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Performance evaluation of rejuvenators in recycled asphalt mixtures based on mechanical and rheological properties

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The use of recycled materials in asphalt mixtures contributes to reducing costs and enhancing the longevity of road infrastructure. However, due to the presence of aged binder in reclaimed asphalt, additives known as rejuvenators are required to restore the lost properties of the binder. This study aims to evaluate the effects of various rejuvenators on the mechanical, rheological, and chemical properties of recycled asphalt mixtures to identify the most effective option for enhancing their durability and performance. Various tests were conducted, including Gel Permeation Chromatography (GPC), Bending Beam Rheometer (BBR), Dynamic Shear Rheometer (DSR), Multiple Stress Creep Recovery (MSCR), and Field Emission Scanning Electron Microscopy (FESEM), to assess the performance of different rejuvenators. The rejuvenators studied include gasoline-engine recycled motor oil with 1 oxidation degree (WEG1), gasoline-engine recycled motor oil with 3 oxidation degrees (WEG3), diesel-engine recycled motor oil with 1 oxidation degree (WED1), diesel-engine recycled motor oil with 3 oxidation degrees (WED3), recycled cooking oil (WCO), and Cyclohexane oil (CY). The BBR test results indicate that rejuvenators with lower oxidation levels and specific compositions, such as WEG1, WCO, and WED1, demonstrated better performance in maintaining stiffness and preventing cracking. Furthermore, the DSR results revealed that all the evaluated rejuvenator compositions exhibited satisfactory performance at high temperatures. The FESEM results also indicate that different rejuvenators can have varying effects on the surface structure and material behavior. Also, the MSCR results show that the best performances are observed among the WEG3 and WED2 samples. Additionally, a Multi-Criteria Decision-Making (MCDM) method was used to select the optimal rejuvenator based on test results. The results showed that WEG3 exhibited the best overall performance in improving the mechanical and rheological properties of recycled asphalt mixtures. As a conclusion, this study showed that rejuvenators, especially WEG3, significantly improve the mechanical, chemical, and thermal properties of reclaimed asphalt mixtures. The results highlight the potential of recycled motor oil-based rejuvenators to enhance durability and support environmental sustainability.

Keywords Recycled asphalt, Rejuvenator, Thermal stability, Multi-criteria decision-making (MCDM), Mechanical properties of asphalt mixtures

In recent years, greater attention to environmental issues and the depletion of natural resources has driven various industries toward using recycled materials and optimizing production processes. One key sector in this regard is road construction and asphalt production. Given the widespread need for construction and continuous road maintenance, recycled asphalt has emerged as a viable solution to reduce the consumption of natural resources and lower operational costs. Recycled asphalt is produced by reprocessing old asphalt mixtures and adding rejuvenators to restore their mechanical and functional properties. Rejuvenators play a crucial role in restoring the primary characteristics of asphalt and improving its durability and flexibility. However, selecting the appropriate type and amount of rejuvenator remains a primary challenge in this process. Factors such as chemical composition, mechanical behavior, and cost must be carefully evaluated to choose the best option.

In addition to cost-saving benefits, recycled asphalt significantly contributes to environmental sustainability. For instance, studies have shown that utilizing recycled asphalt can reduce the demand for virgin bitumen by up

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to 30% and decrease greenhouse gas emissions associated with asphalt production processes by approximately 25%. These environmental benefits make recycled asphalt an essential component in the pursuit of sustainable infrastructure development.

Reclaimed asphalt pavement (RAP) has drawn considerable attention from researchers in recent years. The use of this material in hot asphalt production has increased significantly, with many researchers incorporating high amounts of asphalt chips into hot asphalt mixtures. Utilizing asphalt chips reduces the life cycle costs of asphalt mixtures. Additionally, these chips contribute to the reuse of aggregates and bitumen, playing a significant role in preserving natural resources and promoting sustainable development, while also reducing project costs.

Rejuvenators are substances applied to restore the chemical and performance properties of asphalt mixtures. They are effective because they mitigate the hardening effects and changes in aged asphalt's chemical composition. Chemically, rejuvenators primarily consist of maltenes and aromatic compounds, which replenish the maltenes lost from aged asphalt and restore much of the bitumen's original characteristics. Aromatic compounds improve the rejuvenator's compatibility with asphaltenes in bitumen, ensuring uniform distribution within the mixture¹⁻⁴.

Choosing an appropriate rejuvenator is crucial to ensure that both early-age and long-term performance requirements of recycled asphalt mixtures are met⁵. In the short term, the additive must be capable of quickly interacting with and softening the aged binder present in the RAP to promote effective blending and uniform coating of aggregates⁶. This uniformity helps maintain adequate surface texture and reduces the risk of rutting. However, achieving full and homogeneous diffusion of the rejuvenator throughout the aged binder remains a technical challenge, often leading to localized variations in performance within the mix. As outlined in previous conceptual models⁷, the diffusion of a rejuvenator into the aged asphalt binder generally follows a multi-phase process: initially forming a thin low-viscosity layer on the surface of RAP-coated aggregates; subsequently penetrating the aged binder; gradually reducing the viscosity across binder layers; and finally reaching a state of equilibrium over time as the material stabilizes.

Used motor oil is one product that can serve as a rejuvenator. While the use of recycled motor oils as rejuvenators offers significant benefits in terms of sustainability and waste reduction, potential environmental concerns must be carefully considered. Used motor oils may contain trace amounts of heavy metals and other contaminants that could pose risks of leaching into the soil and groundwater if not properly managed. Therefore, thorough chemical characterization and treatment of these materials are essential before application in asphalt mixtures. Additionally, long-term monitoring and leaching tests should be conducted to ensure that the use of recycled motor oils does not adversely affect environmental quality. Implementing such precautions will help balance the environmental advantages of recycling with the need to protect ecosystems and public health. This oil is used in vehicle engines to regulate temperature and prevent oxidation of metallic surfaces. Over time, the motor oil absorbs impurities like heavy metals and soot (various hydrocarbons) and degrades into used oil, which needs to be replaced. Motor oil is subjected to oxidation and thermal degradation, reducing its quality over time. Additionally, the standard additives, such as detergents, gradually deplete and lose effectiveness. Used motor oil has been identified as a potential rejuvenator in many studies for restoring properties in recycled asphalt mixtures^{8,9}. Several researchers^{10,11} have investigated various rejuvenator types—ranging from waste-derived oils and vegetable-based compounds to synthetic additives—to restore aged asphalt's properties. For example, used motor oil (UMO) has gained significant attention due to its abundance, cost-effectiveness, and chemical similarity to the maltenes fraction of bitumen. Studies have shown that when properly processed and blended, UMO can enhance the rheological and mechanical properties of asphalt binders¹². Other research¹³ efforts have focused on combining UMO with agents like rubber powder, plasticizers, or anti-aging additives to improve performance characteristics and durability of recycled mixtures.

