Evaluation of permanent deformation and durability of epoxidized natural rubber modified asphalt mix

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Abstract. The road distresses have caused too much in maintenance cost. However, better understandings of the behaviours and properties of asphalt, couples with greater development in technology, have allowed paving technologists to examine the benefits of introducing additives and modifiers. As a result, modifiers such as polymers are the most popular modifiers used to improve the performance of asphalt mix. This study was conducted to investigate the use of epoxidized natural rubber (ENR) to be mixed with asphalt mix. Tests were conducted to investigate the performance characteristics of ENR-asphalt mixes, where the mixes were prepared according to the wet process. Mechanical testing on the ENR-asphalt mixes have demonstrated that the asphalt mix permanent deformation performance at high temperature was found to be improved compared to the base mixes. However, the durability studies have indicated that ENR-asphalt mixes are slightly susceptible with the presence of moisture. The durability of the ENR-asphalt mixes were found to be enhanced in term of permanent deformation at high and intermediate temperatures compared to the base asphalt mixes. As conclusion, asphalt pavement performance can be enhanced by using ENR as modifier to face the major road distresses.

1. Introduction

During last decades, a lot of research works has been carried out to modify asphalt and asphalt mix [1-6]. The potential value of polymer modified asphalt (PMA) is that polymers can significantly improve the performance of hot mix of asphalt (HMA) and substantially increase the service life of highway surfaces [8]. Specifically, the addition of polymers significantly improves various asphalt properties, such as elasticity, cohesion, stiffness and adhesive, resulting in a substantial improvement in the performance and quality of asphalt pavements with the HMA being more stable at warmer temperatures and more flexible at colder temperatures. In addition to rutting resistance, a premium polymer can provide a degree of flexibility or elasticity to an HMA, thereby improving the fatigue and thermal cracking characteristics of the asphalt mix.
One of the most well-known, widely used category of polymers is thermoplastic elastomers (TE). TE are polymers with thermoplastic and elastomeric properties [8]. TE polymers derive their strength and elasticity from a physical cross-linking of molecules within a three-dimensional network. Styrene-butadiene-styrene (SBS) is the most commonly known TE that can improve the rheological properties of asphalt [9]. Furthermore, SBS increases binder elasticity at high temperatures and improves flexibility at low temperatures. This improvement led to increased resistance to asphalt rutting at high temperatures, and decrease cracking at low temperatures [10]. Styrene-butadiene-rubber (SBR) is another example of an elastomer polymer and it increases the ductility of asphalt pavement and consequently becomes more flexible and crack-resistant at low temperatures [11]. In addition, other types of additives have also been used as modifiers, i.e., as TE, such as styrene isoprene styrene (SIS), styrene ethylene butadiene styrene (SEBS), ethylene-propylene-diene-terpolymer (EPDM), isobutene-isoprene-copolymer (IIP), crumb-rubber, polybutadiene (PBD), polyisoprene and natural rubber (NR) [7, 12]. However, the major types of styrenic block copolymers such as SBS, SIS and hydrogenated styrene block copolymers (HSBC) have the best modifying potential when blended with asphalt [14-16].

The potential application of epoxidized natural rubber (ENR) as a modifier was realized in the 1980s [15]. ENR is a chemically modified natural rubber created by reacting natural rubber with proxy formic acid [15]. This material has good properties, offering high strength because of its ability to bear strain crystallization, along with increased glass transition temperature. These properties facilitate increased oil resistance, enhanced adhesion properties, damping and reduced gas permeation [16, 17]. According a previous study, ENR is able to increase the viscosity, stiffness and decreased the temperature susceptibility for the binder [18, 19]. The best results were recorded for polymer-modified asphalt containing 6% of ENR [20]. This study was conducted to present a laboratory evaluation of performance of unaged, short and long term aged of base asphalt mix (HMA-0) and asphalt mix modified with 6% of ENR (as a percentage of asphalt by weight) (HMA-6) in terms of dynamic creep, rutting and to evaluate the susceptibility to moisture.

