Smart Green Materials

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Research Article

Effect of Kaolin on Asphalt Concrete Properties Under Aging Conditions

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Articles History: Received: 13 December 2024; **Revised:** 18 December 2024; **Accepted:** 26 December 2024; **Published:** 27 December 2024

Abstract

Asphalt modification is an essential process in enhancing the performance and durability of asphalt mixtures. Recently, many research has been carried out in order to shift the construction industry into a green and sustainable industry. This study investigates the effect of kaolin as a partial replacement for asphalt in asphalt concrete, focusing on its impact on the mechanical properties of the mixture under various aging conditions. The asphalt mixtures were subjected to Marshall stability, resilient modulus, and dynamic creep tests to assess stability, stiffness, and rutting resistance. The results show that the incorporation of kaolin improves the overall performance of the asphalt mixtures, with 6% kaolin replacement providing the most favorable balance between stability, stiffness, and flexibility. Unaged samples with higher kaolin content exhibited increased Marshall stability, resilient modulus, and dynamic creep modulus, indicating enhanced rutting resistance. Long-term aging further enhanced the mechanical properties, with kaolin-modified mixtures showing better performance compared to their short-term aged counterparts. These findings suggest that kaolin can be an effective modifier in asphalt mixtures, offering a sustainable and cost-effective solution to improve the durability and performance of pavement materials.

Keywords: Kaolin, Aging, Industrial Waste, Asphalt

Introduction

Traditional asphalt often exhibits limitations such as poor resistance to rutting, aging, and cracking under high-temperature conditions [1, 2, 3]. To address these challenges, various materials have been explored for asphalt modification, including polymers, rubber, and mineral fillers. Among these, kaolin clay has emerged as a potential alternative due to its abundant availability, low cost, and environmentally friendly properties. Kaolin, a naturally occurring alumino-silicate material, has shown promise in improving the mechanical properties of asphalt by enhancing its stiffness and resistance to permanent deformation. However, the application of kaolin in asphalt modification is still relatively underexplored, and its effects on the long-term performance of asphalt mixtures, especially under different aging conditions, remain unclear. According to [4], in Malaysia, the primary kaolin deposits of different source rocks were found in Ipoh, Bidor, and Sarawak and a secondary kaolin deposit found in the Mersing area.

Despite the potential benefits of kaolin as a modifier, there is a lack of comprehensive studies investigating its impact on the mechanical properties of asphalt under both short-term and long-term aging conditions [5, 6]. Previous research has primarily focused on kaolin's effect on the rheological properties of asphalt, but the correlation between kaolin content and the overall performance of asphalt mixtures, such as stability, stiffness, and rutting resistance, has not been fully addressed [7]. Furthermore, the optimum kaolin content for achieving enhanced performance while maintaining flexibility and durability remains unclear. This study aims to fill this gap by systematically evaluating the effects of kaolin at various replacement percentages on the mechanical properties of asphalt concrete, using a range of testing methods including Marshall stability, resilient modulus, and dynamic creep tests. The findings from this research will provide valuable insights into the feasibility of kaolin as a sustainable and effective modifier for asphalt in pavement applications.

As the construction industry continues to expand at an accelerated pace, the depletion of natural material resources has become a significant concern. The extensive reliance on virgin materials in construction has led to a dwindling availability of these resources, raising alarms about sustainability [8]. Asphalt, a key material in construction, is a black, viscous mixture of hydrocarbons obtained either naturally or as a byproduct of petroleum distillation. It serves as a binder for aggregates in pavement construction, which is essential for road infrastructure globally. However, the quality and performance of pavements often fail to endure over time, with frequent deterioration impacting societal activities and incurring high maintenance and reconstruction costs.

Simultaneously, kaolin-based waste, such as ceramics, is accumulating rapidly, posing environmental and logistical challenges [9]. The limited availability of recyclers accepting ceramic waste often results in these materials being discarded in landfills, contributing to environmental degradation [5]. The primary objective of this study is to discover the recommended percentage of kaolin clay as a partial replacement in asphalt mixtures to enhance their performance and sustainability. By incorporating kaolin clay at varying proportions, the study aims to investigate the performance of kaolin clay-modified asphalt mixtures under different aging conditions, including short-term and long-term aging, to simulate real-life pavement deterioration over time.

The significance of this study presented the effect of kaolin due to its aging condition. It is useful for future research in this field in order to have some or

https://doi.org/10.70028/sgm.v1i2.21

better performance of pavement by using industrial waste which is good for the environment without the needs to use virgin materials.

