumen from Illubirin has a higher fatigue life at a strain level of 2.5 % compared with the 70/100 Petroleum bitumen, while other Natural bitumen has a lower fatigue life. For comparison purposes, the N_f values at 2.5 % and 5 % strain levels are given in Table 8.

3.5.2. Stress-strain response of the Natural bitumen

The stress-strain behaviour of Natural bitumen in comparison with Petroleum bitumen is illustrated in Fig. 7(a). The figure depicts the fatigue failure criterion used to analyse the fatigue behaviour of the binders [52]. The stress exhibited an initial increase followed by a decrease as the strain increased. Furthermore, as the strain amplitude increases, the shear stress

increases for all the binders, indicating that the binder is within the non-linear zone. The maximum stress and corresponding strain values varied among the Petroleum bitumen and various Natural bitumen samples.

As shown in Fig. 7a, the Isowa sourced Natural bitumen reached peak stress faster than the Illubirin and NOPB Natural bitumen comparable to the peak stress of Petroleum bitumen. This indicates that Illubirin has better shear loading resistance compared to other Natural bitumen. On the other hand, the Natural bitumen sourced from Isowa demonstrated the lowest failure strain value among the tested Natural bitumen. In summary, the Natural bitumen from Illubirin could be considered as the source performing closely to the Petroleum binders (60/70 and 70/100).

As shown in Fig. 7(a) the NOPB exhibited a maximum stress at 8.5 % strain, while Isowa sourced bitumen has a maximum stress at

Table 7
Summary of Rheological Data.

Source	Rheological data			
	$G_{g(0^\circ C, 10 Hz)}^+$ 10^8 pa	Phase angle δ	$G_{g(40^\circ C, 0.1 Hz)}^+$ Pa	Phase angle δ
Isowa	5.5	39.6	1200	85.02
Illubirin	6.5	40.6	6148	75.70
NOPB	8.5	39.1	1621	85.34
70/100	9.2	31.7	3110	81.73
60/70	11.2	26.43	9970	84.00

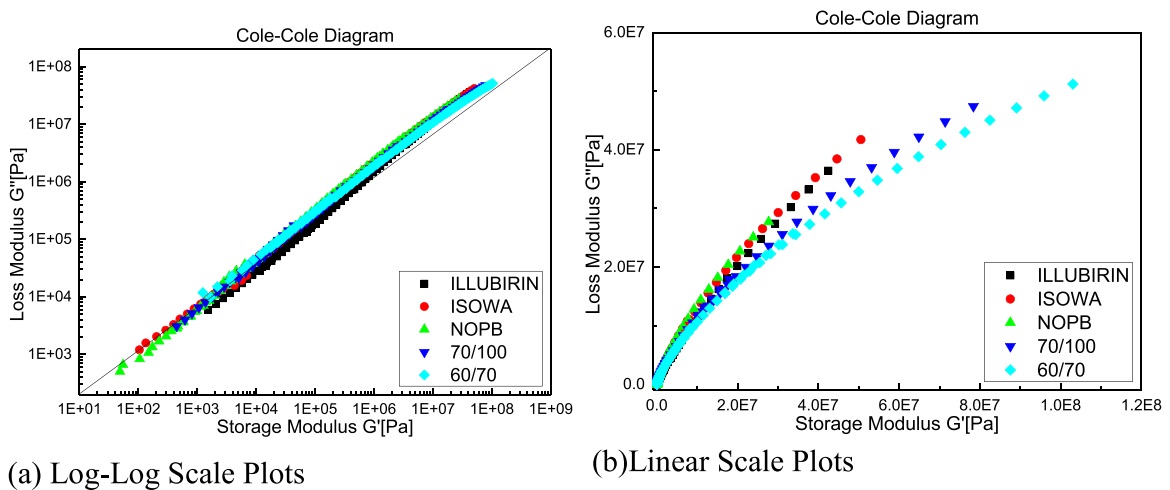


Fig. 5. Cole-Cole Plots for Natural Bitumen and Petroleum bitumen.