Lou et al. (2021) demonstrated that UMOs with high aromatic content could significantly improve the chemical properties of recycled asphalt mixtures¹⁴. Similarly, Guo et al. (2021) developed a new rejuvenating agent by combining used motor oil, plasticizer, and anti-aging additives, which showed promising results for restoring aged asphalt's properties¹⁵. Ziari et al.¹⁶ explored crack behavior in asphalt mixtures containing varying amounts of reclaimed asphalt and fiberglass. Their study assessed mixtures with 0%, 25%, 50%, 75%, and 100% reclaimed asphalt chips and different amounts of fiberglass, using 6% of the rejuvenator Cyclogen. The optimal amount of rejuvenator was determined based on bitumen performance grades. The semi-circular bending (SCB) test evaluated crack resistance at different temperatures (-15°C , 0°C , and 15°C), showing that fiberglass significantly increased fracture energy at all temperatures. Li et al.¹⁷ investigated the effects of recycled edible oil and used motor oil on the properties of asphalt mixtures containing reclaimed asphalt. Their study used Thin Film Oven Test (TFOT) to age bitumen and produced aged asphalt mixtures over various time periods (5, 7, 9, 11, 13, and 15 h). Rejuvenator amounts ranged from 1% to 4%, and physical and chemical properties were evaluated, along with rutting, Marshall strength, and moisture susceptibility tests. Luo et al.¹⁴ introduced a new rejuvenator combining used edible oil and rubber powder. They mixed rubber powder in a 1:4 ratio with used edible oil at 260°C . To assess its performance, 10% of this rejuvenator was mixed with aged bitumen (RTFO + PAV), and PG and FTIR tests were performed. The results demonstrated that this combination was effective in rejuvenating bitumen. Rai et al.¹⁸ investigated the long-term aging effects on rejuvenator properties. Pure bitumen (PG64-22) was aged in the lab using RTFO and PAV, and then mixed with three different commercial rejuvenators at 3%, 6%, and 9% concentrations. The samples were subjected to PAV aging for 5, 10, 15, 20, 40, and 60 h, and Temperature-frequency sweep tests were conducted on the prepared samples. Moniri et al.¹⁹ assessed vegetable- and petroleum-based rejuvenators in asphalt mixtures containing 0% to 100% reclaimed asphalt, showing that fatigue life improved significantly with rejuvenators and that up to 50% reclaimed asphalt could be used without reducing quality. Wang et al.²⁰ provided insight into the diffusion mechanism of rejuvenators and their effect on rheological properties. The study highlighted how rejuvenators could penetrate aged asphalt, improve crack resistance, and optimize asphalt's mechanical behavior through tests such as DSR and BBR. Ziari et al.²¹ evaluated the effectiveness of different rejuvenators in warm-mix asphalt containing high levels of

reclaimed asphalt, emphasizing both mechanical and chemical properties. Increasing RAP content improved rutting resistance and permanent deformation, while rejuvenators like VB and Cyclogen reduced tensile strength, resilient modulus, and strain creep. Truong et al.²² examined the effects of rejuvenators on cracking resistance in high-RAP asphalt mixtures, providing critical insights into how rejuvenators improve performance under various environmental conditions. Kuang et al.²³ analyzed rejuvenators' impact on aged asphalt's performance and microstructure, highlighting their role in restoring flexibility and durability, which are essential for long-term pavement performance. He et al.²⁴ studied the regeneration effects of bio-based rejuvenators for warm-mix asphalt. This research demonstrated how rejuvenators could restore conventional properties of aged asphalt, improving its microstructure and overall performance through tests like DSR and FTIR. Aeron et al.²⁵ examined the effect of optimal dosages of waste engine oil (WEO) and tall oil (TO) rejuvenators on the performance of recycled asphalt binder. The results indicated that the use of 19% WEO and 17% TO significantly enhanced the chemical and rheological properties of the recycled binder, improving its resistance to rutting and fatigue. Ali et al.²⁶ evaluated the mechanical performance of asphalt mixtures containing RAP rejuvenated with WEO. Their findings demonstrated that adding WEO to recycled asphalt mixtures improved Marshall stability by up to 30%, indirect tensile strength by 29%, and moisture resistance by 19%. Deef-Allah et al.²⁷ investigated the impact of using UMO as a rejuvenator in asphalt mixtures. They showed that combining UMO with CRM increased rutting resistance and improved the rheological properties of the asphalt binder.

Although the use of rejuvenators in recycled asphalt mixtures has been extensively studied, several critical aspects remain underexplored. Most previous research has focused on the general performance of rejuvenated asphalt without addressing the variability in rejuvenator source, chemical composition, and oxidation level—factors that significantly influence the final mixture's performance. In particular, the performance differences between rejuvenators derived from gasoline versus diesel engines, or those with varying degrees of oxidative aging, have not been systematically evaluated. Furthermore, limited studies have investigated the influence of moisture content and microstructural effects of rejuvenators on the aged binder. There is also a lack of comparative studies using advanced analytical methods such as GPC and FESEM to correlate molecular-level restoration with macroscopic performance outcomes. These gaps limit the ability to make informed decisions about rejuvenator selection, especially in regions such as Iran where the availability and characteristics of waste oils vary considerably.

To address the identified research gaps, this study investigates the fatigue and cracking behavior of aged bitumen modified with various percentages of recycled and conventional rejuvenators commonly used in Iran. Special attention is given to the type of used motor oil (gasoline- vs. diesel-engine derived), oxidation level (one vs. three degrees), and moisture content as influential variables affecting binder performance. A comprehensive set of laboratory tests—including Gel Permeation Chromatography (GPC), Bending Beam Rheometer (BBR), Dynamic Shear Rheometer (DSR), Multiple Stress Creep Recovery (MSCR), and Field Emission Scanning Electron Microscopy (FESEM)—is used to assess mechanical, rheological, and chemical properties. Furthermore, a multi-criteria decision-making (MCDM) method is applied to identify the most effective rejuvenator for improving the durability and sustainability of recycled asphalt mixtures.

Materials and methods

A total of samples were prepared as shown in Table 1, including unaged (BASE), aged (PAV), and rejuvenated binders. For each rejuvenated sample, 10% by weight of rejuvenator was added to the twice-PAV-aged binder (i.e., 20 g rejuvenator per 200 g aged binder), and mixed at 160 °C for 30 min to ensure homogeneous dispersion. The 10% rejuvenator content was selected based on preliminary trial-and-error testing and supported by findings in previous literature²⁸, aiming to achieve a balance between viscosity reduction and performance recovery without compromising binder durability.

The rejuvenators were derived from waste materials, including used gasoline and diesel engine oils (subjected to 0, 1, and 3 h of heating to simulate oxidation), recycled edible oil from food industries, and a conventional petroleum-based rejuvenator (Cyclogen). Table 1 summarizes the oxidation levels and descriptions for each rejuvenator.

Laboratory samples were prepared by combining aged bitumen with various rejuvenators in fixed proportions. The base binder, PG 64–16, was aged in two stages:

Sample Name	Description	Oxidation degree	Penetration (0.1 mm)	Softening Point (°C)	Viscosity at 135 °C (Pa·s)
BASE	PG 64–16	-	65	48	0.45
PAV	Aged asphalt binder with PAV	-	30	58	1.2
WEG1	Gasoline engine recycled motor oil (no heating)	1	45	53	0.8
WED1	Diesel engine recycled motor oil (no heating)	1	47	52	0.78
WEG2	Gasoline engine recycled motor oil (1 h heating)	2	42	54	0.85
WED2	Diesel engine recycled motor oil (1 h heating)	2	43	54	0.82
WEG3	Gasoline engine recycled motor oil (3 h heating)	3	40	55	0.9
WED3	Diesel engine recycled motor oil (3 h heating)	3	39	56	0.88
WCO	Waste Cooking Oil	-	50	51	0.75
CY	Cyclogen oil (petroleum-based rejuvenator)	-	48	52	0.76

Table 1. Samples examined in this Study.

- Short-term aging was simulated using a Rolling Thin Film Oven (RTFO) according to ASTM D2872, at 163 °C for 85 min, replicating aging during asphalt production and placement.
- Long-term aging was performed using a Pressure Aging Vessel (PAV) at 100 °C and 2.1 MPa for 20 h, in accordance with ASTM D6521. To intensify the aging effect, the PAV process was repeated twice, effectively simulating 5–10 years of field aging.

The results of conventional physical tests, including penetration, softening point, and viscosity at 135 °C, conducted on the base binder, aged binder, and rejuvenated samples, are summarized in Table 1.

