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To cite this article: Panisa Sangnak, Saranagon Hemavibool, Polphat Ruamcharoen & Chor. Wayakron Phetphaisit (02 Dec 2024): Enhancing performance and fuel resistance of modified epoxy asphalt using amine and carboxyl terminated PET co-hardener, Road Materials and Pavement Design, DOI: [10.1080/14680629.2024.2424229](https://doi.org/10.1080/14680629.2024.2424229)

To link to this article: <https://doi.org/10.1080/14680629.2024.2424229>



Published online: 02 Dec 2024.



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Enhancing performance and fuel resistance of modified epoxy asphalt using amine and carboxyl terminated PET co-hardener

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ABSTRACT

In this study, a fuel-resistant modified epoxy asphalt was formulated using a co-hardener containing a commercial amine and carboxyl terminated PET (CTPET). Rheological, physical, mechanical, and fuel-resistance properties were evaluated across various epoxy polymer-to-asphalt ratios. The CTPET co-hardener enables blending with up to 85% epoxy polymer, preserving viscoelastic behaviour comparable to base asphalt. Notably, higher epoxy-to-asphalt ratios enhanced the complex shear modulus, phase angle, rutting resistance, and cohesion bonding. Furthermore, weight loss measurements after immersing the specimens in kerosene at 80°C for 24 h revealed losses of 1.08%, 0.24%, 0.18%, and 0.01% for blends containing 70, 75, 80, and 85 wt% epoxy, respectively, all well below the standard limit of 2%. The material also retained an elongation at break exceeding 1000%. Additionally, the penetration values, softening points, and tensile strengths remained stable before and after immersion, confirming the material's durability and suitability for fuel-resistant applications.

ARTICLE HISTORY

Received 20 May 2024
Accepted 28 October 2024

KEYWORDS

Epoxy asphalt; hardener; modified PET; fuel-resistant performance; kerosene

Introduction

Asphalt plays a crucial role in road construction infrastructure, particularly in sealing concrete joints and serving as a paving material for roads and airport runways. The chemical composition of asphalt primarily consists of asphaltenes, saturates, aromatics, and resin hydrocarbons distilled from crude oil (Aman et al., 2020; Cong et al., 2011; Yu et al., 2020). As a result, asphalt shares significant similarities with conventional hydrocarbon fuel products such as kerosene, gasoline, and diesel in terms of chemical structure and solubility parameters (Du et al., 2021; Xue & Xu, 2023). This makes asphalt easily dissolvable and dilutable by dripping fuel oil, leading to a reduction or loss of bond strength between the asphalt, the aggregate, and the substrate, surface softening, and other deficiencies. The failure of asphalt is further accelerated by temperature fluctuations, humidity, rain, heavy traffic loads, and the emission of oil vapours from vehicles (Du et al., 2021; Liu, Li et al., 2021; Polacco et al., 2011; Xue & Xu, 2023; Yao et al., 2021). Fuel leakages are often identified as a primary cause of premature pavement deterioration in various settings, including airports, intersections, gas stations, parking lots, roads, and industrial areas (El-Rahman et al., 2017; Giuliani et al., 2009; Li et al., 2019). This is especially critical in situations where kerosene spills are a major concern in pavement management. A commonly used and effective solution is the application of concrete pavements, which are designed

to be resistant to hydrocarbons and have high load-bearing capacity (Merusi et al., 2011). However, asphalt is still necessary for sealing joints between concrete road panels, and in many cases, flexible pavements remain preferable. To improve fuel resistance and extend pavement lifespan, modified asphalt has been developed. Numerous research studies have been conducted to improve the fuel corrosion resistance of asphalt pavement. For instance, the incorporation of polymers with carbonyl functional groups in their backbone has shown improvements in fuel resistance (Giuliani et al., 2009; Yao et al., 2021). Additionally, advancements have been made through the inclusion of fuel-resistant modifiers (Liu, Wang et al., 2021) and the use of materials specifically designed to counteract fuel oil corrosion (Li et al., 2019; Rizvi et al., 2019). However, these studies indicated that blending asphalt with thermoplastics, thermoplastic elastomers, and other materials results in only marginal improvements in fuel resistance. It is worth noting that the ASTM D3569-95 standard recommends a weight loss of less than 2% after immersion in fuel oil (ASTM D3569-95, 2000). The weight loss values reported in the aforementioned studies have exceeded this threshold. Furthermore, the incorporation of modified thermoplastic asphalt has not proven to enhance asphalt performance at elevated temperatures. As a result, there is growing interest in introducing thermosetting materials into asphalt to improve both fuel resistance and overall performance.

Epoxy polymer is a promising material that can be blended with asphalt to produce epoxy asphalt. When the epoxy oligomer is added to asphalt, it reacts with its hardener, forming a three-dimensional (3D) crosslinked network. This process enhances the performance of asphalt, providing increased strength, stiffness, rutting resistance, fatigue resistance, water stability, and chemical corrosion resistance (Cong et al., 2019; Liu, Wang et al., 2021; Tian et al., 2022; Yao et al., 2019).

Numerous studies indicate that incorporating epoxy polymers into asphalt or modified asphalt, such as SBS-modified asphalt, significantly improves fuel corrosion resistance at elevated temperatures. Additionally, the inclusion of epoxy polymer mixtures enhances the rheological properties of both pristine and modified asphalt binders (El-Rahman et al., 2017; Kang et al., 2015; Xue & Xu, 2023). However, the performance of epoxy asphalts strongly depends on the amount of epoxy polymer due to its high crosslink density. In particular, an excessive crosslink density in the epoxy polymer can lead to brittleness, which is a significant drawback (Su et al., 2021; Xu et al., 2021). Therefore, improving the toughness of epoxy polymers is essential to ensure their practical application. Research on toughening epoxy polymers has led to various solutions, such as core-shell rubber (CSR) (Su et al., 2021), carbon nanotubes (CNTs) (Domun et al., 2015), and hyperbranched polymer (HBP) (Xu et al., 2016). Carboxyl terminated polyethylene terephthalate (CTPET), prepared from recycled water bottles, contains carboxylic acid groups at the chain termini and has a longer chain compared to amine hardeners. These characteristics make CTPET an excellent choice as both a hardener and a plasticiser, enhancing the flexibility of rigid polymers (Phetphaisit et al., 2015). Additionally, the polar ester molecules and aromatic structure of CTPET provide excellent bonding capabilities, significantly strengthening the bond between the polymer and the substrate while also enhancing the material's chemical resistance (Phetphaisit et al., 2013; Phetphaisit et al., 2023).

This study aims to develop epoxy asphalt with improved fuel resistance and high performance by introducing CTPET as a co-hardener alongside a commercial amine hardener. The research focuses on determining the optimal ratio of epoxy polymer to asphalt. The primary objective is to characterise the performance of the prepared epoxy asphalt by examining its rheological properties, thermal characteristics, and morphology. Additionally, the study presents findings on its fuel resistance, specifically evaluating the physical and mechanical properties before and after immersion in kerosene at various temperatures.

