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Study of the physicochemical properties of bitumen extracted from natural rock deposits

Badanie właściwości fizykochemicznych bitumu pozyskanego z naturalnych złóż skalnych

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ABSTRACT: This study investigates the physicochemical characteristics of bitumen from natural rock deposits and evaluates its potential for road construction following chemical modification. Bitumen was recovered from rock samples using a solvent extraction method, with a yield of 22.5–24.5% by weight. The raw bitumen was subsequently modified with formaldehyde at a temperature of 120°C and a pressure of 15 atm to improve its physicochemical performance. Fourier-transform infrared spectroscopy (FTIR) analysis confirmed the introduction of functional groups such as carbonyl (C=O) and hydroxyl (C–OH), indicating successful chemical modification. Isotopic composition analysis ($\delta^{13}C$) revealed notable heterogeneity between the upper Malta layer and the lower oil-bearing layer, suggesting diverse organic origins and varying degrees of biodegradation within the deposit. Scanning electron microscopy (SEM) was employed to examine the surface morphology of the rock–bitumen interface. The SEM images revealed a cracked, porous structure that may influence the adhesion properties of the bitumen. These results suggest that the modified natural bitumen possesses a reactive chemical structure and complex morphology, making it a viable alternative binder in road construction. The improved structural stability demonstrated in the experiments further supports its suitability for industrial applications, particularly asphalt production and road paving. This research highlights the potential of chemically modified natural bitumen as a sustainable alternative to conventional binders, contributing to the effective utilization of hydrocarbon-rich rocks.

Key words: natural bitumen, solvent extraction, chemical modification, FTIR analysis, SEM-EDS, sustainable binder.

STRESZCZENIE: W niniejszym badaniu przeanalizowano właściwości fizykochemiczne bitumu pozyskanego z naturalnych złóż skalnych oraz oceniono jego potencjalne zastosowanie w budownictwie drogowym po modyfikacji chemicznej. Bitum otrzymano z próbek skalnych metodą ekstrakcji rozpuszczalnikowej, uzyskując wydajność w zakresie od 22,5% do 24,5% wagowo. Surowy bitum został następnie zmodyfikowany formaldehydem w temperaturze 120°C i pod ciśnieniem 15 atm w celu poprawy jego właściwości fizycznych i chemicznych. Analiza metodą spektroskopii w podczerwieni z transformacją Fouriera (FTIR) potwierdziła wprowadzenie grup funkcyjnych, takich jak karbonylowa (C=O) i hydroksylowa (C–OH), co wskazuje na pomyślną modyfikację chemiczną. Analiza składu izotopowego ($\delta^{13}C$) ujawniła znaczną niejednorodność między górną warstwą formacji Malta a dolną warstwą roponośną, co sugeruje zróżnicowane pochodzenie organiczne oraz różny stopień biodegradacji w obrębie złoża. Do zbadania morfologii powierzchni na granicy skała–bitum wykorzystano skaningową mikroskopię elektronową (SEM). Obrazy SEM ujawniły spękaną, porowatą strukturę, która może wpływać na właściwości adhezyjne bitumu. Wyniki wskazują, że zmodyfikowany bitum naturalny charakteryzuje się reaktywną strukturą chemiczną oraz złożoną morfologią, co czyni go realnym alternatywnym spoiwem do zastosowań w budownictwie drogowym. Poprawa stabilności strukturalnej wykazana w eksperymentach dodatkowo potwierdza jego przydatność do zastosowań przemysłowych, zwłaszcza w produkcji asfaltu i materiałów do budowy nawierzchni drogowych. Badanie to podkreśla potencjał chemicznie zmodyfikowanego bitumu naturalnego jako zrównoważonej alternatywy dla konwencjonalnych spoiw, przyczyniając się do efektywnego wykorzystania skał bogatych w węglowodory.