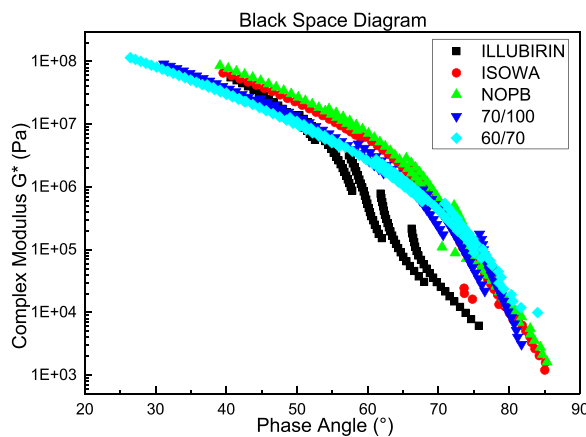


Fig. 6. Black space diagram for Natural bitumen and Petroleum bitumen.

Table 8
Fatigue parameters of binders.

Sample I.D	Damage Level	N_f = at strain level of		$N_f = A[\gamma_{max}]^{-B}$
	Fresh	2.5 %	5 %	
60/100	0.506	8215	1288	$9.509 \times 10^4 (\gamma_{max})^{-2.673}$
70/100	0.452	5227	917	$5.219 \times 10^4 (\gamma_{max})^{-2.511}$
Illubirin	0.397	5555	751	$7.822 \times 10^4 (\gamma_{max})^{-2.886}$
Isowa	0.316	2466	277	$4.442 \times 10^4 (\gamma_{max})^{-3.155}$
NOPB	0.255	3332	728	$2.487 \times 10^4 (\gamma_{max})^{-2.194}$

6.52 % strain, the Illubirin bitumen has a maximum stress at 7.87 % strain, and 70/100 at 8.55 % strain and 60/70 at 9.2 % strain. The sequence of strain at maximum stress, from lowest to highest for Natural bitumen, is Isowa, Illubirin, and NOPB as presented in Table 9. The NOPB required a comparable strain rate to reach its maximum compared to 70/100 petroleum asphalt.

3.5.3. Damage characteristics curves

The damage characteristics curve is a typical way to characterise the fatigue behaviour of binders, as shown in Fig. 7(b). The material integrity of the binder can be determined at any damage intensity of interest. The damage characteristic curves, derived from LAS tests illustrate the properties of the Natural bitumen extracted from both Natural asphalt and Petroleum bitumen, as depicted in In the case of un-damaged samples, the integrity parameter "c" consistently holds a value of 1, and when the damage parameter approaches zero, the material is completely damaged. At any specified intensity level, a higher "c" value indicates a stronger resistance of

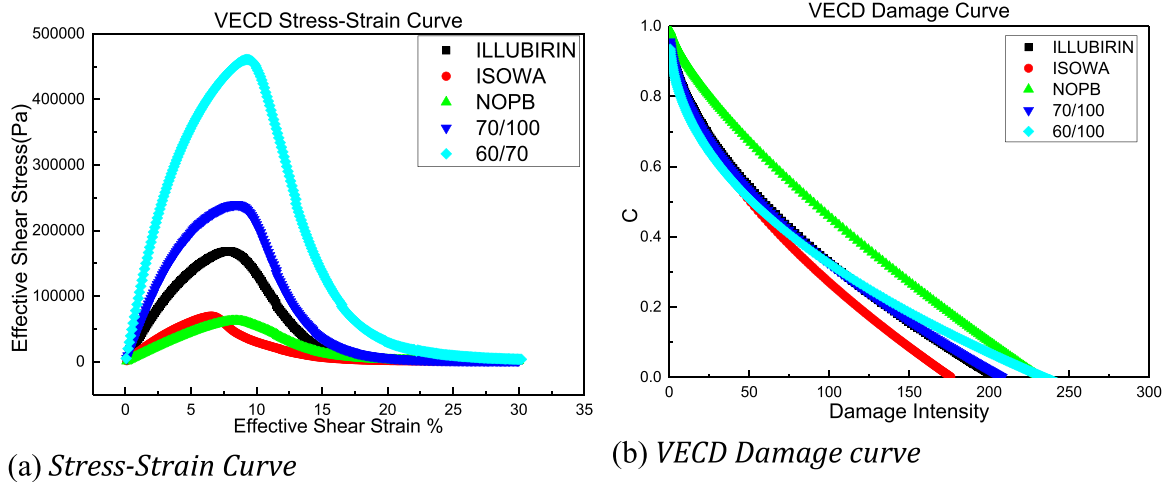


Fig. 7. VECD stress-strain curve and Damage curve.

Table 9
Fatigue failure of binders.

