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Research Article

Improvement of Rheological Properties of Modified Asphalt Treated with Residues of Recycled Rubber from Waste Tires and Oxidized by Air

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Abstract

Asphalt materials loaded with polymer additives have gained particular importance in recent years due to their close association with modification processes and the creation of a clean environment, mainly from plastic wastes in paving and other areas, and they have also caused significant improvement in asphalt properties. It was observed through the research that the rheological properties of the asphalt were improved significantly as added residues of recycled rubber (RRR) from waste tire percentages increased. The observations are apparent from the decrease in permeability of the asphalt and enhance its ductility and elongation. The study focused on modifying the rheological properties of asphalt materials using the residues of the recycled process of rubber (RRR) from waste tires (mainly carbon black, containing residues of rubber extracted from waste tires). The asphalt materials were oxidized in the open system under various conditions of temperature and oxidation time in the presence of a 0.25% (w/w) anhydrous aluminum chloride ($AlCl_3$) catalyst. After determining the optimal conditions for the oxidation process, the added anhydrous $AlCl_3$ catalyst was adjusted to determine its optimal ratio. The modified asphalt samples after oxidation at optimal conditions in the presence of anhydrous $AlCl_3$ catalyst and the recycled rubber (RRR) residues were tested using appropriate measurements. The following measurements of ductility, permeability, softening point, Marshall stability and flow, aging resistance (thin film oven test (TFOT)) and the asphalt content percentages were done, and their results show that the modified asphalt exhibits completely different rheological properties from the original asphalt. The studied N19 and N20 models show availability in paving applications.

Keywords: Rheological properties, oxidation process, asphalt modification, residues of recycled rubber, waste tires

1. INTRODUCTION

Modifying asphalt by adding various polymeric materials, such as polystyrene, polyethylene, polypropylene, natural rubber, and reclaimed rubber from scrap tires, is a modern approach to modification processes [1]. These modifications give the treated asphalt different and excellent properties due to the incomplete dissolution of the additives in the asphalt materials [2]. Among the modern physical methods used in modification processes is adding sulfur to asphalt in various proportions. However, the modification results are temporary (unstable) and change shortly after that due to the phenomenon of phase separation between the additive and asphalt [3]. Several modern chemical methods have emerged for modifying and improving asphalt properties, including treating asphalt material with various polymeric materials. This treatment uses an alkylation catalyst and is conducted between the asphalt and the added polymeric material after suitable treatments to qualify them for the desired chemical reaction [4]. Asphalt with good rheological specifications is very limited so many researchers have resorted to developing rheological properties using various methods, with polymeric material being one of the most critical [5]. In recent years, many research studies have highlighted the influential role of certain polymeric in the modification field [6]. For example, adding tire rubber powder to asphalt mproves its rheological properties, increasing its durability and resistance to external stress and cracks resulting from these stresses [7]. Asphalt, known as the heaviest component produced from crude oil distillation processes, is one of the most complex components, with significant differences between its components being unclear [8]. Asphalt is characterized by its black or dark brown color and high molecular weight compared to other petroleum fractions. Additionally, it has a density ranging from (1.0 to 1.1) g/cm^3 and emits a strong odor when heated [9]. In general, asphalt is a sticky or semi-solid substance with high viscosity at room temperature, obtained through various crude oil distillation processes [10, 11]. The chemical inertness of raw asphalt and its physical properties have made it suitable for multiple uses in construction and industry since ancient times, as its physical properties determine the nature of its use [9], [12].

In the following work, using the waste tire rubber [6], the residues of the recycled rubber (RRR) from waste tires after reclaiming most of their rubber were added to the raw asphalt. The modified asphalt models used in road paving were obtained after measuring various rheological properties such as elongation, permeability, softening point, penetration index, Marshall test, and aging process. The results obtained showed completely different rheological properties from the original non-axial asphalt. This research will provide an economical and efficient approach to improving the rheology of asphalt by recycling waste materials.

