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INVESTIGATION OF PHYSICAL AND CHEMICAL PROPERTIES OF BITUMEN MODIFIED WITH WASTE ENGINE AND INDUSTRIAL OILS

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ABSTRACT

The modification of bitumen with waste oils encourages the transition5 to sustainable production and consumption strategies by increasing resource efficiency and reducing environmental impacts. In this study, bitumen was modified with waste engine oil (WEO) and waste industrial oil (WIO) at the rate of 2%, 2.5%, 3%, 3.5%, and 4% by weight to increase resource efficiency and contribute to sustainable transportation. Physical tests (penetration, softening point, penetration index, rotational viscometer) were applied to the oil modified bitumens, and the chemical composition of the bitumen as a result of the modification was examined using FTIR, SEM, and EDS analyses. Results indicate that modified bitumens exhibit superior performance in cold climate regions. The modification of 4% WIO and WEO into bitumen reduced the mixing temperature by 3.5% and 5%, respectively, and the compaction temperature by 5% and 10%, respectively. The findings of the study indicate that WIO and WEO can be used in bitumen modification to improve its performance. This study contributes to sustainable production practices by not only utilizing waste materials in a sustainable manner but also reducing the environmental impacts of industrial processes.

Keywords: Sustainability, Bitumen, Waste engine oil, Waste industrial oil.

ATIK MOTOR VE ENDÜSTRİYEL YAĞLAR İLE MODİFİYE EDİLMİŞ BİTÜMÜN FİZİKSEL VE KİMYASAL ÖZELLİKLERİNİN İNCELENMESİ

ÖZ

Bitümün atık yağlar ile modifikasyonu, kaynak verimliliğini artırarak ve çevresel etkileri azaltarak sürdürülebilir üretim ve tüketim modellerine geçişi teşvik etmektedir. Bu çalışmada, kaynak verimliliğini arttırmak ve sürdürülebilir ulaşıma katkı sağlamak amacıyla bitüm ağırlıkça %2, %2.5, %3, %3.5 ve %4 oranlarında atık motor yağı (AMY) ve atık endüstriyel yağ (AEY) ile modifiye edilmiştir. Modifiye edilen bitümlere fiziksel deneyler (penetrasyon, yumuşama noktası, penetrasyon indeksi, dönel viskozimetre) uygulanmış ve modifiye bitümlerin kimyasal içeriği FTIR, SEM ve EDS analizleri ile incelenmiştir. Çalışma sonucunda, modifiye edilen bitümlerin hava sıcaklığının düşük olduğu bölgelerde kullanımının daha uygun olacağı tespit edilmiştir. AEY ve AMY ile %4 oranında modifiye edilen bitümlerin karıştırma sıcaklığı sırasıyla %3.5 ve %5 oranında azalırken, sıkıştırma sıcaklığı ise %5 ve %10 oranında azalmıştır. Çalışmadan elde edilen sonuçlara göre AEY ve AMY'lerin bitüm performansını arttırmak amacıyla bitüm modifikasyonunda kullanılabileceği gösterilmiştir. Bu çalışma, atık malzemelerin sürdürülebilir bir şekilde kullanılmasının yanı sıra endüstriyel süreçlerin çevresel etkilerini azaltarak sürdürülebilir üretim uygulamalarına katkıda bulunmaktadır.

Anahtar Kelimeler: Sürdürülebilirlik, Bitüm, Atık motor yağı, Atık endüstriyel yağ.

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1. Introduction

Bituminous binders used in hot mix asphalt (HMA), which exhibit high thermal sensitivity, suffer various thermal changes during the road construction process. In addition, due to the harsh environmental effects and variable traffic loads they are subjected to throughout their service life, changes occur in the properties of bituminous binders both during the construction and use of HMA [1]. This phenomenon, referred to as the aging of the bituminous binder, causes the binder to gradually lose its volatile components and fail to perform as expected [2]. The aging of bituminous binders is also one of the reasons for the deterioration that may occur in the pavement layer [3].

Bituminous binder modification is carried out to enhance the resistance of pavement layers to temperature fluctuations, reduce cracking and deformation to ensure longer service life, increase skid resistance to improve safety, provide protection against environmental impacts, and promote the use of recyclable materials, thereby contributing to environmental sustainability [4].

The use of waste oil in bitumen modification contributes to environmental sustainability [5] while improving the rheological properties and temperature resistance of bitumen, offering a cost-effective solution, enhancing crack and deformation resistance, and facilitating the application process by ensuring mixture homogeneity. Various studies have been conducted considering these effects. The results have shown that bitumen modified with waste engine oil exhibits lower thermal sensitivity, viscosity, and rutting resistance but higher fatigue resistance and temperature sensitivity [6,7]. In their study, Yalçın and Yılmaz [8] aimed to evaluate the physical, mechanical, and rheological properties of waste oil (WO) modification. It was determined that the viscosity and softening point of bituminous binders decreased, while the penetration value increased when modified using waste engine oil and waste vegetable oil. Additionally, as the additive content increased, the complex modulus values decreased for both WO modifications, and the increase in phase angle indicated that the binders exhibited a more viscous behavior. The use of filtered waste engine oil resulted in a 35% reduction in binder hardness compared to base bitumen after short-term aging [9]. In their study, Sani et al. [10] investigated the use of waste engine oil in warm mix asphalt. The performance of modified bitumen (MB), obtained using different percentages of waste engine oil, was evaluated in terms of penetration, softening point, Marshall stability, flow, and stiffness. The results indicated that waste engine oil modification could improve the performance properties of Warm Mix Asphalt, such as stability, flow, and stiffness, while also having a positive effect on penetration and softening point.