Sphalt binder

The asphalt binder used in this study is PG 64 – 16, which is classified for weather conditions within a temperature range of 64 °C to -16 °C. This type of binder is widely used in road construction projects due to its high resistance to deformation and cracking.

Rejuvenators

Three types of rejuvenators were used in this study to enhance the performance of the asphalt samples. These include recycled diesel engine oil, recycled cooking oil, and a commercial rejuvenator (CY):

- Recycled diesel engine oils were selected as rejuvenators due to their chemical composition, particularly the presence of aromatic and maltene fractions, which contribute to the softening of aged bitumen. To simulate varying oxidation levels, the oils were subjected to controlled heating for 0, 1, and 3 h over an open flame. These thermal treatments were used to produce three levels of oxidation, referred to as levels 1, 2, and 3, respectively. Higher oxidation typically increases the oil's hardness and improves the mechanical resistance of the asphalt binder.
- Recycled Cooking Oil: This rejuvenator was selected because of its low cost and easy availability, making it a potential candidate for use in recycled asphalt mixtures.
- Commercial Rejuvenator (CY): CY is a commercially available rejuvenator specifically designed to enhance the mechanical properties of recycled asphalt binders. It features controlled viscosity and resistance to oxidation, making it ideal for use in sensitive pavement projects.

Small beam bending test

This test evaluates the behavior of asphalt binders at low temperatures and assesses their resistance to cracking. Asphalt binder samples are subjected to temperatures of -12 °C and -18 °C, and their bending behavior is examined. The results of this test indicate the flexural modulus and resistance to cracking, which are used to determine the performance of the binder at low temperatures.

For the test, the asphalt binder samples are prepared as small beams (oval or rectangular) with standard dimensions²⁹. The samples are then placed in a Bending Beam Rheometer (BBR). During the test, the samples are subjected to a constant load, and their deflection over time is measured. The measured parameters include the flexural modulus (the binder's resistance to bending deformation), the creep stiffness (measuring the binder's deformation over time under a constant load), and the creep recovery rate (the speed at which the binder returns to its original state after the load is removed)³⁰.

Dynamic shear rheometer (DSR)

The Dynamic Shear Rheometer (DSR) test is used to evaluate the viscoelastic properties of asphalt binders at high and medium temperatures. In this test, asphalt binder samples are subjected to dynamic stress at temperatures of 64 °C and 76 °C to measure their resistance to plastic deformation. These test temperatures were selected based on the performance grade of the base binder (PG 64 – 16) and to assess the behavior of the rejuvenated binders under elevated service temperatures relevant to field conditions. The results provide the shear modulus and phase angle of the asphalt binder, which are essential for assessing its rutting resistance and high-temperature cracking performance^{31,32}.

For this test, asphalt binder samples were tested using an Anton Paar MCR 302 Dynamic Shear Rheometer. The samples were placed as discs between two 25 mm parallel plates with a gap setting of 1 mm, following the standard procedure described in AASHTO T315. The device applies dynamic shear stress to the samples at controlled temperatures, and their viscoelastic response is measured accordingly. The samples are exposed to continuous dynamic strain and shear stress, and the amount of deformation is recorded.

The DSR device uses parallel plates capable of inducing strain and measuring stress at controlled temperatures. It is primarily used to evaluate the behavior of asphalt binders under repeated deformation and alternating stress conditions. The key parameters measured in this test are as follows³³:

- Shear modulus (G): Indicates the binder's resistance to deformation.
- Phase angle (δ): Reflects the viscoelastic behavior of the binder, where $\delta=0$ indicates purely elastic behavior and $\delta=90$ indicates purely viscous behavior.
- Performance temperature range: The range in which the binder performs adequately.

Field emission scanning electron microscope (FESEM)

The FESEM test examines the surface structure of rejuvenated asphalt binders and the distribution of rejuvenator particles in the asphalt mixture. This high-resolution imaging technique provides detailed visuals of the asphalt surface, offering valuable insights into how rejuvenators affect the binder's microscopic structure³⁴. The prepared asphalt binder samples are placed in the Field Emission Scanning Electron Microscope (FESEM). The device uses

electron beams to scan the sample's surface, producing high-resolution images of the surface structure. FESEM has the capability to observe nanoscale details, aiding in the identification of the structure and distribution of rejuvenator particles in the asphalt. The FESEM parameters are as follows³⁵:

- Surface characteristics and particle distribution: Determines the uniform distribution of rejuvenators in the asphalt binder.
- Particle size and morphology: Examines surface structure and changes in particle size.

Gel permeation chromatography (GPC)

Gel Permeation Chromatography (GPC) is used to measure the molecular weight distribution of asphalt binders and rejuvenators. Samples are analyzed at both ambient and elevated temperatures to evaluate molecular distribution changes in the binders³⁶.

The asphalt binder samples are dissolved in suitable solvents and then passed through the columns of the Gel Permeation Chromatography (GPC) device. In this method, the asphalt molecules pass through the column based on their molecular weight; larger molecules elute earlier, while smaller molecules elute later. The data directly reflect the molecular weight distribution of the binder. The GPC parameters are as follows:

- Molecular weight distribution: Measures the quantities of compounds with different molecular weights.
- Maltene-to-asphaltene ratio: Indicates the rheological properties of the binder.

Multiple stress creep recovery (MSCR) test

The MSCR (Multiple Stress Creep Recovery) test assesses the behavior of asphalt binders under repeated loading at high temperatures. This test is used to evaluate the binder's resistance to rutting by measuring its ability to recover after deformation. In the MSCR test, binder samples are subjected to repeated loading at 64 °C and 70 °C, temperatures selected to represent high-temperature field conditions. During testing, each sample is exposed to two stress levels: 0.1 kPa and 3.2 kPa, with 10 cycles applied at each stress level, in accordance with AASHTO T350. Each cycle consists of 1 s of loading followed by 9 s of recovery. The viscoelastic response is recorded after each cycle. The following parameters are derived from the MSCR test³³:

- Creep recovery (R-value): Measures the recovery to the original state after loading.
- Permanent deformation (Jnr): Measures the amount of permanent deformation after removing the load.

These parameters are critical for evaluating the rutting resistance and elastic behavior of asphalt binders under traffic-like conditions.

Results and discussion

This section presents and analyzes the results of the experiments conducted in this research. The primary objective of this chapter is to examine the mechanical and chemical performance of reclaimed asphalt mixtures using various rejuvenators. Several tests were carried out to evaluate different properties of these mixtures. First, the results of each test are presented in tables and graphs, followed by professional and detailed analyses of the performance of each rejuvenator under various experimental conditions. The final goal is to present scientific data and conclusions that will allow the selection of the optimal rejuvenator, which will eventually be done using the Multi-Criteria Decision-Making (MCDM) method. The results from different tests are assessed according to defined criteria, and this data is then analyzed comprehensively to identify the best rejuvenator for reclaimed asphalt mixtures.

Figure 1 illustrates the results of the Bending Beam Rheometer (BBR) test, including the measured stiffness (S) and M-value for the base binder and rejuvenated binders. These parameters are critical for evaluating the low-temperature cracking resistance of asphalt binders.

All binders tested exhibit stiffness values well below the 300 MPa threshold and M-values above the minimum limit of 0.300, as recommended by AASHTO M320. This indicates that all samples meet the basic performance requirements for low-temperature applications. Among the samples, WCO (waste cooking oil) demonstrates the most favorable behavior, showing the lowest stiffness and highest M-value, indicating excellent flexibility and superior resistance to thermal cracking. The rejuvenated binders with engine oil derivatives, particularly WEG1, WED1, and WED3, also show relatively low stiffness and acceptable M-values, suggesting effective restoration of low-temperature performance. WED2, while still meeting the standard criteria, shows a notably higher stiffness value compared to the other rejuvenated samples, which may indicate a slightly stiffer response and reduced flexibility at low temperatures. The base binder (PG 64–16) shows moderate stiffness and a relatively high M-value, and Cyclogen (CY) performs similarly to the base, confirming its typical rejuvenating behavior. Overall, the results in Fig. 1 suggest that all rejuvenators contributed to improving the low-temperature performance of the aged binder to varying extents, with WCO emerging as the most effective rejuvenator in this regard.

