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Laboratory Assessment of Epoxy Asphalt Mixture Incorporating Tire Rubber Waste

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Abstract
The use of epoxy asphalt in road paving is one of the promising solutions for long-life road pavements in service with minimal maintenance. However, the high cost still stands as an obstacle to the widespread use of this high-performance material. The use of tire rubber waste (TRW) is one of the solutions in order to reduce costs, improve the environment, and improve the performance of epoxy asphalt mixtures, in addition to alleviating the brittle behaviour that epoxy asphalt tends to. This study proposes to add TRW in improving epoxy asphalt produced in local laboratories by using phenol Novolac resin as an epoxy curing agent of the epoxy base inside asphalt binder to produce and evaluate improved epoxy asphalt. The percentage of epoxy base used was 25% of the asphalt binder mixed with a 1:1 ratio of epoxy to Novolac using potassium hydroxide (KOH) as a catalyst. Whereas the proportions of added TRW were (1%, 2%, and 3%) of the total mixture weight by using the dry mixing method. The results showed, at its best values at 2% of TRW, that there was an increase in Marshall stability by 10%, and Marshall flow remained within specification limits with a decrease in the value of air voids at the highest bulk density, and a slight decrease in indirect tensile strength by 2%, with remaining excellent resistance to moisture sensitivity at 94%, and improvement in resistance to permanent deformation (rutting) by 14%. This indicates an improvement in the improved epoxy asphalt mixtures by the addition of TRW compared to the reference epoxy asphalt mixtures.

Keywords: TRW ; epoxy asphalt; improved epoxy asphalt; novolac.

الخلاصة: يعد استخدام الإسفلت الإيبوكسي في رصف الطرق أحد الحلول الواعدة لرصف الطرق طويلة العمر في الخدمة بأقل قدر من الصيانة. ومع ذلك، لا تزال التكلفة العالية تشكل عقبة أمام الاستخدام الواسع النطاق لهذه المواد عالية الأداء. يعد استخدام مخلفات مطاط الاطارات (TRW) أحد الحلول من أجل تقليل التكاليف وتحسين البيئة وتحسين أداء خلطات الإسفلت الإيبوكسي، بالإضافة إلى التخفيف من السلوك الهش الذي يميل إليه الإسفلت الإيبوكسي. تقترح هذه الدراسة إضافة مخلفات مطاط الاطارات في تحسين الأسفلت الإيبوكسي المنتج في المختبرات المحلية باستخدام راتنج الفينول نوفولاك كعامل معالجة للايبوكسي داخل رابط الأسفلت لإنتاج وتقييم الإسفلت الإيبوكسي المحسن. كانت النسبة المئوية للايبوكسي المستخدمة ٢٥٪ من رابط الأسفلت والممزوج بنسبة ١: ١ من الايبوكسي إلى نوفولاك وباستخدام هيدروكسيد البوتاسيوم (KOH) كمحفز. بينما كانت نسب مخلفات مطاط الاطارات المضافة (١٪، ٢٪، ٣٪) من وزن الخليط الكلي باستخدام طريقة الخلط الجاف. أظهرت النتائج، بأفضل قيمها عند ٢٪ من مخلفات مطاط الاطارات، أن هناك زيادة في استقرار مارشال بنسبة ١٠٪، وأن تدفق مارشال ظل ضمن حدود المواصفات مع انخفاض في قيمة الفراغات الهوائية عند أعلى كثافة كلية للخلطة، و انخفاض طفيف في مقاومة الشد غير المباشرة بنسبة ٢٪، مع بقاء المقاومة الممتازة لحساسية الرطوبة عند ٩٤٪ ، وتحسين مقاومة التشوه الدائم (Rutting) بنسبة ١٤٪. يشير هذا إلى تحسن في مخاليط الإسفلت الإيبوكسي المحسنة بإضافة مخلفات مطاط الاطارات مقارنة بمخاليط الإسفلت الإيبوكسي المرجعية.