Sample	Condition	Peak stress (Mpa)	Failure strain (%)
60/70	Fresh	0.461	9.2
70/100	Fresh	0.240	8.55
Isowa	Fresh	0.069	6.52
Illubirin	Fresh	0.169	7.87
NOPB	Fresh	0.062	8.26

the material to damage [53].

All the binders attain peak shear stress at different strain amplitudes, and after this point, a gradual decrease in the peak shear stress and an increase in shear strain was observed. The Natural bitumen shows different damage patterns although the binder source from Illubirin shows similar patterns to the Petroleum bitumen. However, the damage characteristic curves are insufficient to give an informed decision on the binder fatigue performance.

3.5.4. Fatigue performance prediction

The NOPB was categorised as a soft binder due to its penetration softening point and viscosity test results, surprisingly exhibiting a higher fatigue life at a low strain level of 5 % compared to Isowa-sourced Natural bitumen. However, at a high % strain level of 5 %, its fatigue life was nearly identical to that of the Illubirin-sourced Natural bitumen as shown in Fig. 8. This suggests that the Natural bitumen from ISOWA tends to have a brittle nature and is more prone to fatigue failure as compared to the Illubirin and NOPB Natural

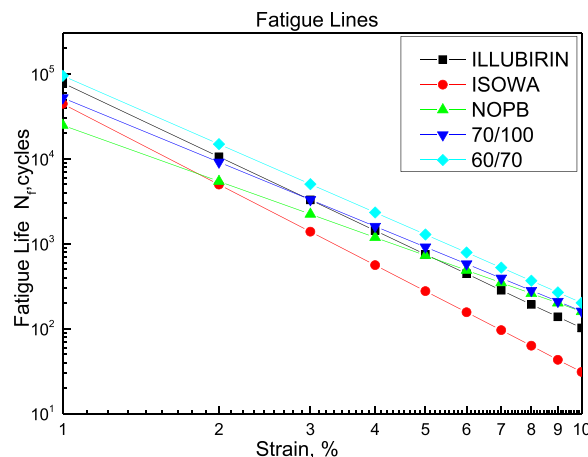


Fig. 8. Fatigue lines.

bitumen. In this study, the reference bitumen was 70/100. At a low strain level, the Illubirin-sourced Natural bitumen demonstrated a higher fatigue life by 6 %. In comparison, at a high strain rate of 5 %, the 70/100 bitumen exhibited a higher fatigue life by 18 %. Blending Iowa-sourced bitumen with NOPB could be a viable strategy to enhance the fatigue life of isowa-sourced bitumen and decrease its sensitivity to fatigue damage. Further experiments is needed to validate this approach. The 60/70 bitumen exhibited

