



# Self-Healing Properties of Recycled Asphalt from Waste Tire Rubber of Waste Oil Pyrolysis

Yankai Qin\*, Yiling Li

School of Civil Engineering, Chongqing University, Chongqing, 400045, China

\*1643644322@qq.com, ly1cqqu2023@163.com

**Abstract.** The waste cooking oil and waste engine oil were combined with waste tire rubber and subjected to pyrolysis to produce the regenerant. The Soluble fractions were separated and characterized through Soxhlet extraction. The self-healing properties of the asphalt were analyzed using a dynamic shear rheometer. The results indicate that both oils were effective in desulfurizing pyrolytic waste tire rubber. The two prepared regenerants can effectively enhance the self-healing properties of recycled asphalt. Furthermore, the waste engine oil-rubber mixture regenerant demonstrates superior self-healing properties.

**Keywords:** waste tire rubber, soluble fraction, asphalt, self-healing performance.

## 1 Introduction

Pavements can improve energy efficiency and serve as a means of transportation. However, asphalt pavements deteriorate over time [1]. The implementation of regeneration and self-repairing techniques can extend the service life of pavements [2]. During the process of asphalt reclamation, regenerant is a frequently used material that helps regulate the balance of components in aged asphalt and restore its performance.

Several researchers have developed recycling agents to enhance the pavement performance of aged asphalt using waste reuse methods. These methods include vegetable oils, waste cooking oils (WCO) [3], other renewable bio-oils, and waste engine oils (WEO) [4]. Furthermore, incorporating waste tire rubber (WTR) into asphalt enhances its rheology and aging resistance, while also mitigating the environmental impact of WTR by decreasing "black pollution". Dong et al [5, 6]. discovered that desulfurization of WTR mixed with WCO can increase the activity of waste rubber/oil rejuvenator (WROR) The WROR is added as a regenerant to improve the performance of aged asphalt. Therefore, blending WTR and waste oil to produce regenerant can provide a solution for both WTCR and WCO, while also turning waste into a valuable resource.

Although rubber-oil mixtures have potential benefits for recycled asphalt, there is limited research on their self-healing properties. To prepare the regenerants, two oils (WCO and WEO) were desulfurized and cracked with rubber powder. The soluble

fractions were separated using Soxhlet extraction. The self-healing properties of each asphalt material were analyzed and tested using the dynamic shear rheometer (DSR).

## 2 Materials and Test Methods

### 2.1 Materials

#### WTR, WCO, WEO, and Asphalt.

Table 1 shows the composition of the 40-mesh waste tire rubber (WTR) used in this study, while Table 2 displays the basic properties of waste cooking oil (WCO) and waste engine oil (WEO). The aged asphalt was produced by subjecting the 70# base asphalt to a membrane oven (TFOT) according to T0610-2011. Table 3 displays the fundamental properties of the asphalt bitumen material.

**Table 1.** Component of WTR.

Rubber hydrocarbon (wt. %)	Carbon black (wt.%)	Mineral filler (wt.%)	Operating oil (wt.%)
56.20	30.49	8.78	4.53

**Table 2.** The main contents of WCO and WEO.

	Flash point (°C)	Saturated fatty acid (wt.%)	Unsaturated fatty acid (wt.%)	light molecular oil (wt.%)
WCO	304	17.54	82.46	-
WEO	218	-	-	89.61

**Table 3.** Basic properties of asphalt.

	Softening point	25 °C Penetration	10 °C Ductility	135 °C Dynamic
70#A	48.0 °C	71.0 mm	62.1 cm	0.436 Pa·s
TFOT	57.2 °C	42.1 mm	8.1 cm	0.533 Pa·s

#### Preparation of Regeneration Agent.

According to Zhao [6], rubber was cracked using liquid phase media (WCO and WEO) at temperatures ranging from 180-260 °C. The WTR was mixed with the two waste oils. Waste cooking oil-rubber mixture and waste engine oil-rubber mixture (WCORM and WEORM) were produced by co-pyrolysis of the mixtures at 300 rpm for 0.5 h, 1 h and 2 h using a screw fan mixer.

#### Preparation of Recycled Asphalt.

This paper presents a study on the self-healing performance of recycled asphalt that was prepared with an optimal yield of 8% recycling agent. The determination of this optimal yield was based on previous experimental work. The aged asphalt was heated to 130 °C and 8% of WCORM and WEORM were added. WCORM and WEORM are

categorized according to solute content, totaling four (A, B, C, D). The asphalt mixture containing the waste rubber oil mixtures was then heated to  $160 \pm 1$  °C and stirred at 300 rpm for 1 h using a screw fan mixer. The corresponding resultant samples are presented in Table 4.