2. RESEARCH METHODS

Materials

The used raw asphalt was Qayara crude asphalt obtained from Al-Qayara refinery Co., (Mosul City, Iraq). The crude asphalt has the following specification characterizations (**Table 1**).

The ground tires were obtained from the general company of Babylon Tire Industrial (Babylon City, Iraq) [13]. The residues from the ground tires were used in the recycled rubber process (RRR). Fluka Company (Switzerland) supplied the anhydrous aluminum chloride ($AlCl_3$). VWR Chemicals (USA) supplied the organic solvent n-hexane.

Table 1. Rheological properties of Qayara crude asphalt

Rheological properties	Measurement range	Mean
Elongation (cm, 25°C)	100+	100+
Softening Point (°C)	50-51	50.5
Penetration Index (PI)	(-1.400)-(-1.426)	-1.413
Permeability, mm (100g, 5s, 25°C)	72.6-74.7	73.7
Asphaltene Ratio (%)	18.3-18.7	18.5

Instrumentation

FTIR JASCO V-630, USA, did the FTIR measurement. The softening point test was done using a ring and ball device instrument, which measures the temperature at which the asphalt sample moves a distance of 2.54 cm when heated at a rate of 5 °C. The softening point of the asphalt was measured in the range between 30-200 °C. The measurement depends on US standards of ASTM (D36-95) [14]. A penetration test was done for solid and semi-solid asphalt materials, and their hardness was indicated using a penetrometer-type YUFENG model in China. The measurement depends on US standards of ASTM (D5/D5MM-13) [15]. Marshall test, which indicates the suitability of asphalt for paving, was done using WYKEHAM FARRANCE's electromechanical TRITECH machine. The measurement depends on US standards of ASTM (D1754-97R) [16]. The thin film oven test (TFOT) was done using TFOT oven model 710812, Japan. The test indicates the effect of aging conditions on asphalt sample [17]. The elongation test was done using a universal testing machine called YUFENG, China. The measurement depends on US standards of ASTM (D113-07) [18]. The blowing device was supplied by Dawson MacDonald Co. Inc. A US electrical shaker was also used by Humber Joe, Germany. Finally, a mixer for the treatment of RRR polymer-raw asphalt was used.

Air Oxidation of Asphalt

A specific weight of raw asphalt was taken and placed in the asphalt processing device. Air was passed over it using an air blower at a constant rate of 120cm^3/min. Air was blown for different durations ranging from 30 to 120 min and at temperatures between 100 and 200 °C. A catalyst of anhydrous aluminum chloride was added using the minimum weight percentage of its optimal catalyst ratio [19].

Determine the optimal catalyst ratio:

Different percentages of anhydrous aluminum chloride catalyst (0.25, 0.5, 1.0, 2.0, and 3.0) % (w/w) were added to determine the optimal percentage of the used catalyst. This stage was done after determining the optimal time and temperature for air oxidation.

Treatment of asphalt with RRR from waste tires:

The raw asphalt was treated with different percentages of residues of recycled rubber (RRR) extracted from waste tires, ranging from 0.5- 4.0% (w/w). The treatment process was done through an air oxidation process[20]. Optimal conditions depend on air oxidation at a temperature of 140 °C for 60 min, and an $AlCl_3$ catalyst is used with 0.25% (w/w).

Separation of asphalt:

The asphalt separation process has been performed for all prepared and original models. A 1.0 g asphalt sample was placed in a 250 ml beaker, and 40 ml of n-hexane was added. The solution was shaken for 3h using an electrical shaker at room temperature. The insoluble asphalt, which remained as sediment, was filtered. The isolated asphalt was washed with n-hexane until its washed solution became colorless. After drying the precipitate at room temperature, the percentage of the oxidized asphalt was calculated [14].

