Effects of Alkylamines-Based and Polyalkylene Glycol-Based Bonding Enhancers on the Performance of Asphalt Binders

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Abstract. The premature deterioration of asphalt pavements usually occurs due to different moisture damage mechanisms resulting in stripping, ravelling, potholes, and disintegration without proper treatment. Numerous efforts have been taken into consideration to improve the bonding between materials, hence prolonging the pavement life. This study evaluates the performance of asphalt binders incorporating Alkylamines-based (ALM) and Polyalkylene Glycol-based (PLG) bonding enhancers. Each bonding enhancer at 0.5% and 1.0% based on the weight of asphalt binder was separately blended with the conventional asphalt binder 60/70 penetration grade using a high shear mixer at 1000 rpm for 30 minutes at 160℃. The physical and rheological properties of modified binders were evaluated through penetration value, softening point, ductility, elastic recovery, rotational viscosity (RV), and dynamic shear rheometer (DSR) tests. Overall, additions of ALM and PLG show identical penetration grade compared to the control sample. Both ALM and PLG showcase a higher ductility and elastic recovery than the neat binder. The DSR test indicates the incorporation of bonding enhancers improves the modified binders' rutting performance. While the application of ALM at 0.5% dosage increased the binder failure temperature out of all the tested samples, where the failure temperature is at 70°C, compared to others at 64°C. Studies at mastics and mixture levels should be conducted to appropriately understand the effect of bonding enhancer on the bituminous materials.

1. Introduction
Traffic volumes have increased in recent decades. The highway system has been subjected to extreme load and environmental demands due to the sudden increase in axial vehicle loads. This demonstrates the need to upgrade the characteristic of current hot mix asphalt (HMA) designs and enhance their efficacy [1, 2]. Hot mix asphalt (HMA) is a mixed substance mainly made up of a binder, aggregates, and mineral filler. Each material's physical and chemical properties have been studied and testified to significantly affect the mixture performance, leading to numerous premature failures [2].
Despite decades of research, there are still constant debates on the influence of asphalt binder rheological properties on pavement distresses. Asphalt binder properties may change during service life due to high axle load, quality of materials, environmental effects, construction methods, and design errors, which decreases the expected performance and service life of flexible pavements [4-8]. In general, road pavement performance is mainly affected by the asphalt binder properties. The rheological properties of asphalt binder are sensitive towards temperature and traffic loading since it is a viscoelastic material [1, 9, 10]. To some extent, the strength of pavement is inadequate to resist the deterioration mechanisms. Whereby, under extreme weather and traffic conditions, a conventional asphalt binder (e.g. 60/70 penetration grade) is inadequate to be used without modification [10, 11]. The viscoelastic characteristics of asphalt pavements that are subjected to varied pavement stresses are dominated by asphalt binders. Rutting and fatigue cracks are significant pavement stressors that afflict asphalt pavements in tropical climates exposed to medium and high temperatures. This is due to the fact that the road's plastic deformation will shorten the pavement's lifespan and create discomfort to road users. In order to increase the service life of an asphalt pavement subjected to various ageing conditions, it is important to select an appropriate asphalt binder with the requisite characteristics to minimize the severity of pavement deterioration [11].

Besides, overloading from heavy vehicles affects pavement strength due to fatigue. A significant interference due to rutting and fatigue cracking caused a permanent failure in the road pavement. The premature deterioration caused by a loss of bond between asphalt binder and aggregate could occur in the presence of different water damage mechanisms [8, 12-14]. Moreover, the presence of water also alters the properties of asphalt binder regarding cohesion behavior. Moisture can cause pavement distress, leading to stripping, ravelling, potholes, and severe disintegration without proper treatment [15, 16]. This distress has contributed to substantial rehabilitation and repair expenses [2, 8, 9, 17, 18]. Based on the previous studies, contributing mechanisms to moisture damage were identified, known as detachment, displacement, spontaneous emulsification, pore pressure, and hydraulic scouring [5, 16, 18, 19]. Moisture damage typically generated by a combination of mechanisms. It is necessary to seek a more fundamental understanding of the moisture damage process by considering micro-mechanisms that influence the adhesive interface between aggregate and asphalt, as well as the cohesion strength and durability of the mastic [9, 16, 18]. Meanwhile, the stripping phenomenon refers to the breaking of the bond between the aggregate and the binder. Anti-stripping agents (ASAs) are the most common way to prevent stripping in a pavement. Hydrated lime is one of the most used ASAs in the United States. Others include liquid ASAs such as amines, diamines, and liquid polymers, as well as solids such as Portland cement, fly ash, flue dust, etc. [20].