Literature Review

Clay has been extensively utilized across various industrial applications due to its numerous advantageous properties. It is broadly classified into four categories: clay, bentonite, industrial kaolins, and palygorskite–sepiolite [10]. Among these, industrial kaolins are particularly significant, with their composition predominantly consisting of kaolinite, accompanied by a smaller fraction of high-purity kaolin minerals. This unique mineralogical composition makes industrial kaolins highly desirable for applications requiring superior quality and performance. Kaolin clay or known as $Al_2Si_2O_5(OH)_4$) composed of SiO_2 (57.63%), Al_2O_3 (37.77%), Fe_2O_3 (0.86%), MgO (0.60%), CaO (0.35%), K_2O (1.80%), TiO_2 (0.61%), P_2O_5 (0.31%) [11]. Annually, approximately 8.5 million metric tons of processed kaolin are produced globally [12]. Historically, kaolin has been widely utilized as a filler material in various industries, including plastics and rubber manufacturing [13] . Additionally, kaolin plays a key role in the production of ceramic goods. High-purity kaolin is essential for producing premium ceramic items such as porcelain, bone china, vitreous sanitary ware, and earthenware. In contrast, lower-quality kaolin is typically employed as a filler in the manufacturing of bricks, pipes, and tiles, highlighting its versatile applications across a range of industries.

Binder will experience two aging phases which is, short-term and long-term aging. The short-term aging refers to construction phase where it will be exposed to 135 to 163 ^{o}C of temperature. While long-term aging demonstrates serviceability towards pavement throughout the year. In both stages, the binder will experience rheological and physiochemical properties such as ductility and adhesion due to age and hardening process [14]. When the binder demonstrates aging, it will affect the asphalt mixture's performance.

According to Ding et al [15], there are four main mechanism of asphalt aging; oxidation, exudation, evaporation and physical hardening. Oxidation is a process of reaction between the binder and available oxygen. It is an irreversible process. Oxidation reaction rate and a number of different reaction will be triggered and enhance more when there are higher energy i.e high temperature and high energy of light such as UV light [16]. Short term and long term aging mechanism will be different due to differences in activation energy level at the different reaction. According to Southern [17], the exposable of binder to high temperature triggers the evaporation process of the volatile components of binder which will change the existing binder properties. Exudation is a process where asphalt discharge oily component into aggregate. The chemistry of asphalt and the porosity of the aggregate itself will determine the exudation rate. Physical hardening is influenced by temperature which will cause molecular structuring up to certain degrees [18].

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In paper manufacturing, kaolin is used as coater and filler. Those undesirable size as it exceed specific size needed for kaolin particle will not be used. Together with waste from the process of kaolin mining, a material with pozzolanic properties can be produced after a heating process [19].

According to Fries et. al [20], the discovery of heating the paper sludges up to certain temperature will be great to be used in the concrete industry. Kaolin mining processes residue shows a lesser degree of temperature to achieve pozzolanic properties, but also show better reactivity compared to the conventional pozzolanic materials [21]. Pozzolan has numerous benefits to offer towards concrete as it contributes higher strength, lower temperature rises and enhanced durability performances.

Kaolin, commonly recognized as a white, soft clay, is highly valued for its plasticity and fine-grained, plate-like particle composition. It forms through the alteration of anhydrous aluminum silicates in feldspar-rich rocks, such as granite, via weathering or hydrothermal processes. The microstructure of kaolin is diverse, consisting of grains with various morphologies, including regular and irregular hexagonal platelets, rolled sheets, co-axial sheets, and occasionally tubes [22]. High levels of amorphous silica and specific surface area enhance kaolin's ability to participate in pozzolanic reactions, making it a natural pozzolanic material with properties well-suited for such applications.

In the construction industry, kaolin has been explored as a partial cement replacement, particularly after undergoing calcination. This approach improves the durability of mortars and cement while reducing the amount of cement required. Cement production, known for its significant energy demands, is a major contributor to CO_2 emissions [23]. By incorporating calcined kaolin into construction materials, not only is the reliance on cement minimized, but the environmental impact, including its contribution to global warming, can also be mitigated. This sustainable approach highlights kaolin's potential to enhance construction practices while addressing critical environmental concerns.

Methodology

Material Preparation

Kaolin, aggregate and binder

In this study, 5M kaolin was utilized as a partial replacement for asphalt in the mixture. The term "5M" denotes the concentration of sulfuric acid used to treat natural kaolin through a reflux process. The kaolin used had a particle size that passed through a 75 µm sieve to ensure uniformity. The aggregates incorporated in this research were of the AC14 grading, indicating a nominal maximum aggregate size of 14 mm in the asphalt mixture. The binder selected for the mixture was asphalt with a penetration grade of 60/70 PEN, known for its consistency and suitability in road construction applications.