Furthermore, in various studies, the combined effects of using waste engine oil alongside different modifiers have been investigated. In their study, Liu, Li, and Wang [11] utilized waste engine oil and polyphosphoric acid for bitumen modification. It was observed that the combined modification improved binder properties, such as rutting resistance and temperature stability, which were weakened by WO modification alone. A mere 2% polyphosphoric acid modification was found to enhance the rutting factor obtained from DSR analysis by 31.2% compared to base bitumen. To enhance the beneficial effects of waste engine oil, phosphogypsum waste was used in bituminous binder modification, demonstrating superior performance in terms of high-temperature deformation resistance, low-temperature crack resistance, and moisture sensitivity [12]. Based on performance test results regarding high-temperature stability, low-temperature crack resistance, and fatigue life, the optimal ratio for the modified asphalt binder was determined to be 2% waste engine oil and 5% phosphogypsum. The combined use of waste engine oil and SBS resulted in a MB with improved fatigue crack resistance and rutting resistance [13]. The combined use of waste vehicle lubricants and waste polymers was found to enhance high-temperature stability and creep resistance [14].

In road construction, the use of different types of waste oils is recommended to reduce the stiffening effect of Reclaimed Asphalt Pavement. Studies conducted for this purpose have shown that waste engine oils can be used as rejuvenating agents in mixtures containing reclaimed asphalt, with notable improvements in moisture sensitivity and stability [15-18]. Additionally, it has been observed that waste engine oils enhance low-temperature properties [19,20]. The similar chemical structure of waste engine oil and asphalt materials allows the recovery of components lost during asphalt aging through waste engine oil modification, thereby improving the properties of aged asphalt in both binder and mixture forms [21-24]. The rejuvenation of Reclaimed Asphalt Binder using waste industrial oil, specifically waste steel rolling oil, has been investigated, showing increased resistance to moisture sensitivity and the ability to reverse aging effects [25].

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This study aims to comprehensively evaluate the potential of using waste motor and industrial oils in the modification of bitumen through a combination of physical and chemical analyses. The investigation will focus on assessing the influence of waste oil additives on key engineering properties of bitumen, with particular emphasis on penetration, softening point, and viscosity, using quantitative data. In addition, the chemical composition of the waste oils and their interactions within the bituminous matrix will be examined to determine their impact on structural integrity and overall material performance. The experimental results will be systematically compared with findings from the existing literature, providing a critical assessment of the viability of WO modification as a sustainable approach to material enhancement and environmental impact reduction.

2. Material and Method

2.1. Bitumen

In the study, AC 50-70 grade bitumen supplied from the Isparta Municipality Asphalt Plant was used. Standard performance tests were conducted on the bitumen, and the values obtained from these tests are presented in Table 1.

Table 1. Properties of asphalt binder

Properties	Standard	Unit	Results
Penetration (25 °C)	TS EN 1426 [26]	0.1 mm	50.48
Softening Point	TS EN 1427 [27]	°C	47.7
Ductility (5cm/min)	TS EN 13589 [28]	cm	>100
Specific Gravity	TS 15326+A1 [29]	g/cm^3	1.005

2.2. Waste oil

Used industrial or motor oils are oils that have been refined from crude oil and, after a certain period of use in industrial or non-industrial applications, particularly for lubrication purposes, lose their original properties. Used vehicle oils (consisting of gasoline engine, diesel engine, transmission and differential, drivetrain, two-stroke engine, hydraulic brake, antifreeze, grease, and other specialized vehicle oils); industrial oils (including hydraulic systems, turbine and compressor, slideway, open-closed gear, circulation, metal cutting and processing, metal drawing, textile, heat treatment, heat transfer, insulation and protective, rust and corrosion, insulation, transformer, mold, steam cylinder, pneumatic system protective, food and pharmaceutical industry, general-purpose, paper machine, bearing, and other specialized industrial oils and industrial greases); special preparations (thickeners, protectants, cleaners, and similar); and contaminated oil products are included in this definition.

Used industrial or motor oils contain various chemical compounds and additives that degrade over time, leading to a significant environmental burden if improperly disposed of. Due to their complex composition and potential toxicity, the recycling and reutilization of waste oils have gained considerable attention in recent years. In particular, incorporating these waste oils as modifiers or rejuvenators in bituminous materials presents a sustainable approach to both managing waste and enhancing the performance of asphalt binders.

According to 2023 data in Turkey, 318 thousand tons of vehicle oil were supplied to the market, with the potential amount of waste motor and transmission oil estimated to be 190 thousand tons. However, in 2023, only 1,731 tons of waste motor and transmission oil were collected by PETDER (Petroleum Industry and Emobility Association) [30].