Experimental

Materials

Asphalt A60/70 was supplied by Tipco Asphalt Co., Ltd, Thailand. EPOTEC YD 128 (Part A: the mixture of Diglycidyl ether of bisphenol A, DGEBA (95%) and Part B: amine-amide based hardener in the ratio

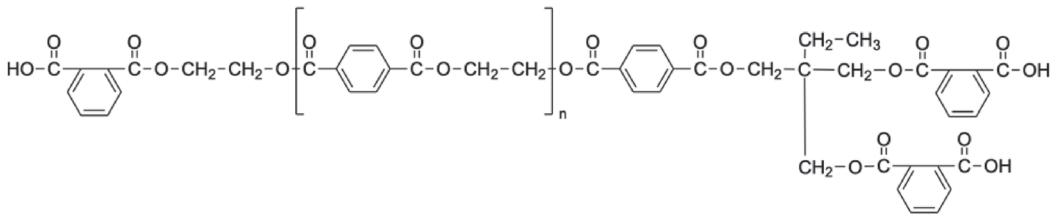


Figure 1. Chemical structure of CTPET.

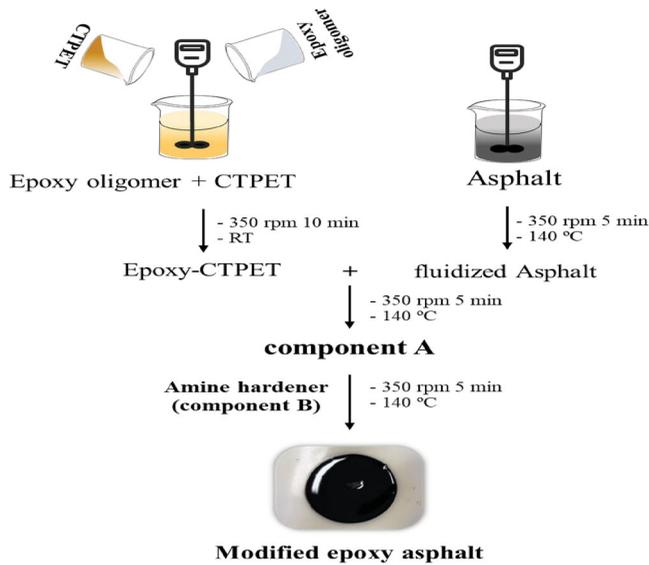


Figure 2. Schematic presentation of the preparation of modified epoxy asphalt products.

of 75:25) was supplied by Richtech Paint Co., Ltd, Thailand. Carboxyl-terminated PET (CTPET) hardener was synthesised in the polymer laboratory at Naresuan University. The synthesis process involved the glycolysis of waste PET using ethylene glycol (EG) and trimethylol propane (THMP), followed by a ring-opening reaction of the resulting intermediate with phthalic anhydride (PA). The weight-average molecular weight (\bar{M}_w) and $T_{g, \text{midpoint}}$ values for the CTPET product, measured by GPC and DSC, were $913 \text{ g}\cdot\text{mol}^{-1}$ and -20°C , respectively. The chemical structure of CTPET is shown in Figure 1 (Phetphaisit et al., 2013; Phetphaisit et al., 2015; Phetphaisit et al., 2023).

Preparation of hot-mixed modified epoxy asphalt

The preparation process of epoxy asphalt is illustrated in Figure 2. The modified epoxy asphalt was composed of components A and B. Component A was prepared by mixing the epoxy oligomer (DGEBA), CTPET, and asphalt. First, the epoxy oligomer and CTPET were mixed for 10 min by stirring at room temperature. Meanwhile, the asphalt was heated to 140°C for 5 min in a container. Then, the epoxy oligomer and CTPET were incorporated into the fluidised asphalt under continuous mixing at 140°C until no apparent clusters could be observed, which took about 10 min. Subsequently, component B, the commercial amine hardener, was added, followed by additional mixing for 30 min at 140°C . After that, the samples were cured at room temperature for 7 days before testing. The content of epoxy resin in the epoxy asphalt product was set at 75%, 80%, 85%, and 90% by weight, as indicated in Table 1. Furthermore, it should be noted that the epoxy resin in EAC consisted of the epoxy

Table 1. Composition (weight%) of the epoxy asphalt in various formulations.

Formulation	AC60/70	EAC15*	EAC30	EAC25	EAC20	EAC15	EAC10
Asphalt (AC 60/70) (wt%)	100	15	30	25	20	15	10
Epoxy resin (wt%)	0	85	70	75	80	85	90

oligomer, amine hardener, and CTPET co-hardener in mole proportions of 2, 0.25, and 0.75, respectively. For comparison, epoxy asphalt with a weight ratio of 85:15, using solely an amine hardener (with an epoxy oligomer to amine hardener ratio of 2:1) and denoted as EAC15*, was also prepared.

Characterisation methods

The glass transition temperature of the epoxy asphalts was determined using a Differential Scanning Calorimeter (DSC) (Mettler Toledo model TGA/DSC1 STAR^e system). For each sample, a temperature scan was conducted in the range from -50°C to 100°C at a heating rate of $10^{\circ}\text{C}/\text{min}$, with a continuous nitrogen flow of 20 mL/min. Afterwards, the temperature was decreased to -50°C at the same rate of $10^{\circ}\text{C}/\text{min}$. Finally, a second heating scan was performed using the same temperature range and heating rate.

Optical microscopy was utilised to assess the dispersion of asphalt within the epoxy polymer matrix. The epoxy asphalt sample was casted onto a thin glass slide and examined using an Olympus microscope equipped with a digital camera (model SZ2-ILST T5 SN 1D02459) at a 40x magnification. Subsequently, the microscope images were analyzed using ImageJ and OriginLab 2023 to determine the particle size of the dispersed phase.

The rheological properties were investigated using a rheometer (HR20, TA Instruments) in a 25 mm parallel plate geometry operating in an oscillation mode. The samples underwent testing with a 1.0 mm gap between the upper and lower plates and an angular frequency of 10 rad/s. A 0.5% strain, representing the linear viscoelastic region of asphalt, was applied within a temperature range of 50°C to 90°C . The complex shear modulus (G^*), phase angle (δ), and Rutting factors ($G^*/\sin\delta$) were evaluated for both asphalt and epoxy asphalt materials. The rheological property tests were carried out in triplicate for each condition.

The bonding interface and extensibility of epoxy asphalt and concrete at ambient temperature were investigated. Test specimens, measuring $30 \times 50 \times 15$ mm, were placed between two concrete blocks with dimensions of $50 \times 50 \times 25$ mm (Liu et al., 2016). The specimens were kept at room temperature for 7 days before testing. Tension tests were conducted at 30°C with a crosshead speed of 0.5 mm/min until the specimens either reached 100% extension or separated. Each formulation was evaluated using three specimens.

To evaluate the fuel resistance of the epoxy asphalt products, observations were made qualitatively by assessing the colour change of the kerosene through visual examination of photographs. A 5 g sample of epoxy asphalt material was poured into a glass container to a height of approximately 1 cm. Subsequently, 20 mL of kerosene was added to the container, which was then placed in an oven at temperatures of 30°C , 70°C , 80°C , 90°C , and 120°C for 24 h. The samples were photographed to document the colour change.