Determination of the rheological properties of the raw and modified asphalt:

The rheological properties of the raw and modified asphalt were determined, including elongation [18], softening point [14], penetration [15], Marshall test [16], and aging test [17] for selected samples.

3. RESULTS AND DISCUSION

Obtaining asphalt with new rheological properties that meet standard specifications, whether for use in paving, grading, or insulation, is crucial. In our study, the treatment of raw asphalt with an air oxidation process and by addition of RRR from waste tires, which is mainly carbon black with rubber residues produced as secondary remnants of rubber extraction processes from waste tires, and is added as the primary modifier for raw asphalt.

Characterization of the added RRR

The FTIR spectrum of RRR from waste tires (**Figure 1** and **Table 2**) shows the absorption frequencies of the remaining rubber's main functional groups at (2920, 2851, and 1456) cm^{-1}, representing the rubber's methylene groups. The band at 1376 cm^{-1} represents the methyl group of the rubber, whereas the peak at 1306cm^{-1} is the methane group of the rubber. The second influential absorption band is at 1582 cm^{-1}, representing the C=C stretching functional group of the carbon black. The third band is at 1148 cm^{-1}, which belongs to the C-O stretching vibration of the carbon black [21]. The FTIR of asphalt-modified RRR gives an almost straight line with no clear absorption peaks, and no functional groups appeared.

Table 2. FTIR adsorption frequencies of RRR additive

Sample	FTIR characteristic functional groups					
	$\gamma(C\text{-}H)_{str}$ methylene-	$\gamma(C\text{-}H)_{str}$ methyl-	$\gamma(C\text{-}H)_{str}$ methine-	$\gamma(C{=}C)_{str}$ trans	$\gamma(C\text{-}C)_{str}$ aliph.	$\gamma(C\text{-}O)_{str}$.
	Wave number υ/cm^{-1}					
RRR from waste tires	2920 2851 1456	1376	1306	963 1582	712	1148

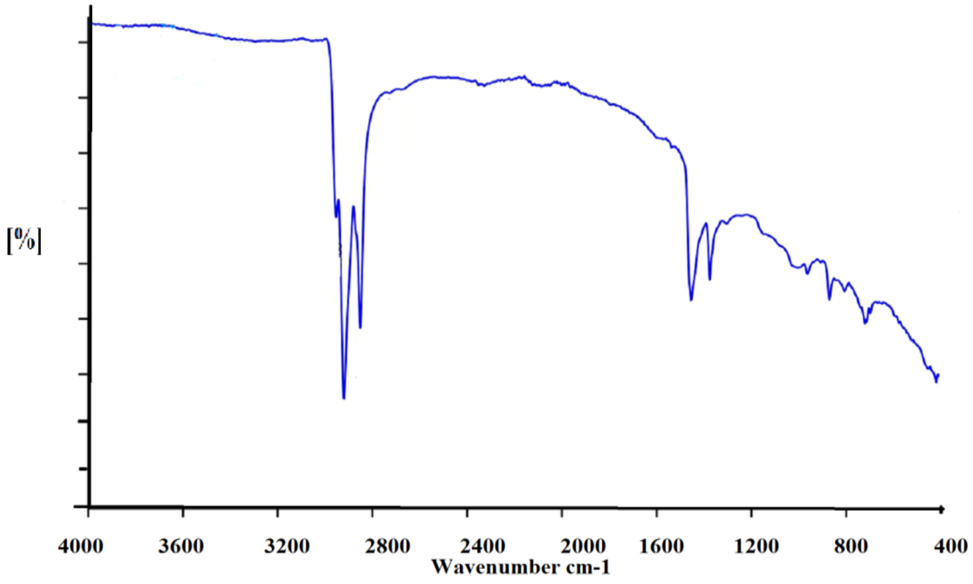

Figure 1. FTIR spectrum of RRR from waste tires

Modification of raw asphalt

Oxidation of asphalt

The oxidation process determined the optimal conditions for modifying asphalt materials. The rheological properties of the oxidized asphalt were studied at different temperatures and time intervals and in the presence of 0.25% (w/w)(**Table 3**).