1. INTRODUCTION

Asphalt concrete can be described as a viscoelastic as well as thermoplastic material [1, 2]. Modification of asphalt binder and asphalt concrete mixtures by different kinds of additives, especially by the polymers, is common [3]. Generally, the thermoset asphalt mixtures are produced of suspended dense structure, which could ensure excellent physical, mechanical, and chemical properties such as water resistance, less heat susceptibility, permanent deformation resistance, and anti-fatigue performance as well as chemical resistance, especially resistance to fuel [4, 5]. Epoxy asphalt is a binder that consist of asphalt binder and epoxy resin cured inside the asphalt due to the reaction of hardener (curing agent) to produce epoxy asphalt [6]. Distinct from other used polymer-modified asphalt binders whose physical properties change with the temperature change, while epoxy asphalt is a thermoset material. Due to the curing process, the epoxy resin after reacted with the curing agent forms a three-dimensional network phase in which asphalt is dispersed [7, 8]. It is known that epoxy asphalt tends to behave towards brittleness due to the effect of epoxy inside the asphalt binder, and one of the proposed solutions is to add materials with rubbery behaviour to improve the epoxy asphalt to make it less brittle. The use of tire rubber waste (TRW) in improving the binder and mixtures of asphalt for paving purposes has been used for a relatively long time ago as a solution that helps in improving the environment and reduces production costs, and this was by using one of two methods, either wet method by dissolving TRW in asphalt binder at elevated temperature or by dry method by adding TRW as a part of mixture aggregate [9-11]. This study proposes to add locally consumed TRW to improve the epoxy asphalt mixtures produced in local laboratories using the dry method and making laboratory evaluated to the produced improved epoxy asphalt mixtures.

2. USED MATERIALS

2.1. Epoxy asphalt

The epoxy asphalt used in this study is an epoxy asphalt produced in the local laboratories from the reaction of the epoxy base with Novolac as curing agent (hardener) material inside the asphalt binder by 25% epoxy of asphalt binder and 1:1 epoxy to Novolac, where the characteristics of its materials are explained in the following items. Table 1 shows the properties of the epoxy asphalt binder and mixtures used in this study.

Table 1 The properties of epoxy asphalt binder and mixtures

Test	Result	Conditions of test	Standard
Penetration	5 mm	100 gm- 25°C, 5 sec.- (0.1mm)	ASTM-D5
Ductility	20 cm	25°C- 5cm/min	ASTM-D113
Softening Point	>100 °C	----	ASTM-D36
Marshal Stability	20 kN	60°C (30-40 min.), 51 mm/min	ASTM D6927
Marshal Flow	3.5 mm	60°C (30-40 min.), 51 mm/min	ASTM D6927
Aire Voids%	4.5%	---------	ASTM D1559
Bulk Unit Weight	2.271 g/cm^3	---------	ASTM D1559
Indirect Tensile Strength	15.66 KPa	25°C, 51 mm/min	ASTM D6931
Tensile Strength Ratio	94%	25°C, 51 mm/min	ASTM D6931
Hamburg Wheel Truck Test (Rutting)	2.62 mm	60°C, @ 10000 Cycles	AASHTO T 324

2.2. Conventional asphalt binder

A conventional bitumen with a penetration degree of 40-50 mm was used, which was brought from the Dora refinery in Baghdad, central Iraq, and a group of tests were conducted on it according to ASTM standards, where its test results are illustrated in Table 2. All tests were performed in the Asphalt Laboratory of the University of Technology, and the National Center for Construction Laboratories in Baghdad.

Table 2 The properties of conventional asphalt binder

<table>
<tr><th>Test</th><th>Result</th><th>Standard limits</th><th>Conditions of test</th><th>ASTM standard</th></tr>
<tr><td>Penetration</td><td>43 mm</td><td>40-50</td><td>100 gm- 25°C, 5 sec.- (0.1mm)</td><td>ASTM-D5</td></tr>
<tr><td>Ductility</td><td>>140 cm</td><td>>100</td><td>25°C- 5 cm/min</td><td>ASTM-D113</td></tr>
<tr><td>Softening Point</td><td>52 °C</td><td>----</td><td>----</td><td>ASTM-D36</td></tr>
<tr><td>Specific gravity</td><td>1.03</td><td>----</td><td>25 °C</td><td>ASTM-D70</td></tr>
<tr><td rowspan="2">Flash and fire points</td><td>295 °C</td><td>> 232 °C</td><td rowspan="2">-----</td><td rowspan="2">ASTM-D92</td></tr>
<tr><td>310 °C</td><td>----</td></tr>
<tr><td rowspan="2">Loss on heating</td><td>66 °C Penetration</td><td>> 55</td><td rowspan="2">163 °C- 50 gm, 5 hr</td><td rowspan="2">ASTM-D1754</td></tr>
<tr><td>60 °C Ductility</td><td>>25</td></tr>
<tr><td rowspan="2">Rotational Viscosity</td><td colspan="2">0.643 Pa. sec @ 135 °C</td><td rowspan="2">------</td><td rowspan="2">ASTM-D4402</td></tr>
<tr><td colspan="2">0.169 Pa. sec @ 165 °C</td></tr>
</table>