Figure 2 illustrates the stiffness values measured for various asphalt binder samples under three levels of deflection (8, 15, and 30 mm). A consistent decreasing trend in stiffness is observed as the deflection increases, which aligns with the known nonlinear viscoelastic behavior of asphalt binders. As deformation levels rise, the internal structure of the binder becomes more mobilized, resulting in a softer response and reduced ability to resist applied loads. This phenomenon is expected under real-world pavement conditions, where increased traffic loading leads to a decline in effective stiffness and greater permanent deformation. Among the samples evaluated, the WED2 sample—modified with diesel-engine recycled motor oil oxidized for 1 h—exhibited a substantial reduction in stiffness across all deflection levels. This sharp decline is likely due to the thermal degradation of key chemical components during the oxidation process. Specifically, the 1-hour oxidation may

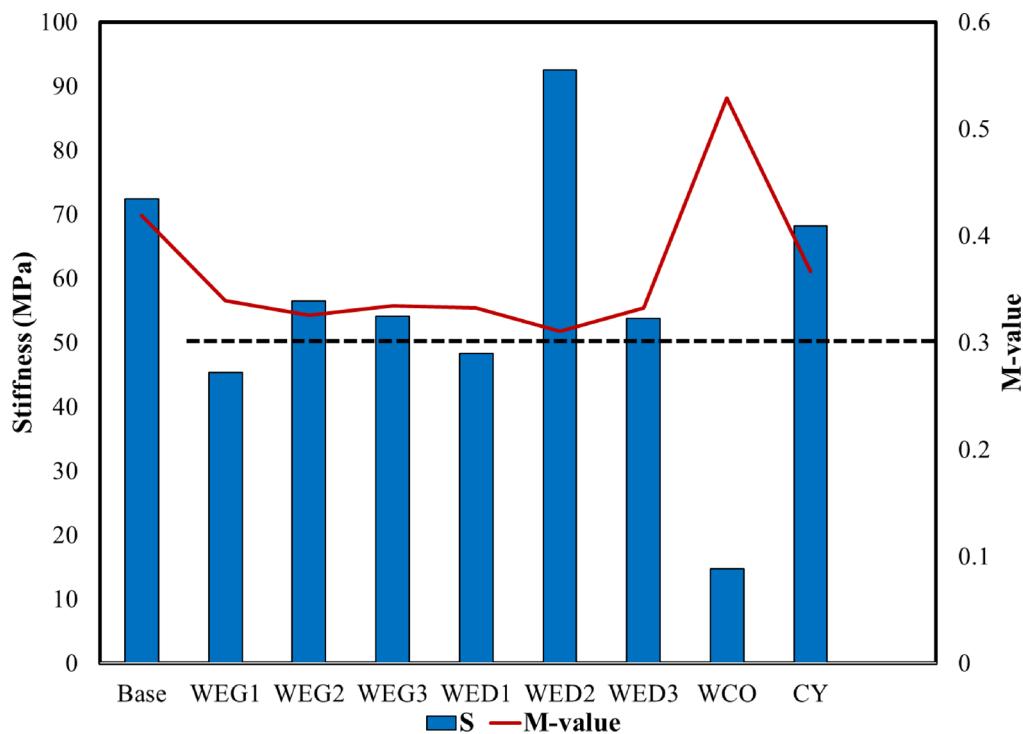


Fig. 1. Stiffness and M-value results of asphalt binders obtained from the BBR test.

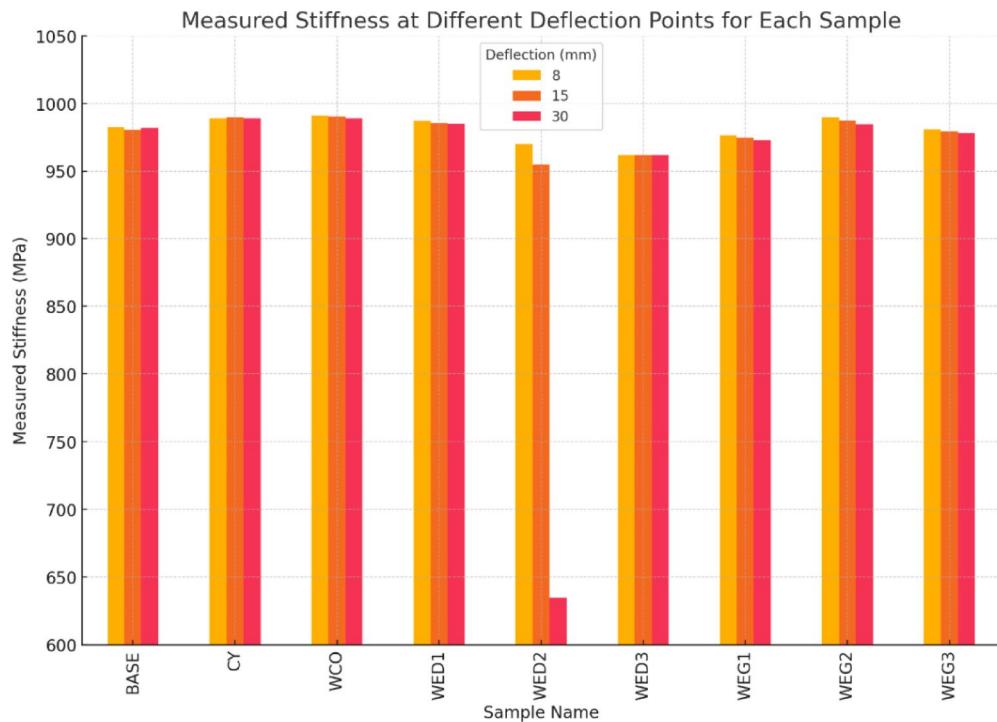


Fig. 2. Measured Stiffness for Different Samples at Deformation Points.

have broken down essential polar compounds in the rejuvenator, reducing its effectiveness in restoring the structural integrity of the aged binder. In contrast, WED1 (no heating) preserved these reactive components, while WED3 (3 h of oxidation) may have undergone further chemical stabilization, enhancing its compatibility with the binder and resulting in improved stiffness. These results suggest the existence of a critical oxidation

threshold, where intermediate heating (such as 1 h) can be detrimental, while either no heating or prolonged heating may yield more favorable mechanical performance.

According to BBR results, binders modified with WED1 and WEG1 (gasoline-engine recycled oil, no heating) exhibited higher stiffness and more favorable m -values, indicating better low-temperature performance and resistance to thermal cracking. In contrast, WED2 also showed significantly lower stiffness in the BBR test, consistent with its poor performance in Fig. 2 under increasing deformation levels. This confirms that rejuvenators with lower oxidation levels or preserved chemical functionality are more effective in improving both the high- and low-temperature behavior of aged binders.