**Table 4.** The used asphalt samples.

Process design	Dose	Name	Penetration (0.1 mm)	Softening point (°C)
TFOT+ WCORM	8%	8%A-TFOT	69.0	48.8
		8%B-TFOT	61.0	50.5
TFOT+ WEORM	8%	8%C-TFOT	75.0	48.5
		8%D-TFOT	71.0	49.1

## 2.2 Test Methods

### Determination of Soluble Fraction.

Previous studies have shown that rubber with a high degree of desulphurization performs well as a regenerant and effectively solves the problem of storage stability [5]. Therefore, the optimal process parameters were determined through Soxhlet extraction combined with the sol fraction. The gum oil mixtures were enclosed in filter paper and underwent Soxhlet extraction with toluene as the extractant for 48 h. The remaining residue was then dried in a vacuum oven at 115 °C for 2 h before. The sol fraction ( $S_0$ ) of the gum-oil mixture was calculated using Eq. (1). For precision, three identical experiments were performed simultaneously for each sample and the average was calculated as the final result.

$$S_0 = \frac{(m_1 - m_2)}{(m_1 - m_0)} \times 100\% \quad (1)$$

Where:  $m_0$  represents the weight of the filter paper (g),  $m_1$  represents the initial weight of wrapped sample (g),  $m_2$  represents the weight of filter extraction residue (g).

### Characterisation of Self-Healing Properties of Asphalt Samples.

Fatigue-healing-fatigue cyclic loading tests were conducted on asphalt specimens using a DSR (ar1500ex Instrument, TA, USA). The strain from fatigue loading was 4.5%, the diameter of the parallel plate was 8 mm, healing temperature was 45 °C, holding time was 20 min, and the thickness of the asphalt film was 2 mm. Fatigue loading was stopped when the complex modulus decreased to 40% of the initial complex modulus. The healing index (HI) [7] was used to determine the level of self-healing for each asphalt material. The Eq. (2) was calculated as follows:

$$HI = \left( \frac{G_2 - G_1}{G_0 - G_1} \cdot \frac{T_1}{T_2} - 1 \right) \times 100\% \quad (2)$$

Where:  $G_0$  represents the initial complex modulus (MPa),  $G_1$  represents the modulus at the starting fatigue (MPa), and  $G_2$  represents the initial modulus for the second

stage (MPa).  $T_1$  and  $T_2$  represent the duration required from start to stop in the first and second stages, respectively (min).

### 3 Results and Discussion

#### 3.1 Sol Fraction

The results of the sol fraction of waste cooking oil-rubber mixtures and waste engine oil-rubber mixtures (WCORM and WEORM) sample means are presented in Fig. 1. The soluble fraction of both rubber-oil mixtures exhibits an increasing and then decreasing trend with increasing temperature and time. This is due to the desulphurization and cracking of WTR produces small molecules of soluble liquid-phase substances that increase the soluble fraction. As time passes, the rate of volatilization for small molecules surpasses the production rate, resulting in a decline. There is no significant difference in soluble content between 260 °C and 220 °C after 2 h for both rubber-oil mixtures. But a difference still exists between 180 °C and 260 °C. Therefore, based on the compatibility of the gum-oil mixtures with asphalt, it can be concluded that the optimal process parameters are 260 °C and a reaction time of 2 h. Two parameters were selected for WCORM (regenerant A and B) for the regeneration of aged asphalt: 260 °C-2 h and 180 °C-2 h. For comparative analysis, WEORM was selected as regenerant C and D using the same parameters.

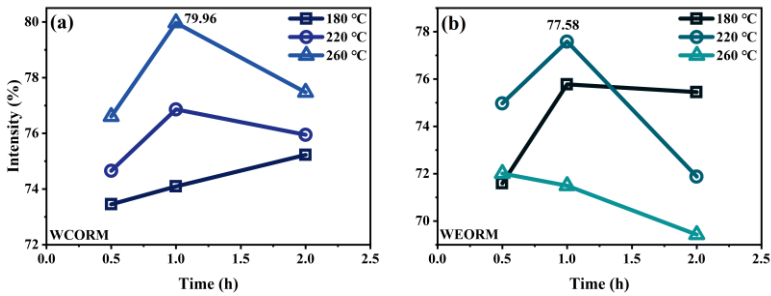


Fig. 1. Sol fraction of regenerants.