2.3. Epoxy resin

The epoxy resin that was used in this study is NITOBOND EP SLOW SET BASE, the supplier is FOSAM COMPANY LTD, have the properties illustrated in Table 3.

Table 3 The properties of epoxy resin

Properties	Value
Appearance	Viscous liquid
Color	White
Odor	Typical
Flash point	> 250°C
Relative density	1.19 @ °C
Solubility (ies)	Insoluble in water

2.4. Curing agent

The Novolac polymer (Phenol formaldehyde solid resin) was used in this research as a curing agent to cure the epoxy resin base inside the asphalt binder to produce epoxy asphalt [12-14], (Figure 1(a)). The Novolac was brought from (State Company for Mining Industries-Bagdad-Iraq) has properties and composition which is illustrated in Table 4. Although phenol groups act as a catalyst to complete the reaction between the hydroxyl alcohol and the epoxy groups, but with the presence of some catalysts such as KOH (Figure 1(b)), the reaction between the hydroxyl group and the epoxy groups is better, more stable, and at a lower temperature. Properties of potassium hydroxide (KOH) are illustrated in Table 5.

Table 4 Properties of Novolac resin

Properties	Unit	Value
Phenol-Formaldehyde	wt.%	100
Density	gm/cm3	1.25
Melting point	°C	85
Thermal conductivity	W/m °C	0.2
Properties medium	-------	Under acidic conditions (HCL)

Table 5 Properties of potassium hydroxide (KOH)

Properties	Unit	Value
Chemical formula		KOH
Molar mass	mol−1	56.11 g
Appearance	--------	white solid, deliquescent
Odor	--------	odorless
Density	g/cm3 (25 °C)	2.12
Melting point	°C	360
Boiling point	°C	1,327
Flash point	-------	Non-flammable

(a) (b)

Figure 1 (a) Phenol Novolac solid resin and (b) potassium hydroxide

2.5. Aggregate

In this study, the crushed natural aggregate was used from the production of the Nabaie quarry in Iraq, west of the capital, Baghdad. Where it is widely used in central Iraq to produce asphalt mixtures. Its physical and chemical properties were as shown in Tables 6. Selected aggregates in the mixtures of reference epoxy asphalt and improved epoxy asphalt followed the Iraqi Standard for Roads and Bridges for a wearing course layer with aggregate of 12.5 mm Nominal maximum size [15], as detailed in Table 7.

Table 6 Properties of aggregate

Test		**sieve size (mm)**	**Apparent Gs**	**Bulk Gs**	**Abs.%**	**Standard**
Specific gravity	Coarse aggregate	12.5	2.674	2.651	0.32	ASTM-C127
		9.5	2.591	2.585	0.09	
		4.75	2.582	2.57	0.18	
	Fine aggregate	4.75– 0.075	2.642	2.615	0.48	ASTM-C128
Angularity	Coarse aggregate	97%	**Limits**			ASTM-D 5821
			Min 95%			
Soundness	Coarse aggregate	3.20%	10-20% Max			ASTM-C88
Equivalent sand (clay content)	Natural (<#4)	84.50%	Min 45%			ASTM-D2419
	Crashed (<#4)	89.60%				
Flat & Elongation aggregate	Flat	0.90%	Max 10%			ASTM-D4791
	Elongation	2.50%				
Toughness, by (Los Angeles Abrasion)	Aggregate Size < 25 mm	21.30%	30 % Max			ASTM-C131

Table 7 Gradation of asphalt mixture

Size of sieves		**% Passing for surface layer (IIIA), according to Iraqi Specification, (SCRB R9, 2003)**		**Work choice**	
Standard Sieve (mm)	English Sieve	Minimum	Maximum	% Passing	% Retaining
19	3/4"	---	100	100	0
12.5	1/2"	90	100	95	5
9.5	3/8"	76	90	83	12
4.75	#4	44	74	59	24
2.36	#8	28	58	43	16
0.30	#50	5	21	13	30
0.075	#200	4	10	7	6
Pan	---				7

2.6. Tire rubber waste additive (TRW)

Tire rubber waste (TRW) was used as a rubber additive to the epoxy asphalt to reduce the brittleness behaviour of the epoxy asphalt. The TRW used in this study is the crumb rubber produced from cutting and fraying locally consumed car tires, which has the properties shown in Table 8 and Figure 2 and has grain size distribution explained in Table 9.