The weight loss of the samples was measured to assess the fuel resistance of the epoxy asphalt. For each formulation, specimens measuring $25 \times 25 \times 10$ mm were immersed in kerosene and maintained at temperatures of 30°C , 70°C , 80°C , 90°C , and 120°C or 24 h (El-Rahman et al., 2017; Giuliani et al., 2009; Yao et al., 2021). The excess oil on the specimens was first blotted using blotting paper. Then, the specimens were placed in a fume cupboard and left until they reached a constant weight. Three specimens were evaluated for each formulation. The weight loss of each specimen was calculated according to Eq. (1):

$$\text{Weight loss (\%)} = \frac{(W1 - W2)}{W1} \times 100 \quad (1)$$

Where w_1 and w_2 are the mass of the epoxy asphalt before and after kerosene immersion, respectively.

The penetration test, conducted according to the ASTM D5-95 standard procedure (Yao et al., 2019), was performed on both the base asphalt AC60/70 and the modified epoxy asphalt products. The samples were tested at 1, 3, 7, 14, and 21 days of age under standard conditions at 25°C, with the depth of penetration measured in units of 0.1 mm. The test was conducted at three random points on each sample, and the average was used for analysis. For the penetration test after immersion in kerosene, the specimens were prepared and left for 7 days before immersion. The samples were then stored at 30°C for 24 h, and the penetration values were compared to those before immersion in kerosene.

The ring and ball softening point was determined according to the ASTM D36-76 methodology (Giuliani et al., 2009). A circular asphalt sample with a thickness of 4 mm was placed in small cylindrical molds with an inner diameter of 20 mm. The test began at a temperature of 5°C and continued until the temperature at which the steel ball penetrated the asphalt specimen was reached. Two specimens were measured for each formulation. To assess the softening point after immersion in kerosene, the specimens were prepared, stored at 30°C for 24 h, and then compared to their softening point values prior to immersion. The tensile adhesion and fuel resistance of the epoxy asphalt were evaluated by measuring the change in tensile adhesion strength before and after exposure to kerosene at various immersion temperatures (30°C, 70°C, 80°C, 90°C, and 120°C) for 24 h. This assessment was performed using an Instron Universal Tester (Model 5965 series) equipped with a 5 kN load cell, with a crosshead speed of 500 mm/min, following the ASTM D 638 standard (Tian et al., 2022; Zhang, Sun et al., 2021; Zhang, Zhang et al., 2021). The samples had dimensions of 30×10×4 mm, and the reported results represent the averages from five specimens tested under each condition.

A t-test analysis was performed using the Data Analysis tool in Microsoft Excel 365 to evaluate statistical differences between the test results of epoxy asphalt before and after immersion in kerosene. The test was conducted at a 95% confidence level.

Results and discussion

Thermal analysis of epoxy asphalt

In this research, asphalt was mixed with epoxy polymers, focusing on high epoxy content to enhance the oil resistance of the asphalt. The crosslinked structure of epoxy polymers is known for providing excellent resistance to solvents and chemical degradation (Jin et al., 2015). However, using only an amine hardener to prepare epoxy asphalt with a weight ratio of 85:15 (denoted as EAC15*) results in a product that is rigid and cures rapidly, solidifying in less than 10 min at 140°C after the addition of the amine. Additionally, the dense network structure formed by the exclusive use of an amine hardener reduces flexibility, making the epoxy asphalt unsuitable for applications requiring flexibility, such as joint sealants. In this study, we found that blending 2 moles of epoxy resin with an amine hardener at a molar ratio of 0.25 and CTPET at a molar ratio of 0.75 helped maintain the mixture in a liquid state, even at temperatures as high as 140°C, allowing for mixing for over 4 h. Furthermore, after curing, the sample retained good flexibility at the operating temperature (30°C).

This result aligns with the DSC analysis shown in Figure 3 and Table 2, where epoxy asphalt with only an amine hardener exhibits a T_g value of 43°C, while the addition of a co-hardener reduces the T_g to -4°C. Blends containing 70–85 wt% epoxy polymer cured with a co-hardener show T_g values ranging from -4°C and -5°C, with minimal variation. This increase is modest compared to AC60/70, which has a T_g of approximately -11°C. This suggests that the epoxy polymer network, formed by amine and ester linkages, slightly restricts the molecular mobility of the asphalt component. The long chains of CTPET extend the crosslink points, allowing the sample to remain flexible even as crosslink density increases with higher epoxy content. However, in epoxy asphalt with 90 wt% epoxy content (EAC10), the T_g significantly increases to around 18°C. This indicates that the high epoxy content increases polymer rigidity by enhancing crosslink density and intramolecular forces while reducing the plasticising effect of the asphalt component (Moser et al., 2024; Si et al., 2018). As a result, a higher T_g is observed.

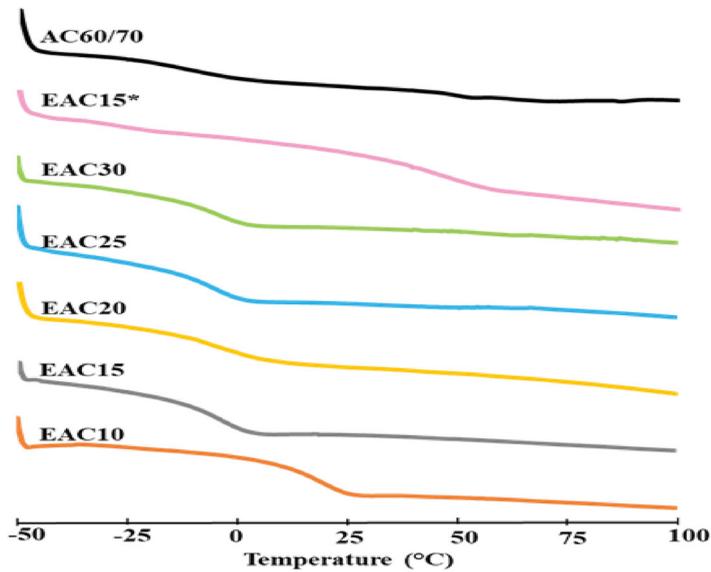


Figure 3. DSC thermograms of asphalt AC60/70 and modified epoxy asphalt with different asphalt contents obtained from the second heating scan.

Table 2. DSC analysis of asphalt AC60/70 and modified epoxy asphalt products.

Sample	AC60/70	EAC15*	EAC30	EAC25	EAC20	EAC15	EAC10
$T_{g,midpoint}$	-10.9°C	42.5°C	-4.9°C	-4.3°C	-4.0°C	-3.6°C	18.5°C

Morphology of epoxy asphalt

The morphology of the epoxy asphalt products was examined using an optical microscope to analyze the matrix and dispersed phases (Figure 4). The epoxy polymer is represented by the yellow phase, while the black inclusions represent the asphalt. It is evident that the asphalt particles are spherical and uniformly distributed within the epoxy polymer across all formulations. The particle size distribution was quantitatively analyzed, as shown in Figure 5. In the system using only amine (EAC15*), the compatibility between the asphalt and epoxy phases was poor. The average diameter of the asphalt phase decreased from $12.1 \pm 7.0 \mu\text{m}$ to $6.1 \pm 2.0 \mu\text{m}$ when switching from an amine hardener to a co-hardener of amine and CTPET, indicating that CTPET significantly enhances compatibility between the asphalt and epoxy resin (Xu et al., 2022). Changing the ratio of asphalt to epoxy resin using a co-hardener resulted in a slight reduction in both the average diameter and distribution of dispersed asphalt particles as the asphalt ratio decreased (Sun et al., 2022; Zhang, Zhang et al., 2021). The particle size for EAC30 exhibited the largest average value, approximately $7.8 \pm 3.5 \mu\text{m}$. For EAC25, EAC20, and EAC10, the average particle sizes were approximately $7.0 \pm 2.3 \mu\text{m}$, $6.9 \pm 2.1 \mu\text{m}$, and $4.9 \pm 1.4 \mu\text{m}$, respectively. In the case of the EA10 formulation, which had the smallest average particle size, it also showed the narrowest particle size distribution. A more uniform distribution with smaller particle sizes is expected to be preferable, leading to higher internal stress concentrations under external loading (Liu, Li et al., 2021). Conversely, formulations with larger average phase sizes and greater phase dispersion may induce lower internal stress development under external forces.