2. Experimental works

2.1 Materials

The asphalt used in this study is 80/100 penetration grade, supplied by the asphalt factory at Port Klang, Malaysia. The ENR was obtained from the Malaysian Rubber Board under the trade name of ENR 50, with 53% epoxidation and passed through 2.36 mm mesh sieve (before shearing). The physical properties of the asphalt and the ENR used are shown in Table 1. The aggregate used for preparing the mix samples was obtained from Negeri Roadstone Sdn. Bhd. Malaysia.

| Material | Properties                        | Value     | Specification |
|----------|-----------------------------------|-----------|---------------|
| Asphalt  | Specific Gravity                  | 1.03      | ASTM D70      |
| 80/100   | Penetration @ 25 °C               | 82        | ASTM D5       |
|          | Softening point (°C)              | 45.7      | ASTM D36      |
|          | Viscosity @ 80 °C (Pa.s)          | 12.6      | ASTM D4402    |
|          | Ductility (cm) @ 25 °C and 5 cm/min | >100   | ASTM D113     |
| ENR      | Size (before shearing)            | 2.36 mm   | -             |
|          | Specific Gravity                  | 0.94      | -             |
ENR-modified asphalt was produced by mixing 6% of ENR (by weight of asphalt) with base asphalt. ENR-modified asphalt was prepared using high shear mixer at 160°C (±1°C) under 4000 rpm of speed for 62 min [20]. ENR was added to the asphalt after the temperature stabilized at 160°C. The asphalt mix design is based on the Superpave volumetric mix design. The design equivalent single axle loads (ESALs) for this study are assumed to be less than 107. This makes the design in the category of 3×106 to 107 ESALs (80 KN/ESAL), or traffic level 4 [21]. Traffic levels are used to determine design requirements such as number of design gyrations for compaction, aggregate physical property requirements, and mix volumetric requirements. The traffic level also determines the level of mix design required. For traffic levels of less than 107 ESALs, a level 2 mix design is recommended [21]. The mix in this study will have a nominal maximum particle size aggregate of 19.0 mm, however, six stockpiles of aggregate consisting of three coarse aggregates and three fine aggregates.

2.2 Asphalt Mix Performance Tests

2.2.1 Dynamic creep test. The dynamic creep test is a testing which also can be implemented using UTM to determine the permanent deformations of asphalt mix in the laboratory. In this study, the dynamic creep test under haversine load pulse was conducted for the base asphalt mixes and ENR-modified mixes in accordance with protocol developed by NCHRP 9-19 Superpave Models [22]. Each category of ENR mixes consists of three replicate samples compacted at 4% air voids with a diameter of 100 mm and 150 mm height were conditioned at provided chamber at a temperature of 40°C for at least four hours before testing is initiated. During initial stage of testing, sample was pre-loaded with conditioning stress at 10 kPa for 120 seconds to ensure the platen is loaded flat on the sample. Then, sample is applied with a haversine wave load cycle which consists of 100 kPa stress pulse with 100 ms pulse width followed by a 900 ms rest period. The test was terminated when the accumulative stain reached 10000 micro-strains or until 10000 cycles whichever come first.

2.2.2 Rutting test. This test simulates wheel passes within a pavement. The procedure is described in BS-598-110 [25]. A laboratory scale of this test was conducted using the Wessex wheel tracking device. Wheel tracking test samples were produced for each mix having the respective design aggregate and OBC obtained earlier. In this study, one mould was used to hold the rut sample in the wheel tracking machine. The height of the mould follows exactly the original slab mould of the Wessex wheel tracking device. Approximately 3,700 gm of mix was compacted to 7±0.5% air voids with a size of 300×300 mm, and a final height of 65±1 mm. The samples were left to cool in room temperature for 24 hours after compaction before calculating the air voids content to meet test requirements. The rut test was conducted at 50°C. Before testing, the samples were conditioned for at least four hours at test temperature. The test was conducted only in dry conditions. The samples were subjected to a simulated trafficking with a simple harmonic motion by applying 520 N load for one hour.

2.2.3 Moisture Susceptibility. It is very important to evaluate the moisture sensitivity of the design mix. This step is accomplished by performing AASHTO Method on the design aggregate structure at the design asphalt binder content [24]. Samples, unmodified and ENR-modified asphalt, with 100 mm in diameter and 63.5±2.5 mm in height are compacted to approximately 7 % air voids. One subset of three samples is the control subset. The other subset of three samples is the conditioned subset. The conditioned subset is subjected to partial vacuum saturation followed by an optional freeze cycle, followed by a 24 hour thaw cycle at 60°C. All samples kept at a temperature of 25°C for a period of 2 hours without soaking, and tested to determine their indirect tensile strengths. The moisture sensitivity is determined as a ratio of the tensile strengths of the conditioned subset divided by the tensile strengths of the dry (control) subset. The minimum criterion is 80 % retained tensile strength [24].
3. Results and Discussion

