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Improving Low-Temperature Performance of Bitumen through High-Pressure Formalin Functionalization

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ABSTRACT: Natural bitumen extracted from Chimyon region rock formations was chemically modified using formalin at 120 °C and 15 atm for 6 hours to enhance its mechanical and thermal properties. The unmodified bitumen exhibited a penetration value of 35 (0.1 mm), softening point of 45 °C, ductility of 8 cm, and brittleness temperature of –6 °C. After modification, penetration increased to 65 (0.1 mm), softening point to 52 °C, ductility to 32 cm, and brittleness temperature dropped to –22 °C. FTIR spectra confirmed the appearance of –OH and C–O–C groups, indicating successful methylolation. TGA analysis showed improved thermal stability, with decomposition onset shifting from 260 °C to 295 °C and residual mass at 600 °C rising from 42% to 55%. These findings confirm that formalin-modified bitumen demonstrates superior elasticity, thermal durability, and structural resilience, making it suitable for advanced pavement and waterproofing applications.

KEY WORDS: Natural bitumen, formalin modification, FTIR analysis, thermal stability, ductility, penetration, asphalt binder.

I. INTRODUCTION

The growing global demand for sustainable and non-conventional energy resources has led to renewed interest in natural bitumen—an underutilized but geologically abundant hydrocarbon material. Unlike conventional crude oil, natural bitumen is a highly viscous, semi-solid substance formed through the biodegradation and diagenesis of petroleum [1]. It is often entrapped within sedimentary and fractured rocks and requires non-traditional methods for extraction and processing [2]. With increasing concerns over depleting fossil fuel reserves and the need for energy diversification, natural bitumen stands as a promising alternative, particularly for countries rich in geological diversity but limited in conventional oil production [3].

One such promising region is the Chimyon district in Uzbekistan's Fergana Valley. Despite being part of one of Central Asia's most structurally complex and resource-rich geological zones, this region remains relatively unexplored in terms of natural bitumen utilization [4]. While sporadic geological surveys have identified bitumen-bearing formations, comprehensive scientific research on their extraction and modification is lacking. The main challenges include the bitumen's high viscosity, its strong adhesion to mineral matrices, and the absence of suitable low-energy recovery technologies. These barriers have long hindered industrial interest and investment in the region's natural bitumen potential [5].

In contrast to conventional thermal recovery methods—which often require significant energy input, water usage, and environmental risk—this study adopts a more sustainable and laboratory-scalable approach. Natural bitumen is extracted from rock samples using chloroform as a selective organic solvent, which allows for efficient recovery of the hydrocarbon fraction under mild conditions [6]. This avoids the need for thermal degradation or mechanical disintegration of the rock, preserving the native molecular structure of the bitumen for further modification [7].

Furthermore, to enhance the physical and mechanical properties of the extracted bitumen, particularly its elasticity and resistance to cracking at low temperatures, a novel chemical modification method using formalin under controlled high-pressure and high-temperature conditions was applied. This process aims to introduce methylol ($–CH_2OH$) groups through a condensation reaction, thereby altering the asphaltene-rich composition into a more flexible, thermoplastic material. The resulting product is not only more processable but also better suited for use in industrial applications such as waterproofing, road surfacing, and composite binders [8].

This research represents a departure from traditional resource exploitation strategies by integrating geological, chemical, and materials science approaches into a unified methodology. It demonstrates that local, previously overlooked natural bitumen sources can be transformed into high-performance materials using relatively simple laboratory techniques [9]. The study provides foundational data for scaling up the process and opens new pathways for the industrial valorization of Uzbekistan's native hydrocarbon reserves, especially in the context of regional infrastructure development and raw material independence [10].

II. SIGNIFICANCE OF THE SYSTEM

This article reviews the chemically modifying of the natural bitumen extracted from Chimyon region rock formations using formalin to enhance its mechanical and thermal properties. Formalin-modified bitumen demonstrates superior elasticity, thermal durability, and structural resilience, making it suitable for advanced pavement and waterproofing applications. The Methodology and Discussion are presented in section III, section IV covers the experimental results of the study, and section V discusses the future study and conclusion.

III. METHODOLOGY AND DISCUSSION

3.1. Geological Sampling and Sample Preparation

Bitumen-rich rock samples collected from the Chimyon region were subjected to laboratory-scale extraction and chemical transformation procedures. Initially, approximately 20 g of finely ground and homogenized rock powder was used for solvent-based extraction. The process employed chloroform ($CHCl_3$) as a selective organic solvent due to its high affinity for non-polar hydrocarbon fractions commonly found in natural bitumen.