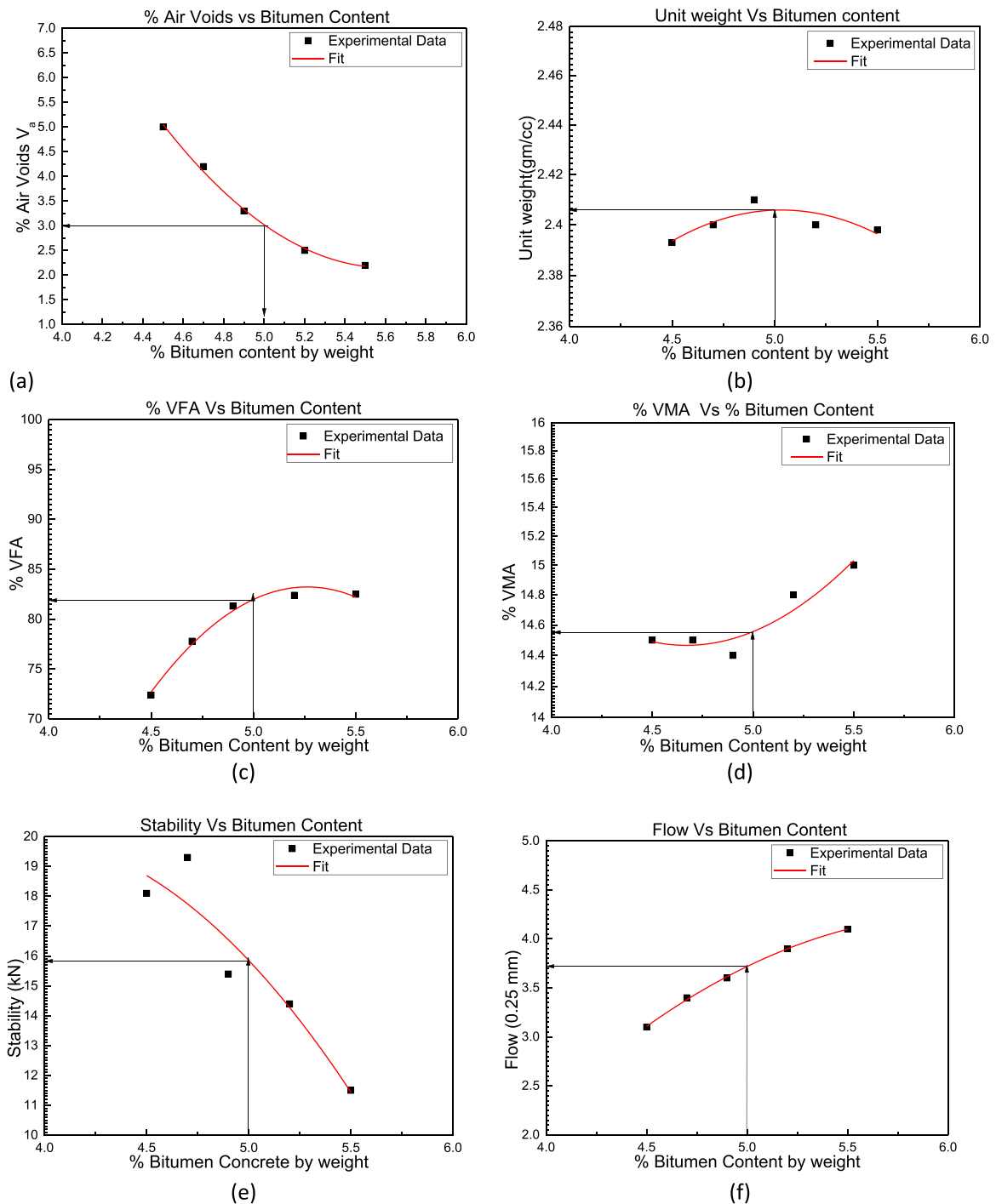


Fig. 9. Stability and Flow Analysis Graphs for optimum binder determination: (a) Relationship between the air voids and bitumen content, (b) Relationship between the asphalt unit weight and bitumen content, (c) Relationship between volume filled with asphalt and bitumen content, (d) Relationship between voids in mineral aggregates and the bitumen content, (e) Relationship between stability and bitumen content (f) Relationship between flow and bitumen content.

certain characteristics at low strain levels, as demonstrated in Fig. 8 and at high strain levels.

In summary, Natural bitumen from Illubirin appears to possess properties similar as 60/70 bitumen concerning fatigue life among the Natural bitumen samples. The laboratory properties of Natural bitumen from Illubirin obtained prompted the trials of using and deploying it for field trials. In addition, the proximity of Natural bitumen from Illubirin to the test section was another factor that was used for the selection. While the NOPB was only used for priming the road before asphalt laying as described in the field application section of this manuscript.

3.6. Marshall flow and stability properties

The Marshall flow and stability properties are crucial to evaluate the performance and behaviour of asphalt concrete mixtures in the Marshall mix design method. The asphalt mixture properties obtained on varying bitumen content for the hot asphalt mixture is presented in Table 10. As shown in Fig. 9 the bitumen content selected for the mixture design was found to be at 5 % bitumen content, which fulfills all the design criteria as shown Table 11.

The Marshall stability and flow results for the optimum binder determination selection using Petroleum bitumen alone are presented in Fig. 9.

The performance of incorporating Natural bitumen by replacing the optimal binder content in the reference mixtures in asphalt mixtures was evaluated. The optimal binder content was replaced with Natural bitumen possessing comparable characteristics in different proportions of 0 %, 5 %, 10 %, 15 %, and 20 %, constituting the maximum allowable within the total mixture. Following that, a set of 15 Marshall samples were manufactured for subsequent testing and analysis.

As presented in Table 12, the quantities of materials converted into grams were utilized to determine the optimal ratios of Natural bitumen. The extraction process was carried out upon incorporating the specified percentages, followed by sieve analyses to ensure compliance with gradation criteria. Furthermore, void analysis, stability, and flow criteria of the asphalt mixtures were evaluated. It was observed that there was an increase in stability as the Natural bitumen content increased, as shown in Table 13. It is noted that mix design A is the control without Natural Asphalt while mix B,C, D, and E are mix designs having varying percentages of Natural asphalt in 5,10,15, and 20 % of the total asphalt mixtures.

