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Physical Properties of Unaged and Short-Term Aged (STA) Liquid Epoxidized Natural Rubber (LENR) Modified Binders

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Abstract

Polymer-modified asphalt (PMA) binders have been utilized extensively to improve the performance and durability of asphalt pavement. Each type of polymer or modifier leads for specific alterations in the performance of the asphalt binder. This study examines how the physical properties of 60/70 penetration grade asphalt binder are influenced by the presence of liquid epoxidized natural rubber (LENR). Specifically, five different proportions of LENR were examined: 0%, 3%, 6%, 9%, and 12% by weight of the asphalt binder. A rolling thin film oven procedure was conducted to replicate the short-term aging of the asphalt binder samples. The unaged and short-term aged samples of LENR-modified asphalt binders were evaluated for physical testing (penetration, softening point, and viscosity). The results show that with the increasing LENR, the penetration reduced, the softening point and viscosity increased. The findings in this study indicated that 3% LENR showed good potential as modifier in asphalt binder.

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1. Introduction

Asphalt concrete mixes have been used in flexible pavements for almost a century, but with increased highway traffic, the necessity for high-quality, long-lasting asphalt mixtures have become vital. Heavy and high-load traffic has been demonstrated to degrade pavements early in their design lives, increasing repair and maintenance expenditures [1]. As a result, various road construction enhancement techniques have been developed, emphasizing improving the technical aspects of current road pavement materials to increase their longevity under increased traffic loads. Furthermore, because the asphalt binder is a scarce and non-renewable resource, asphalt binder modification has become a worldwide priority [2].

Asphalt aging is a key factor in the deterioration and cracking of pavements. It starts during the production of the asphalt mixture and progresses as the material undergoes further oxidation in the field, eventually diminishing the longevity of flexible pavements. Exposure to oxygen, heat, and ultraviolet (UV) light causes asphalt to become brittle and fragile [3]. Short-term aging (STA) happens during the construction phase and is associated with the preparation of asphalt mixtures at elevated temperatures, while long-term aging is caused by external factors like traffic loading, temperature, and environmental conditions [4]. Short-term aging increases the stiffness and complex modulus (G*) of asphalt binders, enhancing their performance at high temperatures [5]. The use of polymers as modifiers in asphalt binders has been a subject of research for an extended period. Polymer modification of asphalt binders has been a long-standing research focus, offering potential solutions to enhance performance and extend pavement service life.

Polymer-modified asphalt (PMA) has enhanced rutting, abrasion, cracking, fatigue, stripping, bleeding, and aging resistance at high temperatures, as well as increased flexibility at low temperatures [6]. The use of polymers improves elasticity, cohesion, stiffness, and adhesion of asphalt [7]. When compared to conventional asphalt binder, PMA has numerous significant advantages. They are more elastic, flexible, resilient, less prone to temperature changes and aging, and have better adhesive and cohesive properties [8]. As a result, the primary goal of polymer-modified asphalt is to increase pavement life service. Many types of polymers have been documented for use in pavement materials, including natural rubber [9]–[11].

Based on the previous research, incorporating natural rubber latex (NRL) into the 60/70 PEN bitumen results in enhanced physical and rheological properties of the binder [10]. As the quantity of NRL increases, it leads to greater stiffness in the mixture, accompanied by increased viscosity and reduced penetration. Similarly, the results of the physical properties, specifically penetration and softening point, showed that the introduction of NRL stiffened the binder, regardless of the mixing speed applied, mainly due to the elastic nature of NRL [12]. When the stiffness of asphalt binder increases, it can lead to several disadvantages, including reduced flexibility, increased susceptibility to thermal cracking and decreased rut resistance. Elevated stiffness levels can reduce the flexibility of asphalt pavement, potentially leading to cracking when subjected to heavy loads or temperature fluctuations [13]. Stiffer asphalt binders tend to be more susceptible to thermal cracking due to their restricted capacity to expand and contract in response to temperature fluctuations, which can result in early pavement damage. Similarly, pavements containing stiffer binders may be at greater risk of rutting, particularly in areas with high traffic volume or heavy loads, as the binder may fail to deform sufficiently under pressure [14]. Rutting in asphalt pavements decreases the structural integrity and overall service life performance of the pavement [15].