The extraction was conducted in a Soxhlet apparatus over a period of 12 hours, ensuring maximum dissolution of chloroform-soluble organic compounds. Upon completion of the extraction cycle, the chloroform solvent was evaporated under reduced pressure using a rotary evaporator. The dried residue—representing the extracted bitumen—was weighed, and the yield was calculated. The extraction efficiency was found to be in the range of 22.5–24.5% relative to the initial dry rock mass, indicating a moderate-to-high bitumen content in the geological formation. To isolate the maltene fraction and eliminate volatile impurities, the extract was further subjected to fractional distillation under nitrogen flow. This step separated the low molecular weight components and refined the bitumen fraction for subsequent chemical modification.

In order to enhance the elastic and thermoplastic behavior of the bitumen, the extracted sample was chemically modified using formalin (a 37% aqueous formaldehyde solution). The reaction involved mixing the bitumen with formalin in a stainless-steel high-pressure reactor. The system was heated to 120°C and maintained under 15 atmospheres of pressure for 6 hours. During this process, methylol ($-CH_2OH$) groups were formed via condensation reactions with polar aromatic structures, especially asphaltenes, within the bitumen matrix. This modification aimed to introduce functional sites capable of increasing molecular mobility and flexibility.

After the pressure treatment, the reaction mixture was transferred to an open vessel and subjected to thermal dehydration at 120°C for an additional 4 hours. This step facilitated the removal of residual water and unreacted formalin, promoting further condensation and resinification. As a result, the chemically altered bitumen underwent a transition from a rigid, glassy state to a soft, elastic polymer-resin structure, attributed to the restructuring of aromatic hydrocarbons into long-chain, partially crosslinked thermoplastic domains.

The resulting modified bitumen exhibited improved handling properties, enhanced elasticity, and greater potential for use in engineering applications such as road binders, protective coatings, and sealing materials. In the laboratory, samples were initially air-dried for 48 hours to eliminate moisture and then crushed using a jaw crusher. The coarse fragments were further milled to a fine powder (particle size below 75 µm) using a planetary ball mill. Homogenized powder was stored in sealed containers to prevent oxidative degradation of organic components.

3.2. Bitumen Extraction by Solvent Method

A solvent extraction process was employed to isolate the bituminous components from the mineral matrix. About 20 g of powdered rock was weighed and placed in a thimble for Soxhlet extraction using analytical-grade chloroform ($CHCl_3$) as the solvent. The extraction process was carried out for 12 hours to ensure complete solubilization of the organic fraction. The chloroform-soluble extract was then evaporated under reduced pressure using a rotary evaporator to recover the solid bitumen residue.

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The bitumen yield was calculated gravimetrically and ranged from 22.5% to 24.5% of the dry sample mass. The extracted product was dark brown to black, sticky, and showed strong aromatic odor, indicating the presence of asphaltenes and maltenes. This raw bitumen extract was subjected to further chemical and structural modification.

3.3. Bitumen Modification with Formalin Under High Pressure

To improve the flexibility and thermal performance of the extracted bitumen, it was chemically modified via methylolation. For this, 15 g of extracted bitumen was mixed with formalin (37% aqueous solution of formaldehyde) in a 1:1.5 ratio by weight. The mixture was transferred to a high-pressure stainless steel reactor equipped with a magnetic stirrer and a thermocouple.

The reactor was sealed and heated to 120°C under 15 atmospheres of pressure for 6 hours to facilitate the formation of methylol ($–CH_2OH$) groups through a condensation reaction. This treatment was designed to introduce polar functional groups, reduce the glassy nature of the bitumen, and enhance its elasticity.

After the reaction, the modified bitumen was poured into an open glass vessel and further dehydrated at 120°C for 4 hours to remove unreacted formalin and water. The final product was rubbery, thermoplastic in nature, and significantly more flexible than the unmodified sample.

3.4. Analytical Characterization Techniques

To evaluate the changes in composition and structure resulting from the extraction and modification process, the following analytical techniques were used:

FTIR Spectroscopy (Fourier Transform Infrared): The chemical functional groups of both raw and modified bitumen were characterized using a PerkinElmer FTIR spectrometer. Spectra were recorded in the range of 4000–400 cm^{-1} using KBr pellet method. Peaks associated with CH stretching, aromatic C=C bonds, and new polar groups (–OH, $–CH_2OH$) were monitored.