Within the scope of the study, the industrial and motor waste oil groups to be used were obtained from Acıöz Petrol Hurdacılık Nakliye Demir Ürünleri San. ve Tic. Ltd. Şti., located in the Selçuklu district of Konya province. The waste oil groups used in the study, namely industrial waste oil and motor waste oil, are shown in Figure 1.

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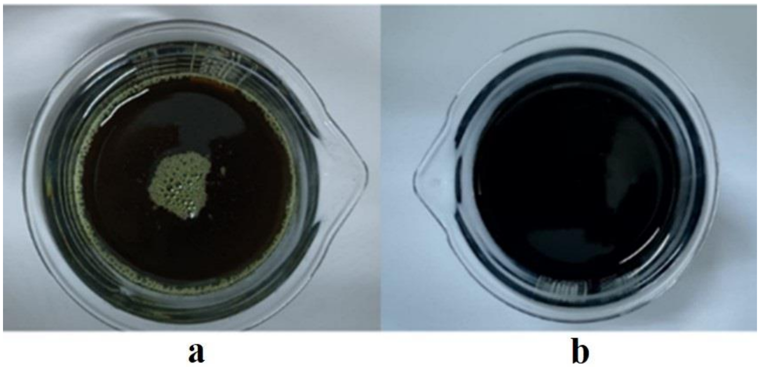

Figure 1. Waste industrial oil (a), Waste engine oil (b)

In the study, the waste industrial and engine oils used were subjected to elemental analysis using the Rotating Disc Electrode Opical Emission Spectrometry (RDE-OES) method (ASTM D6595) (Table 2).

Table 2. The elemental analysis results of the waste industrial and engine oils

	Unit	Calcium (Ca)	Phosphorus (P)	Zinc (Zn)	Ferro (Fe)
Waste Industrial Oil	mg/kg	31,1	181,86	200,6	9,46
Waste Engine Oil		1769,5	847,47	509,79	209,64

2.3. Preparation of Modified Bitumen

For bitumen modification, the bitumen was heated to approximately 145±5 °C, and waste oil from each group was added at weight percentages of 2%, 2.5%, 3%, 3.5%, and 4%. The heated bitumen and waste oil were then transferred to a high-speed mixer mold preheated to 140±5 °C. The high-speed mixer, operating at 1000 rpm, was run for half an hour to ensure a homogeneous mixture of the bitumen and waste oil groups.

Within the scope of the study, the abbreviations for the waste oil groups used in bitumen modification at various weight percentages are provided in Table 3.

Table 3. The abbreviations for the waste oil groups

	Percentages					
	0	**%2**	**%2.5**	**%3**	**%3.5**	**%4**
Waste Industrial Oil	B	I2	I2.5	I3	I3.5	I4
Waste Engine Oil		E2	E2.5	E3	E3.5	E4

The effects of changes in the additive and mixture parameters on the properties of bitumen were determined through Penetration (TS EN 1426), Softening Point (TS EN 1427), and Rotational Viscometer (ASTM D 4402) tests [31], and Penetration Index Analysis was conducted. Additionally, Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and Energy Dispersive Spectroscopy (EDS) analyses were performed on the MB, and the chemical structure of the bitumen was compared with that of base bitumen.

3. Results and Discussion

To investigate the effect of using waste engine and industrial oils in bitumen modification on binder consistency, penetration and softening point tests were conducted first. The results of the penetration test are presented in Figure 2. The increase in penetration value with the rise in waste oil percentage indicates that the MB has transitioned to a softer consistency. According to the penetration results, MB containing 2% by weight of waste oil from each oil group remained within the pure bitumen grade (50/70). Additionally, the specimen I2.5 also stayed within the 50/70 penetration grade. The increase in penetration value with the rise in waste oil percentage suggests that the MB has become softer. Based on these findings, it is concluded that the MB obtained through bitumen modification with each waste

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oil group can be used in cold climate regions. Cold climate bitumens are primarily used in regions exposed to low temperatures, such as highways, roads in mountainous areas, airport runways, and urban roads. These bitumens provide resistance to thermal stresses due to their formulations characterized by high penetration values and low softening points, which enhance their crack resistance. Furthermore, their elastic structure ensures durable performance against frost, icing, and temperature fluctuations.

The physical properties of bitumen intended for use in cold climate conditions play a crucial role in maintaining flexibility at low temperatures and preventing thermal cracking. To this end, bitumens with high penetration values (e.g., 100–150) are typically preferred, as their softness contributes to enhanced elasticity under freezing conditions [32]. Similarly, the softening point of these bitumens is kept relatively low (approximately 35–45 °C), allowing the material to remain workable without becoming brittle [33]. Furthermore, the rotational viscosity at 135 °C is generally limited to below 3000 cP to ensure sufficient workability during production and application phases [34]. Optimization of these parameters enables the material to meet the specific mechanical and environmental demands of cold climate pavements. Recent studies suggest that WO modification may also serve as a promising alternative for enhancing bitumen performance in cold regions. The incorporation of waste lubricating oils has been found to improve low-temperature flexibility and reduce the brittleness of bitumen, offering an environmentally sustainable and economically viable solution for cold climate applications.