In Fig. 3, the results of the DSR (Dynamic Shear Rheometer) test for all the studied composition samples are presented. As observed, all the samples show a linear and upward trend in $G/\sin(\delta)^*$ values and complex shear modulus. This indicates that as the complex shear modulus increases, the $G/\sin(\delta)^*$ value increases similarly for all samples. The linear and upward trends observed in Fig. 3 between the Complex Shear Modulus (X-axis) and $G^*/\sin(\delta)$ (Y-axis) reflect the intrinsic viscoelastic behavior of asphalt binders under dynamic loading. The complex shear modulus represents the total resistance of the binder to deformation under oscillatory shear, combining both elastic and viscous components. Meanwhile, $G^*/\sin(\delta)^*$ is a parameter often used as an indicator of rutting resistance, with higher values correlating to greater elastic response. As the complex shear modulus increases, indicating a stiffer binder, the value of $G^*/\sin(\delta)$ also increases because the binder exhibits more elastic behavior and improved resistance to permanent deformation. The approximately linear relationship arises because both parameters are derived from the same dynamic mechanical data and are inherently related through the phase angle δ , reflecting changes in binder microstructure due to aging or rejuvenation. Additionally, test conditions such as temperature and frequency influence binder stiffness and phase angle consistently, contributing to the observed monotonic increase. This behavior aligns with the polymeric and colloidal nature of asphalt binders, where molecular interactions govern viscoelastic properties and performance.

The graph does not display significant differences between the various samples, suggesting that most of the examined compositions exhibit similar behavior under shear deformation and high temperatures. The WEG1 and WED1 samples perform slightly better compared to other samples, showing higher $G^*/\sin(\delta)$ values at higher complex shear moduli. This implies that these samples have greater resistance to deformation.

The results show that all the studied compositions perform well at high temperatures, exhibiting good resistance to rutting. Samples containing the rejuvenators WEG1 and WED1 may be better options for use in higher temperature conditions.

The results of the samples studied in the present research are presented in Fig. 4. All images exhibit very good surface resolution. These FESEM images are used to examine the microstructures and particle distribution of various rejuvenators within the recycled bitumen. In some samples, such as WED1 and WEG2, particles are well

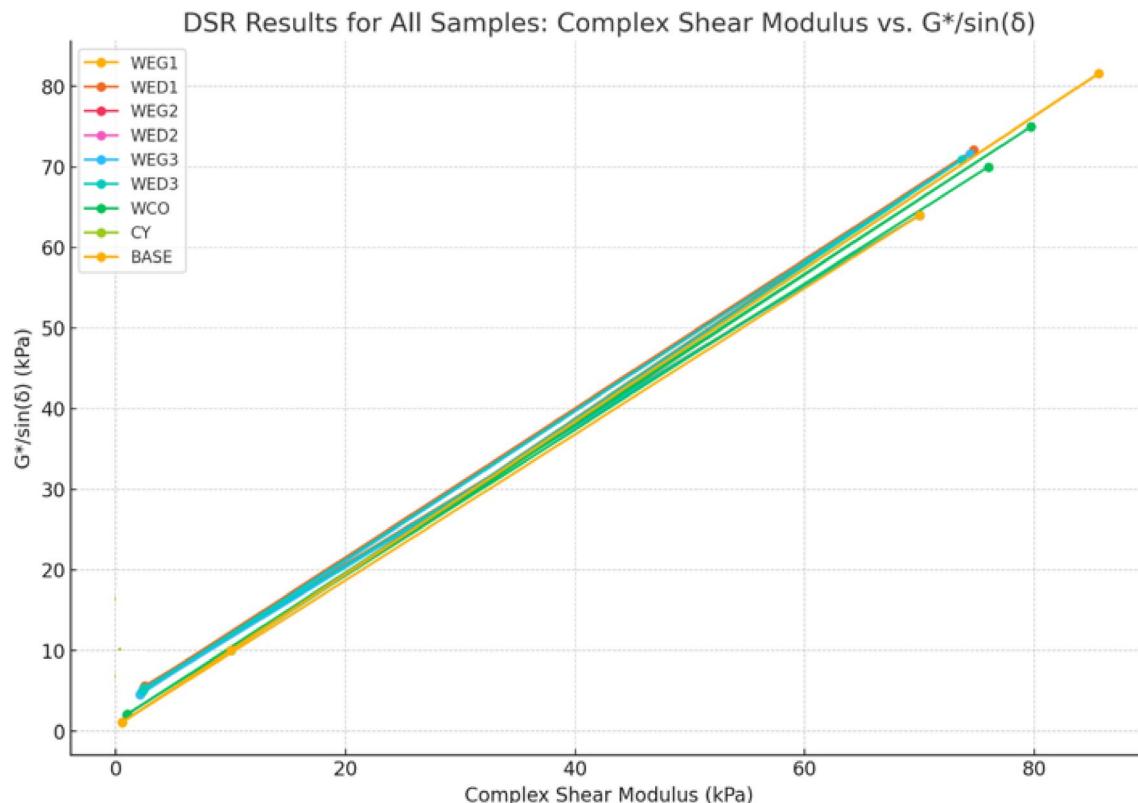


Fig. 3. : DSR Test Results for All Tested Samples.

dispersed, indicating a uniform distribution of the rejuvenator in the binder. This uniform dispersion reflects the ability of the rejuvenators to improve the rheological behavior and enhance the adhesive properties of the binder.

The surface structure varies among different samples. For instance, in the WCO (recycled edible oil) image, a softer and more uniform surface is observed, which may be attributed to the rejuvenating properties of this type of oil. In contrast, the CY, BASE (base binder without rejuvenator), and BASE2 samples display surfaces with larger and more heterogeneous particles. This heterogeneity suggests greater brittleness compared to samples containing rejuvenators.

Samples with different oxidation levels, such as WEG1 and WED3, exhibit varying microstructures. Higher oxidation degrees may lead to the formation of harder and more brittle particles. For example, samples with higher oxidation show increased porosity, indicating chemical changes resulting from oxidation processes.

Samples containing recycled diesel engine oils, such as WED and WEG, demonstrate significant improvement in mechanical and rheological properties due to better particle dispersion and a more uniform surface structure. Samples with recycled edible oil, given their more uniform surface, are likely to exhibit improved adhesive behavior and better resistance to cracking. Control samples BASE, BASE2, and CY, which do not contain rejuvenators, display more brittle structures and weaker mechanical behavior.