Another type of natural rubber product is epoxidized natural rubber (ENR). ENR is chemically modified natural rubber latex (NRL) that has been epoxidized with a proxy formic acid, which provides improved heat and chemical resistance in conjunction with a higher glass transition temperature [2]. However, ENR is in a solid form, making it challenging to blend, and it necessitates elevated temperatures to incorporate it with asphalt binder [16] effectively. Liquid epoxidized natural rubber (LENR) is a new and green replenishable material created by breaking down the high molecular weight of ENR into short lengths of polymeric chains [17]. In contrast to ENR, LENR offers several advantages as modifier due to its lower energy requirements and ease of processing [17], [18]. Additionally, its readily modifiable nature is facilitated by its low molecular weight. Compared to ENR, LENR is softer, stickier, and flows better at higher temperatures [19]. However, there are limited findings available on the use of LENR in pavement materials. Therefore, this study investigates how liquid epoxidized natural rubber (LENR) affects the physical properties of asphalt binder modified by LENR for unaged and short-term aged conditions.

2. Materials and Test Method

2.1 Asphalt Binder

Kemaman Bitumen Company supplied the base asphalt binder (PEN 60/70) used in this study. The basic properties of PEN 60/70 are shown in Table 1. The material complied with Malaysian Standard MS 124, as specified by JKR standard (JKR, 2008).

Table 1 *Properties of asphalt binder PEN 60/70*

Properties	Result	Test Standard	Requirement (MS 124)
Penetration at 25°C (dmm)	63.9	ASTM D5	60-70
Softening point (°C)	50.7	ASTM D36	48-56
Viscosity at 135°C (Pa.s)	0.8	ASTM D4402	3 (Max)

2.2 Liquid Epoxidized Natural Rubber

LENR is a special material manufactured and supplied by the Malaysian Rubber Board (LGM) with 50% epoxidation level of ENR. The preparation of LENR can be obtained from Yusof et al. [17]. Fig. 1 depicts the LENR in a semi-solid state, soft and sticky. Table 2 presents the basic properties of LENR.

Fig. 1 *Liquid epoxidized natural rubber (LENR)*

Table 2 *Properties of LENR*

Properties	Result
Epoxidation level (%)	49.99
Gel content (%)	0.1961
Molecular weight (g/mol)	22171
Density (g/cm^3)	1.0359

2.3 Preparation of LENR Modified Asphalt Binder

LENR was blended with base asphalt PEN 60/70 using a Silverson high shear mixer at 140°C and 4000 rpm for 30 minutes [18]. The base binder was consistently heated until attained a uniform temperature of 130°C, then the binder was modified using LENR at 3%, 6%, 9% and 12% [18]. Subsequently, all the modified asphalt binder and control samples (unaged and short-term aged) were subjected to penetration, softening point and viscosity.

2.3.1 Penetration Test

The penetration test was performed to ascertain the consistency of the asphalt binder. This consistency is often represented as the distance in tenths of a millimetre that a standard needle penetrates perpendicularly into a sample of the material according to the ASTM D5 [20] using the standard of 100 g load, 5 seconds duration, and 25°C testing temperature. Before testing, the asphalt binder was heated to 130°C to soften and then poured about 50 grams into the penetration cup. After cooling, the sample was immersed in a water bath at 25°C for 1 hour.

2.3.2 Softening Point Test

This testing is performed according to ASTM D36 [21] to measure the sensitivity of asphalt binder to temperature fluctuations using the standard protocol. The sample was put into the rings and placed in a 5°C water bath to condition. The steel ball was put on asphalt samples in steel ring brass to assess the degree of softening achieved by the asphalt binder. The thermometer recorded the temperature when the asphalt binder around the ball touched the bottom plate for each ring and ball.