Using different time intervals for the oxidation of raw asphalt samples and at different degrees of temperatures will produce oxidized asphalt that shows different rheological properties (**Table 3**). The asphalt sample N6 has shown good rheological properties (**Table 3**), including softening point and penetration depending on moderate conditions of air oxidation time (60 min) and at (140 °C) temperature. The asphalt ratio % increases with the increase in oxidation time to some extent, then decreases as time increases. The depression in asphalt ratio % occurs due to the condensing reactions and is then followed by dissociation reactions, which are activated as oxidation time is elevated. Further analysis and interpretation of rheological properties will be necessary to understand the full impact of these modifications on asphalt performance.

Table 3. Rheological properties of oxidized asphalt

Sample No.	Time (min)	Temperature (°C)	Ductility (cm,25°C)	Softening Point(°C)	Penetration (mm, 100g, 5s, 25°C)	Asphalt ratio (%)
N0	--------	---------	>100	50.5	42.6	18.5
N1	30	120	91	58.5	40.9	22.00
N2	60	120	>100	63.0	40.0	26.83
N3	90	120	>100	61.5	36.9	27.26
N4	120	120	71	67.0	36.7	31.31
N5	30	140	95	60.5	39.8	22.18
N6	60	140	>100	56.0	42.5	22.91
N7	90	140	>100	59.0	41.3	23.52
N8	120	140	80	63.5	39.8	25.94
N9	30	180	84	63.5	38.5	28.66
N10	60	180	78	63.0	37.8	28.27
N11	90	180	72	65.5	35.3	33.53
N12	120	180	58	68.5	35.0	38.00

N0: Raw asphalt model without any treatment

Catalyst ratio percent determination

Anhydrous aluminum chloride $AlCl_3$ catalyst was used to modify raw asphalt in different ratios %. Different rheological properties were examined on air-oxidized asphalt at 140 °C and for 60min, and their results appear in (**Table 4**).

Rheological properties analyses of air-oxidized asphalt samples at their optimal conditions using different ratio percentages of $AlCl_3$ catalyst have been conducted. It was observed that asphalt sample N14, which uses a catalyst ratio of 0.25% (w/w) (Table 4), is the best catalyst ratio. Its softening point is low (57.5°C) and shows high elongation (+100cm) and permeability (43.6 mm). Therefore, an $AlCl_3$ catalyst with a 0.25% (w/w) ratio can be used to modify asphalt rheological properties.

Asphalt modification by RRR from waste tires

The optimal conditions of the air oxidation process and catalyst ratio percent applied on raw asphalt samples were used to treat asphalt samples with different ratios of RRR obtained from waste tires. The rheological properties of the treated asphalt samples with different ratios of RRR were conducted, and their results are shown in (**Table 5** and **Figure 2**).

Table 4. Rheological properties of air oxidized asphalt samples treated with different catalyst ratios

Sample Number	Catalyst ratio %(w/w)	Softening Point (°C)	Elongation (cm, 25°C)	Permeability, mm (100g, 5s, 25°C)	Asphalt Ratio (%)
N0	0.00	50.5	+100	42.6	18.50
N*	0.00	66.5	89	35.8	27.18
N13	0.10	60.5	93	35.1	26.91
N14	0.25	57.5	+100	43.6	25.26
N15	0.50	58.5	+100	42.4	22.84
N16	0.75	62.5	81	38.6	25.91
N17	1.00	68.5	46	35.2	26.96

N0: Raw asphalt model without any treatment.
N*: Raw asphalt model without catalyst under optimal oxidation conditions.

