

Article

Resource Recovery from Oil-Contaminated Soil Through Organic Phase Extraction for Bitumen-like Material Production

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Abstract

Oil-contaminated soils represent a significant environmental challenge due to the presence of petroleum hydrocarbons and associated pollutants. At the same time, such materials may contain valuable hydrocarbon-rich organic fractions that can potentially be recovered and reused. In this study, the organic phase extracted from oil-contaminated soil was investigated as a secondary hydrocarbon resource for the production of bitumen-like materials. The extracted organic phase was subjected to thermal oxidation to obtain a bitumen-like material, followed by modification using road bitumen (BND 50/70) and an SBS polymer. The resulting bitumen-like material exhibited improved performance after modification, with the softening point increasing from 32.1 to 40.6 °C, the rotational viscosity from 3.11 to 101.5 Pa·s, and enhanced high-temperature rutting resistance. The structural and compositional characteristics of the obtained materials were analyzed using FTIR spectroscopy, nuclear magnetic resonance (NMR), thermogravimetric analysis (TGA), and Saturates, Aromatics, Resins, and Asphaltenes (SARA) fractionation. Physicomechanical and rheological properties were evaluated using penetration, softening point, rotational viscosity, and dynamic shear rheometer (DSR) measurements. The results indicate the presence of aliphatic and aromatic hydrocarbon-rich structures characteristic of petroleum-derived materials. Modification with road bitumen and SBS resulted in redistribution of SARA fractions and improved physicomechanical and rheological properties, including increased softening point, higher viscosity, and enhanced resistance to permanent deformation at elevated temperatures. Overall, the obtained results demonstrate that oil-contaminated soils can be considered a promising secondary source of hydrocarbon-rich raw materials. The proposed approach combines waste remediation with resource recovery and supports the development of sustainable technologies for the utilization of petroleum-containing wastes within a circular economy framework.



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Keywords: oil-contaminated soil; hydrocarbon-rich organic matter; resource recovery; bitumen-like material; waste valorization; circular economy

1. Introduction

The growth in global demand for hydrocarbon raw materials has led to a large-scale expansion of the oil industry, covering all stages from field exploration to oil processing and storage. At each of these stages, technological failures may occur, leading to soil

contamination by petroleum products and negatively affecting ecosystems and human health [1].

Contamination of soils by petroleum hydrocarbons occurs at all stages of oil handling—from extraction to storage and use—resulting in pollution of soils and water resources. These hydrocarbons represent a complex mixture of thousands of aliphatic and aromatic compounds and currently constitute one of the most widespread and serious environmental problems worldwide [2,3]. When entering the soil, petroleum hydrocarbons undergo physical, chemical, and biological transformations, leading to the formation of toxic compounds, land degradation, contamination of groundwater, and disruption of the soil microbial balance [4,5]. To mitigate these adverse environmental impacts, numerous remediation technologies have been developed, which are generally classified into physical, chemical, biological, and combined methods depending on their mechanism of contaminant removal [6,7].

Physical remediation methods include technologies based on the mechanical removal, separation, and isolation of contaminants, such as soil replacement, soil washing, thermal desorption, and electrokinetic treatment [8,9]. Despite their high remediation efficiency and relatively rapid contaminant removal, their application is limited by high energy consumption, substantial operational costs, and the risk of secondary pollution, particularly in the case of thermal technologies [10].

Chemical remediation methods are based on the application of reactive agents capable of transforming, mobilizing, or removing petroleum contaminants from soil. The most widely used approaches include chemical oxidation, chemical leaching, and chemical enhancement, which provide rapid contaminant removal even at high pollution levels [11,12]. However, their application is limited by the high cost of reagents, potential toxicity, and the risk of secondary environmental pollution resulting from the use of aggressive chemicals [13,14].

Biological remediation methods rely on the use of microorganisms, plants, and other living organisms to degrade or remove petroleum contaminants from soil [15]. The most widely applied biological approaches include biostimulation, bioaugmentation, vermi-remediation, and phytoremediation [16]. Although these methods are environmentally friendly and relatively cost-effective, their remediation efficiency strongly depends on the physico-chemical properties of the soil and environmental conditions, and the treatment process generally requires a relatively long time [14,17].

Combined remediation methods integrate physical, chemical, and biological technologies to exploit the complementary advantages of individual approaches, thereby improving remediation efficiency and reducing treatment time [18].

Despite the considerable progress achieved in remediation technologies, increasing attention has been directed toward the processing of petroleum-containing wastes for the extraction of valuable organic components and their subsequent reuse. Such an approach not only reduces the environmental burden but also enables the recovery of valuable organic resources and their return to the circular economy, including their use as feedstocks for the production of bituminous materials and other petroleum-derived products.

Petroleum-containing wastes, such as oil sludges and oil-contaminated soils, represent complex organo-mineral systems containing significant amounts of organic fractions, including aromatic compounds, resins, and asphaltenes, which are similar in composition to the components of petroleum bitumens. Our previous studies have shown that such materials are capable of altering the colloidal structure of bituminous binders and can serve as a source of organic components involved in the formation of composite bituminous systems [19,20].

In mature oil-producing regions, such petroleum-containing wastes may accumulate over long periods of operation and represent not only an environmental liability but also a potential source of recoverable hydrocarbon-rich materials [21]. This issue is particularly relevant to Western Kazakhstan, where oil production has been carried out for more than five decades, resulting in the accumulation of large volumes of oil-contaminated soils in sludge storage sites and disposal areas. Therefore, the development of approaches aimed at resource recovery and waste valorization is of increasing interest within the framework of sustainable environmental management and circular economy principles.

To realize this resource recovery approach, it is necessary not only to recover the organic phase from oil-contaminated soils but also to evaluate its chemical composition and suitability for subsequent utilization in bituminous materials. In this regard, the organic phase was extracted from oil-contaminated soils using organic solvents. However, the structural characteristics of the recovered organic phase and its suitability as a raw material for the production of bituminous binders remain insufficiently studied. Unlike previous studies, which have primarily focused on the remediation of oil-contaminated soils, the present work investigates the structural characteristics of the recovered organic phase and evaluates its potential as a secondary hydrocarbon resource for the production of bitumen-like materials.

Petroleum-containing systems are complex colloidal systems composed of saturates, aromatics, resins, and asphaltenes [22]. Therefore, characterization of these fractions is important for understanding the structure and potential utilization of recovered organic materials.

In this regard, the aim of the present study was to recover the organic phase from oil-contaminated soil, investigate its structural characteristics using FTIR and NMR spectroscopy, and evaluate its potential as a secondary hydrocarbon resource for the production of bitumen-like materials. Comprehensive characterization of the recovered organic phase and the resulting materials allows assessment of the feasibility of converting petroleum-containing wastes into value-added products.

2. Materials and Methods

2.1. Materials

Oil-contaminated soil collected from oilfield facilities in Western Kazakhstan was used as the object of study. The investigated samples represent typical petroleum-containing wastes formed as a result of long-term oil production, transportation, storage, and accidental spills during field operation. Such wastes accumulate over extended periods in oil-producing regions and contain significant amounts of hydrocarbon-rich organic matter that may be considered a potential secondary resource. The investigated soil was characterized by an average moisture content of 14.1% and a mechanical impurity content of 73%. According to previous studies, oil-contaminated soils in Western Kazakhstan are characterized by slightly alkaline pH and high salinity, which are typical features of arid oil-producing regions [23]. The recovered organic phase was further characterized using FTIR and TG analyses.