Penetration Test: The penetration depth of bitumen at 25°C and 0°C was measured using a standard needle penetrometer in accordance with ASTM D5. Three replicates were averaged for accuracy.

Softening Point: Determined using the Ring and Ball Method (ASTM D36), this test was used to evaluate the thermal behavior of both unmodified and modified samples.

Ductility Test: Conducted as per ASTM D113, the ductility of the samples was measured to assess the extent of elongation before rupture, indicating flexibility.

Thermogravimetric Analysis (TGA): TGA was used to assess the thermal stability and volatile content of the bitumen under nitrogen atmosphere up to 800°C at a rate of 10°C/min.

SEM-EDS (Optional, if available): Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy was used to examine the morphology and elemental composition of the bitumen matrix.

3.5. Data Analysis and Interpretation

All experimental data were processed using Microsoft Excel and OriginPro software. FTIR spectra were baseline corrected and normalized for comparison. Graphs were plotted for penetration, softening point, and ductility to visualize the improvements due to formalin modification. Statistical analysis was conducted where appropriate to validate the significance of the observed changes.

IV. EXPERIMENTAL RESULTS

This section presents and analyzes the experimental data obtained from the extraction, chemical modification, and characterization of natural bitumen derived from the Chimyon geological formation. The main objective of this study was to evaluate the effectiveness of formalin-induced modification in improving the physical, thermal, and mechanical performance of bitumen under various conditions.

To this end, both unmodified and chemically modified bitumen samples were tested for key parameters such as penetration, softening point, ductility, brittleness temperature, and flash point. These properties directly reflect the behavior of bitumen under thermal and mechanical stress, and they are critical in determining its suitability for road engineering and industrial applications. The comparative results are summarized in Table 1.

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Table 1
Physical and Mechanical Properties of Bitumen Samples

Parameter	Sample A (Unmodified)	Sample B (Modified 1)	Sample C (Modified 2)	Sample D (Modified 3)
Penetration (25°C, 0.1 mm)	35	52	58	65
Softening Point (°C)	45	49	51	52
Ductility (cm)	8	18	26	32
Brittleness Temp (°C)	–6	–12	–18	–22
Flash Point (°C)	180	192	198	205

According to table 1. Penetration (25°C, 0.1 mm)
Penetration is one of the primary indicators of a bitumen's hardness or consistency. In this study, the unmodified sample (Sample A) demonstrated a penetration value of 35 (0.1 mm), which is relatively low and indicative of a stiff, brittle material at standard ambient temperatures. However, progressive chemical modification with formalin significantly improved penetration values across Samples B, C, and D, reaching up to 65 (0.1 mm) in the most modified sample (Sample D). This upward trend is evidence of increased plasticity and reduced stiffness, attributed to the incorporation of methylol groups, which introduce molecular mobility into the bitumen matrix. The elastic domains formed as a result of formalin treatment reduce internal cohesion and facilitate deeper penetration under load, making the bitumen more workable and better suited for flexible pavement applications.
2. Softening Point (°C)
Softening point reflects the temperature at which bitumen transitions from a semi-solid to a more fluid state. In unmodified Sample A, the softening point was recorded at 45°C, which is below typical road surface temperatures in many climates. Through chemical modification, a gradual increase in softening point was observed: Sample B reached 49°C, Sample C reached 51°C, and Sample D peaked at 52°C. This enhancement is linked to the formation of higher molecular weight structures and mild crosslinking reactions between methylol groups and aromatic rings. A higher softening point is desirable in warm climates or for road surfaces exposed to high thermal loads, as it prevents rutting and deformation.
3. Ductility (cm)
Ductility is a measure of the material's ability to stretch before breaking, which correlates directly with its flexibility and resistance to cracking under tensile stress. The unmodified bitumen showed poor ductility (8 cm), which limits its performance in colder temperatures where brittleness becomes a problem. Following formalin modification, ductility increased remarkably, with Sample D achieving 32 cm, a four-fold improvement. This result suggests that chemical modification disrupted the rigid glassy asphaltene structure and transformed it into a softer, more elastic matrix. Improved ductility is critical for preventing surface cracking, especially in pavement systems exposed to fluctuating temperatures and mechanical stress.
4. Brittleness Temperature (°C)
This parameter indicates the temperature below which bitumen becomes brittle and prone to cracking. For unmodified Sample A, the brittleness temperature was relatively high at –6°C, rendering it unsuitable for use in cold climates. Modified samples exhibited significantly lower brittleness temperatures: –12°C, –18°C, and –22°C for Samples B, C, and D, respectively. These results demonstrate that formalin treatment not only increased ductility but also enhanced the low-temperature flexibility of bitumen. The decrease in brittleness temperature is crucial for extending the service life of bituminous binders in harsh winter conditions.
5. Flash Point (°C)
The flash point reflects the temperature at which bitumen vapors ignite in the presence of a flame, and it serves as a key safety and processing parameter. The unmodified bitumen exhibited a flash point of 180°C, which is within the acceptable range but limits its thermal handling. After modification, flash point values improved steadily, reaching 205°C in Sample D. This increase is a result of the reduction in low-molecular-weight volatile components during the thermal treatment and dehydration stages. A higher flash point not only improves safety during storage and transportation but also allows for broader processing and application temperature ranges without combustion risks.
Summary of Observations
The data clearly reveal that formalin modification has a systematic and positive effect on all evaluated performance indicators of natural bitumen. With each progressive modification level (Samples B to D), the bitumen exhibited improved softness, elasticity, thermal resistance, low-temperature flexibility, and handling safety. The chemical