Table 5. Rheological properties of air oxidized asphalt samples treated with different ratios of RRR from waste tires

Sample Number	RRR waste %(w/w)	Elongation (cm, 25°C)	Softening Point (°C)	Permeability, mm (100g, 5s, 25°C)	Asphalt Ratio (%)
N0	0.0	100+	50.5	42.6	18.50
N18	0.5	100+	56.5	44.1	19.61
N19	1.0	100+	58.0	43.9	20.43
N20	1.5	100+	58.5	42.5	22.52
N21	2.0	100+	59.5	42.4	25.87
N22	3.0	100+	59.0	40.0	26.23
N23	4.0	91	62.5	39.1	27.85
N24	5.0	83	64.0	35.9	28.91

N0: Raw asphalt model without any treatment.

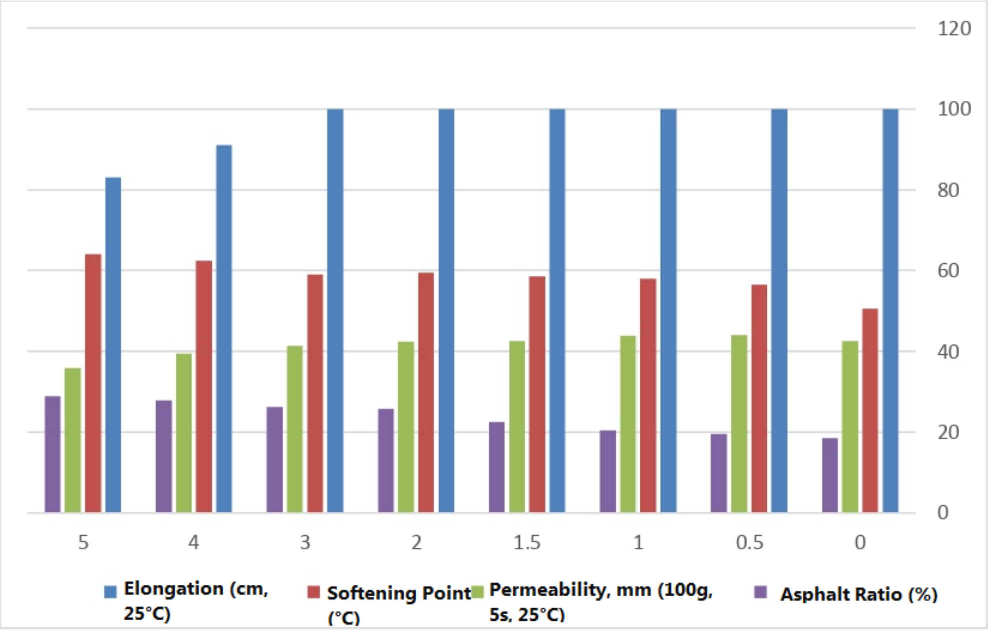

Figure 2. Effect of adding RRR from waste tires on asphalt rheological properties

The results (**Table 5** and **Figure 2**) show that the modification process of asphalt after mixing with RRR from waste tires alongside air oxidation will improve the rheological properties of the raw asphalt. The asphalt samples N18, N19, N20, N21, and N22 mixed with RRR up to an additive ratio of 3.0% (w/w) have shown (**Table 5**) low softening points and high elongation and permeability, which means RRR additive could be added in the ration of 0.5-3.0% (w/w). However, the previous results show improvements in the raw asphalt's rheological properties by adding RRR as external additives and oxidizing it by air. Moreover, both processes must be done on the raw asphalt, and this can be confirmed by repeating the previous modification of adding RRR in the ratio 0.5-3.0% (w/w) on raw asphalt without the air oxidation process, and the results of measuring the rheological properties are listed in (**Table 6**).

Table 6. Rheological properties of asphalt samples treated with different ratios of RRR from waste tires without air oxidation.