The collected soil samples were air-dried, homogenized, and prepared for subsequent extraction and physicochemical characterization.

To investigate the organic and mineral components, Fourier transform infrared spectroscopy (FTIR) was employed to identify functional groups and characterize the chemical structure of the recovered organic phase and the original soil samples.

Thermogravimetric analysis was carried out using a NETZSCH STA 409 instrument (NETZSCH-Gerätebau GmbH, Selb, Germany) to evaluate the thermal behavior and stability of the recovered organic materials.

2.2. Recovery of Hydrocarbon-Rich Organic Phase from Oil-Contaminated Soil

The overall recovery and upgrading procedure used in this study is schematically illustrated in Figure 1. The process consisted of four consecutive stages, including solvent extraction of the hydrocarbon-rich organic phase, thermal oxidation, and subsequent modification with road bitumen and SBS.

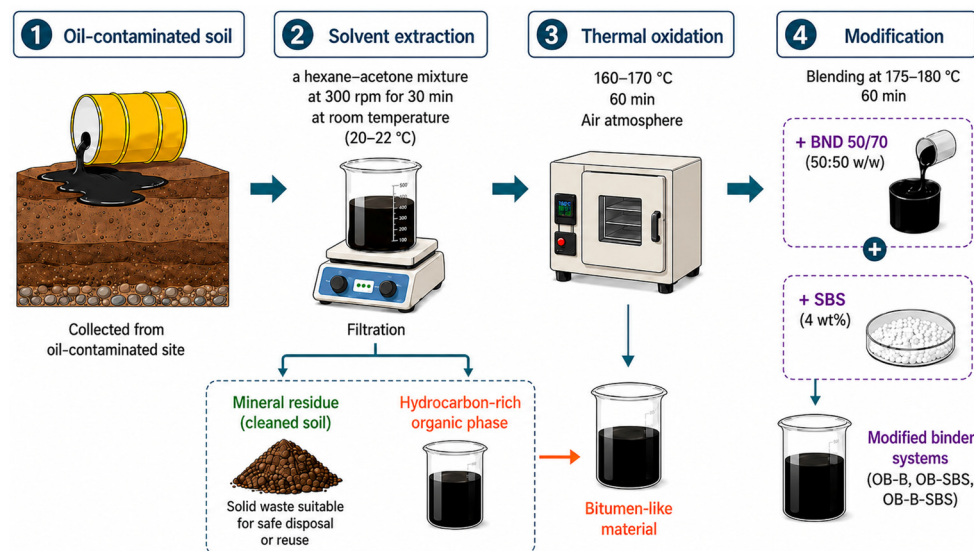


Figure 1. Schematic illustration of the recovery and upgrading process for producing bitumen-like material from oil-contaminated soil. Created with BioRender.com (accessed on 17 July 2026) (License No. BK29BPFK4D).

(1) To isolate the hydrocarbon-rich organic phase from oil-contaminated soil, a solvent extraction method based on the procedure described by Li et al. [24] was employed. This procedure utilized a hexane–acetone mixture (3:1 by volume, 25 vol.% acetone), which provides high extraction efficiency for heavy petroleum fractions. In the present study, this ratio was modified to 1:1 in order to increase the polarity of the solvent system, which improves the extraction of more polar components such as resins. In addition, the presence of acetone may promote the disruption of hydrogen bonds between hydrocarbon-rich colloidal structures and the mineral surface, facilitating the release of bound organic matter during solvent extraction [25].

For the extraction procedure, a 10 g sample of oil-contaminated soil was placed in a glass flask, and 50 mL of a solvent mixture (25 mL of each component) was added. The mixture was stirred on a magnetic stirrer at 300 rpm for 30 min at room temperature (20–22 °C) to ensure efficient contact between the solvent and the contaminated soil. After completion of the extraction process, the mixture was allowed to settle and then filtered through a paper filter (grade F, filtration time ≤ 45 s) to separate the organic phase from the solid residue. The selected hexane–acetone solvent system provided an extraction yield of 19.8%, demonstrating its effectiveness for recovering hydrocarbon-rich organic matter from oil-contaminated soil.

(2) The recovered organic phase was subjected to controlled air oxidation in order to increase its molecular complexity and obtain a more structurally stable bitumen-like material suitable for further characterization and utilization.

The oxidation process was carried out in a laboratory stainless-steel reactor equipped with a mechanical stirrer, an air inlet, a pressure gauge, and a digitally controlled heating system.

A total of 300 g of the organic phase was loaded into the reactor. The sample was preheated to 150 °C under a nitrogen atmosphere to remove residual moisture. After this stage, the nitrogen supply was stopped and air bubbling through the sparger was initiated.

Oxidation was carried out at a temperature of 230 ± 5 °C under atmospheric pressure. The air flow rate was 0.3 L/min, and stirring was maintained at 300 rpm. The duration of the process was 120 min. This duration was selected based on preliminary experimental observations and corresponds to the lower boundary of typical air-blowing processes used in bitumen production, where reaction times generally range from 2 to 4 h and are controlled by monitoring the evolution of physicochemical properties, ensuring sufficient oxidative structuring of the material [26]. Upon completion of oxidation, the air supply was stopped, the sample was cooled in the reactor to 100 °C, then discharged and further cooled to room temperature.

(3) The obtained bitumen-like material served as a model product for evaluating potential approaches to improving the properties of materials derived from recovered hydrocarbon resources.

The stiffness adjustment was carried out by blending the recovered bitumen-like material with BND 50/70 road bitumen at a weight ratio of 50:50. The blending was performed at 160–170 °C under constant mechanical stirring until a homogeneous modified binder system was obtained. This approach allowed increasing the proportion of structure-forming asphaltene components and stabilizing the colloidal system.

The physicomechanical properties of the BND 50/70 road bitumen used as the reference binder are presented in Table 1. The values were obtained from the quality certificate provided by the manufacturer.

Table 1. Characteristics of the reference BND 50/70 road bitumen.

Nº	Property	Value
1	Penetration (25 °C), dmm	63
2	Softening point (Ring and Ball), °C	52
3	Fraass breaking point, °C	−25
4	Flash point, °C	270

Polymer modification was carried out using an SBS (styrene–butadiene–styrene) modifier at a concentration of 4 wt.%. SBS is one of the most widely used polymer modifiers for bituminous binders due to its ability to form an elastomeric network within the bitumen matrix, significantly improving the rheological and performance properties of the material [27,28]. The polymer was introduced into the heated bitumen mixture at a temperature of 175–180 °C, followed by intensive stirring for 60 min.

In addition, a combined modification scheme was implemented, involving the simultaneous introduction of the harder BND 50/70 bitumen and the SBS modifier. This approach was intended to improve the structural stability and physicomechanical properties of the bitumen-like material.

The resulting binder systems were cooled to room temperature and subsequently subjected to the analysis of their physicochemical and performance characteristics.

For clarity and consistency throughout the manuscript, the obtained systems were designated as follows: the oxidized bitumen-like material obtained from oil-contaminated soil was denoted as OB; the binder system prepared by blending with BND 50/70 road bitumen was denoted as OB-B; the SBS-modified system was denoted as OB-SBS; and the combined modified system containing both BND 50/70 and SBS was denoted as OB-B-SBS.