Słowa kluczowe: bitum naturalny, ekstrakcja rozpuszczalnikowa, modyfikacja chemiczna, analiza FTIR, SEM-EDS, zrównoważone spoiwo.

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Introduction

Bitumen is a complex hydrocarbon material widely used in road construction, waterproofing systems, and other infrastructure applications due to its adhesive, water-resistant, and viscoelastic properties (Blab et al., 2024; Alfe et al., 2025). Conventionally, most of the bitumen used in industry is obtained through the fractional distillation of crude oil. However, with rising global concerns over petroleum resource depletion, increasing extraction and processing costs, and environmental sustainability, the exploration of alternative and renewable sources of bitumen has become increasingly relevant (Cheng et al., 2024).

One such alternative is the extraction of bitumen from natural geological formations. Natural bitumen, also referred to as native or rock bitumen, is found in the pores and cracks of sedimentary rocks such as limestone, sandstone, and shale (Dyuryagina et al., 2024). Over geological timescales, organic matter trapped within these rocks undergoes transformation under the influence of temperature, pressure, and microbial activity, leading to the formation of bituminous substances (Dyuryagina et al., 2025). These naturally occurring bitumens vary in composition depending on the source rock, age, and environmental conditions, and are often complex mixtures of high-molecular-weight hydrocarbons, resins, and asphaltenes (Ewa et al., 2024, Guo et al. 2025).

Natural bitumen deposits are of particular interest in countries where petroleum resources are scarce or costly to exploit. Utilizing such local sources can offer economic and logistical advantages, especially in remote or developing regions (Ramdhani et al., 2024). Moreover, natural bitumen may possess distinct physicochemical characteristics that influence its behavior in construction materials, potentially offering enhanced thermal stability, lower oxidative aging, or improved compatibility with certain fillers and aggregates (Demchuk et al., 2024; Shinde et al., 2024).

To assess the practical applicability of natural bitumen in road engineering or other industrial uses, it is essential to study its physicochemical properties comprehensively. These include parameters such as: softening point, penetration value, ductility, flash point, fire point, solubility in organic solvents, and elemental composition (Syrmanova et al., 2024; Temirov et al., 2025). Understanding these characteristics is fundamental for evaluating whether natural bitumen can meet technical standards and perform effectively under varying environmental and mechanical stresses (Zhang et al., 2025).

Furthermore, the chemical composition and microstructural behavior of natural bitumen can significantly influence its interaction with mineral aggregates, moisture sensitivity, and long-term durability in pavement structures. Advanced analytical techniques such as: Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM), are useful tools for identifying functional groups, thermal decomposition behavior, and surface morphology, respectively (Caputo et al., 2020). These analyses provide essential insights into how the molecular structure of natural bitumen affects its macroscopic performance.

In addition, comparing the performance of naturally sourced bitumen with that of conventional petroleum bitumen allows researchers to determine its potential as a viable substitute or additive. It also opens the door to hybrid formulations in which natural and petroleum-derived bitumen are blended to optimize performance characteristics while reducing environmental impacts and material costs (Airey 2003; Temirov et al., 2023a).

This study is thus motivated by the need to explore environmentally sustainable and economically efficient alternatives to petroleum-based bitumen (Lu and Isacsson 1997; Ao et al., 2012). By examining bitumen extracted from natural rock sources and assessing its physicochemical characteristics, the research aims to contribute to the development of local, renewable materials for construction purposes. The outcomes of this work may support future decisions related to road material formulations, infrastructure planning, and resource utilization in regions rich in natural bitumen deposits (Temirov et al., 2023b).

Experimental Part

In this study, two natural rock samples containing bituminous material were collected from a designated geological formation. The bitumen was extracted from the rock matrix using a solvent extraction method. Specifically, chloroform ($CHCl_3$) was used as the extracting solvent due to its high efficacy in dissolving organic bituminous components.