3.1 The design of asphalt mixers

Initially, the bulk specific gravity (Gsb) test of coarse and fine aggregate was calculated for all sieve sizes and shown in Table 2. The apparent specific gravity (Gsa) and effective specific gravity (Gse) were also determined from these tests. Further analysis on the aggregates was conducted as a critical requirement in the Superpave system to analyze the consensus and source aggregate properties in order to achieve a high performance of a HMA pavement. From the results, aggregate from the quarry complied with the specifications set by the standard criteria. Table 2 shows the results of the aggregate property tests.

| Aggregate properties          | Result | Criteria | Standard          |
|------------------------------|--------|----------|-------------------|
| Gsb of coarse aggregate      | 2.58   | -        | ASTM C 127        |
| Gsb of fine aggregate        | 2.61   | -        | ASTM C 128        |
| Flakiness (%)                | 6.00   | <20      | BS 812 : section 105.1 : 1989 |
| Fine Aggregate Angularity (%)| 51.5   | >45      | AASHTO T33        |
| Elongation (%)               | 16.0   | <20      | BS 812            |
| Sand Equivalent Test (%)     | 48.5   | >45      | AASHTO 176        |
| Los Angeles Test (%)         | 32.13  | <45      | ASTM C: 131-81    |
| Soundness Test (%)           | 6.1    | <12      | ASTM C88          |
| Deleterious Materials (%)    | 0.5    | 0.2 to 10| ASTM C142         |

In general, aggregate fulfilled the Superpave mix design requirements and is suitable for use as pavement material. Table 3 shows the aggregate gradation used in this study. Aggregate gradation was prepared using the 19.0 mm nominal maximum size to comply with the Superpave gradation limits.

| Sieve size (mm) | Upper Limit specs. | Lower Limit specs. | Pass (%) | Retained (%) |
|-----------------|---------------------|---------------------|----------|--------------|
| 25              | 100                 | 100                 | 100      | 0            |
| 19              | 100                 | 90                  | 100      | 0            |
| 12.5            | 90                  | -                   | 82       | 18           |
| 9.5             | -                   | -                   | 76       | 6            |
| 4.75            | -                   | -                   | 51       | 25           |
| 2.36            | 49                  | 23                  | 29       | 22           |
| 1.18            | -                   | -                   | 21       | 8            |
| 0.6             | -                   | -                   | 16       | 5            |
| 0.3             | -                   | -                   | 11       | 5            |
| 0.15            | -                   | -                   | 7        | 4            |
| 0.075           | 8                   | 2                   | 4        | 3            |
| Pan             | -                   | -                   | -        | 4            |

The material characterisations, based on standard and Superpave tests are presented in Table 4 [25, 26]. These results compiled with the specifications set by the standard criteria, with considering the binders to be PG 76.
Table 4. Binder properties.

| Binder description (and criteria) | ENR content (by % weight of asphalt) |
|----------------------------------|-------------------------------------|
|                                  | HMA-0                               |
|                                  | HMA-6                               |
| Original binder                  | 0%                                  |
| Penetration at 25°C, 1/10mm      | 82                                  |
| Softening point,°C               | 45.70                               |
| Viscosity @ 135°C (Maximum 3 Pa.s), Pa.s | 0.244 |
| Flash point (Minimum 230°C),°C   | 275                                 |
| Dynamic Shear at 10 rad/s (G*/sinδ), (Minimum 1 kPa), kPa | 0.177 |
| Dynamic Shear at 10 rad/s (G*.sinδ), (Minimum 5000 kPa), kPa | 3962 |

Table 5. Volumetric Properties of all mixes.

| Mix Properties | HMA-0 | HMA-6 | Criteria |
|----------------|-------|-------|----------|
| OBC (%)        | 4.86  | 5.08  | -        |
| Air Voids (%)  | 4.00  | 4.29  | -        |
| VMA (%)        | 14.44 | 17.44 | ≥13      |
| VFA (%)        | 74.14 | 75.00 | 65-75    |

Asphalt mixes were successfully developed in accordance with the procedures described in Superpave mix design standard [21]. In this study, mixes with five percentages (4, 4.5, 5, 5.5, 6%) of asphalt content were prepared (with three replications for every sample) for each unmodified and modified HMA. Specimens were prepared with blended mineral aggregates at an increment of 0.5% of binder (4.0, 4.5, 5.0, 5.5, 6.0%) by weight of aggregate for base and modified HMA.

The optimum binder content (OBC) to obtain acceptable volumetric properties when compared to the established mix criteria is based on the Superpave gyratory compactor (SGC) specimens with 4% air voids. The volumetric properties consists of OBC, effective binder content (Pbe), voids in mineral aggregate (VMA), voids filled with asphalt (VFA), ratio of dust to effective binder content (P0.075/Pbe). The major components in determining stability and durability of Superpave HMA mix consists of VMA, VFA, air voids and dust proportion. Table 5 summarises the volumetric properties of design mixes corresponding to OBC of the mix along with mix design criteria. For all mixes, results showed that the mixes properties satisfy all the criteria set by the Superpave system.