2.3. Investigation of the Main Physicochemical Properties of Bitumen-like Material

The recovered bitumen-like material and the corresponding modified systems were characterized using FTIR, NMR, SARA fractionation, physicochemical testing, rotational viscosity measurements, and dynamic shear rheometry (DSR) in order to evaluate their structural, compositional, and performance-related properties.

FTIR spectra of the bitumen-like material obtained from oil-contaminated soil, as well as its modified compositions, were recorded using a Bruker Alpha II spectrometer equipped with an ATR accessory with a diamond crystal. Prior to measurements, background scanning was performed. The samples were preheated to a fluid state and applied as a thin layer onto the crystal surface. The spectra were recorded in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1} and the accumulation of 32 scans.

Nuclear magnetic resonance measurements were performed at 25 °C using a JEOL JNM-ECA 400 spectrometer. The resonance frequencies were 399.78 MHz for ^1H and 100.53 MHz for ^{13}C nuclei. All samples were prepared in deuterated chloroform (CDCl_3). Chemical shifts (δ) were determined relative to the residual solvent signals of CDCl_3 .

The physicochemical properties of the modified binder systems were studied in accordance with established standard methods: the penetration depth at 25 °C was determined according to ASTM D5, the softening point was measured by the ring-and-ball method in accordance with ASTM D36, and the brittleness temperature was determined by the Fraass method according to ASTM D746.

The chemical composition of binder systems was evaluated using SARA fractionation according to ASTM D4124, which separates bitumen into saturates, aromatics, resins, and asphaltenes.

Rotational viscosity measurements were performed at 135 °C using an RT-1 Plus rotational rheometer equipped with a Thermocell heating system. The measurements were carried out at a fixed rotational speed of 60 rpm under identical testing conditions for all binder systems in order to evaluate their flow behavior and resistance to deformation at elevated temperature.

Rheological characterization was performed using a dynamic shear rheometer (DSR, NETZSCH-Gerätebau GmbH, Selb, Germany) in accordance with ASTM D7175. The rutting parameter ($G^*/\sin\delta$) was determined at 46, 52, and 58 °C to evaluate the high-temperature performance of the investigated binder systems.

3. Results and Discussion

3.1. Recovery and Characterization of Hydrocarbon-Rich Organic Phase from Oil-Contaminated Soil

Figure 2 presents the FTIR spectra of the oil-contaminated soil and the purified organic phase extracted from it. Despite the dominant band of 1000–1050 cm^{-1} , corresponding to Si–O–Si stretching vibrations and confirming the silicate nature of the mineral matrix [29], the spectrum also shows bands at 2920–2850 cm^{-1} characteristic of C–H stretching vibrations of aliphatic hydrocarbons, indicating the presence of hydrocarbon-related components present in the soil [30]. In addition, distinct bands are observed at 1540–1550 cm^{-1} , attributed to the vibrations of C–H and C=C bonds in aromatic structures [31,32]. These spectral features suggest the presence of hydrocarbon-rich organic components exhibiting structural similarities to petroleum-derived materials. However, considering the complex nature of oil-contaminated soils, the extracted organic phase may also contain non-petroleum organic constituents.

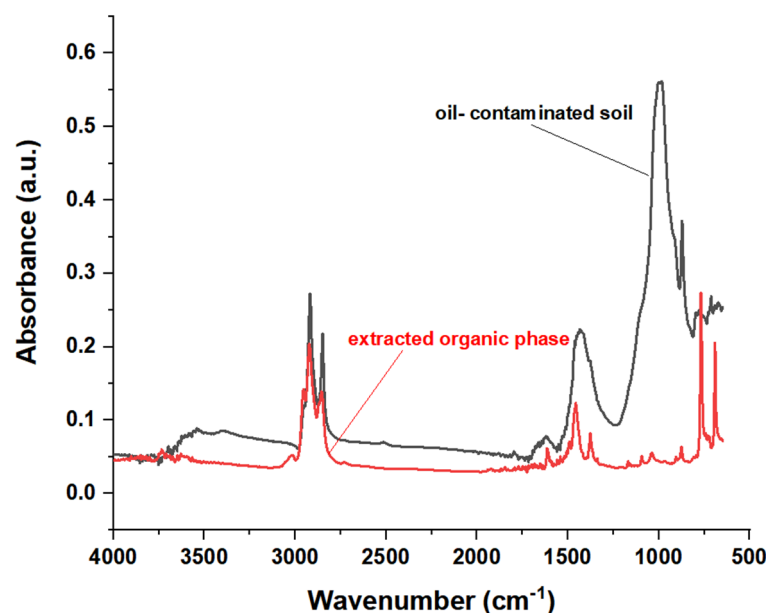


Figure 2. FTIR spectra of oil-contaminated soil and the extracted organic phase, recorded in ATR mode (resolution 4 cm^{-1}).

In the spectrum of the extracted organic phase, the intense band in the $1000\text{--}1100\text{ cm}^{-1}$ region, characteristic of Si–O–Si vibrations, is absent, indicating the effective removal of the silicate mineral component. In addition, the broad band in the $3200\text{--}3600\text{ cm}^{-1}$ region, corresponding to O–H stretching vibrations of hydroxyl groups and adsorbed moisture, is not observed, suggesting the removal of most of the mineral and moisture-associated matrix in the purified phase. The obtained spectrum is predominantly represented by bands characteristic of aliphatic and aromatic hydrocarbon-rich organic structures.

Thus, the results of FTIR spectroscopy and thermogravimetric analysis confirm the efficiency of organic phase extraction from oil-contaminated soil. The absence of bands characteristic of the silicate matrix and hydroxyl groups, together with the reduction in residual mass after thermal decomposition, indicate substantial removal of the mineral component. At the same time, considering the complex nature of oil-contaminated soils, the extracted phase should be regarded as a hydrocarbon-rich organic system rather than a compositionally pure petroleum fraction.

Thermogravimetric analysis (TG), combined with differential thermal analysis (DTA) and derivative thermogravimetric analysis (DTG), is used to determine the thermal stability and phase composition of organo-mineral systems. This method allows the identification of temperature intervals corresponding to dehydration, volatilization, and thermal decomposition of components, making it possible to evaluate the thermal behavior of oil-contaminated soils and the products of their treatment.

On the thermogravimetric curve of the initial oil-contaminated soil (Figure 3), several characteristic stages of mass loss are observed. The first minor mass loss (up to 5%) occurs at temperatures up to $150\text{ }^{\circ}\text{C}$ and is associated with the removal of moisture and volatile organic components. However, this fraction is relatively small and mainly corresponds to light components, while the primary transformations investigated in this study are related to heavier fractions that determine the key physicochemical properties of the material.