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Mix proportion and aggregate gradation

The proportion of aggregates is 95 percent while asphalt is 5 percent. 4 different percentage of Kaolin was added to the asphalt, which is 0%, 3%, 6% and 9% from the weight of asphalt. The particle size distribution was selected according to AC14 gradation limit (Table 1).

Table 1. Gradation limit

Mix	**AC 14**		
BS Sieve	**Minimum (%)**	**Maximum (%)**	**Mass Passing (%)**
20	100	100	100
14	90	100	95
10	76	86	81
5	50	62	56
3.35	40	54	47
1.18	18	34	26
0.425	12	24	18
0.15	6	14	10
0.075	4	8	6

Marshall mix design

The preparation of Marshall specimens was conducted in accordance with ASTM D6927 [24] standards. After raw material preparations, all the materials were mixed according to its proportion at temperature less than 177 °C and compacted by using Marshall compactor at 135 to 155 °C. In this process, Marshall hammer was used to compact the mix inside the standard mould with 75 blows on both sides. Then, the mould was detached from the specimen.

Short-term aging (STA)

The short-term aging process followed the AASHTO R30-02 standard [25]. Loose asphalt mixtures were spread uniformly in a pan to achieve a thickness between 25 and 50 mm. The pan was placed in a forced-draft oven set to a temperature of 135 ± 3 °C for 4 hours ± 5 minutes. To ensure consistent aging throughout the mixture, stirring was performed at 60 ± 5 minute intervals. After the aging process, the mixtures were compacted using a Marshall compactor to produce specimens that adhered to testing requirements.

Long-term aging (LTA)

The long-term aging procedure was performed in accordance with AASHTO R30-02 standards [25]. Specimens that had previously undergone short-term aging were placed in a pre-heated forced-draft oven maintained at a temperature of 85 °C. The specimens were kept in the oven for 5 consecutive days to simulate long-term aging effects. After the 5-day period, the oven was turned off and allowed to cool naturally to room temperature. The specimens were then removed from the oven and tested within 24 hours to ensure compliance with the standard's requirements. This process aimed to replicate the long-term oxidative aging of asphalt mixtures in field conditions.

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Experimental

This study investigates the effect of kaolin as a filler on the properties of asphalt concrete under various aging conditions (Figure 1). The research methodology begins with the preparation of raw materials, including the gradation of aggregates through sieve analysis to achieve the desired particle size distribution. Asphalt of grade 60/70 PEN is then mixed with varying percentages of kaolin (0%, 3%, 6%, and 9%) using a asphalt mixer to ensure uniform distribution. Specimens are prepared and categorized based on three aging conditions: unaged (UA), short-term aging (ST), and long-term aging (LT), with 24 samples for each condition. The prepared samples undergo a series of tests, including the Marshall stability test to evaluate structural integrity, the resilient modulus Test to determine elasticity and load-bearing capacity, and the dynamic creep test to assess rutting resistance. The results are analyzed to understand the influence of kaolin content and aging on asphalt concrete performance, providing insights into potential applications and long-term durability improvements. Figure 2 displays the equipment used in this study.

Stability Test

Marshall Stability test determined the maximum load that can be sustained by mixture at the loading rate of 50.88mm/m. The loading increased until it reached maximum loading. Greater than that, the load continued to decrease and ended while the maximum loading was recorded. Specimens went immersion process in the water bath at 60 ± 1 °C. It immersed for 45 minutes. Then, the prepared samples were tested by Marshall stability machine, The stability value of every sample was identified first based on ASTM D6927 [24]. Samples weight were recorded after removing from the water bath and after applying load. Losses of the weight of samples were also identified from this procedure.

Resilient Modulus Test

The resilient modulus test was conducted to evaluate the stiffness of the asphalt mixtures under varying conditions, including density, moisture, and stress levels. The test employed an Asphalt Universal Testing Machine (UTM) to measure the indirect tensile strength of the specimens under repeated loading. The specimens were conditioned for 4 hours at specified temperatures of 25 °C and 40 °C before testing. This procedure adhered to the ASTM D7369 standard [26], ensuring accurate and consistent evaluation of the material's stiffness properties under controlled laboratory conditions.