Table 8 Chemical composition of tire rubber waste

Chemical composition (%)	**Value**
Acetone extract	15.5
Ash content	6.0
Carbon black	29.5
Rubber hydrocarbon	49.0

Figure 2 Tire rubber waste (TRW)

Table 9 Grain sizes distribution of tire rubber waste

Sieve No.	Sieve Size (mm)	% Retaining by weight
No. 40	0.425	41
No. 50	0.3	25
No. 80	0.180	20
No. 200	0.075	14

3. LABORATORY WORK

3.1. Sample preparation

To describe the mixing operation of conventional asphalt with the hardener (Novolac) and epoxy resin to obtain epoxy asphalt and improved epoxy asphalt, the following steps were adopted in sequence:

1. Novolac is ground by using the electric mill to be used as a dry powder curing agent with a conventional bitumen binder.
2. The epoxy and the curing agent (Novolac) that used 25% of asphalt binder, and the ratio of epoxy to Novolac is (1:1).
3. The bitumen binder (40-50) was raised it a temperature to around 150 ° C in the oven, then an appropriate amount of heated asphalt was placed in the propeller mixer. Then Novolac was added gradually to the bitumen binder to avoid agglomeration of polymer with continuous mixing at 170 ° C for 15 minutes to achieve good mixing and dispersion of the polymer into the bitumen binder.
4. An appropriate amount of epoxy base is raised its temperature to not less than 80 C° in the oven.
5. 0.03% Potassium Hydroxide (KOH) catalyst of the wight of epoxy asphalt is added to the asphalt/Novolac blend and the appropriately heated epoxy is added to the asphalt/Novolac blend and continue mixed automatically by the shear mixer for 1 min to produce homogenous epoxy asphalt.
6. A portion of the fine aggregate remaining on the sieve # 200 equivalent to (1%, 2%, and 3%) of the total weight of the mixture was replaced by tire rubber waste, mixed with the mixture aggregate by using dry mixing method.
7. The produced epoxy asphalt is added to the preheated aggregate to about 160C° to produce the samples of epoxy asphalt and improved epoxy asphalt mixtures for Marshal samples.
8. Finally, the samples after compacted by 75 blows on both faces of Marshal samples by using Marshal Hammer, then samples are cured in the oven for 5 hours with a temperature >155C° to be ready for tests [16].

3.2. Experimental tests

3.2.1. Marshall test

To evaluate asphalt mixture performance, by this study, the properties of improved epoxy asphalt mixtures compared with reference epoxy asphalt mixtures were evaluated. The tests were performed at 60C°according to the Marshall stability test AASHTO T245 (AASHTO 2014). The Marshall stability, and flow results were used for the evaluation process of the performance of improved epoxy asphalt [17]. Where the ratio of bitumen that used was the optimum ratio calculated (5.1%) of Marshal sample weight by using Marshal design method procedure. The samples were prepared by using the procedure that described in the item 3.1. above.

3.2.2. Indirect tensile strength test (IDT).

IDT strength values, as well as laboratory mix design test, it is used to assess the quality of the asphalt mixtures in bearing cracking or fracturing. IDT test was implemented depending on ASTM D6931 - 17 “Standard Test Method for Indirect Tensile” (IDT) Strength of Asphalt Mixtures on the reference epoxy asphalt and the improved epoxy asphalt.