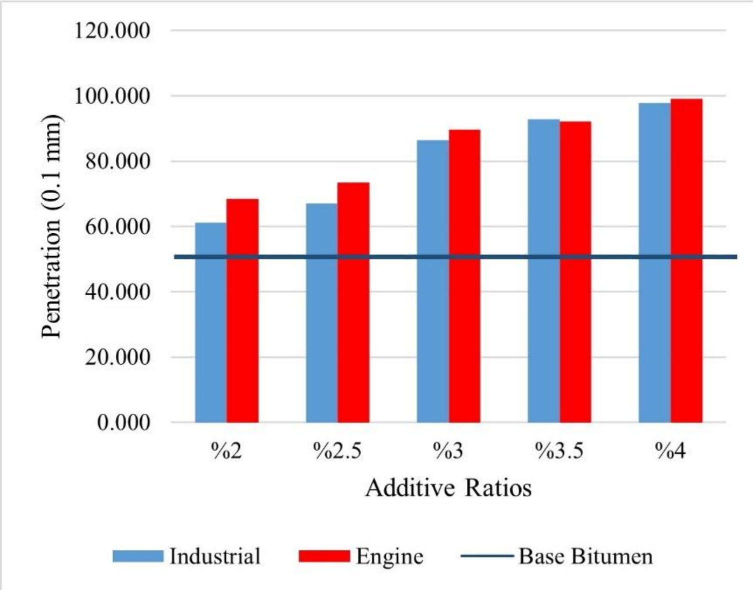

Figure 2. Penetration test results

According to the data obtained from the experimental study, it is observed that bitumen modified with 2.5% by weight of waste oil from each waste oil group has a softening point similar to that of base bitumen (Figure 3). Additionally, in general, the softening points of waste oil-MB are lower compared to base bitumen. As the percentage of waste oil by weight increases, a decrease in the softening point value is observed. The suitability of bituminous binders for specific regions can be inferred based on their softening points. Accordingly, it is concluded that bitumen modifications using waste oil are suitable for use in cold climate regions.

In their study, Gökalp et al. [35] a 2% WO modification resulted in a 12.6% increase in the penetration value compared to base bitumen, while a 4% modification yielded a 33.5% increase. Regarding the softening point, the changes were recorded as a 9.2% decrease for the 2% modification level and an 18.4% decrease for the 4% modification level relative to the base bitumen. In this study, a 2% WO modification resulted in a 45.4% increase in the penetration value compared to base bitumen, while a 4% modification led to a 96.2% increase. As for the softening point, a 2% modification showed a 4.61% increase relative to the base bitumen, whereas a 4% modification resulted in a 1.7% decrease.

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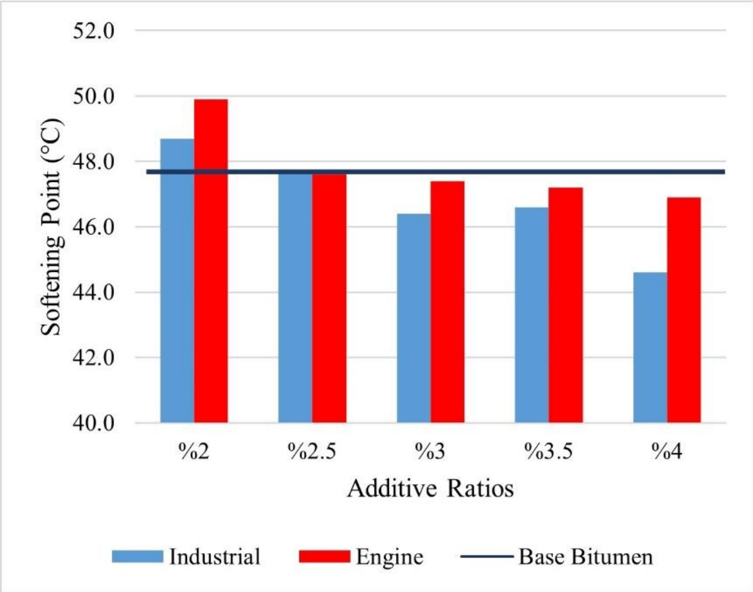

Figure 3. Softening point test results

To determine the temperature sensitivity of bituminous binders, various methods and indices are used. One of these is the penetration index. The calculation of the index utilizes the penetration value and softening point value of the bituminous binder. The penetration index values were calculated using the Equation 1 [36].

$$(20\text{-PI})/(10+\text{PI}) = 50.[\log(800) - \log(\text{Pen}) / (\text{TRB}-25)] \tag{1}$$

In this equation, Pen 25 ºC represents the penetration value at 25°C, and TRB denotes the softening point. According to the obtained data, the penetration index value for each waste oil group modification falls within the expected range (Figure 4). In particular, it has been observed that the temperature sensitivity of pure bitumen is improved through WO modification.