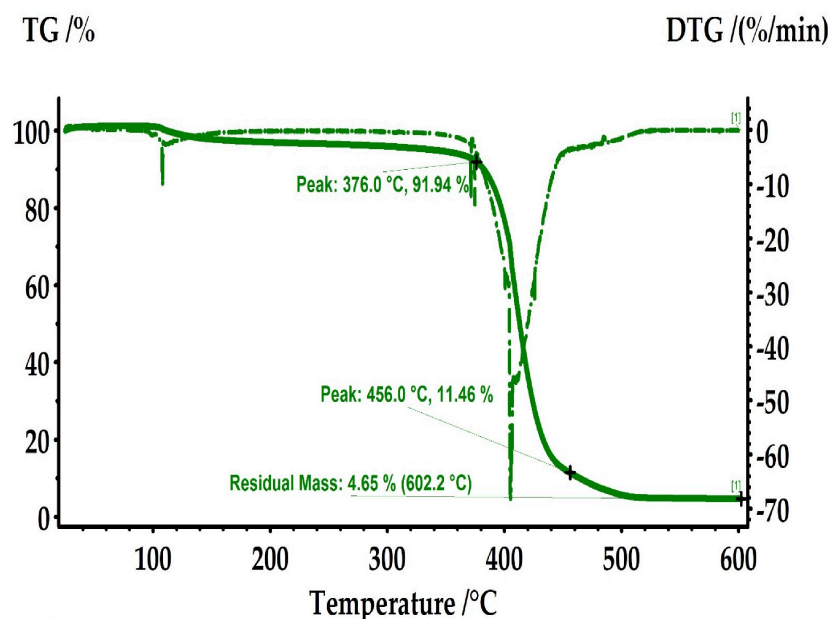


Figure 3. TG curve of oil-contaminated soil, recorded under a nitrogen atmosphere in the temperature range of 50–600 °C at a heating rate of 20 K/min.

The main mass loss occurs in the 350–500 °C range, where pronounced thermal effects are observed on the DTA and DTG curves. The peaks at 405 °C and 492 °C correspond to the decomposition of the organic phase, which includes both aliphatic and aromatic hydrocarbons associated with the mineral matrix. Such behavior is typical of oil-contaminated soils and is associated with the thermal decomposition and desorption of hydrocarbon components within this temperature interval, which is consistent with literature data [33].

The total mass loss is about 95.35%, while the residual mass is 4.65%, indicating that the sample was predominantly composed of thermally decomposable organic matter with a limited amount of thermally stable inorganic residue.

Thermogravimetric analysis of the purified organic phase (Figure 4) shows that the main mass loss occurs in the temperature range of 200–350 °C, which is associated with the thermal decomposition and evaporation of medium- and high-boiling organic components.

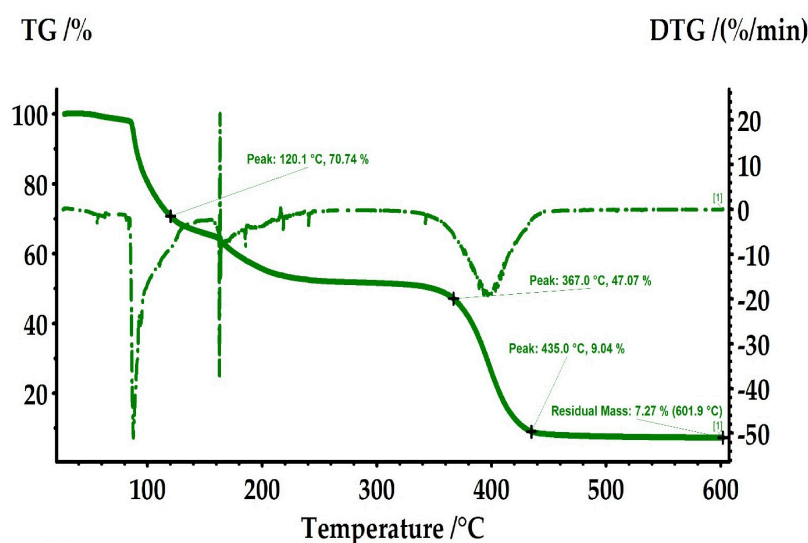


Figure 4. TG curve of the organic phase obtained from oil-contaminated soil, recorded under a nitrogen atmosphere in the temperature range of 50–600 °C at a heating rate of 20 K/min.

Quantitative analysis of the TG curves reveals significant differences in the thermal behavior of the initial oil-contaminated soil and the extracted organic phase. For the purified organic phase, a mass loss of about 53% is observed already at 367 °C, while in the range of 367–435 °C an additional ~38% is removed, reflecting the sequential thermal decomposition of medium- and high-boiling organic components.

In contrast, for the initial soil about 92% of the initial mass is retained up to 376 °C, indicating stabilization of the organic phase by the mineral matrix. The main mass loss occurs in a higher temperature range (up to ~456 °C), where the organic phase associated with the mineral matrix decomposes.

Thus, extraction leads to the concentration of the organic fraction and the elimination of the mineral influence, which is confirmed by the shift in the temperature intervals of decomposition and the change in the character of the thermogravimetric curves.

These results indicate that oil-contaminated soil contains a considerable amount of recoverable hydrocarbon-rich organic matter that may be regarded as a potential secondary resource.

3.2. Structural Characterization of Recovered Organic Material and Its Modified Forms

The recovered oxidized product, hereafter referred to as the bitumen-like material (OB), was obtained from the hydrocarbon-rich organic phase extracted from oil-contaminated soil. The term bitumen-like material is used because the obtained product exhibits characteristic chemical, thermal, physicochemical, and rheological features commonly associated with petroleum-derived bituminous materials, although it differs from conventional paving bitumen in its origin and composition. The recovered bitumen-like material (OB) was further investigated to evaluate its structural characteristics and potential for subsequent utilization. Preliminary physicochemical testing indicated that the material exhibited relatively low viscosity and limited resistance to deformation at elevated temperatures, suggesting the need for additional structural modification.

To evaluate the possibility of improving the performance characteristics of the recovered material, different modification approaches were investigated. Blending with the harder road bitumen (grade BND 50/70) was expected to increase structural stiffness and resistance to deformation due to the incorporation of a more structured binder component. In addition, modification with SBS (4 wt.%) was applied because this polymer is widely used for improving the rheological and operational characteristics of bituminous materials [34]. According to the literature, SBS contents in the range of 3–5 wt.% are considered optimal for enhancing elasticity and resistance to thermo-oxidative aging without excessive viscosity increase [34,35].

A combined modification approach involving both BND 50/70 and SBS was also investigated in order to evaluate the possibility of obtaining a more balanced combination of physicochemical properties.

Considering the observed changes in material behavior, further structural characterization was performed using FTIR and NMR spectroscopy. These methods provide information on the major functional groups and structural fragments present in complex hydrocarbon-rich systems and allow comparative evaluation of compositional changes occurring during modification.

The FTIR spectra of the bitumen-like material obtained from oil-contaminated soil and its modified forms (Figure 5) demonstrate a set of functional groups typical of bituminous binders. In all samples, intense bands are observed at 2920 and 2850 cm^{-1} , corresponding to C–H stretching vibrations in aliphatic fragments [36], as well as bands at 1460 and 1375 cm^{-1} associated with the deformation vibrations of CH_2 and CH_3 groups. The presence of a band in the region of about 1600 cm^{-1} indicates the presence of aromatic structures [19].