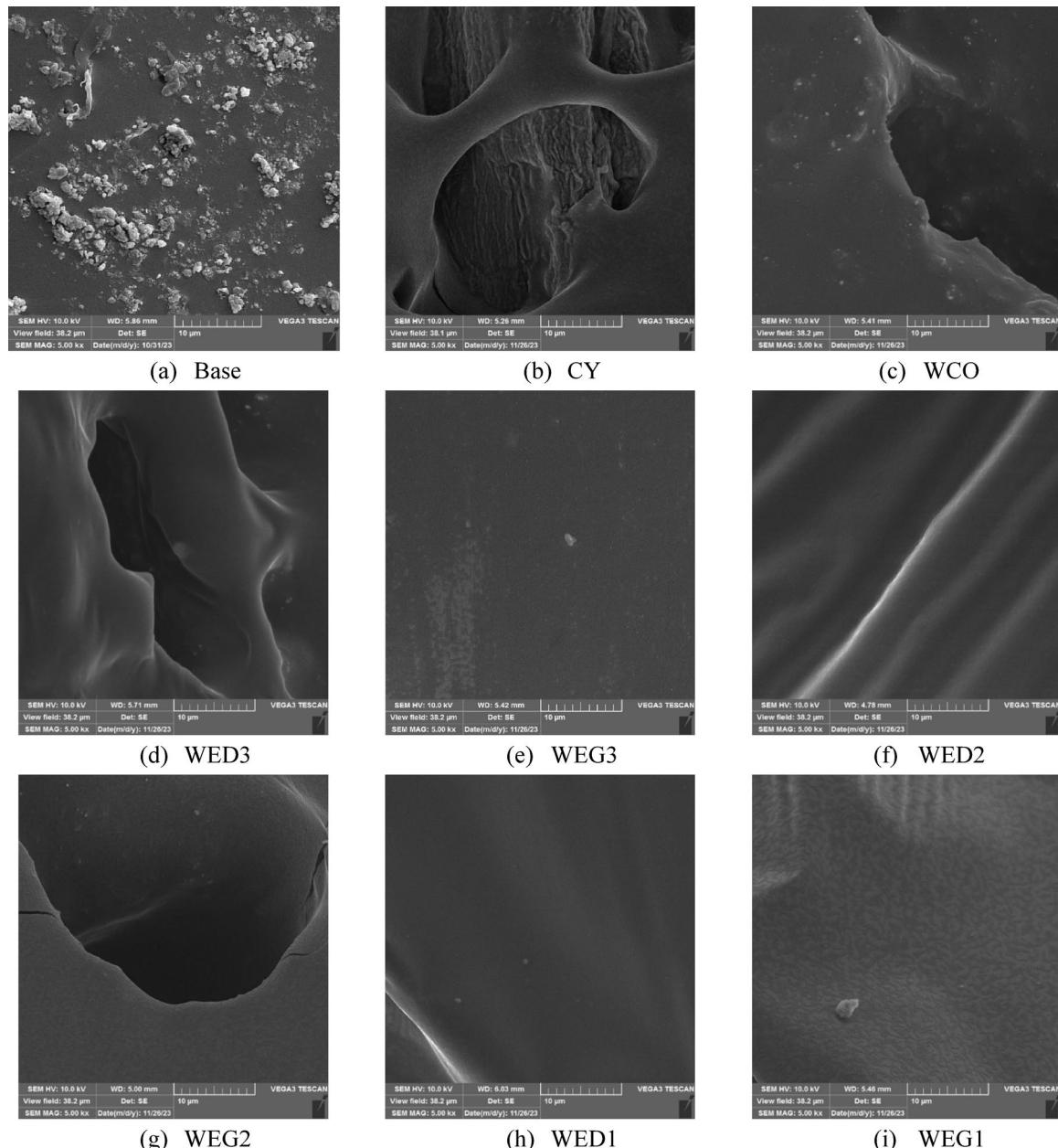


Fig. 4. Field Emission Scanning Electron Microscope (FESEM) results.

The FESEM images clearly illustrate that different rejuvenators can have varied effects on the surface structure and material behavior. These results can serve as important inputs for comparing the mechanical and chemical performance of the rejuvenators in the final analysis.

Figure 5 illustrates the molecular weight distribution of three key parameters: number-average molecular weight (Mn), weight-average molecular weight (Mw), and z-average molecular weight (Mz) for the different studied compositions.

- Mn (Number-average molecular weight): This parameter reflects the average molecular weight of the compounds. The various samples of recycled engine oils (WEG1, WEG2, WEG3) exhibit different Mn values, ranging from 1100 to 1400 g/mol, indicating variations in their chain structures. This suggests that the chemical composition and the length of molecular chains differ across the samples.
- WCO (Recycled cooking oil) and BASE (Control bitumen): These two samples show lower Mn values compared to the other rejuvenators. This lower molecular weight suggests that these samples contain shorter or less complex molecular chains, which could impact their rheological and mechanical properties differently than the engine oils.

The variation in molecular weights across different samples provides insight into how the molecular composition of rejuvenators affects the behavior and performance of reclaimed asphalt mixtures.

Mw represents the average molecular weight considering the heavier molecules' contribution. The Mw values in different oils range from 1550 to 1900 g/mol. This is higher than Mn, indicating that heavier molecules have a more significant effect on this parameter.

Mz, on the other hand, reflects the tendency toward very high molecular weight molecules. The Mz results show that all compounds have higher Mz values compared to Mw and Mn. This range is between 1700 and 2100 g/mol for the various compounds, indicating the presence of long-chain molecules in their structures.

Overall, the GPC data analysis for selecting the best rejuvenators, in terms of molecular weight and its distribution, shows that recycled motor oils with different oxidation levels in various compositions yield diverse results, which can influence the final properties of the recycled asphalt binders.

The mechanical behaviors observed in the BBR and DSR tests are directly influenced by the chemical interactions between rejuvenators and aged asphalt molecules. This interaction reduces internal stresses and improves crack resistance, as evidenced by the higher $G/\sin(\delta)$ values in DSR tests. The lack of sufficient chemical interaction limited their ability to restore the viscoelastic properties of aged asphalt, resulting in lower rutting and cracking resistance.

The Multiple Stress Creep Recovery (MSCR) test was conducted on all binder samples, starting with a 200-second pre-load conditioning phase, followed by 10 loading cycles at each stress level of 0.1 kPa and 3.2 kPa. Each cycle consisted of 1 s of creep loading and 9 s of recovery to assess the binders' resistance to permanent deformation under repeated stress. Figure 6 presents the cumulative deformation (%) of various binder samples as a function of time under the MSCR test. This test evaluates the ability of asphalt binders to resist permanent deformation under repeated loading, especially at elevated temperatures. From the graph, it is evident that the Base binder (aged binder without any rejuvenator), along with samples such as WCO and WEG3, exhibit the

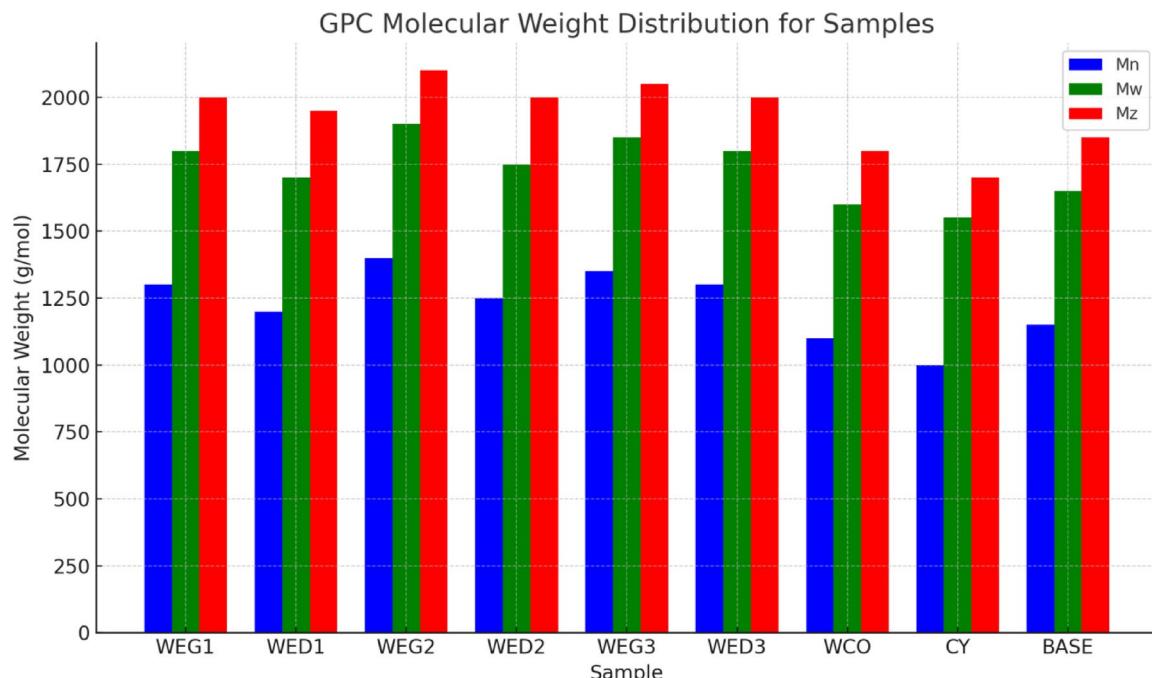


Fig. 5. - Graph of Molecular Weight Distribution for Three GPC Test Parameters.