Under severe environmental conditions and the traffic volume, the application of conventional asphalt binder is inappropriate to withstand deteriorating factors, internally and externally. It is closely related to the effect of binder properties on road pavement performance [9]. To improve pavement performance, investigators are currently looking at various modifiers generally classified as elastomers, plastomers, nanomaterials, and waste materials are blended with asphalt binders [21]. Different amounts and combinations of additives have been used to improve asphalt binders’ resistance to permanent deformation, cracking’s, moisture damage, etc. [6, 21]. The utilization of various substitute materials enhances the binder properties, such as elasticity, recovery, adhesive/cohesive strength, hence improve pavement durability. Polymers are regarded as the most effective modifying agents for bitumen modifiers. Various polymers, including polyethylene, polypropylene, styrene-butadiene styrenen (SBS), styrene-butadiene rubber (SBR), polyphosphoric acid (PPA), ethylene bis-stearamide wax (EBS), and crumb rubber, have been adopted [7, 22, 23].

On the other hand, applications of anti-stripping agents, surfactants, bonding initiators could also be used to improve the long-term performance and durability of asphalt pavement [23, 24]. Moisture infiltration through joints and fractures can make a pavement more vulnerable to disintegration over time. Anti-stripping additives have been utilized to improve the physicochemical bonds between bitumen and aggregates by decreasing the bitumen surface tension, resulting in improved bonding and water resistance. The thermodynamic phenomena of physicochemical adhesion between aggregate and
asphalt binder are regulated by the free energy of the material's surface. The addition of hydrated lime to the asphalt mixture can improve the asphalt binder's adherence to the aggregate [27].

This study aims to evaluate the physical and rheological properties of modified asphalt binder using bonding enhancers and compared it with the conventional binder. Two bonding enhancers identified as Alkylamines-based (ALM) and Polyalkylene Glycol–based (PLG) were incorporated to improve the molecular bond between materials in asphalt mixtures. All binder specimens' physical and rheological properties were evaluated via penetration value, softening point, ductility, elastic recovery, rotational viscosity (RV), and dynamic shear rheometer (DSR) tests. Subsequently, impact on the performance grade of each binder based on its failure temperature at elevated temperatures was also observed.

2. Materials and methods

2.1. Materials

A Conventional binder 60/70 penetration grade was used as a control sample. Two additives, namely Alkylamines-based (ALM) and Polyalkylene Glycol–based (PLG) bonding enhancers at 0.5% and 1.0% based on the weight of asphalt binder, were adopted as recommended by the manufacturer. Rheological properties and chemical compositions of the bonding enhancers are shown in Table 1. Table 2 shows the properties of the control binder.

| Table 1. Physical and chemical properties of bonding enhancers |
|---------------------------------------------------------------|
| Properties | ALM [28] | PLG [29] |
| Physical Form | Dark brown liquid | Dark brown liquid |
| Specific Gravity | 1.09 | 1.04 |
| Recommended Dosage (based on the weight of bitumen) | 0.25 – 1.0% | 0.25 – 1.0% |
| Composition | Alkylamines, Alkanol amines, Alkylene amines | Polyalkylene glycol mixture, Glycol ethers |

| Table 2. Physical properties of 60/70 Penetration grade asphalt binder |
|---------------------------------------------------------------|
| Test | Standard Test | Spec. Requirement | Unit | Test Results |
|-----------------------------------|-----------------|-------------------|------|--------------|
| Penetration Value | ASTM D5 | 60-70 | dmm | 66 |
| Softening Point | ASTM D36 | 42-52 | °C | 45.5 |
| Penetration Index (PI) | - | -2 to 2 | - | -1.774 |
| Ductility | ASTM D113 | >100 | mm | 138 |
| Elastic Recovery | ASTM 6084 | >10% | % | 10 |

Prior to the test, both ALM and PLG modified asphalt binders with 0.5%, and 1.0% dosages were prepared using a high shear mixer at 1000 rpm for 30 minutes at 160°C [15].

2.2. Experimental Methods

The softening point test was conducted following the ASTM D36 [30] using the ring and ball apparatus. During the test, samples were heated, and the temperature at which the ball touched the base plate was recorded. The consistency of asphalt binder samples was measured using the penetration test. The test was conducted by determining the vertical penetration depth in tenths of a millimeter at 25°C as specified by ASTM D5 [31]. The penetration value obtained was also used to classify the bitumen in a range of penetration grades. The test was performed on both; control and modified asphalt binders to determine the temperature sensitivity of each specimen in terms of penetration index (PI). Asphalt binders with low PI have high-temperature sensitivity indicating rapid consistency changes with temperature variations. The PI measures the temperature susceptibility of an asphalt binder which is
derived mathematically from the standard penetration and softening point values as given in Equation 1 [30, 31].

\[
PI = \frac{1952 - 500 \times \log (Pen_{25}) - 20 \times SP}{50 \times \log (Pen_{25}) - SP - 120}
\]

where:
- Pen_{25} = Penetration at 25°C
- SP = Softening point.