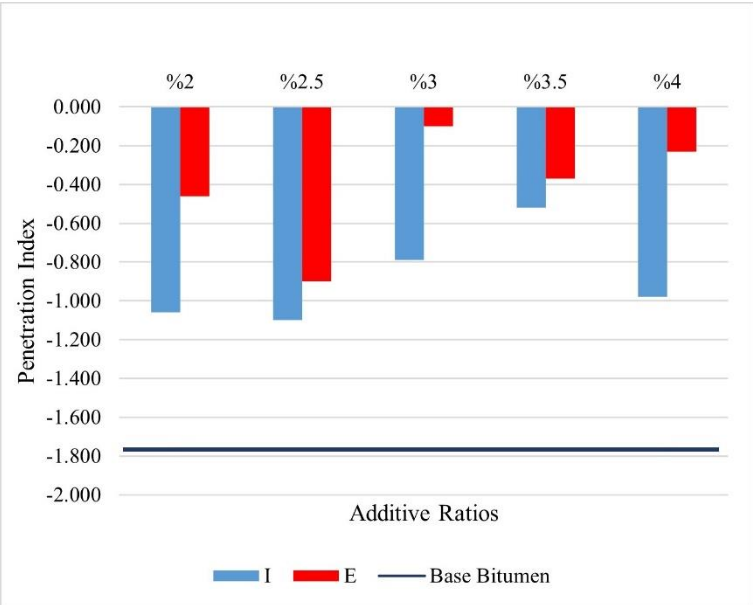

Figure 4. Penetration index results

To determine the mixing and compaction temperatures of the bituminous binders used in the study, a rotational viscometer test was conducted at two different temperatures: 135°C and 165°C. According to the results, the lowest mixing and compaction temperatures were observed at a 4% additive ratio for both waste oil group modifications. The lowest mixing and compaction temperatures were found in the E4 bituminous binder (containing 4% waste engine oil). As shown in Figures 5 and 6, the mixing

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temperature for pure bitumen is 162.8°C, and the compaction temperature is 154.8°C. As seen in Figure 5, the mixing temperatures for waste industrial oil modification are 161.2°C for I2, 159°C for I2.5, 158.9°C for I3, 158.3°C for I3.5, and 157.6°C for I4. Similarly, the compaction temperatures are 151.9°C for I2, 149.8°C for I2.5, 148.6°C for I3, 147.5°C for I3.5, and 146.8°C for I4.

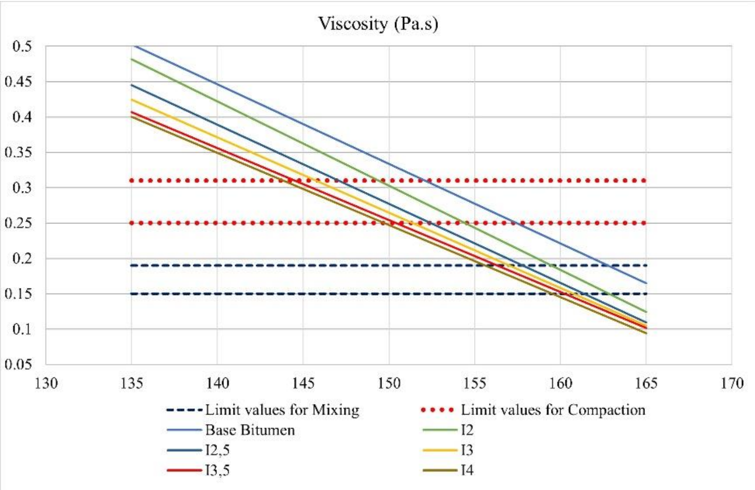

Figure 5. Rotational viscometer values for waste industrial oil modification

As shown in Figure 6, the mixing temperatures for waste engine oil modification are 160.3°C for E2, 158.2°C for E2.5, 157.1°C for E3, 156°C for E3.5, and 155.1°C for E4. The compaction temperatures are 147.8°C for E2, 144.5°C for E2.5, 142.5°C for E3, 141.8°C for E3.5, and 139.4°C for E4. For the highest additive ratio of 4% in waste industrial oil modification (I4), the mixing temperature decreased by approximately 3.5%, and the compaction temperature decreased by approximately 5%. In waste engine oil modification, for the highest additive ratio of 4% (E4), the mixing temperature decreased by 5%, and the compaction temperature decreased by 10%. When evaluating all WO modifications, the lower mixing and compaction temperatures indicate improved workability compared to pure bitumen. The increased workability allows for pavement production and construction activities at lower temperatures, reducing the cost of pavement construction and energy consumption through the modification of bituminous binders with waste oils.

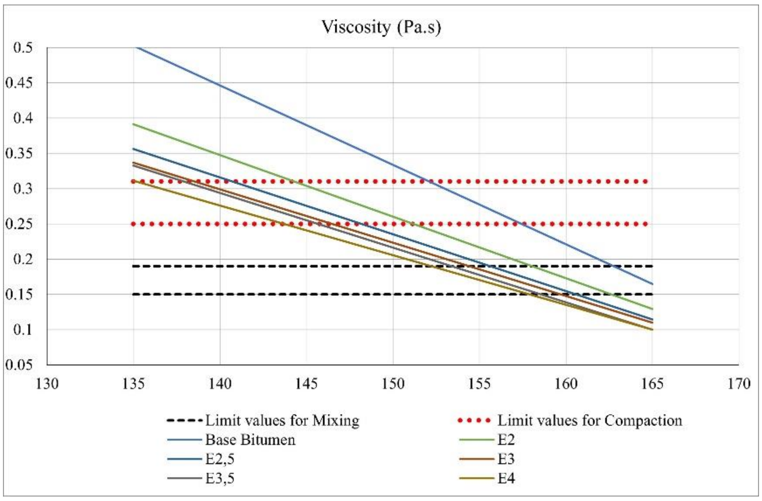

Figure 6. Rotational viscometer values for waste engine oil modification

To determine the chemical structure of the bituminous binders used in the study, FTIR analysis was performed using the JASCO FT/IR-4700 device. The FTIR analysis was conducted in the mid-infrared region (7800-350 cm^{-1}) at room temperature, within a wavenumber range of 400-4000 cm^{-1}, using the FT/IR-4700 device with a resolution higher than 0.4 cm^{-1}. The analysis was completed by measuring the spectrum against percent transmittance and absorbance. FTIR analysis was applied to pure bitumen and the MB samples with the lowest and highest weight percentages for each waste oil group, namely