Approximately 100 g of crushed rock sample (particle size <2 mm) was placed into a Soxhlet extraction apparatus and continuously extracted with analytical-grade chloroform for 8 hours. The extraction process was carried out under controlled heating using a reflux condenser and heating mantle set to maintain the solvent's boiling point (61.2°C). After extraction, the chloroform solution was subjected to rotary evaporation at reduced pressure using a Buchi Rotavapor R-300 to recover the solvent and concentrate the bituminous extract. The yield of chloroform-soluble material was 22.5–24.5% by weight of the original rock sample.

The obtained bitumen extract was then subjected to further fractional separation to isolate the maltene and asphaltene fractions. Asphaltenes were precipitated by adding *n*-heptane at a 1:40 ratio and allowing the mixture to stand for 24 hours.

The soluble maltene fraction was separated by filtration, while the asphaltene residue was washed and dried.

To enhance the elasticity and flexibility of the extracted bitumen, it was chemically modified through reaction with formaldehyde. In a stainless-steel high-pressure reactor (Parr Instrument, 300 mL capacity), 50 g of extracted bitumen was mixed with a formalin solution (37% aqueous formaldehyde) at a 1:1 molar ratio relative to reactive aromatic units. The reaction was conducted under 15 atm pressure at a temperature of 120°C for 6 hours. This process led to the formation of methylol ($–CH_2OH$) functional groups via electrophilic substitution of the aromatic nuclei present in the asphaltene structures.

After completion of the methylolation reaction, the product was transferred to a glass reaction vessel and subjected to thermal dehydration in an open system at 120°C for 4 hours to remove water molecules and promote condensation reactions between methylal groups. This step converted the methylenated asphaltenes into a cross-linked polymeric network, transforming their rigid, glassy microstructure into a more flexible and elastic matrix, akin to that of petroleum-based, polymer-modified binders.

In addition, the presence and distribution of the ^{13}C isotope were determined using ^{13}C Nuclear Magnetic Resonance (NMR) spectroscopy. The analyses were performed on a Bruker AVANCE 400 MHz spectrometer, operating at a frequency of 100.6 MHz for ^{13}C nuclei, using deuterated solvent ($CDCl_3$). Chemical shifts were referenced to tetramethylsilane (TMS = 0 ppm) as the internal standard. The observed signal peaks provided information on the presence of ^{13}C nuclei and their chemical environment. Where necessary, the $^{13}C/^{12}C$ isotope ratio was further examined using isotope ratio mass spectrometry (IRMS).

As a result of these modifications, the naturally extracted bitumen was transformed into a material with enhanced viscoelastic behavior, improved adhesion potential, and better performance characteristics suitable for road engineering applications.

To evaluate the chemical structure and surface morphology of both the extracted natural bitumen and conventional petroleum bitumen (DIPPEL oil), Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) analyses were conducted. FTIR spectra were recorded using a Thermo Scientific Nicolet iS50 spectrometer equipped with an attenuated total reflectance (ATR) accessory. Each sample was scanned over the range of 4000–500 cm^{-1}, with a resolution of 4 cm^{-1}, averaging 32 scans per measurement to ensure signal stability. Spectra of the chemically modified bitumen, as well as the reference petroleum binder, were processed and compared to identify characteristic absorption bands, including aliphatic, carbonyl, and hydroxyl functional groups.

SEM imaging was performed using a JEOL JSM-IT300 scanning electron microscope operated at an accelerating voltage of 15 kV. The bitumen samples were air-dried and mounted on aluminum stubs with carbon adhesive. To minimize charging, a thin gold coating (~10 nm) was sputtered on the sample surface prior to imaging using a Quorum Q150R ES coater. Micrographs were captured at magnifications ranging from 500× to 2000×, allowing detailed observation of surface features such as cracks, folds, microvoids, and layer uniformity. Comparative analysis between the natural and standard bitumen samples enabled identification of microstructural irregularities and processing-induced differences relevant to binder performance.