The ductility test measures the tensile properties of bituminous materials, particularly their ability to undergo plastic deformation before failure or to be elongated under traffic load without fracture. The elongation of an asphalt binder measures it before breaking based on the ASTM D113 [29]. Two ends of a briquet asphalt binder specimen are pulled apart at a specified uniform speed (5 cm/min ± 5.0 %) and temperature (25 ± 0.5°C) while continuously immersed in water. This method also can be used to measure the elastic recovery of an asphalt binder based on the recoverable strain after splitting the elongated briquet specimen at temperature 25 ± 0.5°C and speed 5 cm/min ± 5.0 %. Subsequent estimation of elastic recovery percentage then calculated using Equation 2 [30].

\[
\% \text{ Elastic recovery} = \left( \frac{E - X}{X} \right) \times 100
\]

where:
- E = Original length of the specimen (cm)
- X = Elongation of the specimen with severed ends just touching (cm).

Unmodified asphalts rarely show elastic recoveries of more than 10%. The elastic recovery of polymer-modified materials depends on the type and amount of polymer used.

Then, the rotational viscosity (RV) test was carried out using a Brookfield viscometer to determine the flow resistance of asphalt binder for both modified and unmodified samples at elevated temperatures between 120°C and 180°C at 10°C increments. The mixing and compaction temperatures of each binder were determined at selected viscosity limits. The experiment was performed in accordance with ASTM D4402 [34]. Viscosity readings in centipoises were obtained using spindle No. 27. The rotational viscometer determines the torque required to rotate the spindle at a constant speed shear rate of 20 rpm while immersed in the asphalt binder sample.

The rheological properties of asphalt binders measured using a HAAKE RheoStress 6000 dynamic shear rheometer (DSR) based on ASTM D7175 [35]. Changes in complex modulus (G*), phase angle (δ), rutting resistance factor (G*/sinδ), and performance grade temperature were observed using the temperature sweep mode. The test was conducted under various elevated temperatures between 46°C to 82°C at 6°C increments for all samples. During the test, all samples were tests at a frequency of 10 rad/s (1.59 Hz). The modified binder’s susceptibility towards rutting and fatigue cracking was assessed.

3. Results and Discussion

3.1. Physical properties of asphalt binders

Table 2 shows the softening point and penetration values of the control binder, as well as ALM and PLG modified binders with different dosages. The penetration test determines the consistency of control and modified asphalt binders, while the softening point test confirms the maximum service temperature of asphalt binders. Figure 1 shows the penetration of modified asphalt binders decreases upon the addition of ALM and PLG compared to the conventional control base. However, both bonding enhancers do not change the grade of the modified binders, which remain within 60 to 70 penetration value.
The softening point can be regarded as an indirect test control of high-temperature rigidity. The test results show that both bonding enhancers have resulted in similar softening points, except the modified binder prepared using 0.5% ALM. Figure 1 shows the highest softening point is attained by 0.5% ALM modified asphalt binder at 47.5°C, while the 1.0% ALM recorded the lowest softening point at 45°C. Thus, a binder with softening point above this range would perform well at high temperatures. A binder with a high softening point is preferred to be used for pavement constructions at high-temperature climate areas.

**Figure 1.** Results of the softening point and penetration tests.

The penetration index (PI) is determined based on the conducted test. Figure 2 indicates that the PI ranges between -2.0 to +2.0, except the sample of 1.0% ALM. In general, those values are in line with the standard requirement for the application on bitumen in road construction [33]. The finding illustrates that the resulting penetration index increases from -1.774 for conventional binders to -1.437 for 0.5% ALM and -1.703 for 0.5% PLG modified binders, while it decreases to -1.959 for 1.0% PLG. The PI for 1.0% ALM (-2.093) is out of the acceptable range. This suggests that incorporating ALM and PLG bonding enhancers is appropriate for higher temperature sensitivity in hot weather countries. The 1.0% ALM–based bonding enhancer is not suitable for low-temperature conditions due to its rapid brittleness, which susceptible to transverse cracking. Asphalt cement with PI below -2.0 exhibits brittleness at low temperatures and susceptible to transverse cracking in a cold climate. Permanent deformations through rutting at elevated temperatures have been established for PI beyond 1.0 [33]. Therefore, the consistency of modified asphalt binder’s changes with the function of temperature.