Dynamic Creep Test

The dynamic creep test was conducted to assess the permanent deformation characteristics of the specimens, specifically their resistance to rutting. Samples were conditioned for 4 hours in an Asphalt Universal Testing Machine (UTM) at a temperature of 40 °C. It is also important to note that all tests, including those conducted using the UTM, were performed after the machine had been properly calibrated to ensure the accuracy and reliability of the results. Following

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conditioning, a 300 kPa applied stress was used to simulate the loading conditions. The test was performed in accordance with the BS EN 12697-26 standard [27], ensuring the proper methodology for evaluating the rutting potential and deformation behavior of the asphalt mixtures under dynamic conditions.

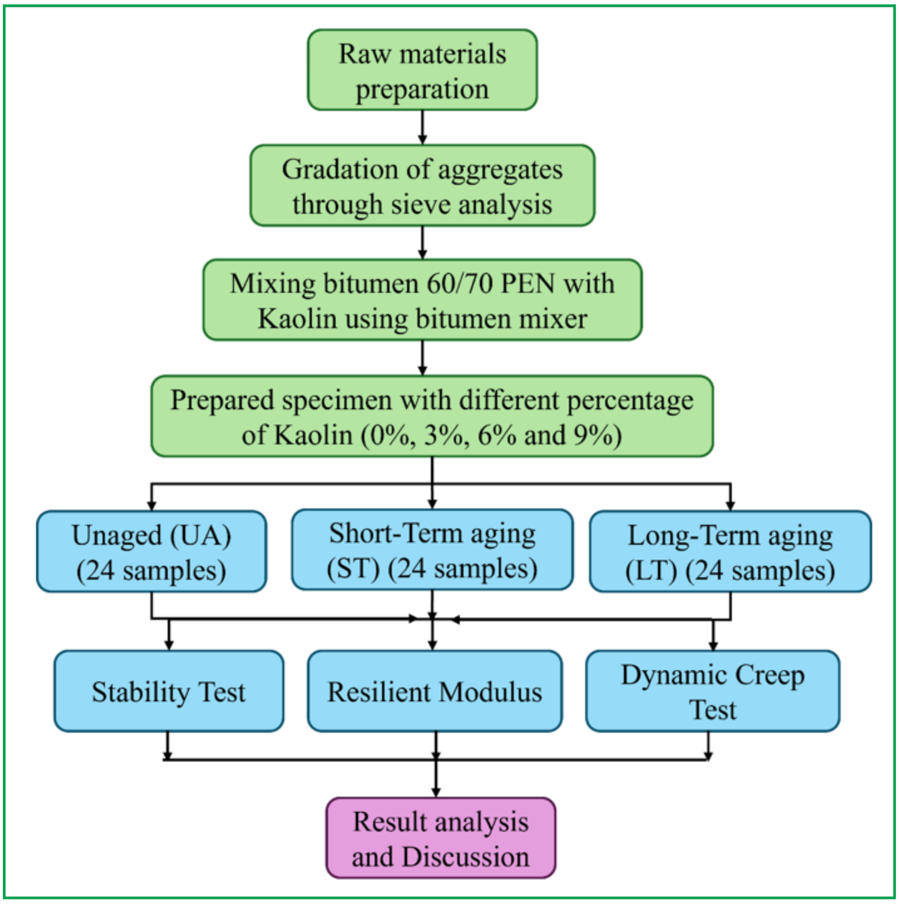

Figure 1. Flowchart of study

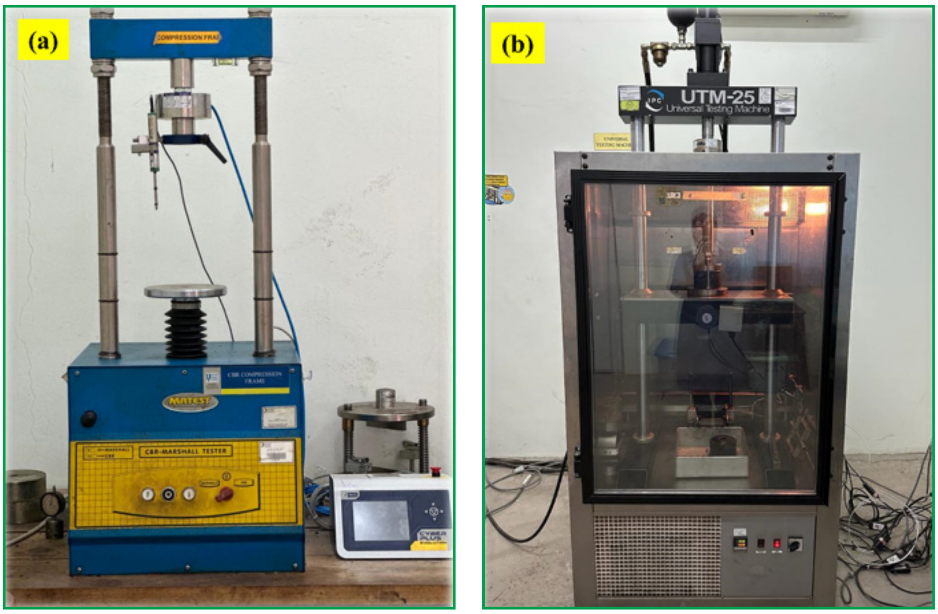

Figure 2. (a) Marshall stability machine and (b) UTM used in this study

Results and Discussion

Stability

The Marshall stability results exhibit a clear trend based on the varying percentages of kaolin and aging conditions (Figure 3). For the unaged (UA) samples, stability consistently increases with higher kaolin content, peaking at 9% kaolin with a value of 18,540 N, which represents a substantial improvement of 25% compared to the control sample (0% kaolin). In short-term aged (ST) and long-term aged (LT) conditions, a similar upward trend is observed, with stability reaching its highest values at 9% kaolin, reflecting an increase of 30% and 35%, respectively, compared to the control samples under the same conditions. Notably, despite some reduction in stability due to aging, the values remain above the minimum standard requirement of 8000 N, indicating that the kaolin-modified mixtures maintain satisfactory performance across all conditions.