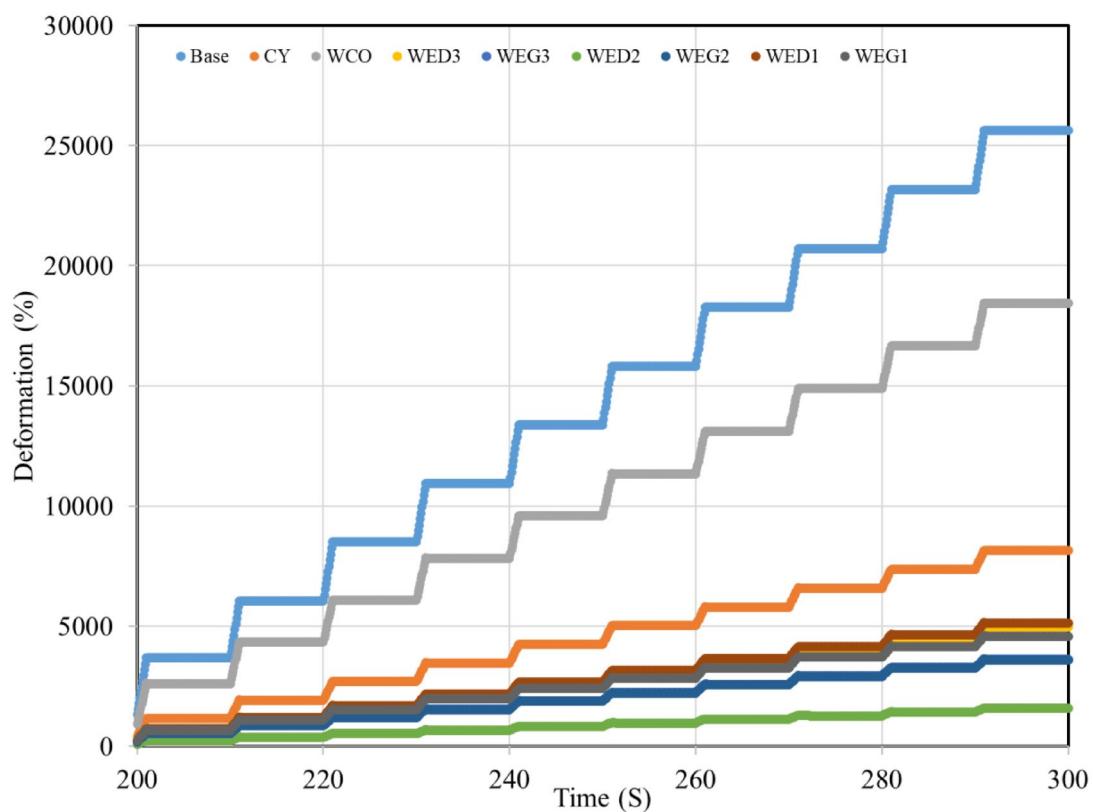


Fig. 6. MSCR Test result.

highest levels of cumulative deformation. These samples show a sharp, stepwise increase in deformation across loading cycles, indicating poor rutting resistance and limited elastic recovery. This behavior suggests that these binders have insufficient capacity to withstand high-temperature stresses, particularly under repetitive loading conditions. The Base binder, in particular, displays the weakest performance, which aligns with expectations given its fully aged and unrejuvenated state.

Conversely, samples modified with rejuvenators such as WED2, WEG2, and WED1 demonstrate substantially lower cumulative deformation, indicating superior resistance to permanent deformation and better elastic recovery. Their flatter deformation curves imply that these rejuvenators effectively restored the binder's viscoelastic balance, likely by improving the maltene-to-asphaltene ratio and enhancing network homogeneity. The improved performance in these samples may also be attributed to better particle dispersion, as observed in FESEM images, and the chemical properties of diesel-derived rejuvenators with moderate oxidation levels. Interestingly, although WCO (recycled edible oil) showed promising low-temperature behavior in BBR tests, it performs poorly in MSCR, emphasizing that rejuvenator effectiveness can vary significantly with temperature and loading condition. Overall, the MSCR results confirm that the choice of rejuvenator type and oxidation level plays a critical role in enhancing high-temperature rutting resistance of aged asphalt binders.

The results of the MSCR test, summarized in Table 2, provide valuable insights into the rutting resistance and elastic recovery of the aged and rejuvenated binder samples under two stress levels (0.1 kPa and 3.2 kPa). The non-recoverable compliance (Jnr) reflects the permanent deformation after each loading cycle, while the percent recovery (R%) indicates the binder's ability to return to its original shape.

The Base binder exhibited the highest Jnr values (6.72 at 0.1 kPa and 7.59 at 3.2 kPa), coupled with extremely low R% values, confirming its weak rutting resistance and poor recovery capacity due to aging. In contrast, WED2 showed the best performance, with the lowest Jnr values (0.40 and 0.47) and the highest R% values (27.25% and 11.22%), demonstrating excellent elasticity and stability under stress. Other effective rejuvenators include WEG2 and WED1, which also presented low Jnr and relatively high R% values, indicating balanced mechanical performance.

The Jnr difference (the increase in Jnr from 0.1 kPa to 3.2 kPa) highlights the stress sensitivity of each sample. A lower difference suggests more consistent performance across varying loads. Again, WED2 and WEG2 performed well, while samples like WCO and CY had significantly higher Jnr values and lower R%, indicating poor high-temperature performance and higher susceptibility to rutting under heavy loads. These results reinforce the importance of selecting rejuvenators not only based on viscosity or chemical compatibility, but also on their ability to improve binder performance under repeated stress.

Statistical tests play a vital role in validating experimental findings and assessing the significance of observed differences. In Table 3, the means and standard deviations of Jnr and R (%) are reported. To investigate whether

Sample	Stress (kPa)	Jnr	R (%)	Jnr difference
Base	0.1	6.72240	2.32401	12.89451
	3.2	7.58922	0.04937	
WEG1	0.1	1.23348	11.75454	10.00066
	3.2	1.35683	4.05310	
WEG2	0.1	0.94945	15.78428	12.05340
	3.2	1.06389	5.61187	
WEG3	0.1	0.93627	13.80339	14.95393
	3.2	1.07628	4.83070	
WED1	0.1	1.27707	12.68845	19.56190
	3.2	1.52689	3.73714	
WED2	0.1	0.40162	27.25244	16.55636
	3.2	0.46812	11.22422	
WED3	0.1	1.31152	9.85078	12.34709
	3.2	1.47346	3.62686	
WCO	0.1	4.64194	3.69322	17.92326
	3.2	5.47393	0.65619	
CY	0.1	2.06878	7.56605	16.98587
	3.2	2.42018	1.44286	

Table 2. MSCR results showing Jnr, R%, and Jnr difference for binder samples at two stress levels.

Parameter	Mean	Standard Deviation
Jnr (0.1 kPa)	2.17	2.1
Jnr (3.2 kPa)	2.49	2.41
R (0.1 kPa)	11.64	7.39
R (3.2 kPa)	3.91	3.33
Jnr difference	14.81	3.17

Table 3. Mean and SD of Jnr and R (%) at two stress levels.

	0.1 Kpa	3.2 kPa
Mean	2.171392222	2.494311111
Variance	4.424430327	5.784498977
Observations	9	9
Pearson Correlation	0.99948691	
Hypothesized Mean Difference	0	
df	8	
t Stat	-3.123502102	
P(T <= t) one-tail	0.007076783	
t Critical one-tail	1.859548038	
P(T <= t) two-tail	0.014153565	
t Critical two-tail	2.306004135	

Table 4. Paired t-test results for Jnr at 0.1 and 3.2 kPa.

the results under stress levels of 0.1 kPa and 3.2 kPa are significantly different, a Paired Two Sample for Means test was conducted for both Jnr and R. The results of these tests are presented in Tables 4 and 5. Since the p-values are less than 0.05, the null hypothesis is rejected, indicating that the two stress levels lead to statistically significant differences. Specifically, the 3.2 kPa stress level causes greater permanent deformation in the samples compared to 0.1 kPa.