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I2, I4, E2, and E4. As shown in Figure 7, the bituminous binders modified with 2% and 4% waste industrial oil exhibit similar and symmetrical transmittance peaks compared to base bitumen.

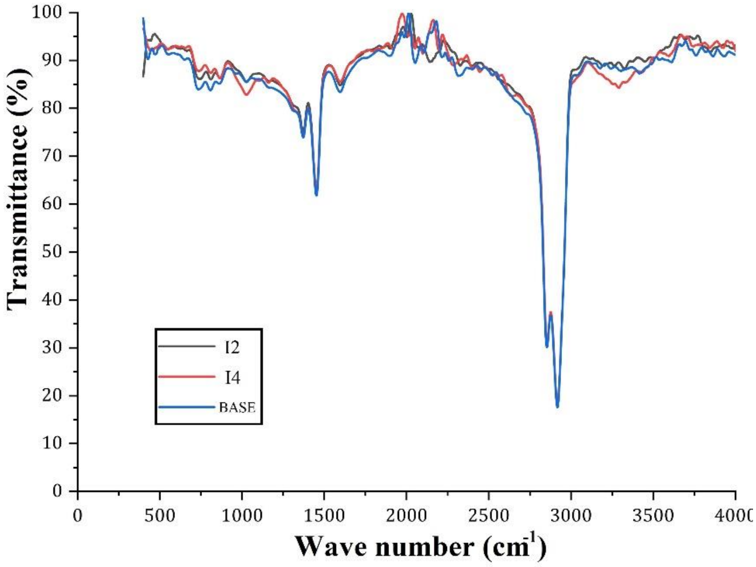

Figure 7. FTIR spectra of waste industrial oil modification

In Figure 8, the bitumen modified with 2% and 4% waste engine oil shows similar and symmetrical characteristics to base bitumen. Based on the evaluation of the FTIR analysis results, it is observed that waste industrial and engine oil modifications are compatible with the bituminous binder. There is no change in the chemical structure of the MB, indicating that the WO modification results from a physical process.

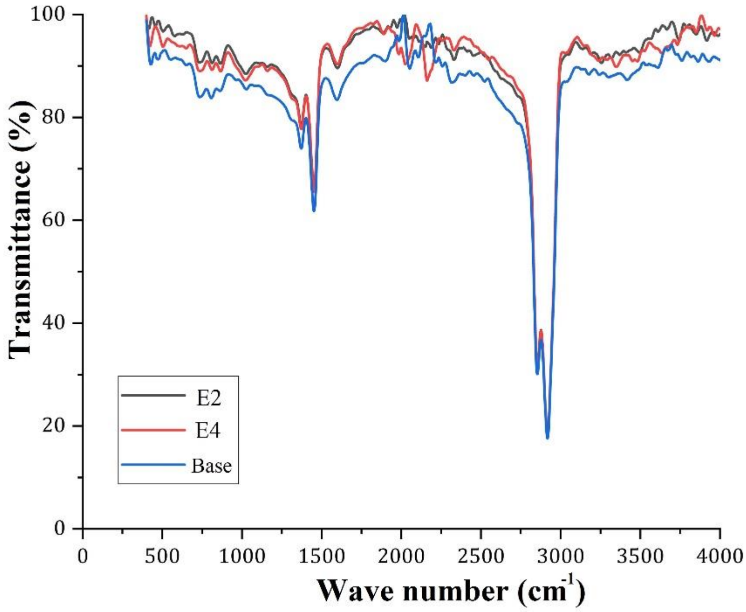

Figure 8. FTIR spectra of waste engine oil modification

To determine the morphological properties of the pure and waste oil-modified bituminous binders used in the study, SEM analysis was performed using the FEI Quanta FEG 250 electron microscope. Additionally, to determine the elemental analysis values of the bituminous binders used in the study, EDS analysis was conducted using the EDAX/EDS detector integrated into the SEM device. The results of the SEM and EDS analyses were presented together on a single figure for each bituminous binder and evaluated collectively. Within the scope of the study, the data obtained from the RDE-OES