2.3.3 Viscosity Test

The viscosity test is used to determine the workability of the asphalt binder. This test was conducted on the control and modified asphalt binder according to ASTM D4402 [22]. About 20 grams of asphalt binder were poured into the viscosity mold and tested at 135°C and 165°C.

2.4 Rolling thin film Oven

Rolling thin film oven (RTFO) aims to imitate short-term asphalt binder aging when exposed to high temperatures during manufacturing and placement aging at the premix facility. This testing was conducted following the standard ASTM D2872 [23]. 8 bottles with 35 g of asphalt binder in each bottle were rotated for 85 minutes at a temperature of 163°C with 4000 mL/min airflow. After a brief ageing time, the samples were tested for physical tests (penetration, softening and viscosity) to compare with the unaged sample.

3. Results and Discussion

3.1 Penetration Test

The consistency of base binder and the LENR-modified binder was assessed using the penetration test. Fig. 2 presents the result of penetration of the asphalt binder for unaged and short-term aged samples. The base asphalt binder is 60/70 penetration grade, characterized by a penetration value of 63.9 dmm. After the addition of LENR, it can be seen that the penetration value decreased to 55.5 dmm for 3%, and slightly increased to 57.2 dmm (6%), 58.3 dmm (9%), and 60.3 dmm (12%) respectively for unaged condition. The same trend was observed for STA condition samples. This shows that LENR considerably influences the penetration values of the modified binders compared to the base binder. The stiffness enhancement of the LENR-modified binder can be attributed to the reinforcing impact of incorporating rubber products into the base binder. An earlier investigation similarly noted that the enhanced stiffness of the latex-modified binder could be attributed to the asphalt base binder's strengthening achieved by adding natural rubber latex (NRL) [12]. The substantial decrease in penetration values could prove advantageous in fortifying the modified binder's ability to withstand temperature-related issues, potentially leading to improved durability and performance throughout its service life [15]. However, the findings for 6% to 12 % of LENR contradict Al-Mansob et al. [18], where the proportion of LENR increases, the penetration decreases. The differences in these findings show that different sources of asphalt binder and different densities of materials can produce unique interactions for polymer-modified asphalt [24]. The penetration of unaged LENR or ENR-modified binders is typically lower compared to base asphalt, reflecting greater stiffness and reduced flowability. This enhancement is attributed to the elevated stiffness and elasticity imparted by LENR or ENR, which significantly improve the asphalt binder resistance to deformation. However, after short-term aging, the penetration of ENR-modified binders increases, indicating that aging impacts the binder's structure. This change may stem from modifications in the polymer network or oxidation processes that alter the binder's consistency, making it softer and more penetrable [16], [25].