Throughout the process, the temperature and pressure were carefully monitored using a digital thermocouple and pressure gauge. The chemical structure and phase transitions of the modified bitumen were later confirmed using spectroscopic and thermal analysis techniques (discussed in later sections).

Results and Discussion

To assess the chemical characteristics of the extracted bitumen, the carbon isotopic composition ($\delta^{13}C$, %) of its main fractions was analyzed. Two samples were taken from two zones of the Chimiyon field: the oil-bearing (lower) layer and the Malta (upper) layer. The results are presented in Table 1.

Table 1. Carbon isotopic composition ($\delta^{13}C$, %) of bitumen fractions from the Chimiyon field

Tabela 1. Skład izotopowy węgla ($\delta^{13}C$, %) frakcji bitumu ze złoża Chimiyon

Component (fraction)	$\delta^{13}C$ [%] – oil (lower)	$\delta^{13}C$ [%] – Malta (upper)
Paraffin–naphthenic hydrocarbons	28.0	28.8
Aromatic hydrocarbons	28.6	28.8
Benzene resins	26.4	27.2
Alcohol–benzene resins	28.9	28.7

The data indicate that the carbon isotopic composition in all fractions is slightly heavier (i.e., more enriched in ^{13}C) in the upper Malta layer compared to the lower oil-bearing zone. The paraffin–naphthenic hydrocarbons showed an increase from 28.0% to 28.8%, which may reflect secondary alteration or biodegradation processes in the upper layer. Aromatic hydrocarbons remain relatively stable (28.6% to 28.8%), indicating their structural resistance to transformation.

More noticeable differences are observed in benzene resins, with $\delta^{13}C$ increasing from 26.4% to 27.2%, suggesting higher degrees of oxidation or polymerization in the upper horizon.

Conversely, alcohol–benzene resins exhibited a slight decrease in $\delta^{13}C$ values (28.9% to 28.7%), possibly due to their greater polarity and solubility under near-surface conditions.

In summary, the isotopic analysis confirms geochemical differentiation between the layers, reflecting varying degrees of maturity and transformation of the organic matter, which may influence the functional and processing behavior of the extracted bitumen.

To evaluate the chemical structural differences between the extracted bitumen and conventional petroleum-based bitumen, Fourier-transform infrared (FTIR) spectroscopy was employed. The resulting spectra are presented in Figure 1, where the blue curve represents the modified bitumen extracted from natural rock, and the red curve corresponds to conventional bitumen (DIPPEL oil) used as the reference.

The FTIR spectra of both samples display several characteristic absorption bands commonly associated with hydrocarbon-based materials:

- 2920 cm^{-1} and 2850 cm^{-1}: Strong absorption peaks attributed to the asymmetric and symmetric stretching vibrations of aliphatic $–CH_2$ and $–CH_3$ groups. These bands are present in both samples, confirming their hydrocarbon backbone;
- 1450 cm^{-1} and 1375 cm^{-1}: Bending vibrations of $–CH_2$ and $–CH_3$ groups. These peaks are slightly more pronounced in the extracted bitumen, indicating a higher content of saturated hydrocarbon chains;
- 1700–1710 cm^{-1}: A distinct carbonyl (C=O) stretching vibration observed in the red spectrum (extracted bitumen), which is absent or significantly weaker in the conventional bitumen. This peak is a clear indicator of oxidized functional groups, such as: ketones, aldehydes, or carboxylic acids, likely formed during the formalin-induced chemical modification process;
- 1600 cm^{-1}: Aromatic C=C stretching vibrations. Both samples exhibit this peak, though it is slightly more intense in the extracted bitumen, suggesting a greater presence of aromatic structures or increased conjugation after reaction;
- 1030–1100 cm^{-1}: C–O stretching vibrations associated with alcohols, ethers, or ester groups. These bands are more prominent in the extracted bitumen, reflecting the formation of methylol ($–CH_2OH$) or ether linkages due to formaldehyde reaction under elevated temperature and pressure;
- 700–900 cm^{-1}: Out-of-plane C–H bending vibrations typical for substituted aromatic rings. Slight shifts and changes in intensity between the two spectra may indicate subtle structural rearrangements or substitutions during modification.