**Figure 2.** Penetration index (PI).
3.2. **Ductility and elastic recovery**

The ductility requirement in pavements and road constructions should not be less than 100 cm [36], and elastic recovery should not be less than 10% [37]. Figure 3 shows the ductility and elastic recovery of both ALM and PLG modified binders are superior than the control binder with an average ductility of more than 150 cm (sample remains intact when the test stopped even after being stretched to 150 cm), indicating high resilience to break upon reaching the maximum length, while the elastic recovery is more than 10% compared to the control binder. In summary, the addition of bonding enhancer enhanced the ductility and elasticity of asphalt binders, which is essential to improve the resistance to fatigue cracking, hence extending the pavement service life and durability.

![Figure 3](image)

**Figure 3.** Results of the (a) ductility test and (b) elastic recovery test.

3.3. **Rotational viscosity (RV)**

At high temperatures, the viscosity of asphalt binders is vital since it indicates the binder's ability to pump through the asphalt binder plant and for the production of asphalt mixtures. Rotational viscometer (RV) test was carried out to evaluate the properties of asphalt binders at elevated temperatures (120 to 180°C) with 10°C temperature increments. This research also aims to determine the mixing and compaction temperatures of asphalt mixtures by measuring the viscosity of asphalt binders. Figure 4 shows the addition of ALM and PLG bonding enhancers altered the viscosity of binder, with slight differences in production and compaction temperatures. Considering the temperature analysis at the same viscosity limit line for mixing (0.17±0.02 Pa.s) and compaction (0.28±0.03 Pa.s) processes [30,32]. The presence of ALM and PLG bonding enhancers shown that the required mixing temperatures are within 160°C that is comparable with the mixing temperature of the conventional binder, except for 0.5% ALM modified binders. The 0.5% ALM requires higher mixing temperature, which is 170°C. The 0.5% ALM also showcases higher stiffness than other modified binder, e.g., 0.5% PLG, 1.0% PLG, and 1.0% ALM. Thus, it can be concluded that the optimum temperature for the mixture production is 160°C. Meanwhile, the compaction temperatures for all tested samples are between 150°C and 160°C. The addition of 0.5% ALM-based and 1.0% PLG-based binders do not change the physical properties in term of penetration value and softening point. One of the key factors in controlling the mechanical performance of asphalt mixes is the mixing and compaction temperatures. Therefore, temperature affects the reduction in viscosity for both conventional and modified binders. The addition of bonding enhancers helps to improve the viscoelasticity behavior of asphalt binders. This leads to superior pavement structural integrity, while reducing the viscous components and increasing their elasticity [33].
3.4. Rutting resistance and failure temperature

The complex modulus (G*) and phase angle (δ) values derived from DSR results are used as indicators of permanent deformation based on Superpave specifications. Asphalt binder is a visco-elastic material whose δ defines the ratio of its elastic to viscous response [39]. The response of the binder G* against temperatures alludes to its elastic storage and viscoelastic dissipation properties. Based on Figure 5 (a), high G* value of ALM and PLG indicates adequate rigidity to sustain rutting, while a lower value suggests asphalt susceptibility to permanent deformation. Similarly, the δ refers to the lag in strain relative to stress of materials whereby the δ for a purely elastic material is 0°, while a purely plastic material shows an angle of 90° [40].
Additionally, from Figure 5 (b), the $\delta$ of the control asphalt binder was witnessed to continuously increase with temperature elevations, which infers dominant viscous behavior. The modified binder with 1.0% PLG sustained the highest $\delta$, while the binder with 1.0% ALM had the lowest $\delta$ from 58°C to 82°C. Hence, the addition of ALM and PLG to binders evidently increased the phase angle, at elevated temperature conditions. Such occurrence attributed by the presence of interconnected polymer networks within asphalt binder which enhances its viscous component leading to increments in its plasticity.

![Figure 5](image_url)

**Figure 5.** Effect of ALM-based and PLG-based bonding enhancers on (a) $G^*$ and (b) $\delta$

Meanwhile results obtained in Figure 6 (a) indicated that the incorporation of 0.5% ALM and 0.5% PLG, as well as 1.0% ALM resulted in a noticeable increase in the binder rutting resistance $G^*/\sin\delta$ values albeit with the exception of the 1.0% PLG which produced relatively lower $G^*/\sin\delta$. This infers that the addition of ALM and PLG bonding enhancers increases binder $G^*/\sin\delta$, thus highlighting its resistance to rutting.