3.7. Cantabro Loss test

Cantabro Loss tests were conducted to understand the impact of moisture on the ravelling resistance of hot mix asphalt that were modified with Natural bitumen. This was done for both before and after exposure to moisture damage. The incorporation of Natural bitumen extracted from Illubirin into dense-graded asphalt concrete mixtures was observed to enhance the cantabro loss properties of the asphalt as shown in Fig. 10. The addition of 5 % Natural bitumen into the mixture was observed to improve the properties; however, even with 10 %, similar strength is exhibited compared to 0 %, aligning with the results obtained in the Marshall test in the previous section. The reference mixtures without Natural bitumen and other mixtures with Natural bitumen under dry conditions exhibited a Cantabro loss values below the maximum Cantabro loss criteria. The results of these tests are illustrated in Fig. 10.

Considering the moisture effect on the asphalt, it was observed that an increase in the proportion of Natural bitumen in the hot mixtures resulted in enhanced resistance to moisture effects. Specifically, mixtures containing 5 %, 10 %, and 15 % Natural bitumen displayed enhanced resistance compared to the mixture without Natural bitumen. However, the mixture containing 20 % Natural bitumen exhibited relatively poor performance compared to mixture A. Under wet conditions, the addition of 5 % into the control mixture showed more resistance to degradation during the conduct of the cantabro test, followed by 10 % and 15 % Natural asphalt. It can be seen that both the control mixtures, mixtures B, C and D, performed fairly similarly, with the exception of mixture E, which demonstrated the lowest performance in this pattern.

Further analysis was done to assess the moisture effect on the performance of natural asphalt using the Cantabro Loss Moisture Index (CLMI)[54]. It is noted that the CLMI provides a better assessment of the moisture effect on asphalt-modified Natural bitumen and is the ratio of the Cantabro loss percentage in the dry condition to that in the wet condition. The moisture effect on samples with

Table 10
Asphalt parameters.

ID	Asphalt content (%)	V _a (%)	Unit wt. (gm/cc)	VFA	VMA (%)	Stability (K.N)	Flow(mm)
1	4.5	4	2.393	72.4	14.5	18.3	3.1
2	4.7	3.5	2.4	75.9	14.5	19.3	3.4
3	4.9	2.7	2.42	81.3	14.4	15.4	3.6
4	5.2	2.6	2.407	82.4	14.8	14.4	3.9
5	5.5	2.2	2.398	85.3	15	11.5	4.1

Note:Va: Air voids; VMA: Voids in minerals aggregates; VFA: Voids filled with bitumen.

Table 11
Summary criteria of selected bitumen content [42].

Parameter	Design	Criteria
Flow	3.7	2–4 mm
% Air Void	3 %	3–5 %
Volume filled with asphalt	81 %	75–82 %
Stability	15.5 %	≥ 3.5 kN
% Bitumen content	5 %	5–8 %

Table 12
Optimal Design matrix for binder replacement using Natural bitumen.

Mix design	% of NA in Mix.	NA Composition		Wt. of PB(g)	% N.B	% P.B.
		Sand(g)	NB(g)			
A	0	0	0	60	0	5
B	5	60	7.2	52.8	0.6	4.4
C	10	120	14.4	45.6	1.2	3.8
D	15	180	21.6	38.4	1.8	3.2
E	20	240	28.8	31.2	2.4	2.6

Note: NA: Natural asphalt, NB: Natural bitumen, PB Petroleum bitumen.

Table 13
Summary data for asphalt-modified Natural bitumen.

Mix design	% NA	Bitumen content (%) 5–8	VFA 75–82 %	Air void 3–5 %	Stability (kN) ≥ 3.5 kN	Flow (mm) 2–4(mm)	Gradation limit WE/NWE	Criteria
								P/F
A	0	5	81.3	3	15.4	3.6	WE	Pass
B	5	5	76.8	3.6	21.4	3.2	WE	Pass
C	10	5	78.1	3.4	22.7	3.7	WE	Pass
D	15	5	65.9	5.9	21.3	3.20	NEW	Fail
E	20	5	85.6	2.3	20.7	3.7	NEW	Fail

Note: WE: Within the gradation envelope, NWE: Not Within the gradation envelope, NA: Natural Asphalt; VFA: Volume filled with asphalt, P: Pass, F: Fail.