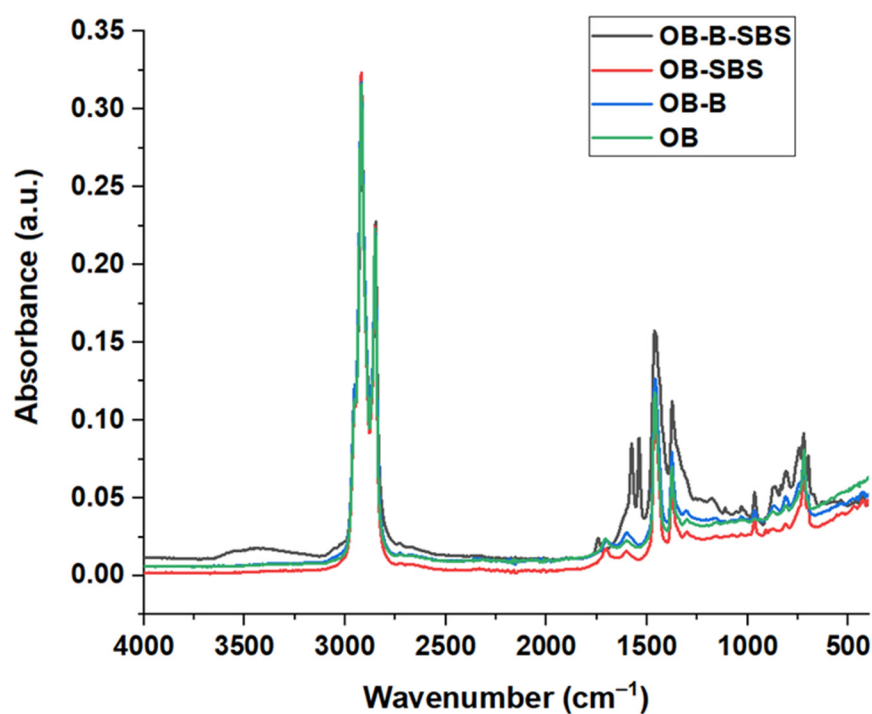


Figure 5. FTIR spectra of OB and its modified systems, recorded in ATR mode (resolution 4 cm^{-1}).

The absence of an intense band in the $1000\text{--}1100\text{ cm}^{-1}$ region, characteristic of Si–O–Si vibrations [37], indicates the effective removal of the mineral matrix and suggests enrichment in organic components. Thus, the obtained product exhibits structural features typical of bituminous materials; however, considering its origin, it should be regarded as a hydrocarbon-rich organic system with properties similar to conventional bituminous binders.

At the same time, it should be noted that the relatively smooth and well-defined spectral features may not fully reflect the intrinsic compositional complexity of soil-derived organic extracts. Such systems may still contain structurally diverse components, including soil-associated organic matter, which can overlap within the same spectral regions. Therefore, the FTIR data should be interpreted as indicative of dominant hydrocarbon-rich structures rather than as evidence of a chemically uniform or purely petroleum-derived material.

The FTIR spectra obtained in the present study indicate that blending with BND 50/70 did not result in the appearance of new absorption bands corresponding to new functional groups. However, it is accompanied by changes in the relative intensity of the bands, reflecting a redistribution of molecular fragments and an increase in the proportion of high-molecular-weight components in the system.

In the samples modified with 4% SBS (OB-SBS), the appearance or intensification of a band in the $\sim 966\text{ cm}^{-1}$ region is observed, which is characteristic of the butadiene segment of SBS, confirming the presence of the polymer phase in the binder matrix [38]. At the same time, the bands corresponding to aromatic and aliphatic fragments are preserved, indicating the formation of a composite structure without destruction of the original binder matrix.

Combined modification with BND 50/70 and SBS demonstrates the most pronounced changes in the region of polymer markers while preserving the characteristic features of the organic matrix, indicating successful incorporation of the modifying components into the recovered material.

The FTIR spectroscopy results were complemented by ^1H and ^{13}C NMR analysis, providing additional information on the distribution of major structural fragments within the recovered material and its modified forms.

In the ^1H NMR spectra (Figure 6a), the region 0.5–1.0 ppm was assigned to protons of methyl groups in saturated compounds (γ -position relative to the aromatic ring), while the range 1.0–2.0 ppm corresponds to protons of methylene and methine groups of aliphatic chains. Signals in the 2.0–4.0 ppm region are associated with protons in the α -position relative to aromatic rings, carbonyl groups, or heteroatoms. Aromatic protons are located in the 6.0–9.0 ppm range [39]. The absence of pronounced intensity in the 4.5–6.0 ppm interval indicates a low content of olefinic fragments in the studied material.

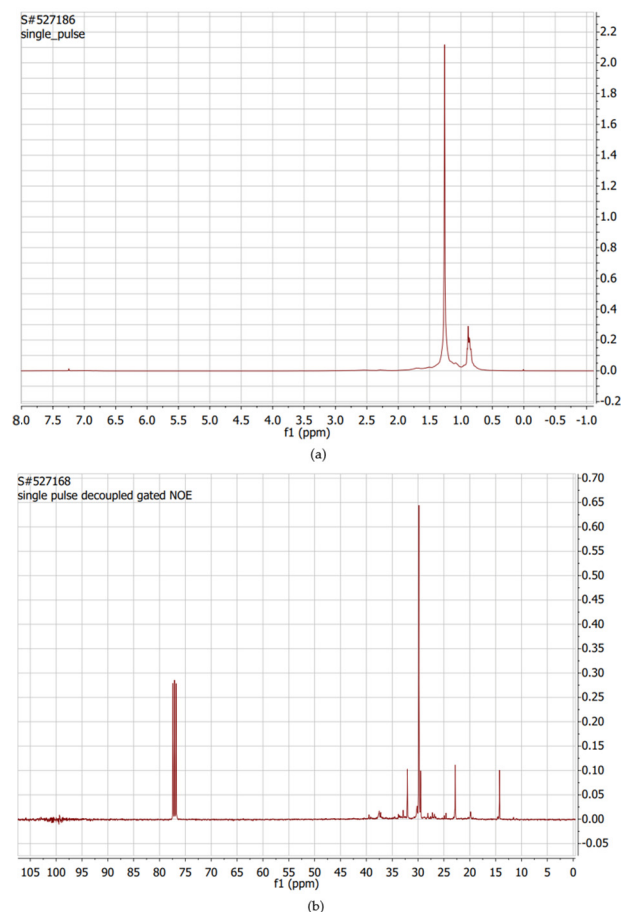


Figure 6. (a) ^1H and (b) ^{13}C NMR spectra of OB, recorded in CDCl_3 at 25 °C (399.78 MHz for ^1H and 100.53 MHz for ^{13}C).

Signals in the ^{13}C NMR spectra (Figure 6b) in the 7–50 ppm region correspond to aliphatic carbons of saturated structures, whereas the 110–160 ppm range reflects the presence of aromatic carbon atoms [40]. The 170–200 ppm region may be associated with carbonyl groups, although their contribution in the studied samples is insignificant.

At the same time, it should be noted that NMR spectra provide averaged structural information and may not fully resolve the compositional complexity of soil-derived organic systems, which can include a mixture of hydrocarbon-rich organic components and natural organic matter.

Thus, the ^1H and ^{13}C NMR data indicate that the obtained material exhibits a mixed aliphatic–aromatic structure characteristic of hydrocarbon-rich bituminous systems, while retaining a complex and heterogeneous molecular composition associated with its origin.

The analysis of the ^1H and ^{13}C NMR spectra (Figure 7) of the mixture of the bitumen-like material obtained from oil-contaminated soil with BND 50/70 road bitumen (OB-B) showed the preservation of aliphatic and aromatic regions characteristic of binder systems, without the appearance of new functional signals. In the ^1H NMR spectrum, the region 0.5–2.0 ppm, corresponding to methyl and methylene protons, predominates, whereas the

intensity of the aromatic region (6.0–9.0 ppm) changes only slightly, indicating the absence of chemical transformations and confirming the physical nature of the blending process.