Rheology properties of epoxy asphalt

The complex shear modulus (G^*) is an indicator of a material's resistance to shear deformation under an external load, representing the ratio of peak stress to peak strain (Li et al., 2022). An increase in G^*

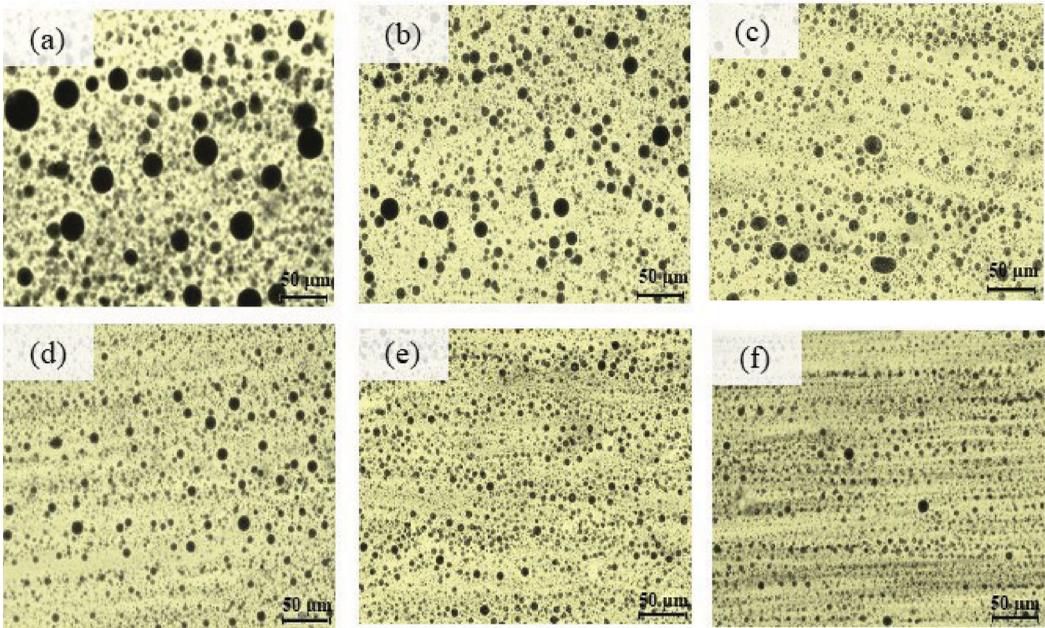


Figure 4. Optical microscope images of modified epoxy asphalt products with different asphalt contents: (a) EAC15*; (b) EAC30; (c) EAC25; (d) EAC20; (e) EAC15; (f) EAC10.

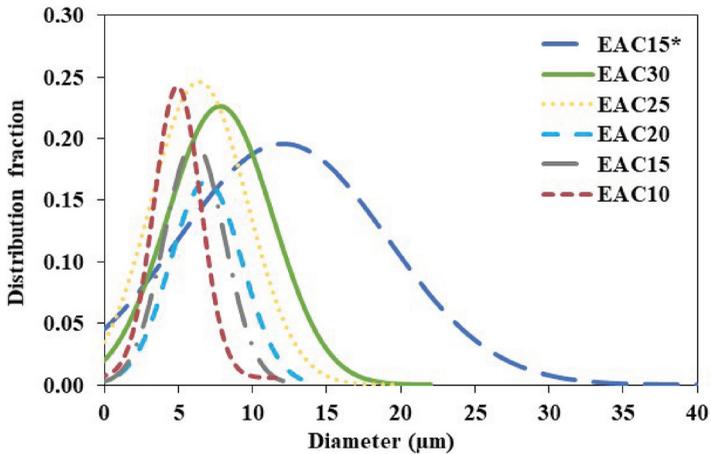


Figure 5. Particle size distribution of modified epoxy asphalt products with different asphalt contents: (a) EAC30; (b) EAC25; (c) EAC20; (d) EAC15; (e) EAC10.

corresponds to higher asphalt stiffness and a greater ability to resist flow deformation. At 50°C, the G^* of AC60/70 was measured at 19.7 kPa. The incorporation of epoxy polymer containing CTPET co-hardener with asphalt enhances the modulus of the sample. EAC10 exhibits the highest modulus at 34.5 kPa, followed by EAC20, EAC25, EAC15, and EAC30, with corresponding values of 24.9, 21.6, 19.7, and 11.8 kPa, respectively (Figure 6(a)). As the temperature rises, both asphalt and epoxy asphalt products soften, leading to reduced elastic behaviour and decreased resistance to deformation. However, at the same temperature, epoxy asphalt products show a higher complex shear modulus (G^*) than the original AC60/70 asphalt, particularly in the 70–90°C range. This suggests that the epoxy polymer in the asphalt effectively enhances its resistance to external loads at high temperatures. This improvement is

attributed to the formation of a strong crosslinked structure within the epoxy polymer, incorporating tertiary amine linkages from the amine hardener and ester/amide linkages from the CTPET hardener (Xue & Xu, 2023). As a result, the epoxy asphalt material becomes less susceptible to shear deformation (Xue & Xu, 2023; Liu, Li et al., 2021).

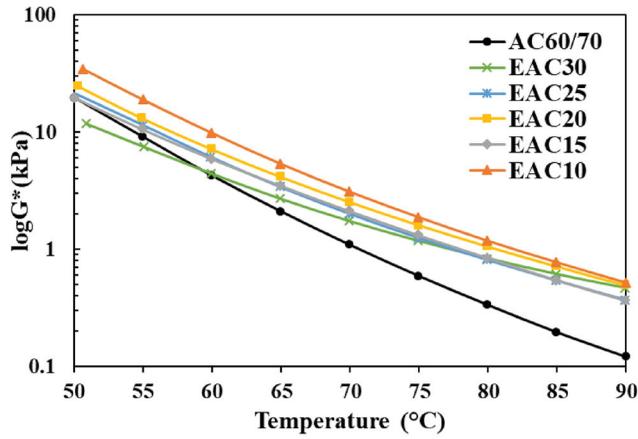
In terms of rheological properties, the phase angle (δ) represents the phase difference between stress and strain during oscillation, offering insight into the viscoelastic behaviour of a material. A phase angle close to 90° indicates a more viscous material, while phase angles near 0° signify a more elastic material (Liu, Li et al., 2021; Li et al., 2022). As shown in Figure 6(b), at the same temperature, the phase angles of the epoxy asphalt products decreased significantly compared to the original AC60/70 asphalt. Additionally, the phase angle tends to decrease as the asphalt ratio increases, ranging from 10 to 30 parts by weight. This suggests that the incorporation of epoxy polymer containing CTPET increased the elastic component of the asphalt.