3.2 .3 Indirect tensile strength ratio test (TSR)

Moisture susceptibility of asphalt mixtures was assessed by indirect tensile strength test (IDT) according to AASHTO T 283-14. Where the samples prepared with 7% air voids were separated to make two groups, each group consisting of three samples, one of the groups was kept in the vacuum (97.3–98.7) kPa for around 15 min to be saturated of (55%–80%), while the second group kept in the room temperature.
After that, the samples of the second group remain in the water at normal pressure for half an hour, then the samples are submerged in a water bath with a temperature of 60 °C for a time of 24 hours. After that, the samples are submerged in water with a temperature of 25° C for a time of 2 hours. Finally, the indirect tensile test values are calculated using a standard load machine at a load rate of 50 mm/min to calculate the TSR % by using the Equation 1:

$$TSR\ \% = RT2/RT1 x 100 \quad (1)$$

Where RT1 is the average indirect tensile strength of dry specimens (MPa), RT2 is the average indirect tensile strength of conditional samples (MPa), and TSR is the average tensile strength ratio (%).

3.2.4. Wheel Track Test

For evaluating the permanent deformation's resistance of reference and improved epoxy asphalt mixtures, a wheel tracking test was used according to AASHTO T 324-2011, where the used test samples were prepared by using Superpave samples that have dimensions of 150 mm in diameter x 50 mm depth as shown in Figure 3 [18]. The test was conducted at 60 °C, 0.7 MPa. The samples were prepared by using a Superpave gyratory compactor in the asphalt laboratory of the university of technology, where the air voids of prepared samples were 4%, and the wheel track test was performed in the civil laboratory of Karbala university.

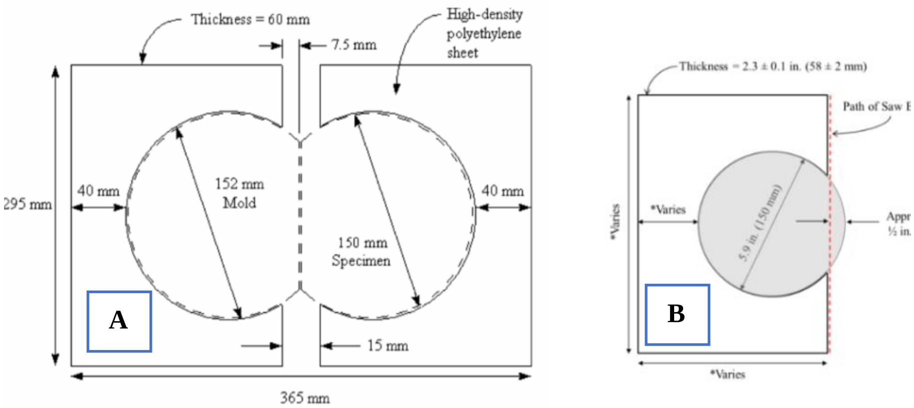

Figure 3 Cylindrical steel compaction mold and sample

4. RESULTS AND DISCUSSION

4.1. Marshall properties

It is clear that from Figure 4 that adding tire rubber waste (TRW) to epoxy asphalt using dry mixing method by replacing the fine aggregates remaining on sieve # 200 with a quantity of TRW equivalent to (1%, 2%, 3%) of the total weight of the mixture, led to a decrease in the values of Air Voids (Av%), where it reached of 1% and 2% TRW, about 3.4%, and 4.1%, compared with 4.5% for reference epoxy asphalt, but the Av% increased to 5.5% at 3% of TRW due to a decrease in the workability of mixtures led to difficulties during the mixing and compaction process when preparing the test specimens.

In the same context mentioned above, the bulk unit weight increased by adding TRW and reached (2.310, and 2.291) g/cm3 for TRW ratios (1% and 2%), respectively, compared with 2.271 g/cm3 of reference epoxy asphalt, but it decreased to reach 2.233 g/cm3 when adding 3% TRW as shown in Figure 5. The addition of TRW to epoxy asphalt improved Marshall's stability values (Figure 6), especially for TRW ratios (1% and 2%), through which Marshall's stability reached (22 kN) compared with (20 kN) for reference epoxy asphalt, while it decreased to (15 kN) of (3%) TRW. This decrease is also due to the decrease in the workability of the mixtures during the process of preparing the test specimens, while the flow values, were not significantly affected by the addition of TRW to epoxy asphalt, except for 3%, which witnessed a relatively large decrease to reach (2.6 mm) compared to (3.5 mm) for epoxy asphalt without additive, but they all remain within the acceptable specifications. (Figure 7).