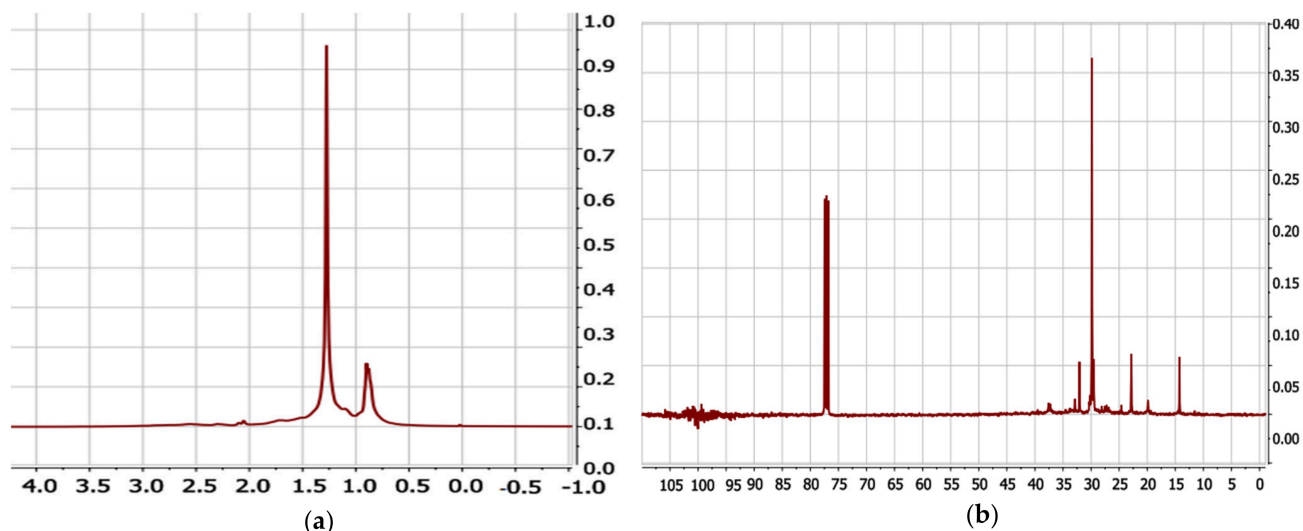


Figure 7. (a) ^1H and (b) ^{13}C NMR spectra of the OB-B, recorded in CDCl_3 at 25 °C (399.78 MHz for ^1H and 100.53 MHz for ^{13}C).

A similar pattern is observed in the ^{13}C NMR spectra: the predominance of signals in the aliphatic range (7–50 ppm) is preserved, while aromatic carbon atoms remain present in the 110–160 ppm region. The redistribution of relative intensities between these regions reflects changes in the ratio of structural fragments as a result of blending; however, the absence of new signals indicates the formation of a homogeneous composite system without chemical reactions between the components. It should be noted that the NMR spectra reflect averaged structural features of the system and may not fully capture the complexity of interactions between components, especially considering the heterogeneous nature of the initial organic phase.

In the ^1H NMR spectrum of the binder system modified with SBS—OB-SBS (Figure 8a), a signal is observed in the aromatic region with a maximum at ~6.9–7.0 ppm, which corresponds to protons of aromatic structures and may be associated with the presence of styrenic fragments of the polymer [41].

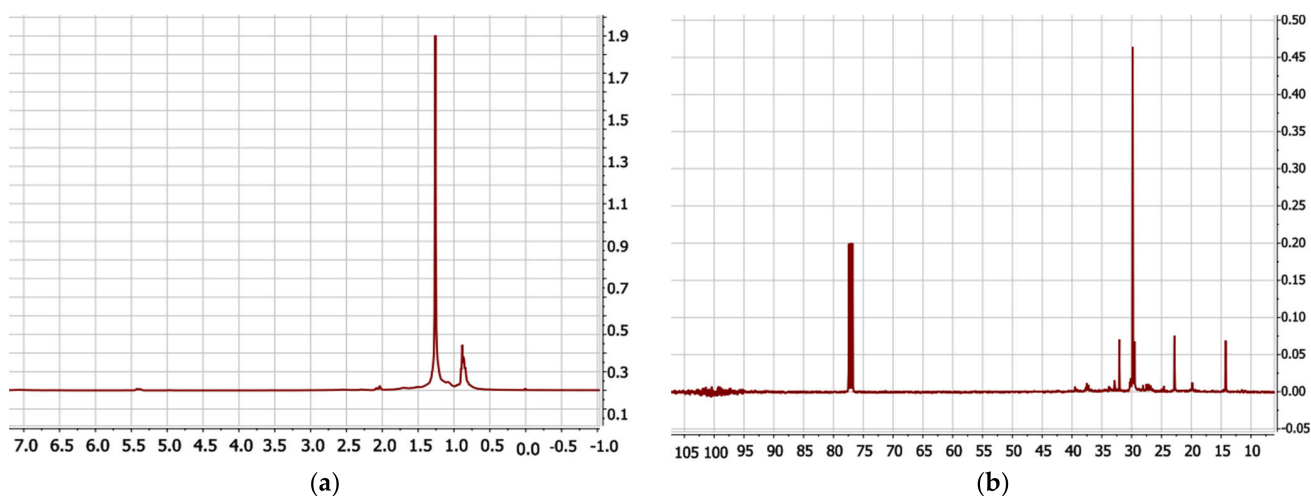


Figure 8. (a) ^1H and (b) ^{13}C NMR spectra of the OB-SBS, recorded in CDCl_3 at 25 °C (399.78 MHz for ^1H and 100.53 MHz for ^{13}C).

For the combined modified binder system, OB-B-SBS (Figure 9a), this signal becomes more pronounced, indicating an increased contribution of aromatic structures in the system. The enhancement of the aromatic region may result from the combined effect of a stiffer binder matrix and the presence of styrenic blocks of SBS, while the overall spectral pattern remains typical of binder systems.