The enhanced stability with increasing kaolin content can be attributed to its filler properties, which improve the interlocking mechanism and binder stiffness within the asphalt matrix. The superior performance of kaolin-modified mixtures under unaged and aged conditions highlights its ability to mitigate the adverse effects of oxidative aging. The decreasing stability from UA to ST and LT conditions, although expected due to binder hardening and loss of flexibility, is less pronounced in mixtures with higher kaolin content. This suggests that kaolin contributes to improved resistance against aging-induced degradation. However, beyond 6% kaolin, the rate of stability improvement diminishes, potentially indicating an optimal kaolin threshold where further additions may lead to particle clustering and reduced effectiveness. These findings underscore the importance of optimizing kaolin content to balance mechanical performance and durability, aligning with sustainable pavement practices [28].

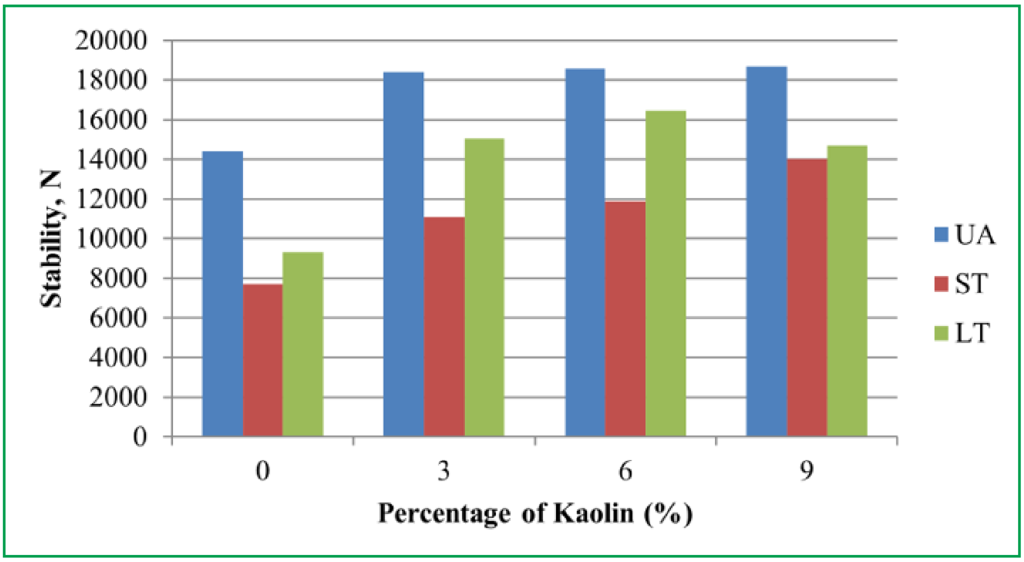

Figure 3. Marshall stability vs % kaolin on aging condition

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Resilient Modulus

Conditioned at 25 °C

Figure 4 demonstrates the relationship between the resilient modulus at 25 °C and different kaolin replacement percentages under various aging conditions. The results show a consistent upward trend in resilient modulus with increasing kaolin content, indicating improved stiffness of the asphalt mixture. For unaged samples, the resilient modulus increased by 23.3%, 24.7%, and 39.4% at 3%, 6%, and 9% kaolin replacement, respectively, compared to the control sample. The highest modulus for unaged samples was recorded at 9% kaolin, signifying its superior stiffness. Similarly, long-term aged (LT) samples exhibited higher resilient modulus values than short-term aged (ST) samples, with the highest values again achieved at 9% kaolin replacement. The consistent improvement in resilient modulus across all aging conditions confirms the positive impact of kaolin on the stiffness and structural integrity of asphalt mixtures, meeting and exceeding typical standard requirements for pavement applications.