Mostly, the two indicators of rutting potential that typically adopted are binders with high temperature susceptibility and binders that less harden upon oxidation. However, the culmination of these two issues could be mitigated with polymer and ASA modifications used in the binder to generally stiffen it, and reduce its temperature susceptibility, thus providing a more elastic and as well a less viscous material [15]. The nucleophilic lone nitrogen present in alkyl-amines also suggests that amines in bitumen may have the tendency to react and donate electrons with the negatively charged carboxylic groups of bitumen (COO) which helps strengthens its bonding and stiffness. Whereby, the PLG’s high viscous index can potentially help in providing more stiffness within bituminous phases. Hence resulting in more rigidity and improved $G^*/\sin\delta$ of asphalt binders. Similar observations of amine-based liquid anti-stripping agent on Advera-modified binder drew a conclusion that 0.5% dosage anti-stripping agent could effectively enhance the fatigue and low temperature resistances of Advera-modified binder. However, the rutting factor of the binder decreased when the amount of anti-stripping agent was increased [15]. This suggests that although the addition of ASA in bituminous phases does increase its rutting and fatigue resistance, such improvement is also sensitive to the ASA dosage and concentration as was observed in this study.
Figure 6. (a) Rutting resistance and (b) failure temperature of ALM-based and PLG-based bonding enhancers.

According to Superpave specifications, an asphalt binder’s failure temperature is defined as the temperature wherein the G*/sinδ ratio is less than 1.0 kPa. High failure temperature implies asphalt binder’s low susceptibility to deformation under elevated pavement temperatures and vice versa. Based on Figure 6 (b), the failure temperature response of an asphalt binder to ALM and PLG additions exhibited similar failure temperatures of 64°C for both PLG dosages and 1.0% ALM. Meanwhile the failure temperature of 0.5% ALM occurred at 70°C. This finding suggests that incorporating a bonding enhancer did not necessarily change the binder rate of failure except for 0.5% ALM. This was anticipated since only the 0.5% ALM was witnessed to exhibit better penetration and softening performance. Similar findings have been reported for the rheological and moisture susceptibility of water bearing warm asphalt mixtures containing liquid antistrip additives (ASA), which suggested that although ASA incorporation in warm mix asphalts lead to a minor enhancement in the rutting resistance of asphalt binders at high temperature, it had no obvious influence on the performance grade of binders [15]. Additionally, amine-based liquid anti-stripping additives of various mix dosages were also reportedly observed to cause more remarkable performance grade changes in PG 70-28 and PG 76-28 polymer modified binders than that of PG 64-22 [15].

In summary, based on the collected experimental results, the G* of both ALM and PLG at 0.5% was sufficient to resist rutting. Subsequently, the G*/sinδ of the ALM and PLG at 0.5% dosage was also indicate adequate resistance to rutting better than the control binder and both ALM and PLG at 1.0%.

4. Conclusions
In this study, the effect of elevated temperature on the rheological properties of modified binders incorporating Alkylamines-based and Polyalkylene Glycol-based bonding enhancers was analyzed by performing both physical characterization of penetration, softening point, ductility, elastic recovery, rotational viscosity, and permanent deformation test via the dynamic shear rheometer (DSR). The following conclusions were drawn from the study:

1. The addition of ALM and PLG bonding enhancers in bitumen does not change the penetration grade of asphalt binders, while more or less comparable softening point threshold. Both test results highlight that the ALM at 0.5% exhibited the highest softening and lowest penetration in comparison to the other tested samples.

2. Additionally, the impact of both the ALM and PLG resulted in an increase in its ductility and elasticity. This suggests that both modifiers possess elastomeric tendencies to improve the bitumen’s recovery after permanent deformation due to temperature and stress loading impacts.
3. Nonetheless, both ALM and PLG additions resulted in comparable production temperature in comparison to the control sample.

4. An increase in rutting resistance of both ALM and PLG was observed whereby both ALM and PLG sustained higher G*/sinδ relative to that of the control binder, while only ALM at 0.5% increased the binder failure temperature out of all the tested samples.

In summary, the results indicated that the flexibility and elastic recovery of asphalt binder can be improved via incorporation of ALM-base and PLG-base bonding enhancers, which are induced by a better bonding characteristic. It translated into greater resistance to permanent deformation based on the binder rheological indicators. In addition to that, further studies should be conducted to investigate its long-term ageing properties and considerations on the potential influence of bonding enhancers on mastics are essential to improving the mixture performance.

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