Multi-criteria decision-making (MCDM)

The multi-criteria decision-making (MCDM) method was employed to identify the optimal rejuvenator for improving the properties of recycled asphalt mixtures. This approach utilized laboratory data and key criteria, including thermal stability, strain resistance, adhesion enhancement, and reduction of aging effects. By assigning

	0.1 kPa	3.2 kPa
Mean	11.63524	3.914701111
Variance	54.55663969	11.11883938
Observations	9	9
Pearson Correlation	0.991973404	
Hypothesized Mean Difference	0	
df	8	
t Stat	5.648823145	
P(T <= t) one-tail	0.000241041	
t Critical one-tail	1.859548038	
P(T <= t) two-tail	0.000482081	
t Critical two-tail	2.306004135	

Table 5. Paired t-test results for R (%) at 0.1 and 3.2 kPa.

Sample	Thermal stability	Strain resistance	Adhesion improvement	Aging effects reduction	Environmental desirability	Availability	Final score
WEG1	0.85	0.7	0.9	0.8	0.75	0.7	0.765
WEG2	0.8	0.65	0.88	0.78	0.7	0.65	0.695
WEG3	0.88	0.75	0.8	0.85	0.85	0.8	0.865
WED1	0.8	0.65	0.95	0.7	0.65	0.6	0.655
WED2	0.75	0.6	0.9	0.68	0.6	0.55	0.635
WED3	0.9	0.6	0.75	0.9	0.8	0.75	0.785
WCO	0.75	0.6	0.85	0.65	0.85	0.85	0.735
CY	0.7	0.55	0.8	0.6	0.6	0.6	0.635
Base	0.7	0.55	0.8	0.55	0.55	0.6	0.455

Table 6. The results of sample analysis using the MCDM method.

appropriate weights to each criterion, MCDM facilitates the selection of the best rejuvenator based on their overall performance. The criteria and their respective weights were defined as follows: environmental desirability, reflecting the impact of rejuvenators on the environment in terms of emissions, recyclability, and ecological effects, weighted at 0.15; availability, indicating the ease of procurement and market abundance, weighted at 0.10; thermal stability, representing the ability to withstand high temperatures without significant weight loss, weighted at 0.30; strain resistance, denoting tolerance to deformation under loading conditions, weighted at 0.25; adhesion improvement, referring to uniform particle distribution and enhanced bonding within the asphalt mix, weighted at 0.25; and reduction of aging effects, measuring the rejuvenator's capacity to reduce the absorption of carbonyl and sulfoxide groups, which serve as indicators of binder aging, weighted at 0.20.

For performance analysis, ten different samples—WEG1, WEG2, WEG3, WED1, WED2, WED3, WCO, CY, and Base—were evaluated as shown in Table 6. Each rejuvenator was normalized across the aforementioned criteria with scores ranging from 0 to 1. According to the MCDM results, WEG3 achieved the highest overall score of 0.865, demonstrating superior performance particularly in thermal stability, strain resistance, and aging reduction. WED3 ranked second with a score of 0.785, excelling in environmental desirability and aging effect reduction. WCO placed third with a score of 0.735, mainly due to its high marks in environmental desirability and availability.

In this paper, the comprehensive results of tests performed on recycled asphalt mixtures containing various rejuvenators were thoroughly and scientifically analyzed. These tests included rheological, thermal, and chemical evaluations, aimed at determining the impact of rejuvenators on the mechanical performance and durability of asphalt mixtures.

A study by He et al. explored the diffusion mechanism of rejuvenators and their effect on the rheological properties of asphalt mixtures. This research highlighted that rejuvenators improve asphalt's resistance to cracking and strain by penetrating the aged asphalt binder. Similarly, in the present study, the LAS (Linear Amplitude Sweep) test revealed that rejuvenators such as WEG1 and WEG3 showed excellent performance in enhancing strain resistance and maintaining the structural integrity of asphalt mixtures.

The findings of this study align with previous research, such as that conducted by Lou et al. (2021), which highlighted the significance of high aromatic content in rejuvenators for improving chemical properties of recycled asphalt mixtures. Similar to their results, WEG3 demonstrated superior performance in restoring viscoelastic properties. However, this study expands upon their findings by providing a detailed molecular weight analysis that connects the chemical composition of rejuvenators to specific mechanical behaviors observed in BBR and DSR tests.

In comparison to Ziari et al. (2020), who emphasized the role of fiberglass in enhancing fracture resistance, the current study highlights the comparable impact of oxidation levels in motor oil-based rejuvenators. For

instance, WED3 exhibited crack resistance similar to mixtures containing fiberglass, suggesting that rejuvenator oxidation levels play a critical role in performance enhancement.

Furthermore, unlike Haghshenas et al. (2018), who reported long-term superiority of petroleum-based rejuvenators, this study demonstrates that vegetable-based rejuvenators, such as WCO, can perform adequately in specific conditions, particularly in terms of adhesion and environmental sustainability.

Ultimately, from the comparative results and various tests, WEG3 was found to be the leading rejuvenator, excelling in thermal and mechanical resistance while also showing superior performance in reducing asphalt binder aging and enhancing particle adhesion. This study demonstrated that using the right rejuvenators, such as WEG3, can significantly enhance the quality and durability of recycled asphalt mixtures, reducing maintenance costs and extending the lifespan of road infrastructure. Overall, the results from this chapter emphasize the critical role of selecting optimal rejuvenators for improving the efficiency and longevity of asphalt mixtures under diverse environmental and mechanical conditions.

Conclusion

This study investigated the impact of various rejuvenators on the mechanical, chemical, and thermal properties of reclaimed asphalt mixtures using GPC, FESEM, and BBR tests.

Key outcomes include:

- Mechanical performance: Rejuvenators WEG1, WEG3, and WED3 notably improved strength and resistance to deformation-induced cracking, with WEG3 providing the best long-term durability.
- Adhesion and microstructure: FESEM results showed that WED1, WED3, and WEG1 enhanced bitumen particle distribution and adhesion, improving mixture durability, especially WEG1's strong bitumen-aggregate bonding.
- Optimal rejuvenator selection: WEG3 was identified as the most effective rejuvenator, offering extended lifespan, lower maintenance costs, and better asphalt performance under harsh conditions.
- Environmental benefits: Use of recycled rejuvenators, particularly WEG3, reduces energy consumption and greenhouse gas emissions, supporting sustainable pavement technologies.

This work advances the field by integrating molecular weight analysis and statistical validation, demonstrating that recycled materials like motor oil and cooking oil can perform as well or better than commercial rejuvenators. The findings emphasize the critical role of chemical composition and oxidation level in selecting optimal rejuvenators for recycled asphalt mixtures.

Future research

While this study identified WEG3 as the most effective rejuvenator among those tested, future research could explore a broader range of waste-derived oils and bio-based rejuvenators to assess their environmental impact, economic feasibility, and long-term field performance. Additionally, incorporating aging simulations and field trials over extended periods would help validate laboratory results under real-world conditions. Investigating the compatibility of rejuvenators with different types of reclaimed asphalt pavement (RAP) and binders from various sources is also recommended. Moreover, integrating advanced modeling techniques, such as machine learning, could enhance the prediction of performance outcomes based on rejuvenator properties. Finally, the development of standardized protocols for rejuvenator selection and dosage optimization remains an important area for future work to ensure consistency and reliability in asphalt recycling practices.

Data availability

We are unable to share the research data at this stage because these datasets are actively being analyzed for a separate manuscript currently in preparation. To protect the novelty of our ongoing work and prevent premature disclosure, we will consider data sharing requests after the completion and publication of both related studies. Researchers interested in the data may contact the corresponding author for future access arrangements.

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Declarations

Competing interests

The authors declare no competing interests.

Additional information

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