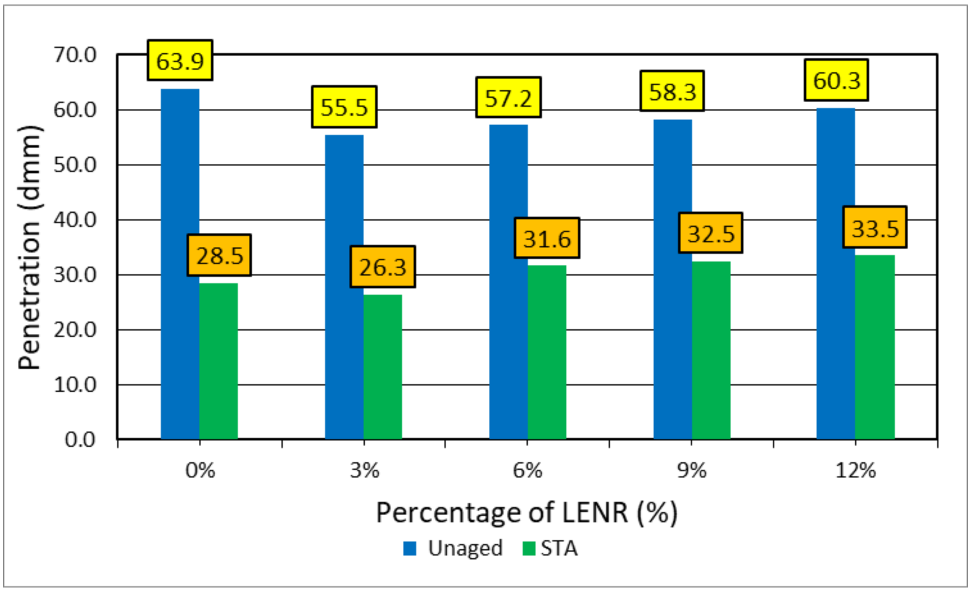

Fig. 2 *Penetration test result for unaged and short-term aged samples*

3.2 Softening Point Test

Fig. 3 displays the outcomes of the softening point test for both unaged and short-term aged samples. The softening point test was employed to ascertain the highest service temperatures that the asphalt binder can withstand. It is evident that there is an increasing trend in the softening point temperature within the range of 0% to 3% LENR modification, while the binders modified with 9% to 12% LENR exhibit a decreasing trend in softening point temperature. The softening point test results reveal that when a 3% ratio of LENR by asphalt weight is introduced to both unaged and short-term aged (STA) binders, there is an initial increase in softening point temperature, followed by a gradual decrease, as depicted in Figure 4. In unaged binders, the softening point values for 6%,9% and 12% are 61.9°C, 60.6°C, and 58.9°C, whereas for short-term aged binders, the softening point reduced to 52.4°C, 52.0°C, and 51.1°C, respectively. The decrease in penetration can be attributed to the hardening effect caused by the addition of ENR to the base asphalt, which also increases the softening point value [25]. This indicated that incorporation of LENR into asphalt improves the binder's stiffness and elasticity, especially at elevated temperatures.