The increasing resilient modulus with higher kaolin replacement reflects the material's effectiveness in enhancing the stiffness of asphalt mixtures. This behavior is attributed to kaolin's fine particle size and its ability to improve the asphalt-filler matrix, which reduces deformation under repeated loading. The unaged samples displayed significant gains in modulus, highlighting kaolin's immediate contribution to mechanical strength. However, the results also reveal that aging plays a crucial role in further improving modulus, as LT samples exhibited higher values than ST samples. This indicates that kaolin-modified binders are less susceptible to aging-induced deterioration, which often leads to reduced flexibility and increased brittleness. While higher stiffness is advantageous for rutting resistance, excessive stiffness at higher kaolin contents (9%) could compromise flexibility, potentially leading to cracking under thermal or load-induced stresses. Therefore, the balance between stiffness and flexibility must be carefully considered to optimize kaolin content for durable and resilient pavements.

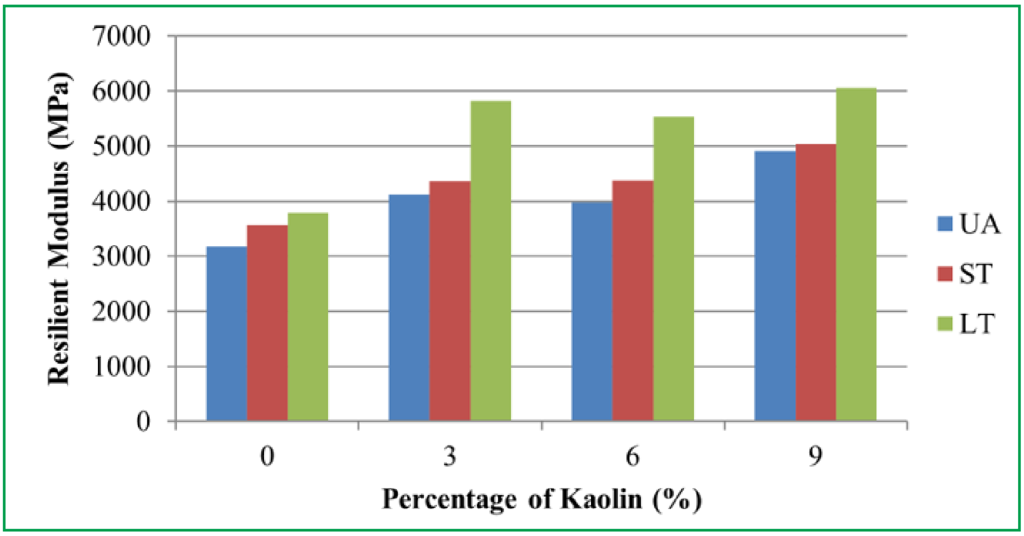

Figure 4. Resilient modulus vs % Kaolin on aging condition at 25 °C

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Conditioned at 40 °C

Figure 5 illustrates the relationship between resilient modulus at 40 °C and varying kaolin replacement percentages under different aging conditions. Resilient modulus values at 40 °C were generally lower than those at 25 °C due to the increased temperature, which reduces the stiffness of asphalt mixtures. For unaged samples, the modulus increased significantly by 94%, 130.12%, and 112.25% for 3%, 6%, and 9% kaolin replacements, respectively, compared to the control sample. The highest resilient modulus for unaged samples was observed at 6% kaolin, followed by a decline at 9% kaolin. In contrast, for short-term and long-term aged samples, the modulus consistently increased with kaolin content, with the highest values recorded at 9% replacement. Additionally, long-term aged samples outperformed both unaged and short-term aged samples, reflecting the superior rutting resistance of the aged mixtures.