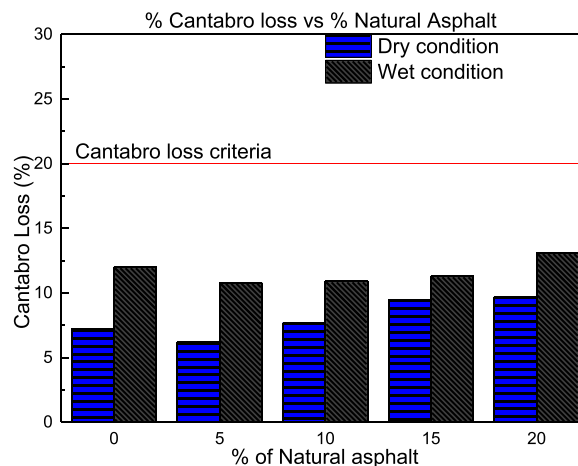


Fig. 10. Cantabro loss % of asphalt-modified Natural bitumen.

15 % Natural bitumen shows the highest resistance to moisture compared to samples with 0 %, 5 %, 10 %, and 20 %.

As can be seen from the analysis above, different percentages of Natural bitumen inclusion into the asphalt mixtures provide a slightly different assessment. In order to have a better understanding, the statistical tool was used to identify the most suitable conditions for the mixtures.

3.8. Statistical analysis

The influence of Natural bitumen with varying percentages on the Marshall stability and flow values was studied using the analysis of variance ANOVA tool kit [55]. The fundamental parameters that describe either to accept or reject the criteria are the p-value and F-value. The p-value determines the significance of and impact of increased Natural bitumen on the outcome in the Marshall criteria. Hence if the p-values are less than 0.05 for the confidence level of 95 %, it should be accepted that the percentage of Natural bitumen influenced the selected criteria. While the F-value categorizes variables based on their degree of significance, and with a higher value indicating greater influence on the overall performance of the mixture.

The p-value for the flow, air void and unit weight were found to be higher than 0.05, which means that adding Natural bitumen did not affect their values. As shown in Table 15 the p-value for stability and void filled with asphalt(VFA) is less than 0.05, indicating that the addition and gradual increase in the percentage of Natural bitumen significantly impacts the strength properties. The F value for VFA is the highest which shows that the addition of Natural bitumen has a significant influence on the VFA of the asphalt mixtures which can also be seen in Table 10.

In summary, the statistical analysis indicates that the addition of Natural asphalt to the asphalt mixtures has significant influences on the voids filled with asphalt which is an important factor in determining the performance and durability of asphalt mixtures.

4. Field performance evaluation

As discussed in the previous sections and asphalt modified with Natural bitumen demonstrates comparable performance to conventional hot mixtures in the laboratory. The following section presents findings of using mix design A (control) and mix design B(5 % Inclusion of Natural asphalt) (see Table 12) in the previous section.

4.1. Asphalt material production/ Laying

The second phase of the study was to use the best mix design meeting all criteria i.e. mix design B design by incorporating Natural asphalt into the hot mixtures for field trials. Monitoring of the sections were done through visual inspection at both sections. Mix design A and mix design

B, were prepared at the plant for the trial in accordance with Federal Ministry of Works specifications [56]. The Natural asphalt was mixed at the site at 170 °C and construction was done at a temperature between 120 and 140 °C (see Table 12). To ensure proper adhesion between the wearing course and the base course layer, an emulsion prime coat was sprayed at a rate of approximately 0.4–0.61 l per square meter. Finally, a hand roller was used to compact the layers effectively with about 10–15 passes, according to maintenance practice in Nigeria as shown in Fig. 11. A total of 30 sections(were placed on the road with a low-traffic single carriage road.

4.2. Visual Inspection

As shown in Table 16, the visual inspections of the patched areas were done by examining all the 30 sections at different durations. It was observed that after 6 months there was no defect on the patched surface of the both types of sections were the mixtures A and mixtures B were used. However, after 24 months of weathering and traffic, gradual defects were observed mostly at the edges of the patched sections.

About 65 % of the test sections utilizing mixture A failed and about 70 % of the test sections having mixture B had also failed. In summary, since both A and B mixture types yielded similar outcomes, it is reasonable to conclude that mixture B with Natural asphalt neither exhibited inferior nor superior performance compared to petroleum bitumen asphalt mixtures.