Sample Number	RRR waste %(w/w)	Elongation (cm, 25°C)	Permeability, mm (100g, 5s, 25°C)	Softening Point (°C)	Asphalt Ratio (%)
N25	0.5	100+	40.6	61.0	25.23
N26	1.0	91	38.9	62.5	27.15
N27	1.5	80	35.9	64.0	30.95
N28	2.0	63	33.2	68.0	35.14
N29	3.0	50	30.0	71.0	36.30

The rheological properties of asphalt samples measured without air oxidation (Table 6) show a decrease in values of most rheological properties, especially elongation values, which means air oxidation of asphalt is a critical process. It demonstrates the role of air oxidation in improving the rheological properties of the modified asphalt. Air oxidation is one of the most important processes used in organic reactions on an industrial and laboratory scale. Usually, air is used as a source of oxidation due to its ease of handling and economical availability, as air is one of the cheapest oxidizing agents. It only requires minimal costs associated with its handling. Furthermore, air oxidation leads to the following reactions [20].

$$\text{Oils} \xrightarrow{\text{air oxidation}} \text{Resins} \xrightarrow{\text{air oxidation}} \text{Asphalt}$$

Applications of modified asphalt

Referring to the results of the rheological properties in (**Table 5**), the asphalt modification, which consists of adding RRR from waste tires alongside air oxidation, was improved. The elongation values for the modified samples were within the standard specifications for paving asphalt, as outlined in (**Table 7**), up to an additive ratio of 3.0% (w/w), accompanied by excellent permeability and softening point values. However, at a ratio of 4.5% (w/w), there was a decrease in elongation values beyond the specified limits for paving asphalt. This variation in rheological properties when using waste is attributed to greater blending of the materials, thus increasing homogeneity between the two materials and positively affecting the rheological properties of the asphalt materials. Additionally, it is noted from the results (**Table 5**) that the best samples (N18, N19, N20, N21, and N22) are suitable for paving. The studied samples of modified asphalt show that some treated samples can be used as paving asphalt after undergoing engineering tests. It was also found that some other samples exhibited high softening point values and low permeability and elongation values (**Table 5**), qualifying them for use in producing mastics used as moisture barriers; other samples can be used as asphalt for flooring (**Table 7**), which shows the standard specification for asphalt used in producing mastics according to ASTM D491-88, as well as the Iraqi standard specification for asphalt used in paving, and the Iraqi standards for asphalt used in tiling according to the state cooperation of road and bridges (S.C.R.B).

Table 7. Standard specifications for asphalt used in mastic production [22], in paving [23], and in tiling according to S.C.R.B [24]

Rheological Measurement	For Mastic ASTM D491-88	For Paving S.S. No. 1196	For Tiling S.C.R.B sect. R9
Softening Point (°C)	54 - 65	57 - 66	51-60
Permeability, mm (100g, 5s, 25°C)	20 - 40	18 - 40	40-50
Elongation (cm, 25°C)	15 – no upper limit	10 – no upper limit	100 – no upper limit
Stability (KN)	------	------	7 – no upper limit
Creep (mm)	------	------	2-4

Marshall Test

The Marshall test is a popular and confirmed method for measuring the load and flow rate of tested asphalt samples. By applying pressure to sample [16], the test indicates how suitable the asphalt is for paving. When the sample begins to deform, stability and flow measurements are taken using the device's specific gradients. Stability refers to the ability of the asphalt mixture to resist deformation resulting from repeated transportation loads. It depends on internal friction and cohesion.

Conversely, flow measures the vertical deformation in the Marshall model at the point of failure. High flow values indicate that the asphalt mixture is more susceptible to permanent deformation, while low values suggest the presence of a few voids leading to initial cracking. The Marshall measurements (**Table 8**) show the stability and flow values for the best-modified asphalt samples obtained compared with the raw asphalt according to the standard specifications of the Iraqi Roads and Bridges Authority (S.C.R.B). It is evident from (Table 8) that the stability values for the raw asphalt were within the specifications of the Iraqi Roads and Bridges Authority as paving asphalt. However, the stability values for the modified asphalt (N18 and N19) samples were much better, indicating improved resistance to deformation.