The improved resilient modulus at 40 °C with increasing kaolin content indicates the material's capacity to enhance rutting resistance, particularly in high-temperature conditions. The peak performance at 6% kaolin for unaged samples suggests an optimal content where kaolin's stiffening effect is maximized without compromising the binder's flexibility. However, the decline at 9% kaolin for unaged samples could be due to particle aggregation, which diminishes the uniformity and effectiveness of the binder-filler interaction. In aged samples, the continuous improvement up to 9% replacement highlights kaolin's role in mitigating the effects of aging, possibly by forming a more stable binder matrix that resists deformation under sustained loading. Nonetheless, while higher kaolin content enhances stiffness and rutting resistance, excessive stiffness, particularly at elevated temperatures, may increase the risk of fatigue cracking. Therefore, balancing stiffness and flexibility remains critical for optimizing asphalt mixture performance under varying environmental and traffic conditions [5].

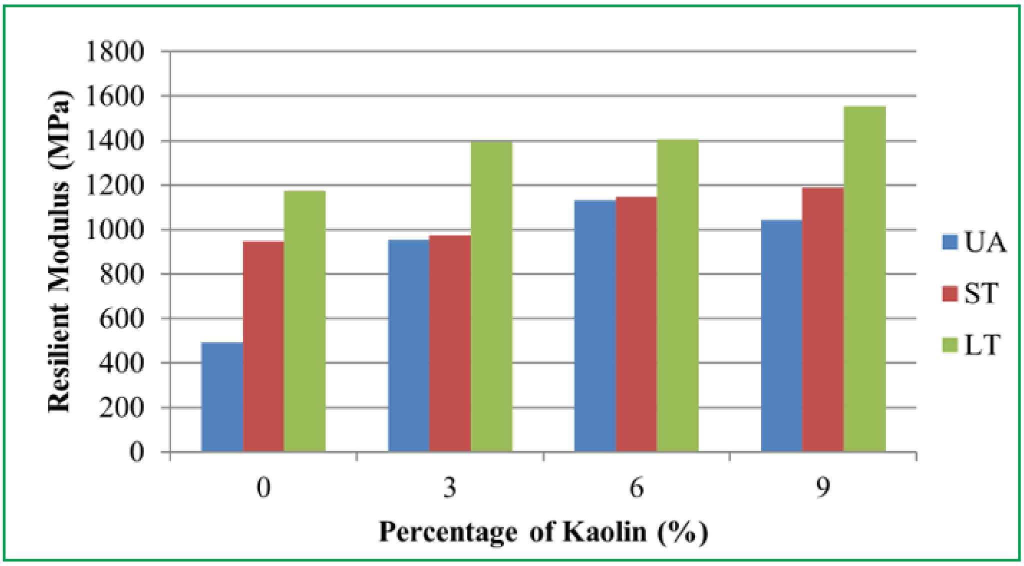

Figure 5. Resilient modulus vs % kaolin on aging condition at 40 °C

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Dynamic Creep

Figure 6 presents the dynamic creep modulus for various kaolin replacement percentages and aging conditions. The results show that the creep modulus of unaged samples increased with higher kaolin content, with 9% kaolin replacement achieving the highest modulus, 89% higher than the control sample. Interestingly, 3% kaolin replacement resulted in a 24% lower creep modulus compared to the control, whereas 6% kaolin replacement showed a significant improvement of 41% above the control sample. For aged samples, long-term aged specimens exhibited consistently higher creep modulus values compared to short-term aged ones for 0%, 3%, and 6% kaolin replacements. The peak creep modulus for short-term aging occurred at 9% kaolin replacement, while for long-term aging, the highest value was achieved at 6% replacement. These results align with the standard expectations for materials with improved rutting resistance, as a higher creep modulus indicates reduced susceptibility to permanent deformation under repeated loading.

The dynamic creep test results reveal the effectiveness of kaolin in improving rutting resistance by increasing the creep modulus, particularly at higher replacement levels. The lower modulus observed at 3% kaolin replacement for unaged samples suggests that this level of modification may not provide sufficient stiffness to counteract deformation. However, the substantial improvements at 6% and 9% replacements demonstrate the benefits of kaolin in enhancing the structural integrity of asphalt mixtures. The optimal performance at 6% kaolin for long-term aged samples indicates that aging amplifies the positive effects of kaolin, likely due to its role in stabilizing the binder matrix. On the other hand, the reduced performance of 9% kaolin in long-term aging may indicate diminishing returns or particle aggregation, which could compromise the material's effectiveness. The balance between stiffness and flexibility is critical, as excessive stiffness at higher kaolin contents might lead to cracking under thermal or mechanical stress, despite the improved rutting resistance [5]. These findings emphasize the importance of optimizing kaolin content for durable and resilient pavements.