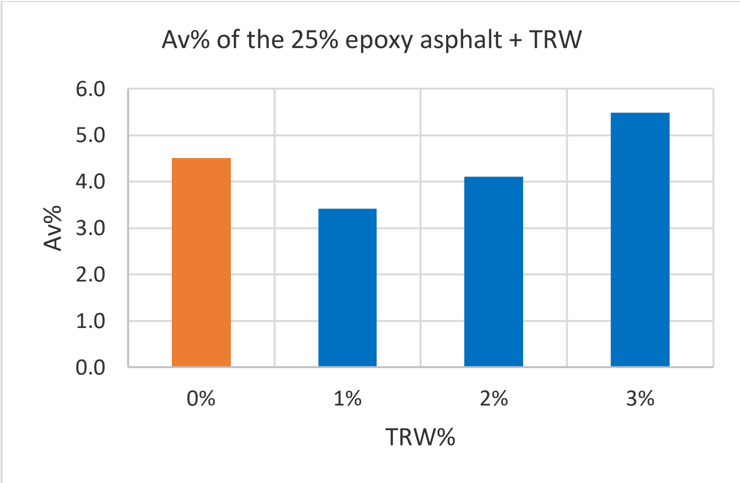

Figure 4 Av% of EA + % of TRW

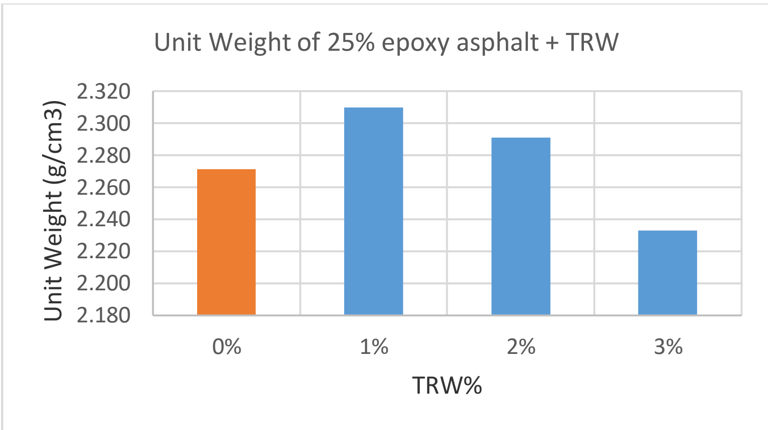

Figure 5 Bulk Unit Weight of EA + % of TRW

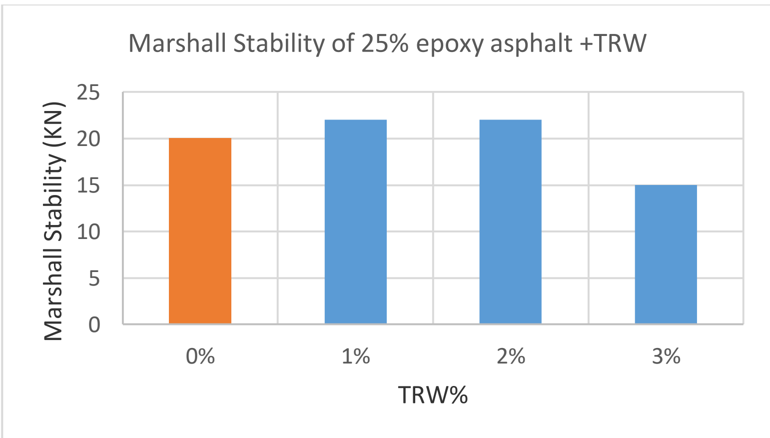

Figure 6 Marshall Stability of EA + % of TRW

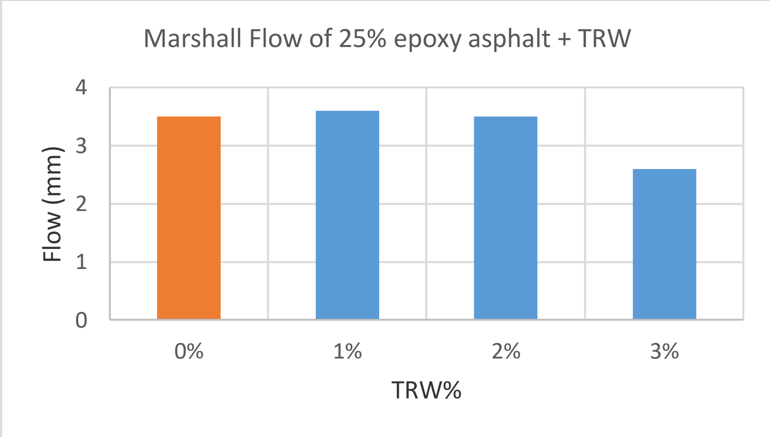

Figure 7 Marshall Flow of EA + % of TRW

4.2. Indirect tensile strength (ITS)

Figure 8 explain a relationship between IDT of reference epoxy asphalt mixtures samples and improved epoxy asphalt mixtures by adding TRW in ratios (1%, 2%, and 3%) of the total mixture weight. Where it is noticed that there was a decrease in the indirect tensile strength values, which amounted to (15.45, 15.4, 15.1) Kpa for TRW ratios in epoxy asphalt (1%, 2%, and 3%), respectively, compared with the value of indirect tensile strength value of epoxy asphalt without additive, which was (15.66) Kpa. The presence of waste tire rubber particles as part of the aggregate reduced the area of aggregate attached to the epoxy asphalt binder, which slightly affected the tensile strength of the improved epoxy asphalt mixture.