Table 8. Illustrates stability and flow values for the raw and the best modified asphalt samples, with specifications of the Iraqi Roads and Bridges Authority (S.C.R.B)

Sample Number	RRR ratio%	Modified asphalt	
		Stability (KN)	Flow (mm)
N0	4.5	10.6	5.3
N18		15.4	3.2
N19		14.6	3.9
N23		9.8	5.4
N24		8.4	6.6
Standard Specifications of State Cooperation of Iraqi Roads and Bridges Authority (S.C.R.B)		7 The minimum	2-4

The flow value (**Table 8**) for the raw asphalt was outside the specifications of the Iraqi Roads and Bridges Authority as paving asphalt, explaining why roads paved with this asphalt without treatment experience rutting, leading to poor economic consequences. On the other hand, the flow values for modified asphalt samples were significantly lower, making them more resistant to rutting and indicating fewer voids in the mixture.

In conclusion, the modified asphalt samples are more suitable for paving purposes than the raw asphalt, as they meet the required specifications for stability and flow according to the Iraqi Roads and Bridges Authority. This reduces the susceptibility of paved roads to deformation and rutting, thereby increasing their stability and durability.

The modified samples (N23 and N24) show poor stability, and flow values (**Table 8**) are not satisfactory in terms of elongation, permeability, and softness (**Table 5**). They also did not meet the specifications of the Iraqi Roads and Bridges Authority (**Table 7**).

Aging test (thin film oven test (TFOT))

For studying the effects of the aging conditions on the modified asphalt samples, a furnace test [16,17] was conducted on thin asphalt membranes according to the ASTM D1754-97R using thin film oven test (TFOT) for the best examined samples (N18 and N19) and their results are presented in (**Table 9**).

The modified asphalt samples (**Table 9**) show that the degree of susceptibility to aging conditions, such as temperature and oxygen exposure, is generally low. It indicates that modified asphalt samples have high resistance to stress and fewer cracks, leading to a longer service life. The literature suggests that the lack of susceptibility of modified asphalt samples to aging conditions is attributed to the composition of polymer additives. These additives improve the mechanical properties of asphalt, such as strength, stress resistance, and resistance to thermal cracking, while increasing its resistance to rutting [25, 26].

Table 9. Rheological specifications of raw and modified asphalt samples measured before and after aging test (TFOT) with their difference

Sample Number	Rheological Properties	Before Test	After Test	Difference
N0	Elongation	+100	+100	
	Softness	50.5	53.5	3.5
	Permeability	42.6	40	2.6
	Weight loss percentage	------	0.06	
N18	Elongation	+100	+100	
	Softness	56.5	58.5	2.0
	Permeability	44.1	42.3	2.2
	Weight loss percentage	------	0.03	
N19	Elongation	+100	+100	
	Softness	58	60.5	2.5
	Permeability	43.9	40.5	3.4
	Weight loss percentage	------	0.04	

4. CONCLUSIONS

The study shows the rheological properties of Qayara crude asphalt, including softening point, elongation, penetration index, permeability, and asphalt ratio %, were improved after conducting air oxidation on the raw asphalt using 0.25% (w/w) anhydrous aluminum chloride at 140 °C and for 1 h. Moreover, Residual recycled rubber (RRR) from waste tires was added to the asphalt up to an additive ratio of 3.0% (w/w). The blend improves the rheological properties of the modified asphalt system. Some selected samples show better Marshall values than raw asphalt, indicating the potential use of this additive in paving operations that suit our country's environment and climate. The study demonstrated that modified asphalt samples with RRR from waste tires are less affected by aging conditions than raw asphalt. Finally, the study revealed that the asphalt ratio (%) generally increased with increased air oxidation time and the polymer additive ratio.

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