It is observed that the original asphalt shows a slight increase in phase angle (δ) as the temperature rises. However, when asphalt is blended with the epoxy polymer at a 30:70 weight ratio, the material exhibits a significant reduction in δ with increasing temperature. A similar trend is observed with a 25:75 blend, though the decrease in δ is less pronounced. On the other hand, when asphalt is blended in ratios of less than 20%, δ remains relatively stable, even at elevated temperatures of up to 90°C . Considering the relationship between δ and morphology, it can be suggested that the dispersed asphalt phase exhibiting diverse and larger particle sizes in epoxy asphalt EAC30 contributes to improved elastic behaviour as the temperature rises, leading to a reduction in phase angle. Meanwhile, the presence of a well-dispersed and smaller-sized asphalt phase helps maintain stable viscoelastic behaviour despite temperature fluctuations, resulting in minimal changes in phase angle.

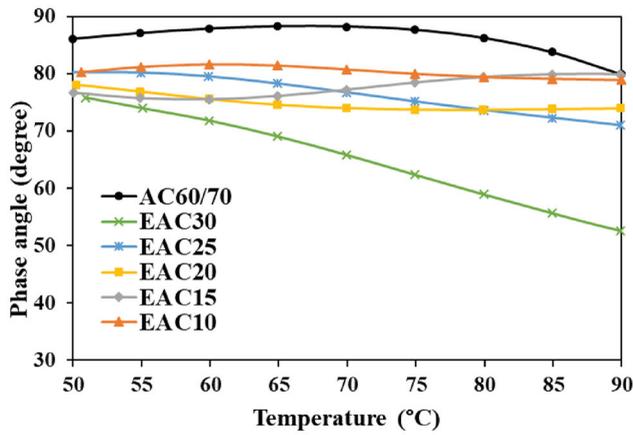
Rutting factors ($G^*/\sin\delta$) are used as indicators of asphalt's resistance to permanent deformation, with higher values reflecting superior rutting resistance performance (Liu, Li et al., 2021). As illustrated in Figure 6(c), the rutting factors for each epoxy asphalt sample declined with increasing temperature, highlighting a shift in the asphalt's viscoelastic behaviour. Specifically, both the complex shear modulus and the proportion of the elastic component in the asphalt decreased as the temperature rose, implying a reduction in the asphalt's anti-deformation and deformation recovery abilities. At the same temperature, epoxy asphalt products exhibited higher rutting factors compared to AC60/70 asphalt. Additionally, the critical temperature corresponding to a $G^*/\sin\delta$ value greater than 1 kPa, which indicates the rutting resistance, ranged from 78°C to 82°C for all epoxy asphalt products with co-hardener. This is higher than the base asphalt's critical temperature, measured at 71°C (Wei et al., 2022). These findings suggest that incorporating epoxy polymer with an amine-CTPET co-hardener significantly enhances the high-temperature stability of asphalt, although this effect diminishes at even higher temperatures.

Bonding behaviour of epoxy asphalt

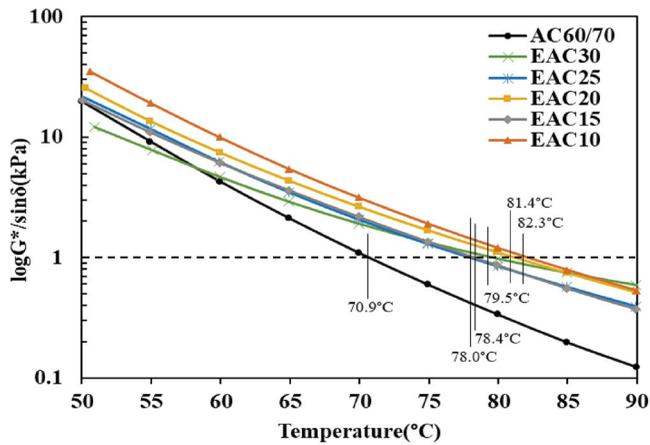
This study aimed to investigate the bonding behaviour between epoxy asphalt and concrete, as well as within the epoxy asphalt itself, focusing on failure behaviour for potential application as a joint sealant. Figure 7 illustrates the stress–strain curves of epoxy asphalt products for adhesion with cement at 30°C . The research measured the force required to stretch the epoxy asphalt by 100% or separate it from concrete, representing cohesion bond failure and adhesion bond failure, respectively (Bionghi et al., 2021). The experimental results revealed consistent changes across all formulations. Initially, the epoxy asphalt exhibits a period of linear elastic deformation during elongation, followed by a decrease in stress during the relaxation stage within the 10–80% elongation range, before entering a stable stage (Cai et al., 2020). Furthermore, the results identified two distinct types of failure. Materials containing 15–30 wt% of asphalt displayed similar trends, allowing the epoxy asphalt to stretch beyond 100% with cohesion bond failure behaviour resembling that of AC60/70 asphalt. It was observed that the cohesion strength of the epoxy asphalt products was higher, indicating superior cohesion bonding compared to the base asphalt. Additionally, the cohesion strength increased as the asphalt ratio



(a)



(b)



(c)

Figure 6. (a) Complex shear modulus (b) Phase angle, and (c) Rutting factors of asphalt AC60/70 and modified epoxy asphalt products.

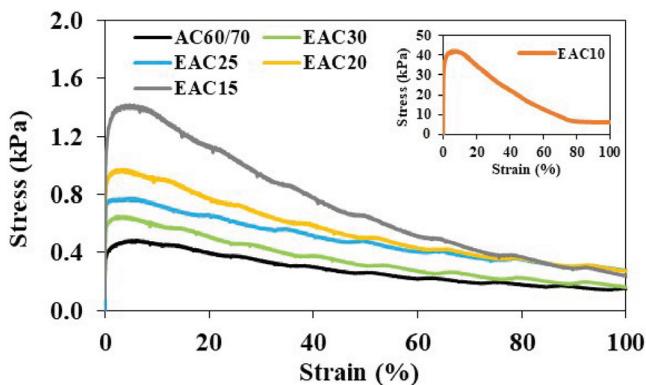


Figure 7. Bonding strength between concrete and modified epoxy asphalt products with varying asphalt contents.



Figure 8. Failure images for bonding between modified epoxy asphalt products and concrete.

decreased. Notably, the formulation containing 10 wt% of asphalt exhibited higher cohesion within the epoxy asphalt than adhesion between the epoxy asphalt and cement. The force required to separate this adhesion exceeded 40 kPa, indicating the high cohesion strength of the epoxy asphalt. This observation aligns with the DSC data, which show that a higher T_g value corresponds to increased crosslinking density and/or cohesive strength, resulting in greater polymer rigidity (Chen et al., 2023; Moser et al., 2024; Si et al., 2018). The formulation containing 10% asphalt by weight exhibited a higher T_g value than the other formulations, indicating stronger cohesive properties. Consequently, separation occurred at the interface between the epoxy asphalt and the cement surface, as shown in Figure 8.