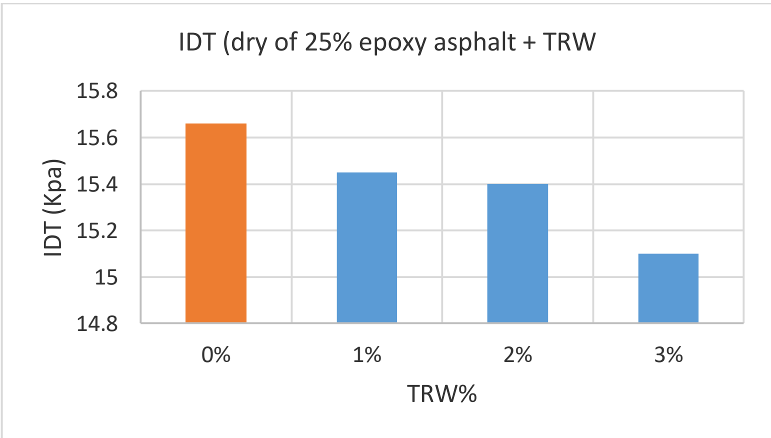

Figure 8 Indirect Tensile Strength of epoxy asphalt + % TRW

4.3. Moisture susceptibility

Figure 9 illustrate the values of Tensile Strength Ratio (TSR%) that resulting from the ratio of dividing IDT values of the samples (conditional) by IDT values of the samples (unconditional) under standard test conditions for epoxy asphalt with TRW ratios (1%, 2%, and 3%) compared with pure epoxy asphalt samples under the same conditions. Where it is noticed that the addition of TRW had no influence on the quality of resistance of improved epoxy asphalt mixtures to water sensitivity, and the TSR values remained at around 94% for all mixtures. This indicates the very good resistance of the epoxy asphalt mixtures to moisture due to the good bonding strength, which increases the resistance to stripping and peeling [19].