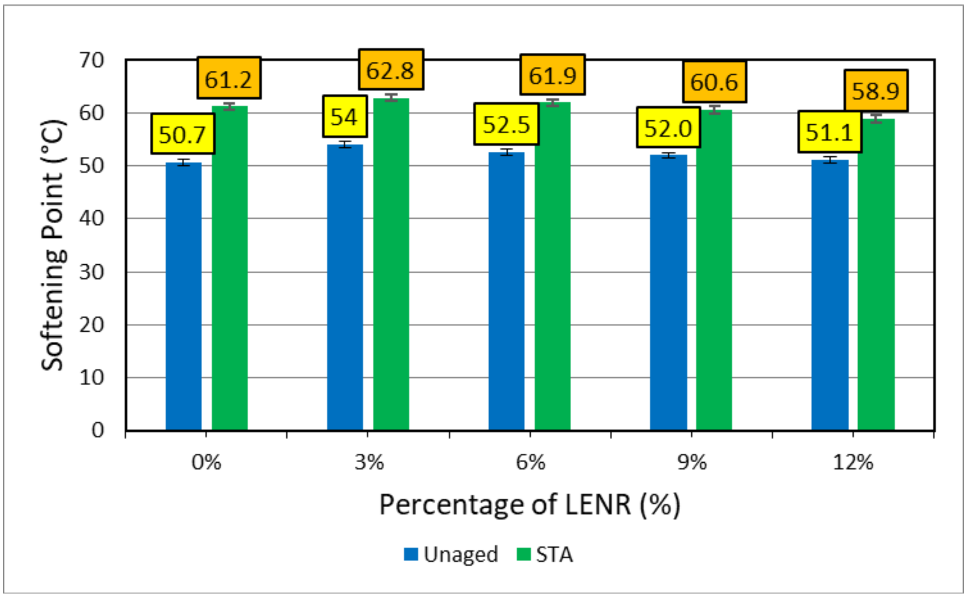

Fig. 3 *Softening point test result for unaged and short-term aged samples*

3.3 Viscosity test

The study investigates the viscosity of LENR-modified asphalt binders at weight ratios of 3%, 6%, 9%, and 12% for unaged and short-term aged binders using a Thermal Brookfield Viscometer at temperatures of 135°C and 165°C. The findings indicate that the viscosity of the LENR-modified asphalt binders surpasses that of the unmodified asphalt binder, as illustrated in both Fig. 4 and Fig. 5. Viscosity values consistently increase as the ratio of LENR added to the asphalt binders increases, and this trend is observed for both temperature conditions. Previous studies have also reported similar discoveries, demonstrated increased bitumen viscosity when modified with latex [26]. At 135°C, there is an increasing trend in viscosity values for unaged samples, which are 6.1%, 13.4%, 14.7%, and 18.7% for 3%, 6%, 9%, and 12% of LENR, respectively. For short-term aged samples at the same temperature, the viscosity values are 1.952, 2.086, 2.110, and 2.184 Pa.s, respectively. At 165°C, it was observed that the viscosity values for both conditions (unaged and short-term aged) also exhibit an increasing trend. At 165°C, the viscosity values for unaged samples are 42.0%, 26.1%, 24.9%, and 17.1% for 3%, 6%, 9%, and 12% of LENR, respectively. The viscosity values for short-term aged (STA) samples at the same temperature are 0.612, 0.641, 0.735, and 0.808 Pa.s, respectively. The abrupt increase in viscosity can indeed have an adverse impact on asphalt mixtures with aggregates. High viscosity makes it challenging for the mixer to spray and mix the asphalt with the aggregate effectively. This situation may lead to potential clogs or disruptions in the mixture system [14].