3.2 Dynamic creep test
This test is used to determine the resistance of HMA mixes to plastic deformation. The dynamic creep curve consists of three parts: primary, secondary and tertiary. The accumulated strain is recorded at each load cycle, and in this test, termination occurred when the numbers of cycles reaches 10000 cycles. As the loading period is required to terminate at 10000 cycles, tests showed that not all
specimens failed before reaching the maximum number of cycles. The tests were conducted on the unaged and aged mixes. Under the 10000 load cycles, all axial strains exhibit curve relationship with load cycles in the strain versus load cycles plot. In this test, mixes where subjected to short and long term oven ageing to evaluate the influence of the added ENR on the asphalt mix, and the results were compared before and after short term ageing (STA) and long term ageing (LTA) ageing.

Figure 1 shows the results of comparison between the accumulated strain of unaged HMA-0 and HMA-6. The dynamic creep for the HMA-0 mix is extremely more than for HMA-6; this behaviour can be attributed to the influence of the ENR on the binder, and thus, on the mix. The mechanical properties of the HMA-6, at high temperatures, rely on the properties of the binder, especially for the permanent deformation. It is expected that the modified mix resistant to permanent deformation will increase. Therefore, adding ENR to asphalt mixes significantly decreased its susceptibility to permanent deformation.

The behaviour of a polymer on asphalt mix can be compared with a previous research with using various percentages of styrene butadiene styrene (SBS) as modifier [27]. Khodaii and Mehrara [27] found that adding 5% SBS to the asphalt increased the resistance of the asphalt mix to permanent deformation.

Figures 2 and 3 show the permanent deformation results for the mixes before and after STA and LTA. As expected, the results of both HMA-0 and HMA-6 mixes show the same behaviour of accumulated strain. The HMA-0 and HMA-6 after STA and LTA ageing produced less accumulated strain compared with the unaged mixes. However, the effect of STA and LTA on the permanent deformation performance of the HMA-0 mixes is high compared with HMA-6, indicating that ageing has less influence on the mixes with polymer. Table 6 shows the ageing index of the ultimate strains for the dynamic creep test. Compared with HMA-0, the accumulated strain for HMA-6 decreased more than three times after STA and six times after LTA.
Figure 2. Dynamic Creep Curves for HMA-0 before and after oven ageing.

Figure 3. Dynamic Creep Curves for HMA-6 before and after oven ageing.

Table 6. Ageing index for the dynamic creep test results.

| Mix            | Ultimate Strain % | Ageing index % |
|----------------|-------------------|----------------|
| Unaged-HMA-0   | 3.334             | 1.00           |
| STA-HMA-0      | 0.657             | 5.07           |
| LTA-HMA-0      | 0.267             | 12.48          |
| Unaged-HMA-6   | 0.348             | 1.00           |
| STA-HMA-6      | 0.248             | 1.40           |
| LTA-HMA-6      | 0.170             | 2.05           |
3.3 Rutting test
The rutting test is a simulative test, also known as torture test conducted using the dry wheel tracking device. The test temperature of 50°C was chosen to simulate extreme environmental conditions for the HMA mixes. The wheel passes over HMA specimens and terminates after the test duration reaches 45 minutes. The result of rutting deformation is visible from the wheel tracking test conducted on the specimen as shown in Figure 4. In general, HMA-6 mixes exhibited better rutting resistance compared to HMA-0 mixes as shown in Figure 4. The maximum rutting values for HMA-6 mixes ranged from 2.05 mm to 3.24 mm, while HMA-0 mixes exhibited high rutting, with the maximum rutting values ranging from 3.07 mm to 5.71 mm. This obviously indicates the high resistance of HMA-6 mixes to rutting compared to HMA-0.

On the other hand, it was found that the STA and LTA caused a significant impact on the rutting resistance for both HMA-0 and HMA-6, which is expected. STA and LTA decreased the rutting susceptibility for both mixes, HMA-0 and HMA-6. However, for HMA-6 showed more resistance to ageing impact. Table 7 shows the ageing indexes for all mixes. The process of oxidation in binders (ageing), form polar compounds that tend to increase the amount of asphaltenes. These asphaltenes contribute to a solid structure of asphalt binder that leads to increased binder stiffness and viscosity. However, in term of post curing, there is no visible swelling in the modified HMA, this is because of the existing of the epoxide in ENR which prevents the swelling of the polymer or the ENR modified asphalt. In addition, the ageing of asphalt mix was associated with the degradation of the ENR polymer and oxidation of asphalt, leading to a decrease in polymer molecular weight and an increase in the content of polar oxygen-containing molecules and asphalt molecular weight. Therefore, the general trend of ENR modified asphalt was to became more resistance to ageing effect because the asphalt content in HMA-0 is less than in HMA-6 [28-31].