#### 3.2 Self-Healing Properties of WCORM Recycled Asphalt

The healing index (HI) is a critical indicator for assessing the self-healing properties of asphalt. HI measures the change in the dynamic shear modulus of asphalt before and after healing [7]. A lower HI index indicates better self-healing performance. Fig. 2 shows the self-healing capacity of the WCORM recycled asphalt and raw asphalt samples. Fig. 2a and Fig. 2b demonstrate that the HI index of WCORM reclaimed asphalt with different solubles content after addition of regenerants A and B is 8.68% for 8% A-TFOT and 12.60% for 8% B-TFOT. This indicates that both regenerants can improve the self-healing performance of recycled asphalt. The healing index of the 8% A-TFOT rubber-oil mixture exceeded that of the base asphalt. This suggests that the rubber-oil mixture had a high degree of rubber powder cracking after 2 h of high-temperature

cracking, while still maintaining a certain sol-gel content. The addition of the regenerant improved the fluidity and promoted the self-healing phenomenon of the asphalt.

### 3.3 Self-Healing Properties of WEORM Recycled Asphalt

Fig. 2 illustrates the self-healing capacity of the WEORM reclaimed asphalt and raw asphalt samples. As shown in Fig. 2e and Fig. 2f, the addition of regenerants C and D restores the performance of aged asphalt. The HI of WEORM reclaimed asphalt with 8% C-TFOT and 8% D-TFOT is 6.46% and 6.73%, respectively. Both regenerants improved the self-healing properties of the reclaimed asphalt. Furthermore, both materials exhibited a higher HI compared to the base asphalt and WCORM recycled asphalt. This may be due to the complex chemical composition and the presence of large amounts of light oil in WEO.

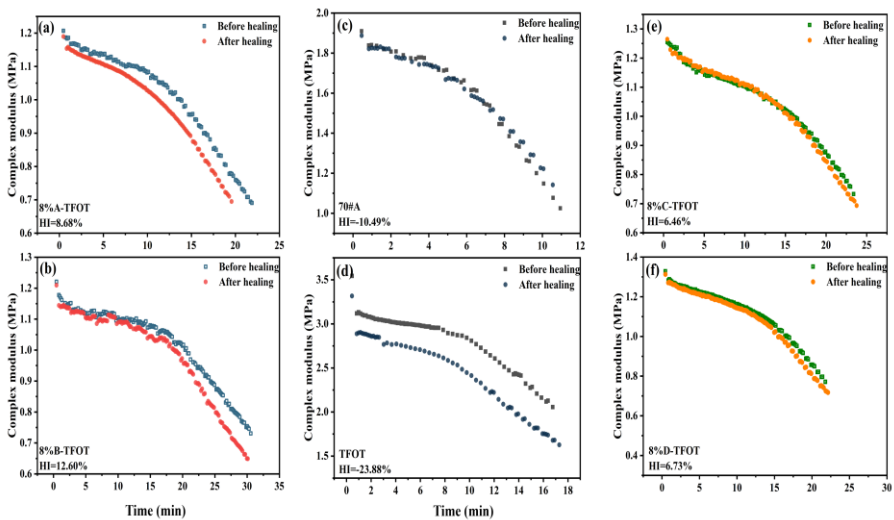


Fig. 2. Self-healing properties of WCORM and WEORM recycled asphalt with asphalt materials.

## 4 Conclusion

The study investigated the preparation of regenerants from two oils (WCO and WEO) blended with WTR, and the sol fraction was used for characterization. The self-healing properties of each asphalt sample were tested using DSR and evaluated using the HI.

Both WCO and WEO were effective in desulfurizing the pyrolysis waste tire rubber powder. Two liquid-phase media can achieve a high degree of rubber powder desulfurization and a high density of sol distribution at 260 °C for 2 hours.

WCORM regenerant can effectively enhance the self-healing performance of recycled asphalt. Treatment with regenerant at 260 °C-2 h can provide asphalt with even better self-healing performance.

WEORM rejuvenator can provide better self-healing ability. The strong self-healing performance at 180 °C-2h and 260 °C-2h may be attributed to the high concentration of light oil fraction in WEO and the high degree of desulfurization in WEORM.

Pre-treated waste rubber oil has potential as a reagent for asphalt regeneration. Further research should focus on the micro-mechanisms of interaction between waste rubber-oil mixtures and aggregates.

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