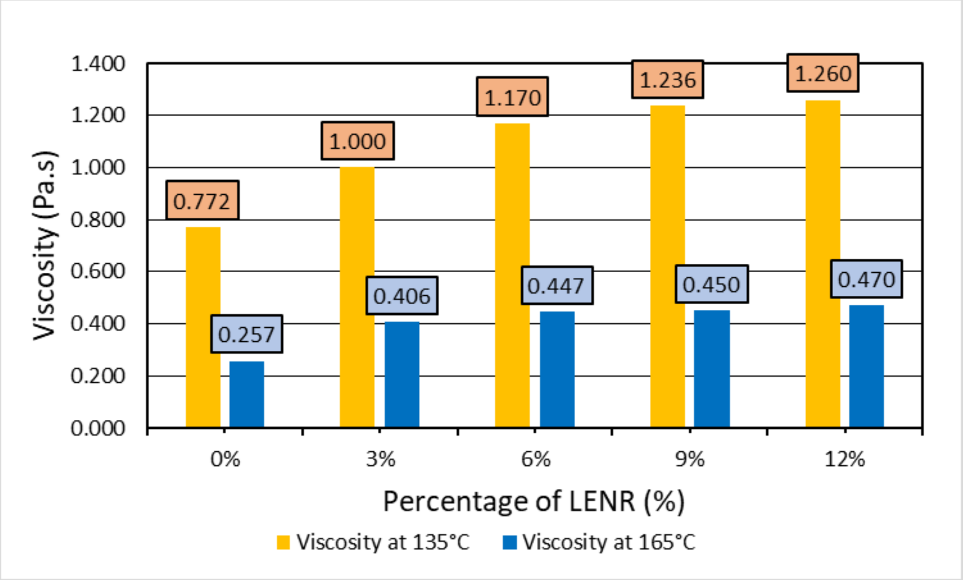

Fig. 4 *Viscosity test result for unaged samples*

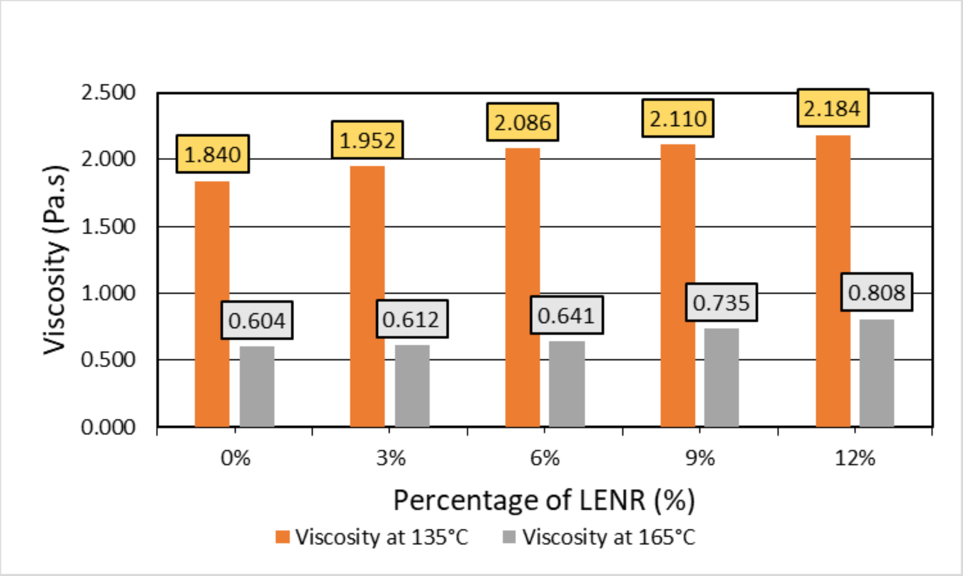

Fig. 5 *Viscosity test result for short-term aged samples*

3.4 Mass Loss

This test aims to assess how heat and air affect the mobility of an asphalt binder film and to collect residue for subsequent examinations. This procedure is denoted as per ASTM D2872. Table 3 displays the mass loss outcomes for various percentages of LENR in the asphalt binder following the RTFOT process. The specified criteria for determining the acceptable mass loss percentage of asphalt binder align with the standards outlined in ASTM D2872. The base binder exhibits a mass loss of 0.236%, which is lower than the mass loss values observed for the 6%, 9%, and 12% of LENR-modified asphalt, which are 0.332%, 0.328%, and 0.308%, respectively. A negative value indicates a loss of mass compared to the initial mass after undergoing the rolling thin film oven test. However, the negative symbol was removed in this study to simplify the data. The findings show that among the various percentages of LENR added to the asphalt binder, 3% of LENR demonstrates the lowest percentage of loss, measuring 0.171%. In contrast, asphalt binders containing 6% of LENR have the highest loss percentage among the different LENR content levels, measuring 0.332%. Indeed, all the other percentages of LENR-modified asphalt can be considered suitable for pavement construction since they have a mass loss of less than 1%, which aligns with the standard specification. When evaluating various modified binders for LENR, a 3% content appears most appropriate as it exhibits the lowest mass loss value.

Table 3 *Mass loss of LENR-modified asphalt*

Mix Type	**Mass before**	**Mass after**	**Percentage Loss (%)**	**Specification < 1.0%**
0% LENR	160.133	160.040	0.236	Pass
3% LENR	137.698	137.638	0.171	Pass
6% LENR	138.627	138.501	0.358	Pass
9% LENR	166.151	166.036	0.328	Pass
12% LENR	165.936	165.827	0.308	Pass

3.5 Determination of Optimum LENR Content

Table 4 presents the ranks and scores for u-modified and LENR-modified binder. Various physical characteristics such as softening point, penetration and viscosity of the modified bitumen were evaluated to ascertain the optimal LENR content. The scores range from 1 to 6, where 1 signifies excellent performance across all six samples (base asphalt binder, 0%, 3%, 6%, 9%, and 12% LENR), and 6 represents the poorest performance. Once the scoring is finished, the total score is calculated by adding individual values. A lower total score indicates better performance, while a higher score reflects worse performance. The results showed that the 3% LENR content performed excellently, meeting all the required parameters.