5. Conclusions

The research presents the assessment of the performance of Natural asphalt as an alternative material for low-volume roads. Furthermore, the results of the field trial using Natural bitumen-modified asphalt mixtures with Petroleum asphalt on low-volume roads are compared. The fatigue performance of the Natural bitumen sourced from Illubirin was found to be comparable to that of the Petroleum bitumen. However, the fatigue performance of the Natural bitumen from Isowa and the plain binder was not as promising in terms of having desired stiffness values. An easy-to-implement ranking criteria based on Marshall mix design was developed for the selection of the percentage of Natural asphalt to be added to the hot mixtures. Based on the results obtained and discussions presented, the following conclusions have been drawn.

- i. The viscoelastic properties of the Natural bitumen obtained from the Natural asphalt were found to be comparable to those of the Petroleum binder. The results of the physical and rheological tests conducted, when compared to the Petroleum binder, fell

Table 14
Cantabro loss test.

Mix design	% of NA. in Mix.	Average % Cantabro Loss		Index		
		Dry	Wet	Dry	Wet	CLMI
A	00.00	7.20	12.00	1.00	1.00	1.667
B	05.00	6.20	10.80	0.86	0.90	1.742
C	10.00	7.70	10.90	1.06	0.90	1.416
D	15.00	9.50	11.30	1.31	0.94	1.189
E	20.00	9.70	13.10	1.34	1.09	1.351

Table 15
ANOVA analysis of the parameters with different percentages of Natural bitumen.

Parameter	S.S.	df	M.S.	F	p-value
V _a (%)	358.496	9	101.124	3.14328	0.11418
Unit Wt (gm/cc)	394.4154	9	144.4152	4.62128	0.0638
VFA (%)	11,869.581	9	11,404.12	196.00954	0.000001
VMA (%)	304.076	9	53.824	1.72063	0.226
Stability (kN)	547.365	9	265.225	7.52038	0.02536
Flow(mm)	356.544	9	106.276	3.39719	0.10255

Note: V_a: Air voids; VMA: Voids in mineral aggregates.

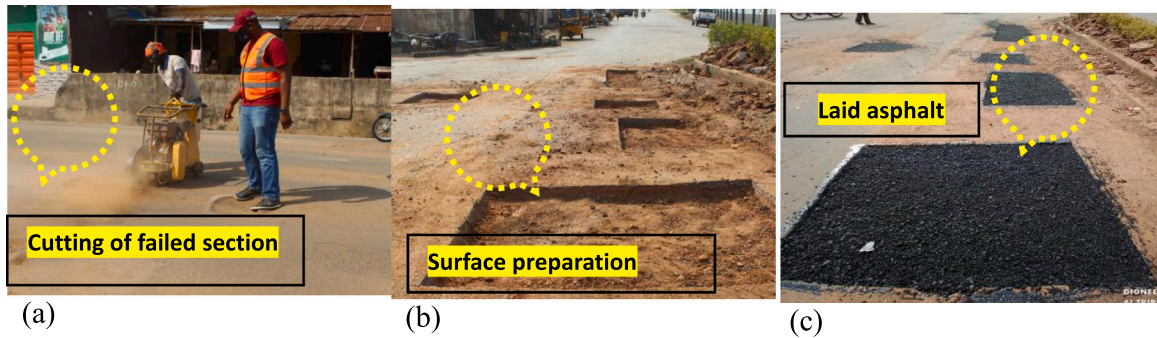


Fig. 11. The construction process of using Natural bitumen and Petroleum bitumen.: (a) cutting off the failed portions, (b) prepared surface ready for priming, (c) Asphalt material laid and ready for compacting.

within the specified limits for road application. Besides, it was reported that there is a high variability among properties of the Natural bitumen and aggregates obtained from Natural asphalt collected at different locations.