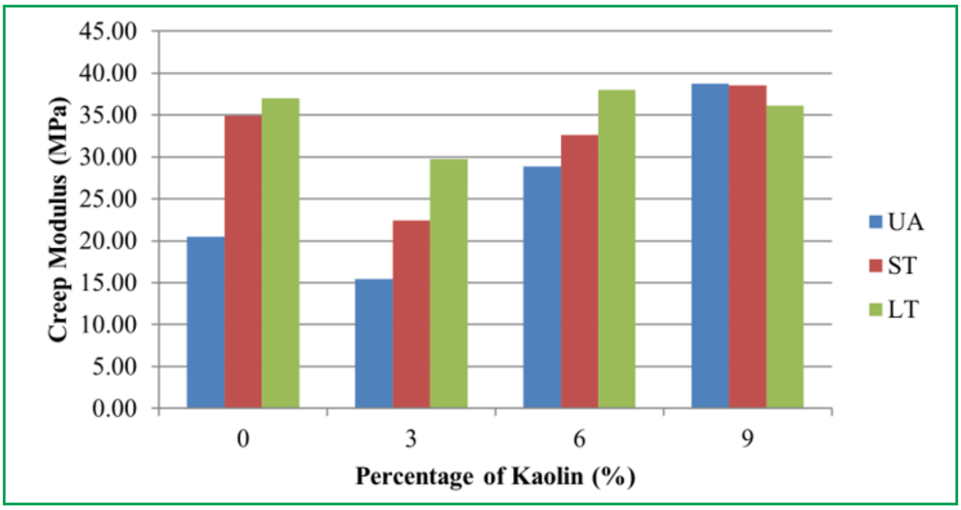

Figure 6. Dynamic creep modulus vs % Kaolin on aging condition

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CONCLUSIONS

The results of the Marshall stability, resilient modulus, and dynamic creep tests collectively highlight the significant impact of kaolin replacement on the mechanical properties of asphalt mixtures. A strong correlation exists among the three tests, indicating that higher kaolin content generally enhances the stability and stiffness of the mixtures, contributing to improved rutting resistance and durability. Marshall stability results show consistent increases with kaolin replacement, particularly under unaged conditions, while resilient modulus measured at both 25 °C and 40 °C and dynamic creep tests reinforce this trend by demonstrating superior stiffness and reduced deformation under both aging conditions. Notably, 6% and 9% kaolin replacements emerge as critical points, with 6% exhibiting optimal performance in most cases, particularly for long-term aging. The dynamic creep test complements the findings of the resilient modulus by confirming that mixtures with higher creep modulus resist permanent deformation more effectively, correlating with the increased stability and stiffness observed in the other tests. Together, these results underline the compatibility and reinforcing effects of kaolin in asphalt mixtures across a range of performance indicators.

In conclusion, the incorporation of kaolin as a partial replacement in asphalt mixtures enhances mechanical performance, particularly in terms of stability, stiffness, and rutting resistance. While kaolin content above 6% provides higher short-term benefits, such as improved stability and creep modulus, the long-term performance suggests that 6% is the optimal replacement level, balancing stiffness, flexibility, and durability. Higher percentages, such as 9%, may introduce diminishing returns due to potential particle aggregation, which could compromise the uniformity of the binder-filler matrix. While the incorporation of kaolin in asphalt mixtures has shown significant improvements in mechanical properties and durability, certain limitations must be acknowledged. The addition of kaolin, particularly at higher percentages, can lead to increased brittleness and reduced flexibility, which may impact the mixture's ability to accommodate thermal and traffic-induced stresses over time. These findings underscore the importance of optimizing kaolin content to achieve a sustainable and high-performing asphalt mixture. Furthermore, the results emphasize the critical role of aging conditions in evaluating material performance, as long-term aging highlights the resilience and stability imparted by kaolin-modified mixtures. Overall, this study demonstrates the feasibility of kaolin as a cost-effective and sustainable filler material, contributing to the development of durable and rut-resistant pavements.

ACKNOWLEDGEMENT

The authors highly appreciate the Malaysian Ministry of Higher Education and Universiti Malaysia Pahang Al-Sultan Abdullah (UMPSA) for supporting this research through the research industrial grant (UIC240831).

Conflicts of Interest

The authors declare no conflicts of interest.

Author Contributions

Zuraidah Hashim: conceptualization, methodology, writing-original draft preparation. **Wan Noor Hin Mior Sani:** data curation, supervision. **Safety Husna Pangestika:** visualization. **Nik Nur Dina Nik Azmi:** investigation. **Mohd Hazree Hashim:** writing-reviewing and editing.

Data Availability Statement

The data used to support the findings of this study are included within the article.

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