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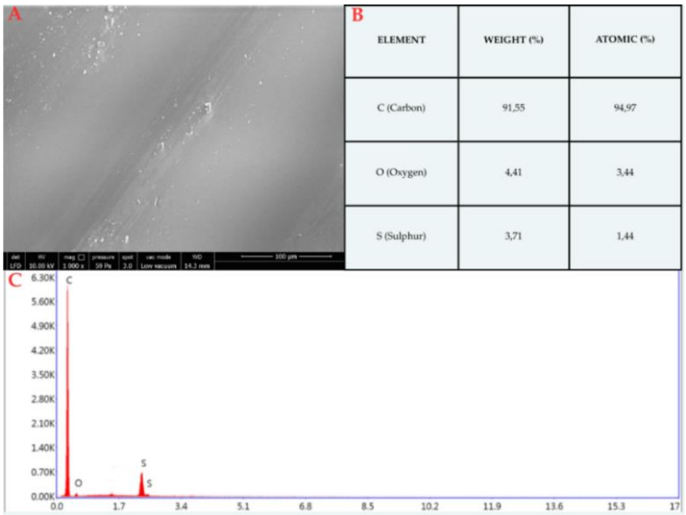
elemental analysis applied to waste industrial and engine oils align with the results of the EDS analysis. When the obtained data are evaluated, it is observed that the WO modification occurred homogeneously.

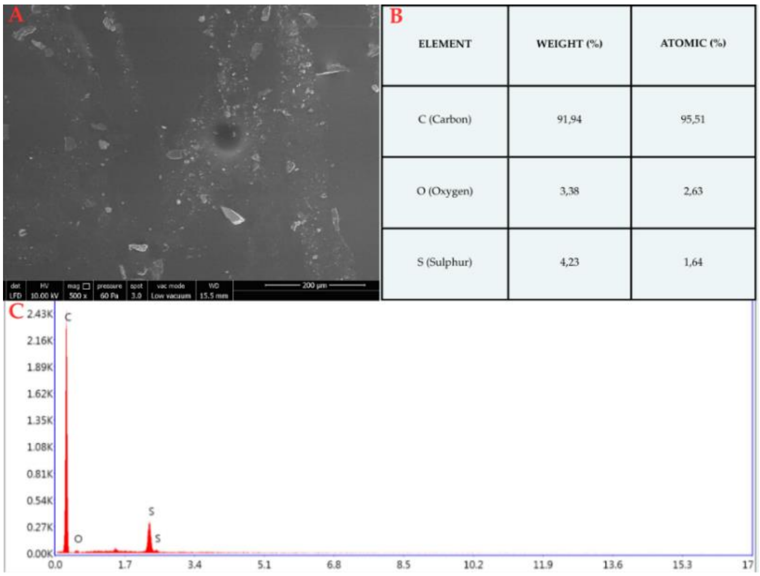
According to the SEM image shown in Figure 9, the surface morphology of the base bituminous binder exhibits a uniform and homogeneous structure.



ELEMENT	WEIGHT (%)	ATOMIC (%)
C (Carbon)	91,55	94,97
O (Oxygen)	4,41	3,44
S (Sulphur)	3,71	1,44

Figure 9. SEM images (A) and EDS analysis results (B and C) of base bitumen

As shown in the SEM image in Figure 10, the surface morphology of the I2 sample exhibits noticeable changes when compared to that of the base bitumen.



ELEMENT	WEIGHT (%)	ATOMIC (%)
C (Carbon)	91,94	95,51
O (Oxygen)	3,38	2,63
S (Sulphur)	4,23	1,64

Figure 10. SEM images (A) and EDS analysis results (B and C) of I2

According to the SEM image presented in Figure 11, a distinct pattern formation is observed on the surface morphology of the I4 sample. It has been noted that the pattern becomes more pronounced with the increase in the modification ratio of the waste industrial oil. Furthermore, the waste industrial oil appears to be uniformly dispersed within the bituminous binder without causing any agglomeration. According to the EDS results for sample I4 in Figure 11, different elements that are not present in the structure of base bitumen are observed. The obtained results are in agreement with the findings from the RDE-OES method conducted in this study.

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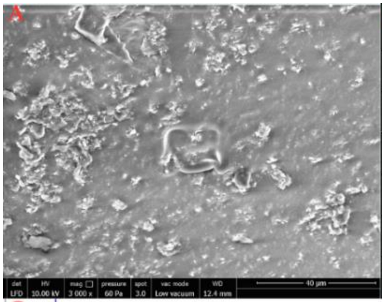

ELEMENT	WEIGHT (%)	ATOMIC (%)
C (Carbon)	86,96	91,44
O (Oxygen)	8,44	6,66
S (Sulphur)	3,31	1,31
Ca (Calcium)	0,32	0,10
Na (Natrium)	0,53	0,29
Si (Silisium)	0,14	0,06

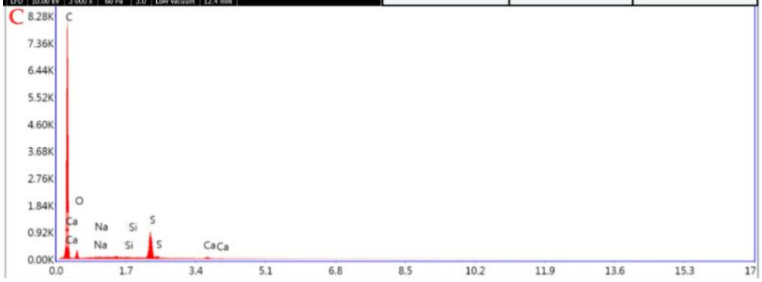

Figure 11. SEM images (A) and EDS analysis results (B and C) of I4

As shown in the SEM image in Figure 12, the surface morphology of the E2 sample exhibits noticeable changes when compared to that of the base bitumen.