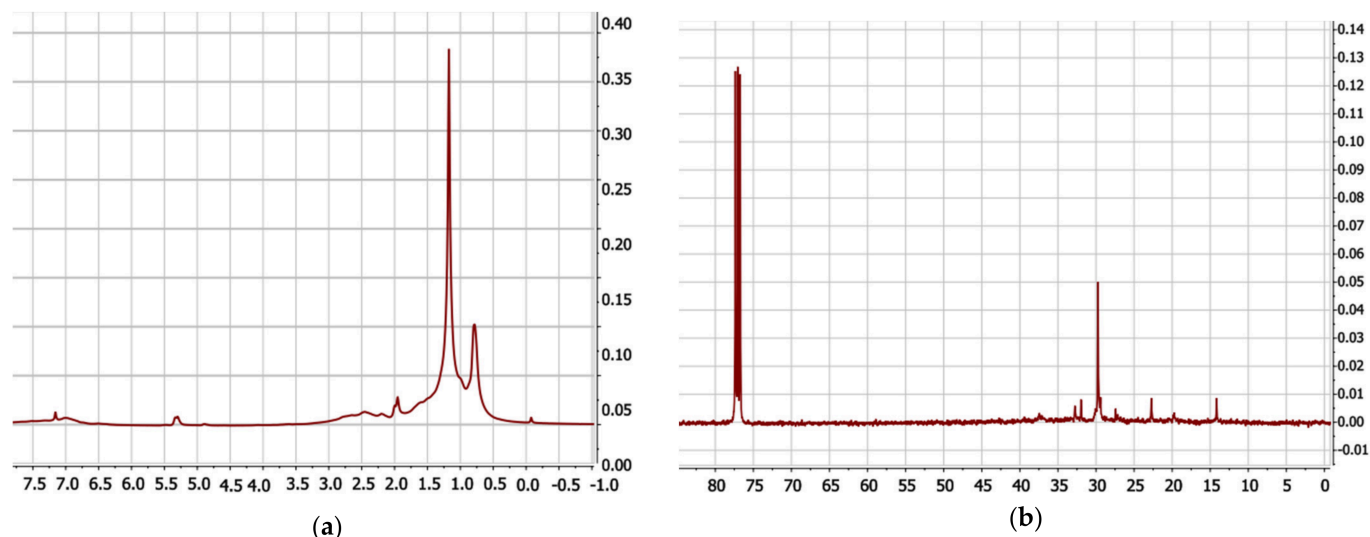


Figure 9. (a) ^1H and (b) ^{13}C NMR spectra of the OB-B-SBS, recorded in CDCl_3 at 25 °C (399.78 MHz for ^1H and 100.53 MHz for ^{13}C).

In the ^{13}C NMR spectra of the SBS-modified samples (Figures 8b and 9b), the characteristic distribution of signals in the aliphatic (7–50 ppm) and aromatic (110–160 ppm) regions is preserved. At the same time, a more pronounced intensity of signals is observed in the aromatic range, which may be associated with the presence of styrenic blocks of SBS. The absence of signals in new chemical shift regions confirms that the modification is not accompanied by the formation of additional functional groups and is predominantly compositional in nature.

At the same time, the observed spectral features should be interpreted as indicating structural changes in a complex system, rather than the formation of a chemically homogeneous structure.

Overall, the combined FTIR and NMR results indicate that the thermal oxidation treatment of the recovered organic phase did not result in substantial alteration of its primary hydrocarbon framework. The recovered material retained the characteristic aliphatic and aromatic structural fragments typical of bituminous systems, while subsequent modification with BND 50/70 and SBS was predominantly compositional in nature and was not accompanied by the formation of new dominant functional groups. These findings support the potential use of the recovered material as a bitumen-like binder.

The obtained structural data confirm the predominantly compositional character of the modification and substantiate the need for further investigation of the physicomechanical and performance properties of the resulting binders. At the same time, the observed spectral intensity changes should be interpreted with caution, since the NMR spectra provide averaged structural information and may not fully capture the complexity of interactions in such a complex system.

3.3. Physicomechanical and Rheological Properties of Recovered Bitumen-like Materials

The analysis of physicomechanical properties (Table 2) revealed a consistent change in properties at different stages of modification. The OB is characterized by a high penetration

depth (286.5×10^{-1} mm) and a relatively low softening point (32.1 °C), indicating its increased softness and limited thermal resistance.

Table 2. Main physicommechanical characteristics of the studied bituminous binders.

Nº	Parameter	OB	OB-B	OB-SBS	OB-B-SBS
1	Penetration at 25 °C, 0.1 mm	286.5 ± 1.1	284.0 ± 1.2	308.0 ± 0.9	321.0 ± 1.0
2	Softening point (Ring and Ball), °C	32.1 ± 0.1	34.7 ± 0.2	37.7 ± 0.2	40.6 ± 0.4
3	Fraass brittleness temperature, °C	-50.0 ± 0.5	-45.0 ± 0.5	-47.0 ± 0.3	-43.0 ± 0.3
4	Rotational viscosity at 135 °C, Pa·s	3.11 ± 0.07	6.78 ± 0.05	14.31 ± 0.05	101.50 ± 0.05

Values are presented as mean $\pm$ standard deviation ($n = 3$).

Blending with the harder BND 50/70 bitumen (OB-B) leads to a decrease in penetration to 284×10^{-1} mm and an increase in the softening point to 34.7 °C, indicating an increase in the structural stiffness of the system. The introduction of SBS into the bitumen-like material (OB-SBS) is accompanied by an increase in the softening point to 37.7 °C and a change in penetration to 308×10^{-1} mm, reflecting the redistribution of structural components and possible polymer-induced structuring within the binder system, which contributes to improved thermal resistance.

The most pronounced effect is observed for the combined modified binder system (OB-B-SBS), where the maximum softening point (40.6 °C) is achieved while maintaining acceptable penetration values (321×10^{-1} mm). This indicates an effective combination of increased matrix stiffness and the modifying influence of the polymer phase.

The Fraass brittleness temperature ranged from -50 to -43 °C. Although the modified binders exhibited slightly higher Fraass temperatures, the obtained values indicate that satisfactory low-temperature flexibility was maintained. Since the Fraass brittleness temperature is a widely accepted indicator of the low-temperature cracking resistance of bituminous binders, the results suggest that the modification did not significantly impair their resistance to thermal cracking [42,43].

In addition, rotational viscosity measurements performed at 135 °C demonstrated a significant increase in viscosity after modification of the binder systems. The initial bitumen-like material (OB) exhibited the lowest viscosity (3.11 Pa·s), whereas blending with BND 50/70 increased the viscosity to 6.78 Pa·s. Modification with SBS resulted in a more pronounced increase in viscosity (14.31 Pa·s), which may be associated with polymer swelling and increased resistance to flow within the binder matrix. The combined modified system (OB-B-SBS) exhibited the highest viscosity value (101.5 Pa·s), indicating substantial structural reinforcement and possible polymer-induced structuring of the system.

To further evaluate the rheological behavior of the obtained binder systems, Dynamic Shear Rheometer (DSR) measurements were performed. The rutting parameter ($G^*/\sin\delta$) was determined at different temperatures and used as an indicator of resistance to permanent deformation at elevated temperatures. The results are presented in Table 3.

Table 3. Rheological properties of the investigated binder systems determined by DSR.

Sample	$G^*/\sin\delta$ at 46 °C, kPa	$G^*/\sin\delta$ at 52 °C, kPa	$G^*/\sin\delta$ at 58 °C, kPa
OB	1.40 ± 0.05	0.7 ± 0.08	0.4 ± 0.05
OB-B	1.70 ± 0.05	0.9 ± 0.08	0.5 ± 0.05
OB-SBS	2.6 ± 0.08	1.6 ± 0.07	0.8 ± 0.07
OB-B-SBS	4.2 ± 0.08	2.2 ± 0.08	1.3 ± 0.09

Values are presented as mean $\pm$ standard deviation ($n = 3$).

The unmodified OB and OB-B samples exhibited $G^*/\sin\delta$ values above 1.0 kPa only at 46 °C, indicating limited resistance to rutting at elevated temperatures. In contrast, the SBS-

modified systems demonstrated improved rheological performance. The OB-SBS sample maintained $G^*/\sin\delta$ values above 1.0 kPa up to 52 °C, whereas the combined modified system (OB-B-SBS) retained values above this criterion even at 58 °C.

These results demonstrate that SBS modification substantially improves the high-temperature performance of the recovered bitumen-like materials and enhances their resistance to permanent deformation under elevated service temperatures.

The observed changes in physicochemical and rheological properties are consistent with the compositional differences identified by FTIR and NMR analyses. Together, these results indicate that the properties of the recovered bitumen-like material can be effectively adjusted through modification with road bitumen and SBS.