Oil resistance of epoxy asphalt

Table 3 presents the weight loss of epoxy asphalt products after 24 h of immersion in kerosene at various temperatures, compared with asphalt 60/70 and epoxy asphalt prepared solely with an amine hardener (EAC15*). It was found that the immersion of asphalt 60/70 in kerosene at 30°C resulted in immediate dissolution. When comparing formulations with the same asphalt-to-epoxy ratio (15:85) using two different hardeners, it was observed that using only an amine hardener (EAC15*) significantly reduced asphalt dissolution compared to asphalt 60/70. However, this performance was inferior to the combination of amine and CTPET co-hardener (EAC15). The use of amine-only formulations caused asphalt to peel from the specimens during testing at room temperature. In contrast, formulations with co-hardeners exhibited asphalt shedding at around 80°C (Table 4). Although employing amine hardeners improves the kerosene resistance of epoxy asphalt, the resulting samples lack the flexibility needed for use as joint sealants. Conversely, formulations using CTPET as a co-hardener at the same mixing ratio maintain appropriate flexibility, making them more suitable for this application.

Table 3. Weight loss (%) of asphalt and modified epoxy asphalt products after immersion in kerosene at different temperatures for 24 h.

Samples	Average weight loss (%)				
	30°C	70°C	80°C	90°C	120°C
AC60/70	100 ± 0.00	100 ± 0.00	100 ± 0.00	100 ± 0.00	100 ± 0.00
EAC15*	0.21 ± 0.01	0.55 ± 0.01	1.03 ± 0.02	2.05 ± 0.01	4.41 ± 0.11
EAC30	0.00 ± 0.00	0.66 ± 0.09	1.08 ± 0.10	1.97 ± 0.09	16.54 ± 0.81
EAC25	0.00 ± 0.00	0.12 ± 0.01	0.24 ± 0.09	0.89 ± 0.01	1.98 ± 0.18
EAC20	0.00 ± 0.00	0.09 ± 0.00	0.18 ± 0.01	0.58 ± 0.11	1.20 ± 0.12
EAC15	0.00 ± 0.00	0.00 ± 0.00	0.01 ± 0.00	0.03 ± 0.01	0.49 ± 0.12
EAC10	0.00 ± 0.00	0.03 ± 0.01	0.14 ± 0.04	0.35 ± 0.03	1.13 ± 0.12

Table 4. Fuel corrosion photographs of asphalt and modified epoxy asphalt products after immersion in kerosene at different temperatures for 24 h.

Temperature	Samples		
	AC60/70	EAC15*	EAC15
30°C			
80°C	-		

Blending asphalt with epoxy polymers containing CTPET hardener in various weight ratios of epoxy polymer (70–90 wt%) prevented asphalt separation from the test specimens. The weight loss decreased as the ratio of epoxy polymers increased, due to the higher proportion of the oil-resistant epoxy network structure, except at 90% epoxy polymer content. This exception may be attributed to the rigid network structure of the epoxy polymers (high T_g), which could reduce interfacial adhesion between the epoxy and asphalt (Si et al., 2018). Consequently, asphalt in rigid epoxy asphalt products may dissolve more easily than in more flexible formulations, resulting in higher dissolution values for rigid products (EAC15* and EAC10) compared to flexible ones (EAC15–EAC25). Increasing the immersion temperature from 70°C to 120°C for 24 h led to greater asphalt separation. However, the weight loss remained below 2%, complying with the ASTM D3569-95 standard for oil-resistant joint sealants, except for the EAC30 formulation at 120°C. These findings suggest that incorporating CTPET co-hardener into epoxy asphalt improves kerosene oil resistance, especially as the proportion of epoxy resin increases. The optimal ratio was found to be a 15:85 weight ratio of asphalt to epoxy resin, which exhibited minimal asphalt separation (less than 0.03%) even under testing conditions at 90°C.

Table 5 presents a t-test evaluating the statistical differences in epoxy asphalt before and after immersion in kerosene at temperatures ranging from 80°C to 120°C for 24 h. A comparison of epoxy asphalt mixtures with the same weight ratio (15:85), labelled EAC15* and EAC15, reveals a significant difference in weight loss under kerosene exposure at 120°C, with p -values of 0.0078 and 0.1529, respectively (Alamri et al., 2020; Rizvi1 et al., 2019; Shi et al., 2022). These results indicate that incorporating a co-hardener composed of amine and CTPET improves the oil resistance of epoxy asphalt. Although

Table 5. T-test statistical analysis of epoxy asphalt weight loss after immersion in kerosene at 80°C to 120°C for 24 h.

Samples	p-Value			Are the Results Significantly Different at 95% Confidence?		
	80°C	90°C	120°C	80°C	90°C	120°C
EAC15*	0.0588	0.0016	0.0078	no	yes	yes
EAC30	0.0587	0.1488	0.0152	no	no	yes
EAC25	0.2284	0.0715	0.0577	no	no	no
EAC20	0.0515	0.1193	0.0635	no	no	no
EAC15	0.0635	0.2048	0.1529	no	no	no
EAC10	0.1772	0.0544	0.0674	no	no	no

weight loss increases with higher soluble asphalt content, the performance remains strong at kerosene immersion temperatures below 90°C, as suggested by *p*-values greater than 0.05.

It can be inferred that the reduced solubility of the modified epoxy asphalt products can be attributed to the crosslinked network of the epoxy polymer and the physical interactions between the epoxy functional groups and bitumen. Additionally, previous research has noted that incorporating polymers with functional groups, such as ester groups ($-O-C=O$) and aromatic rings, into asphalt can create strong interactions with polar components and the aromatic/resin fraction, thereby enhancing oil resistance (Giuliani et al., 2009; Li et al., 2019; Liu, Li et al., 2021; Merusi et al., 2011; Rizvi et al., 2019). CTPET contains a substantial amount of ester groups and aromatic functionalities. As the epoxy polymer ratio increases, the content of these functional groups in the epoxy asphalt products also increases. This enhancement correlates with improved oil resistance in the epoxy asphalt products.

Penetration and softening point of epoxy asphalt before and after fuel immersion

Figure 9 displays the penetration values of base asphalt and epoxy asphalt products after being stored at room temperature for various durations. It was observed that the penetration value of the base asphalt, after being left for 1 d, was 69 dmm. Furthermore, the penetration of the material decreased over time with the extended storage period. Specifically, after 21 days of storage, the base asphalt exhibited a reduced penetration value of 53 dmm, indicating a trend towards increased hardness and consistency over time. In the epoxy asphalt mixture, the penetration value of the material decreased proportionally with a reduction in the asphalt ratio (Xue & Xu, 2023). This reduction in penetration is attributed to the restricted molecular mobility caused by the enhanced formation of the epoxy network structure (Liu, Li et al., 2021; Yao et al., 2019). Nevertheless, after 21 days of storage, it was discovered that the formulation with 30 wt% asphalt had a penetration value close to that of the base asphalt, around 51–53 dmm. Formulations with 15–25 wt% asphalt had penetration values, ranging from 33–34 dmm. The formulation with 10 wt% asphalt had the lowest penetration value of 13 dmm.