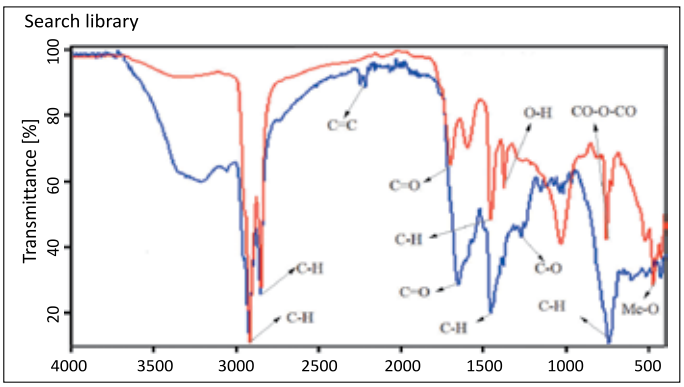

Figure 1. FTIR spectra comparison of extracted bitumen (blue line) and conventional bitumen (red line)

Rysunek 1. Porównanie widm FTIR bitumu ekstrahowanego (niebieska linia) i bitumu konwencjonalnego (czerwona linia)

According to Figure 1, the spectral differences between the two samples confirm that the extracted bitumen underwent chemical transformation during the treatment process. The appearance of new functional groups, particularly carbonyl- and hydroxyl-containing moieties, supports the formation of a cross-linked polymer-like structure resulting from methylolation and subsequent dehydration reactions. These chemical changes are consistent with improved physical performance, such as: enhanced elasticity, adhesion, and resistance to oxidative aging.

Thus, the FTIR analysis provides strong evidence for the successful functional modification of natural bitumen, making it a promising alternative to conventional petroleum bitumen in construction and road engineering applications.

As shown in Figure 2, the SEM image of standard petroleum-derived bitumen shows a smooth, compact, and uniform surface morphology. The texture appears continuous with minimal surface irregularities, indicating a well-processed and homogeneously structured matrix. This type of microstructure is typical for bitumen obtained via industrial distillation and refining processes, where impurities are minimized and colloidal stability is well maintained. The absence of visible microcracks or granularity suggests good cohesion and thermal processing, contributing to predictable mechanical performance under normal pavement conditions.

In contrast, the SEM image of bitumen extracted from natural rock reveals a rough, cracked, and heterogeneous surface. The microstructure displays folded layers, grooves, and microvoids, which are likely the result of natural geological processes and the influence of chemical extraction. These features indicate a less uniform internal structure, possibly due to high asphaltene content, partial oxidation, or mineral residue embedded in the bitumen matrix. Such morphology may contribute to increased brittleness or variable mechanical response, especially under temperature fluctuations. Additionally, comparison with the provided SEM images – IMG1 (first) showing a normal or ordinary appearance with a smoother, uniform gray surface, Carbon-Potassium (C-K) displaying red-hued fine structures possibly due to carbon-potassium