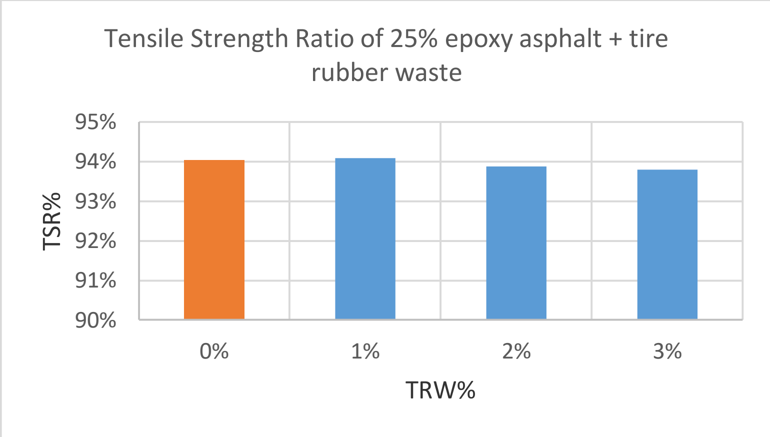

Figure 9 Tensile Strength Ratio of epoxy asphalt + % TRW

4.4. Permanent deformation (rutting) resistance

To determine the resistance of the improved epoxy asphalt by adding tire rubber waste to permanent deformations especially at high temperatures compared to the resistance of reference epoxy asphalt to permanent deformations under the same condition, used the Hamburg Wheel Track test by using cylinder specimens compacted by using superpave gyratory compactor according to (BS EN 12697-22). (Figure 10) illustrate the reaction of rutting depth and repetition cycles when a standard load at a temperature of 60 °C is applied, where it is noticed that the value of the rutting depth after 10,000 cycles of repeat the standard load was 2.25 mm for samples of 2% TRW of improved epoxy asphalt which showed good results in other performance tests comparing with a rutting depth of 2.62 mm for reference epoxy asphalt. Using TRW with epoxy asphalt mixtures as additive by using the dry method by replacing a portion of the fine aggregate showed a slight improvement in the mixture's resistance to permanent deformations. The ability of tire rubber waste to improve the resistance of asphalt mixtures to permanent deformations has enhanced the ability of the improved epoxy asphalt to resist this type of deformation, thus enhancing its performance [11].

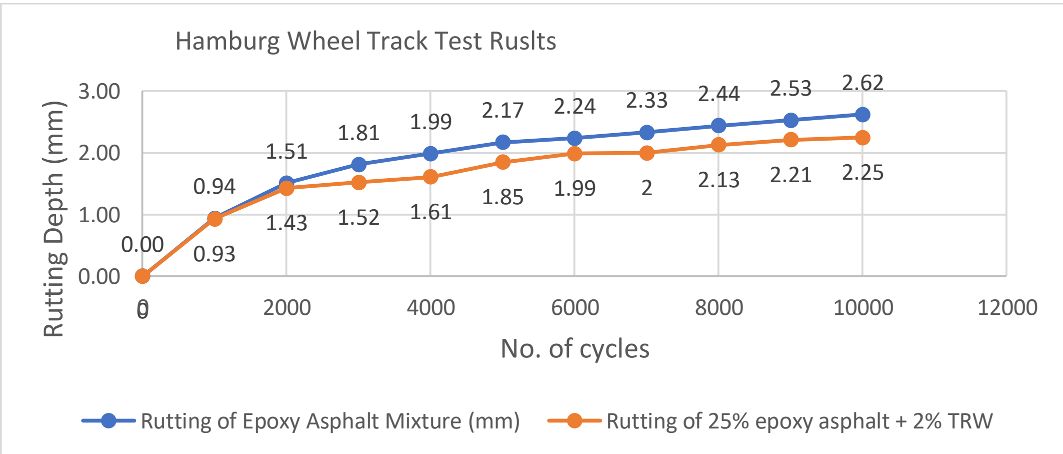

Figure 10 Hamburg Wheel Track Test results of 25% epoxy asphalt + 2% TRW

5. CONCLUSIONS

The following conclusions can be drawn from the work presented in this study:

1. The addition of tire rubber waste (TRW) to improve the epoxy asphalt mixtures produced from this study using the dry method slightly lowered percentage air voids (Av%) because of the increase of bulk unit weight of the improved epoxy asphalt mixtures, but after 2% TRW, the results were reversed.

2. Addition of tire rubber waste (TRW) to improve the epoxy asphalt mixtures produced from this study using the dry method slightly improved the Marshall stability values by 10% especially for the ratios 1% and 2% of TRW, while the Marshall stability decreased by 25% of 3% TRW.
3. The addition of tire rubber waste (TRW) to improve the epoxy asphalt mixtures produced in this study using the dry method led to a very slight reduction of about 2% of the indirect tensile values compared to the epoxy asphalt.
4. The addition of tire rubber waste did not affect the water sensitivity of the improved epoxy asphalt mixtures compared with the excellent water impact resistance of the epoxy asphalt mixture.
5. The excellent permanent deformation strength of epoxy asphalt mixture is slightly improved around 14% for improved epoxy asphalt mixture of 2% TRW.
6. The addition of 2% TRW to the epoxy asphalt mixtures produced in this study by the dry mix method gave the best results for improving the epoxy asphalt mixtures.

7. Recommendations

The use of tire rubber waste in improving epoxy asphalt is justified to improve the behaviour of epoxy asphalt, which tends to behave brittle, which weakens its resistance to low-temperature conditions, and although we used the dry mixing method in this study, we recommend using the wet mixing method and conducting the necessary tests to get better results for both binder and mixtures of epoxy asphalt.

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