The results depicted in Figure 10 were obtained by measuring the penetration value of epoxy asphalt specimens stored for 7 days and subsequently immersed in oil at 30°C for 24 h. The findings indicate that all formulations exhibited a modest increase in penetration, ranging from 0% to 14%, compared to their values before oil immersion (Van Rooijen et al., 2004). This observed rise in penetration suggests that the asphalt, following immersion in kerosene, facilitates the movement of the epoxy network, leading to enhanced chain mobility during penetration testing. However, it is noteworthy that the epoxy network, particularly when combining amine linkage and ester linkage, plays a crucial role in maintaining the hardness and consistency of the epoxy asphalt after kerosene immersion. As a result, the penetration values show only a small change.

The softening point of the base asphalt was observed to be 48.6°C. Increasing the epoxy polymer content in the mixture from 70 wt% to 85 wt% did not significantly affect the softening point, which remained within the range of 48.6–49.1°C. This suggests that blending epoxy polymer with a CTPET co-hardener into asphalt, even at a high proportion of up to 85 wt%, does not substantially impact the thermal stability of the modified asphalt compared to the base asphalt (Liu, Li et al., 2021). However,

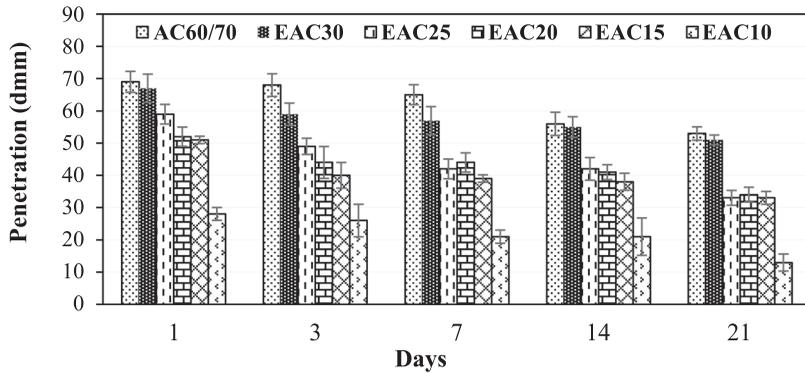


Figure 9. Penetration of asphalt and modified epoxy asphalt products for 1, 3, 7, 14, 21 days.

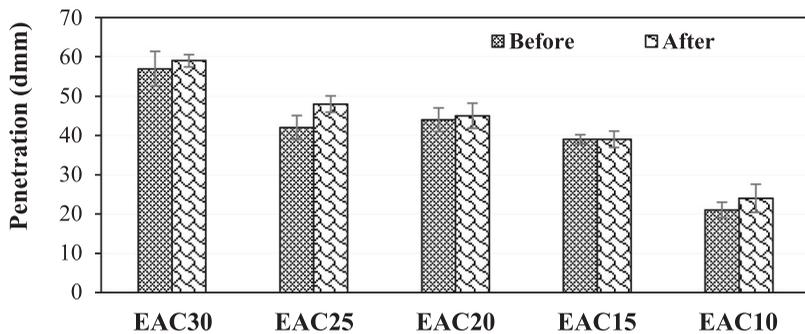


Figure 10. Penetration of modified epoxy asphalt products after being stored for 7 days followed by an immersion in kerosene at 30°C for 24 h.

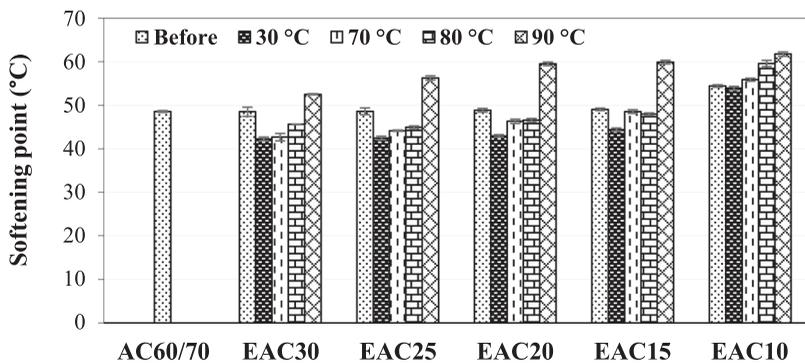


Figure 11. Softening point of asphalt and modified epoxy asphalt products before and after immersion in kerosene at different temperatures for 24 h.

in the formulation containing 90 wt% epoxy polymer, the softening point increased notably to 54.5°C. This represents an approximately 11% increase compared to the base asphalt, indicating an elevated softening temperature (Figure 11).

Measuring the softening point of the epoxy asphalt products after immersion in kerosene oil at various temperatures for 24 h indicated that the specimens became softer compared to those measured before immersion, except for the specimens immersed in oil at 90°C, which exhibited a higher softening point (Figure 11). When the softening point of the specimens was measured after immersion in oil

at 30°C, all specimens showed a reduction in the softening point by approximately 9–12% (15–30% by weight of asphalt) (Van Rooijen et al., 2004). Although no asphalt leakage was observed during the immersion of the epoxy asphalt specimens in kerosene at 30°C, the penetration of kerosene into the epoxy asphalt may disrupt the intermolecular structure of the asphalt, resulting in a decrease in viscosity and temperature stability (Xue & Xu, 2023). At higher oil temperatures, increased asphalt leakage may release aromatic and saturated hydrocarbon components, leading to an increase in viscosity and hardness of the specimens (Li et al., 2019).

Consequently, the softening point of the specimens increases as the immersion temperature rises. For specimens immersed in kerosene at 90°C, the softening point increased by 15–40% compared to their values before immersion, ranging from 53°C to 62°C.

Tensile properties of epoxy asphalt before and after fuel immersion

In this research, the samples were prepared by mixing at 140°C for 30 min, followed by a 7-day period at room temperature before undergoing tension testing. Figure 12 illustrates the tensile strength and elongation at break of the epoxy asphalt products at various asphalt ratios. It was observed that mixing the epoxy polymer and asphalt in a 70:30 ratio significantly enhanced the tensile strength, increasing it from 0.03 MPa to 0.22 MPa. The tensile strength of the specimens remained relatively consistent, ranging from 0.28 to 0.38 MPa, as the asphalt proportion was reduced from 25 wt% to 15 wt%. However, increasing the epoxy polymer content to 90 wt% resulted in a remarkable six-fold increase in tensile strength compared to the specimens with 85% epoxy polymer. While reducing the asphalt proportion may enhance tensile strength, blending epoxy polymer and asphalt in a 70:30 ratio significantly reduced specimen elongation, decreasing from 7000% to 2800%. Increasing the epoxy polymer proportion led to denser cross-linking within the specimens, with asphalt acting as a plasticiser and flexible linkages from the CTPET contributing to improved elongation at break. Specimens containing 75% and 80% epoxy polymer exhibited notable increases in elongation, reaching 3600% and 4100%, respectively, along with corresponding improvements in tensile strength. However, excessive epoxy polymer content (beyond 85 wt%) made the specimens more prone to separation, resulting in hard and brittle fracture behaviour and elongation of less than 700% when the epoxy polymer content was 90%.

Figure 13 shows the stress–strain relationship of modified epoxy asphalt products. The curves of the samples show an initial increase, signifying a linear elastic stage. All samples exhibit elastic behaviour up to approximately 1% elongation. After reaching the yield point, the tensile stress gradually decreases as the material transitions into plastic behaviour due to a pronounced necking effect (Cong et al., 2019; Liu et al., 2016). An analysis of the correlation between tensile properties and morphology revealed that incorporating asphalt at a 30 wt% ratio resulted in varied asphalt particle sizes, which were notably larger than those observed at other epoxy polymer-to-asphalt ratios.