- ii. The naturally occurring Plain Binder (NOPB), in its natural state, exhibits characteristics of soft bitumen based on the softening point and penetration test values. This makes it suitable for potential applications as an asphalt extender or a viscosity reducer. However, further studies are needed to explore the possibility of utilizing NOPB as a rejuvenator and binder for warm mix asphalt.
- iii. The Marshall stability values increased in Natural bitumen-modified asphalt mixtures with the increase in Natural bitumen percentage. Nevertheless, it was found that the addition of 10 % of Natural bitumen met all the Marshall mixture design criteria.
- iv. The Cantabro loss percentage decreased in value as the percentage of Natural bitumen in the hot mixtures increased. This improvement reached up to 5 % in dry conditions and up to 15 % in wet conditions.
- v. The statistical analysis revealed that the modification of asphalt with Natural bitumen significantly influences the Voids in Mineral Aggregates (VMA) and Marshall stability values compared to other parameters.
- vi. Field trials show that a mixture prepared with Natural asphalt designed using the proposed framework performs similar to Petroleum bitumen. Further investigations revealed that, after six months, sections modified with Natural asphalt exhibited no signs of deterioration. Subsequent inspections after 24 months showed a similar rutting trend to commonly used petroleum binders.

Table 16
Some selected locations where Natural bitumen and Petroleum bitumen were used.

Site	6 months	12 Months	24 Months	Remarks
1 (PNA)	 No defects	 No defects	 Defects mostly at the edges of patched sections	The sections that underwent treatment with Petroleum asphalt-modified Natural bitumen exhibited no signs of distress at both the 6-month and 12-month marks. However, after 24 months, there was a gradual emergence of potholes along the edges of the patched sections.
2(PA)	 No defects	 No defects	 Defects mostly at the edges of patched sections	The sections that underwent treatment with Petroleum asphalt displayed no signs of distress at both the 6-month and 12-month. Moreover, even after 24 months, there was no gradual formation of potholes along the patched sections.
3 (PNA)	 No defects	 No defects	 Minimal defects on the patched sections	The sections that received treatments with both Petroleum asphalt-modified Natural bitumen showed no signs of distress at both the 6-month and 12-month. Furthermore, even after 24 months, there was no formation of potholes along the patched sections.
4(PA)	 No defects	 No defects	 Defects on the patched sections	The sections patched with Petroleum asphalt exhibited no signs of distress at both the 6-month and 12-month intervals. However, after 24 months, a significant formation of potholes was observed along the patched sections.

Note: PA: - Petroleum bitumen, PNA: Petroleum bitumen modified Natural asphalt.

In summary, utilizing Natural bitumen in pavement construction offers the advantage of reducing dependency on Petroleum-sourced bitumen for countries like Nigeria. In the laboratory, the incorporation of Natural asphalt enhanced certain Marshall properties in comparison to petroleum bitumen, while in the field, performance shows that the behaviour is comparable. The use of Natural asphalt may provide a good alternative to local road industries.

Future recommendations

- The recyclability of Natural asphalt should be evaluated to help predict a more practical replacement of aged Natural bitumen with new Petroleum bitumen.
- The environmental, economic, and life cycle cost of using Natural asphalt for pavement construction should be considered to have field-based experimental data.
- The cohesion and adhesion properties of Natural bitumen on aggregates should be evaluated for better performance.
- Life cycle assessment should be conducted on using Natural asphalt for pavement construction in real-life conditions.
- The rejuvenating potentials of Natural Bitumen in old asphalts to improve the performance of asphalt should be evaluated.
- The interaction between PB and NA using FTIR and other available tools and its effects on physical properties such as softening point, viscosity, ductility, penetration values, and rheological properties should be studied.

CRediT authorship contribution statement

Poot Marco: Writing – review & editing, Supervision, Methodology. **Duna Samson:** Writing – review & editing, Supervision, Formal analysis, Conceptualization. **Cor Kasbergen:** Writing – review & editing, Resources, Project administration. **Daniel Akinmade:** Writing – review & editing, Validation, Methodology, Investigation, Data curation, Conceptualization. **Sandra Erkens:** Writing – review & editing, Supervision, Resources, Methodology. **Kumar Anupam:** Writing – review & editing, Supervision, Methodology, Conceptualization.

Funding

This research received a grant from Nigerian Building and Road Research Institute in Nigeria, Tu-Delft Global Initiative in the Netherlands and TUDELFT is acknowledged for providing the study facilities.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: AKINMADE OLUWATOSIN DANIEL reports financial support was provided by TU-DELFT GLOBAL INITIATIVE. AKINMADE OLUWATOSIN DANIEL reports financial support, administrative support, equipment, drugs, or supplies, and travel were provided by Nigerian Building and Road Research Institute. AKINMADE OLUWATOSIN DANIEL reports financial support, administrative support, article publishing charges, equipment, drugs, or supplies, statistical analysis, and writing assistance were provided by Delft University of Technology. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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