3.4. Compositional Changes in Recovered Bitumen-like Materials According to SARA Fractionation

To further investigate the compositional characteristics of the recovered bitumen-like material and its modified forms, SARA fractionation was performed to determine the distribution of saturates, aromatics, resins, and asphaltenes. Since the balance between these fractions strongly influences the physicochemical and rheological behavior of hydrocarbon-rich materials, SARA analysis provides valuable information regarding compositional changes occurring during modification.

The results of the fractional composition analysis are presented in Table 4. Modification of the recovered material with road bitumen and SBS resulted in noticeable redistribution of the major SARA fractions.

Table 4. Distribution of SARA fractions in recovered bitumen-like materials and modified systems.

Nº	Content, wt. %	OB	OB-B	OB-SBS	OB-B-SBS
1	Asphaltenes	26.5 ± 0.1	21.3 ± 0.1	21.0 ± 0.2	18.1 ± 0.1
2	Resins	34.4 ± 0.2	32.2 ± 0.3	36.8 ± 0.2	35.7 ± 0.3
3	Saturates	12.5 ± 0.1	14.8 ± 0.1	13.1 ± 0.1	13.8 ± 0.2
4	Aromatics	26.6 ± 0.1	31.7 ± 0.2	29.1 ± 0.4	32.4 ± 0.3

Values are presented as mean ± standard deviation ($n = 3$).

The addition of road bitumen resulted in a decrease in the asphaltene content from 26.5 to 21.3 wt.% accompanied by corresponding changes in the aromatic and resin fractions. These changes indicate a redistribution of the major components within the hydrocarbon-rich matrix and suggest modification of the compositional balance of the recovered material.

A similar trend was observed for the SBS-modified systems. In particular, the resin content increased to 36.8 wt.% while the asphaltene fraction decreased to 21.0 wt.%. According to literature data [32], SBS modification is commonly associated with redistribution of maltene components and interactions between the polymer phase and aromatic-resin fractions. Such changes may contribute to the improved physicochemical and rheological properties observed for the modified materials.

Despite the decrease in asphaltene content after modification, the OB-B-SBS sample exhibited the highest rotational viscosity and $G^*/\sin\delta$ values. This indicates that the performance of the modified binders is governed not only by the asphaltene content but also by the overall balance of the SARA fractions. In particular, the aromatic fraction serves as the dispersion medium for SBS swelling, improving polymer–bitumen compatibility and promoting the formation of a more homogeneous binder structure [34,43]. Therefore, the enhanced rheological performance of the OB-B-SBS sample may be attributed not only to changes in the asphaltene content but also to the redistribution of the SARA fractions following modification. Furthermore, according to the literature [44], the formation of a three-dimensional elastomeric SBS network within the bitumen matrix con-

tributes to increased viscosity, a higher complex shear modulus, and improved resistance to permanent deformation.

It should be noted that the economic feasibility of the proposed approach should be considered in the context of the growing demand for bituminous materials and the limited availability of conventional petroleum resources [45]. For example, in Kazakhstan, annual bitumen production capacities are estimated at approximately 1.0 million tons, whereas the demand of the road construction sector reaches about 1.3 million tons, resulting in a persistent supply deficit that is partially compensated through imports [46].

At the same time, large-scale transport infrastructure development programs, including the construction and reconstruction of thousands of kilometers of highways, continue to increase the demand for bituminous materials. Under such conditions, the recovery of hydrocarbon-rich organic matter from oil-contaminated soils may represent an additional source of raw materials while simultaneously reducing the environmental burden associated with the long-term accumulation of petroleum-containing wastes.

Therefore, despite the energy-intensive nature of certain processing stages, the proposed approach should be considered not only from the perspective of direct production costs but also in terms of resource recovery, waste valorization, and long-term environmental sustainability.

From an environmental perspective, solvent extraction is considered a promising technology for the treatment of petroleum-contaminated wastes, combining relatively low energy consumption, mild operating conditions, and the recovery of valuable hydrocarbons as secondary resources [47,48]. An additional advantage of this approach is the possibility of regenerating and reusing organic solvents, thereby reducing solvent consumption, minimizing secondary waste generation, and improving the overall sustainability of the process [48,49]. Furthermore, the recovery of the hydrocarbon-rich organic phase from oil-contaminated soil not only reduces the amount of petroleum-containing waste but also enables the recovered hydrocarbons to be reused as a feedstock for the production of bitumen-like materials, in accordance with the principles of resource recovery and the circular economy [47]. When implemented in closed processing systems, solvent recovery and recycling may also contribute to minimizing volatile organic compound (VOC) emissions to the environment [48,49].

The proposed process employs conventional unit operations, including solvent extraction, oxidation, and blending, which are widely used in the petroleum industry. Therefore, the approach has potential for further scale-up after process optimization and techno-economic evaluation.

The primary objective of the present study was not the complete environmental remediation of oil-contaminated soil, but the recovery and valorization of the hydrocarbon-rich organic phase as a potential secondary resource for the production of bitumen-like materials. In recent years, research on petroleum-containing wastes has increasingly shifted from conventional disposal strategies toward resource recovery and waste valorization, recognizing these materials as valuable secondary feedstocks rather than solely hazardous wastes [50,51]. Within this context, the proposed approach aims to recover valuable hydrocarbons while simultaneously reducing the amount of petroleum-containing waste requiring further treatment. Nevertheless, the environmental assessment of the treated soil, including the determination of residual petroleum hydrocarbon content and its compliance with regulatory requirements for reuse or disposal, was beyond the scope of the present study and requires dedicated future investigation.

4. Conclusions

The present study demonstrated the feasibility of recovering a hydrocarbon-rich organic phase from oil-contaminated soil with an extraction yield of 19.8 wt.% using a white spirit–acetone solvent system. FTIR, NMR, and thermogravimetric analyses confirmed the presence of structural fragments characteristic of petroleum-derived materials and the effective separation of the organic fraction from the mineral matrix.

The recovered organic phase was successfully converted into a bitumen-like material through controlled oxidation. SARA fractionation demonstrated that modification significantly altered the composition of the recovered material. In the OB-B-SBS system, the asphaltene content decreased from 26.5 to 18.1%, while the aromatic fraction increased from 26.6 to 32.4%. These compositional changes were accompanied by a substantial improvement in performance, including an increase in the softening point from 32.1 to 40.6 °C, rotational viscosity from 3.11 to 101.50 Pa·s.

Although the obtained results demonstrate the feasibility of the proposed approach, further studies are required to evaluate its large-scale implementation, economic viability, and the environmental characteristics of the treated mineral residue.

Overall, the obtained results indicate that oil-contaminated soils should be considered not only as environmentally hazardous wastes requiring remediation but also as a potential source of recoverable hydrocarbon-rich materials. The proposed approach combines waste remediation with resource recovery and may contribute to the development of sustainable technologies for the utilization of petroleum-containing wastes within a circular economy framework.

5. Patents

The results presented in this study are related to Utility Model Patent No. 12106 (Republic of Kazakhstan), entitled “Method for Producing Oxidised Bitumen”, published on 24 April 2026.

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Abbreviations

The following abbreviations are used in this manuscript:

NMR	nuclear magnetic resonance
TGA	thermogravimetric analysis
FTIR	Fourier transform infrared spectroscopy
SARA	Saturates, Aromatics, Resins, Asphaltenes
SBS	Styrene–Butadiene–Styrene
BND	Bitumen for Road Applications (road petroleum bitumen)

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