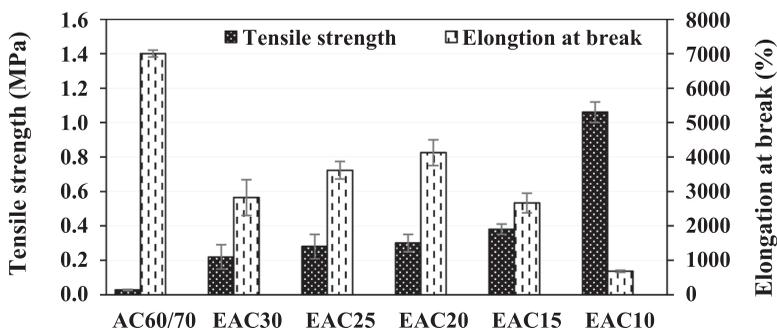


Figure 12. Tensile strength and elongation at break of modified epoxy asphalt products with varying asphalt contents.

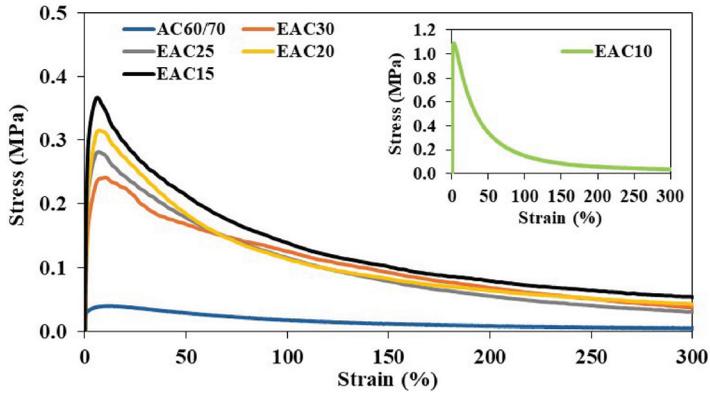
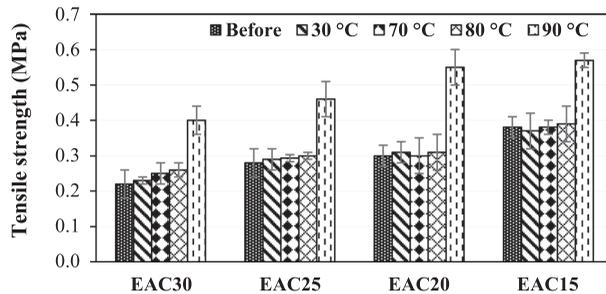
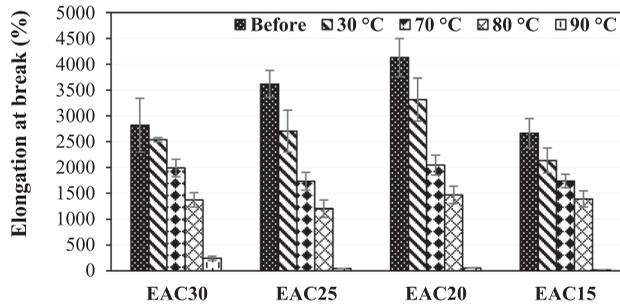


Figure 13. Stress-strain curve of modified epoxy asphalt products with varying asphalt contents.



(a)



(b)

Figure 14. (a) Tensile strength and (b) Elongation at break of modified epoxy asphalt products before and after immersion in kerosene at different temperatures for 24 h.

Consequently, these specimens exhibited limited stretching capabilities under moderate force. In contrast, reducing the asphalt ratio decreased the size of the dispersed asphalt phase, affecting the force required for specimen stretching. Notably, specimens with 10 wt% asphalt content had the smallest particle sizes in the dispersed phase, with minimal size distribution. These specimens exhibited the highest modulus values, reflecting maximum hardness, but showed minimal elongation before failure.

The tensile properties of the epoxy asphalt products after aging in kerosene at various temperatures are depicted in Figure 14. The tensile strength remains consistent with the pre-immersion values when the materials are subjected to temperatures below 80°C. However, there is a comparatively shorter strain failure after aging. At higher kerosene aging temperatures, increased strain failure was

observed. This could be attributed to the dissolution of aromatic and saturated hydrocarbon compounds from the asphalt in kerosene, potentially diminishing the viscoelastic behaviour of the epoxy asphalt materials and consequently reducing their elongation properties (Li et al., 2019). Nevertheless, the three-dimensional structure of the epoxy polymer maintains the strength of the samples, resulting in consistent tensile force. However, after immersion in kerosene at 90°C, the samples exhibited increased hardness, accompanied by a rise in the softening point value. This could be due to the loss of flexible asphalt components and an increased presence of the rigid cross-linked epoxy polymer.

Conclusions

To develop a fuel-resistant material with properties similar to asphalt, a new epoxy asphalt specifically designed for use in areas exposed to fuel or fuel vapours was evaluated. Simultaneously, the performance of epoxy asphalt using a synergistic co-hardener, composed of both amine and acid hardeners, was compared with that of base asphalt and epoxy asphalt formulated with only an amine hardener. The main conclusions of this study are summarised as follows:

- (1) The addition of epoxy cured with a co-hardener at a substantial ratio of up to 85 wt% maintained the asphalt's thermal properties, with a glass transition temperature (T_g) below 0°C, which is lower than the T_g of epoxy cured with only an amine hardener (above 40°C). Furthermore, the co-hardener significantly improved compatibility with asphalt, as evidenced by the reduction in dispersed phase size from $12.1 \pm 7.0 \mu\text{m}$ to $6.1 \pm 2.0 \mu\text{m}$.
- (2) Compared to base asphalt, the epoxy asphalt exhibited improvements in complex shear modulus and rutting resistance, accompanied by a reduction in phase angle. These improvements were proportional to the increased epoxy resin ratio, indicating enhanced resistance to shear deformation under external loads, greater elasticity, and improved resistance to permanent deformation at elevated temperatures. Additionally, the cohesion bonding strength of the epoxy asphalt increased, suggesting a shift from cohesion failure to adhesion failure as the epoxy content increased. This shift can be attributed to the enhanced crosslinking network and the higher ester and aromatic group content derived from CTPET, which strengthened the interaction between the epoxy resin and asphalt.
- (3) Blending a modified epoxy resin with a concentration of at least 75 wt% effectively enhanced fuel resistance, particularly at higher immersion temperatures. Notably, asphalt loss remained below 2%, even when tested at 120°C. However, within this ratio range, 80 wt% epoxy resin provided the optimal balance between tensile strength and elongation, both before and after immersion in kerosene. A recommended immersion temperature of no more than 80°C ensures optimal material performance.

Author contribution

P.S.: Experiment, Formal analysis, Writing-original draft. S.H.: Conceptualization, Methodology, Review & Editing. P.R.: Conceptualization, Methodology, Experiment, Review & Editing. CW.P.: Conceptualization, Resources, Methodology, Validation, Writing-original draft & Editing, Supervision. All authors have read and agree to the published version of the manuscript.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

This research was funded by Naresuan University, Thailand (grant number R2566B031).

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