An improvement in the performance of the asphalt mix has been observed on addition of SBS [32]. Improved resistance to rutting of the pavement was observed with SBS modification, compared with the base asphalt mix. On the other hand, an eight years old SBS modified asphalt pavement was found to have experienced intensive oxidative age hardening. Polymer modified asphalt mixes were found to be stiffer, thus, increased rutting resistance of the HMA [33].

![Rutting Test Results for unaged and aged mixes.](image)
Table 7. Ageing index for the rutting test results.

| Mix            | Rutting depth (mm) | Ageing index % |
|----------------|--------------------|----------------|
| Unaged-HMA0    | 5.71               | 1.00           |
| STA-HMA0       | 4.07               | 1.40           |
| LTA-HMA0       | 3.07               | 1.86           |
| Unaged-HMA6    | 3.24               | 1.00           |
| STA-HMA6       | 2.55               | 1.27           |
| LTA-HMA6       | 2.05               | 1.58           |

3.4 Moisture Susceptibility

Indirect tensile strength (ITS) results of each mix under the dry and conditioned case were averaged based on results of three specimens [24]. The results for ITS test, are listed in Table 8. According to the AASHTO standard, all mixes tested were prepared to have air voids content in the range of 7±0.5%. The trend showed that ITS of all specimens decreased from the dry to conditioned specimens implying the presence of deterioration in the mixes which affects the strength of the HMA mix.

The tensile strength ratio (TSR) result is an indication of the HMA mix susceptibility to moisture damage. HMA-0 and HMA-6 mixes met the required minimum 80% TSR value as specified in AASHTO T283. The indirect tensile strength test showed that HMA-6 mixes exhibit higher tensile strength values than HMA-0 before and after conditioning, this showed that HMA-0 is more likely to be susceptible to hydrolysis. Khudyakova, Strizhev and Mashkova [34] investigated the chemical nature of the hydrolysis of asphalt and concluded that asphalt–mineral adhesiveness is primarily determined by the chemical composition of the dispersion medium (i.e. resins, aromatics and saturates), and by the concentration of the polar compounds capable of forming bonds with acid-based minerals which are not destroyed by water. In a previous study, it was found that asphalt mix modified with SBS (HMA-SBS) reduced the susceptibility of HMA to moisture damage [29]. It was found that the SBS-HMA asphalt samples achieved a higher TSR value than the unmodified HMA, 88.9% and 76.7% respectively.

Table 8. Moisture susceptibility test results.

| Mix     | ITS- Dry (Kpa) | Air void (%) | ITS-Conditioned (Kpa) | Air void (%) | TSR (%) |
|---------|----------------|--------------|-----------------------|--------------|---------|
| HMA-0   | 374.65         | 6.60         | 357.00                | 6.70         | 0.86    |
|         | 397.72         | 7.03         | 318.18                | 6.99         |         |
|         | 401.47         | 6.85         | 335.86                | 7.29         |         |
| HMA-6   | 424.94         | 6.48         | 386.56                | 7.12         |         |
|         | 419.63         | 7.02         | 363.56                | 7.17         | 0.89    |
|         | 443.53         | 6.98         | 394.04                | 7.04         |         |

4. Conclusions

The mechanical performance of HMA-6 mixes for unaged, STA and LTA ageing were discussed in this study and compared to the performance of HMA-0 mixes. The mechanical performances which were investigated consisted of dynamic creep, rutting, and moisture susceptibility. In addition, the mixes were subjected to short term ageing and long term ageing. Permanent deformation, at 40 and
50°C, of ENR-HMA is dominated by the ENR content. Where, ENR-HMA produced less deformation comparing with base-HMA. This is due to the presence of highly elastic rubber in HMAs at high temperature. The effect of the ageing on the permanent deformation performance of the base-HMA is extremely high comparing with ENR-HMA, indicating that ageing has less effect on HMA with the existence of the ENR. Moisture damage has reduced after modifying the mixes with ENR, indicating that ENR-HMA is less susceptible to moisture damage comparing with the base-HMA. This improvement is not significant, but the results still within the standard requirements.

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