ELEMENT	WEIGHT (%)	ATOMIC (%)
C (Carbon)	88,88	93,69
O (Oxygen)	4,88	3,86
S (Sulphur)	5,17	2,04
Ca (Calcium)	0,65	0,21

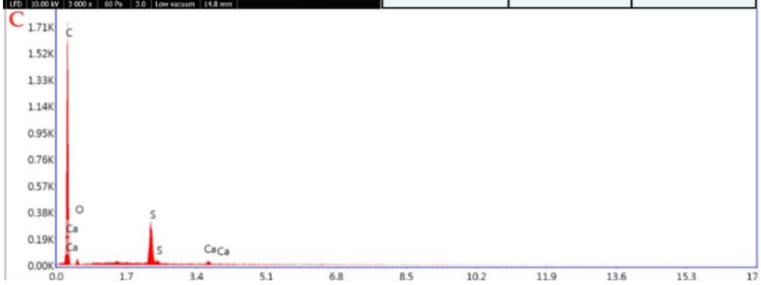

Figure 12. SEM images (A) and EDS analysis results (B and C) of E2

According to the SEM image presented in Figure 13, a distinct pattern formation is observed on the surface morphology of the E4 sample. It has been noted that the pattern becomes more pronounced with the increase in the modification ratio of the waste engine oil. Furthermore, the waste engine oil appears to be uniformly dispersed within the bituminous binder without causing any agglomeration. Furthermore, comparative analysis demonstrated noticeable differences in the surface pattern formations between waste industrial oil and waste engine oil. According to the EDS results for sample E4 in Figure 13, different elements that are not present in the structure of base bitumen are observed. The obtained results are in agreement with the findings from the RDE-OES method conducted in this study.

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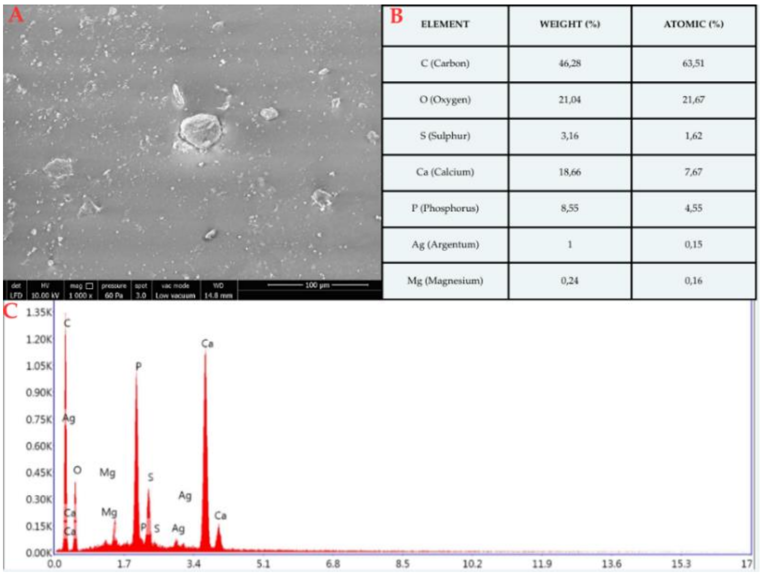

ELEMENT	WEIGHT (%)	ATOMIC (%)
C (Carbon)	46,28	63,51
O (Oxygen)	21,04	21,67
S (Sulphur)	3,16	1,62
Ca (Calcium)	18,66	7,67
P (Phosphorus)	8,55	4,55
Ag (Argentum)	1	0,15
Mg (Magnesium)	0,24	0,16

Figure 13. SEM images (A) and EDS analysis results (B and C) of E4

4. Conclusion

The fact that sustainable transportation provides environmental and economic benefits forms the cornerstone of this study. The analyses conducted with this focus aim to contribute to sustainable development goals and guide future transportation strategies.

•It is concluded that the MB obtained through WO modification can be used in cold climate regions.

•In particular, it has been observed that the temperature sensitivity of pure bitumen is improved through WO modification.

•WO modification has increased the workability of the bituminous binder. Thus, working at lower temperatures will reduce pavement construction costs and energy consumption.

•When the elemental analysis results are evaluated, it is observed that waste industrial and engine oil modifications are compatible with the bituminous binder, and the modification process is a result of a physical change.

•The study supports the suitability of using waste industrial and engine oils, which are difficult to store and reuse, in bitumen modification.

In conclusion, the role of waste oils in bitumen modification holds significant potential for environmental sustainability and asphalt performance improvements. The use of waste oils in the asphalt industry not only contributes to solving environmental problems but also enhances the physical and chemical properties of asphalt. Therefore, further research on the effects of waste oils in asphalt modification will increase knowledge in this field and contribute to the development of sustainable practices.

This study has yielded promising results regarding the use of waste oil-MBs, particularly in asphalt mixtures. The findings demonstrate the potential of waste oil additives to enhance the physical properties of bitumen, indicating that such modifications may contribute significantly to sustainable material applications. In light of these results, more comprehensive and long-term studies are planned, particularly focusing on the rejuvenation of aged bitumens using waste oils. Such investigations could provide substantial benefits by both reducing environmental waste and improving the performance of pavement materials.

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