Table 4 *Score and ranking of conventional and LENR-modified binders*

Test	0% LENR	3% LENR	6% LENR	9% LENR	12% LENR
Penetration @ Unaged	5	1	2	3	4
Penetration @ STA	5	1	2	3	4
Softening Point @ Unaged	5	1	2	3	4
Softening Point @ STA	5	1	2	3	4
Viscosity @ Unaged	1	2	3	4	5
Viscosity @ STA	1	2	3	4	5
Mass loss	2	1	5	4	3
Total score	24	9	19	24	29
Rank	3	1	2	3	4

Notes: 1 = good performance, 5 = poor performance

Given the objective of enhancing the physical properties of bitumen by adding LENR, a percentage of 3% is deemed optimal in this study. The unmodified bitumen exhibits a penetration value of 63.9 dmm in the penetration test. The existence of LENR is demonstrated to enhance the hardness of bitumen in the unaged and short-term aged specimens. The addition of LENR reveals specific upward trends in the penetration results. The lowest penetration values were obtained at 3%. The findings indicate that the initial addition of LENR leads to an increase in the softening point. This suggests that incorporating a significant quantity of LENR may not be advantageous for enhancing the softening point of asphalt binder. When 3% of LENR is incorporated, the softening point reaches its peak temperature at 54°C. A stiffer matrix will increase challenges in softening the mixture, consequently leading to a higher softening point [27].

The viscosity test shows that the viscosity of LENR-modified asphalt binder samples decreases as the temperature rises under unaged and short-term aged conditions. This phenomenon might be attributed to the diminished cohesion of the binder, especially at elevated temperatures, resulting in a softened binder [18]. It is also worth noting that all the modified binders exhibited higher viscosity than the base asphalt, and the viscosity of the modified asphalt increased as the LENR content increased. At 3% LENR modification, the modified binders exhibited the lowest viscosity, with enhancements of 29.5% and 6.1% for unaged and short-term aged conditions, respectively, compared to the base asphalt at 135°C. Conversely, the most significant increase in viscosity among the modified binders was noted in the case of the 12% LENR modified asphalt binder, showing a rise of 0.488 Pa.s compared to the base asphalt. As the binder undergoes aging, it experiences mass loss due to the volatilization of its viscoelastic components, as stated by Neto et al. [28]. Consequently, lower mass loss signifies enhanced resistance to permanent deformations and pavement fatigue. Upon analysis, mixtures modified with 3% LENR content exhibited the least mass loss, indicating greater durability against pavement deformations and fatigue [28].

4. Conclusion

This study assessed the impact of varying quantities of LENR added to asphalt binder on its physical properties. This research revealed that incorporating LENR as an additive in asphalt binder enhances its characteristics by increasing stiffness and offering improved resistance to temperature-related issues. Considering the outcomes of this study, it is advisable to employ LENR as a modifier for asphalt binders to achieve improved elasticity, penetration, softening point, and viscosity. The optimal recommendation is 3% LENR as the ideal content, as the results suggest that asphalt binder modified with 3% LENR demonstrates superior outcomes in physical properties compared to unmodified asphalt binder. It also appears that adding up to 3% of LENR blends effectively with the asphalt binder compound and enhances the softening point, surpassing the conventional bitumen. The results presented in this paper cover the preliminary stages and require further analysis. Therefore, the performance of LENR-modified binder in asphalt mixture would be studied.

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Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Nurul Hidayah, Khairul Nizam Mohd Yunus, Azuan Poharan, Madi Hermadi, Nurul Hayati Yusof, Mazlina Mustafa Kamal;* ***data collection:*** *Nor'Yuhanis Roslan, Azuan Poharan;* ***analysis and interpretation of results:*** *Nor'Yuhanis Roslan, Nurul Hidayah Mohd Kamaruddin;* ***draft manuscript preparation:*** *Nor'Yuhanis Roslan, Nurul Hidayah Mohd Kamaruddin. All authors reviewed the results and approved the final version of the manuscript.*

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