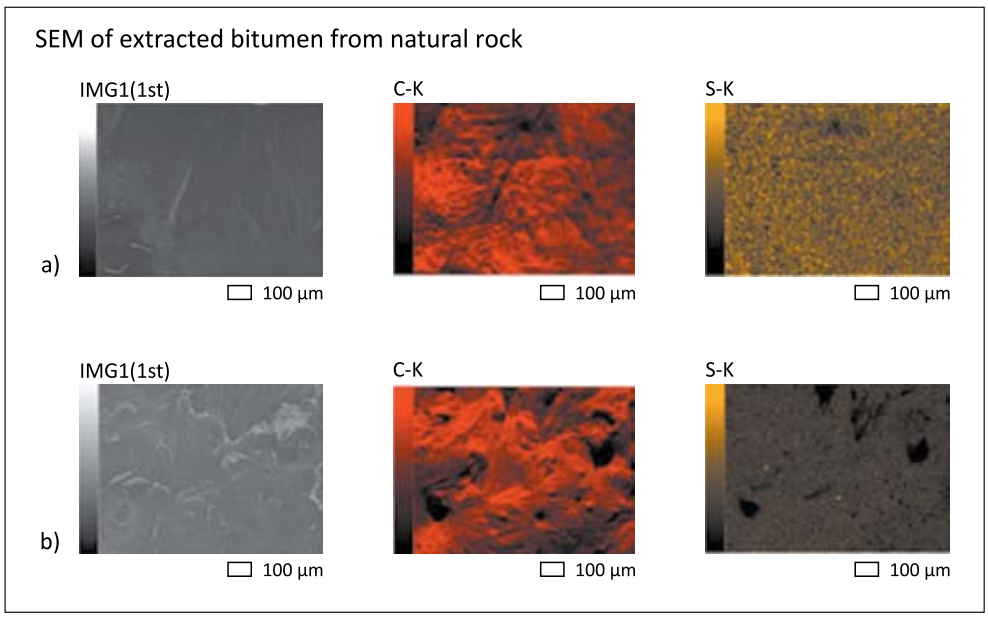

Figure 2. SEM images showing the surface morphology of (a) standard petroleum bitumen and (b) bitumen extracted from natural rock

Rysunek 2. Obrazy SEM przedstawiające morfologię powierzchni (a) standardowego bitumu naftowego oraz (b) bitumu pozyskanego z naturalnej skały

influence, and Sulfur-Potassium (S-K) exhibiting yellow-gold fine structures potentially linked to sulfur-potassium effects – highlights differences in surface texture and composition. These variations may reflect different preparation methods or chemical treatments applied to the samples.

Comparative Interpretation

The distinct differences between the two samples highlight the influence of origin and processing.

Standard bitumen presents a refined and stable morphology, suitable for consistent performance in pavement applications.

Extracted bitumen shows signs of natural heterogeneity and microstructural irregularities, which can affect its elasticity due to the uneven distribution of asphaltenes and maltenes, leading to localized stiffness variations and reduced molecular mobility within the binder matrix.

These morphological features should be considered during further modification or blending processes, especially when targeting high-performance or climate-adapted bituminous materials.

Conclusion

The results of the study confirm that bitumen extracted from natural rock deposits possesses distinct physicochemical and structural characteristics that differentiate it from conventional petroleum-derived binders. Carbon isotope analysis of bitumen fractions from the Chimiyon field revealed slight enrichment in ^{13}C isotopes in the upper Malta layer compared to the lower oil-bearing zone, with paraffin–naphthenic hydrocarbons increasing from 28.0% to 28.8%, and benzene resins shifting from 26.4% to 27.2%, indicating progressive oxidative transformation or biodegradation.

The FTIR spectra of extracted natural bitumen (blue) and standard bitumen (red) show very similar functional groups, with overlapping peaks for C–H (2900–3000 cm^{-1}), C=O (1700 cm^{-1}), C=C (1600 cm^{-1}), and C–O/O–H (1000–3500 cm^{-1}). This suggests a close chemical composition, with minor differences possibly arising from impurities or processing, indicating similar material properties.

SEM imaging further demonstrated pronounced microstructural differences between natural and conventional bitumen samples. While standard bitumen exhibited a smooth and compact morphology, the extracted sample displayed grooved, cracked, and heterogeneous surfaces, likely resulting from mineral inclusion, asphaltene concentration, and partial oxidation effects. Such heterogeneity may influence the mechanical response and compatibility in road material applications.

Collectively, the findings validate the feasibility of using natural rock-derived bitumen as a base material for modified binders, especially after tailored chemical treatment. The observed enhancements in chemical functionality and structural complexity underscore its potential for sustainable infrastructure solutions in resource-limited regions.

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