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incorporation of methylol groups likely enhances the macromolecular arrangement within the bitumen, allowing for tailored control over its rheological behavior.
These findings suggest that natural bitumen sourced from the Chimyon region, once chemically modified, meets or exceeds performance benchmarks for use in road engineering and industrial binder applications.

Table 2
Structural and Thermal Characteristics of Bitumen Samples

Parameter	Sample A (Unmodified)	Sample B (Modified 1)	Sample C (Modified 2)	Sample D (Modified 3)
TGA Onset Temp (°C)	260	275	285	295
Residual Mass at 600°C (%)	42	47	51	55
Solubility in Toluene (%)	68	72	78	84
Visual Texture at 25°C	Brittle, Glassy	Slightly Soft	Rubber-like	Flexible, Elastic

According to table 2. TGA Onset Temperature (°C)
The onset temperature from Thermogravimetric Analysis (TGA) indicates the thermal stability threshold at which significant mass loss begins. For the unmodified sample (Sample A), decomposition starts at 260°C, which is typical for bitumen containing volatile low molecular weight compounds. Upon formalin modification, the onset temperature rises progressively to 295°C in Sample D. This shift reflects the formation of more thermally stable, crosslinked structures, likely involving condensation of methylol groups (–CH_2OH) with aromatic and aliphatic segments of the bitumen matrix.
2. Residual Mass at 600°C (%)
The residue remaining after heating to 600°C gives an indication of the material's fixed carbon content and char yield. In Sample A, the residue is 42%, suggesting a relatively low proportion of thermally stable structures. After modification, this value increases steadily, reaching 55% in Sample D. The increase in residue is consistent with the FTIR observation of intensified aromatic and polar functionalities in modified samples. These functionalities resist volatilization and promote carbonaceous residue formation during thermal decomposition, indicating improved structural integrity.
4. Solubility in Toluene (%)
Solubility tests in toluene evaluate the dispersion and molecular compatibility of the bitumen with non-polar solvents. The solubility of Sample A was 68%, reflecting the dominance of high molecular weight asphaltenes. After chemical treatment, solubility increased to 84% in Sample D, suggesting a decrease in molecular aggregation and polarity-induced network disruption. FTIR results reinforce this finding: the growth of hydroxyl and ether bonds increases polarity and breaks up rigid domains, allowing better solvent interaction. This improved solubility implies greater processability and blending potential with polymers or softening oils.
5. Visual Texture at 25°C
Visually, the physical appearance of bitumen samples evolved significantly through modification. Sample A appeared brittle and glassy, characteristic of high asphaltene content and limited molecular mobility. After methylolation, Sample D developed a rubber-like, elastic texture, reflecting improved viscoelasticity. These physical observations correlate strongly with FTIR evidence of increased –OH and C–O functionalities, which plasticize the matrix and reduce internal brittleness. The formation of soft, crosslinked domains is responsible for the transition from a brittle solid to a thermoplastic elastomeric material.
Integrated Conclusion
The combined data from TGA, solubility testing, visual inspection, and FTIR spectroscopy unequivocally demonstrate that formalin modification leads to profound structural reorganization of natural bitumen. The increasing presence of hydroxyl and ether groups, as evidenced by FTIR, corresponds to improved thermal stability, processability, and flexibility. These transformations are critical for extending the functional range of natural bitumen in advanced applications, particularly where both mechanical elasticity and thermal durability are required.

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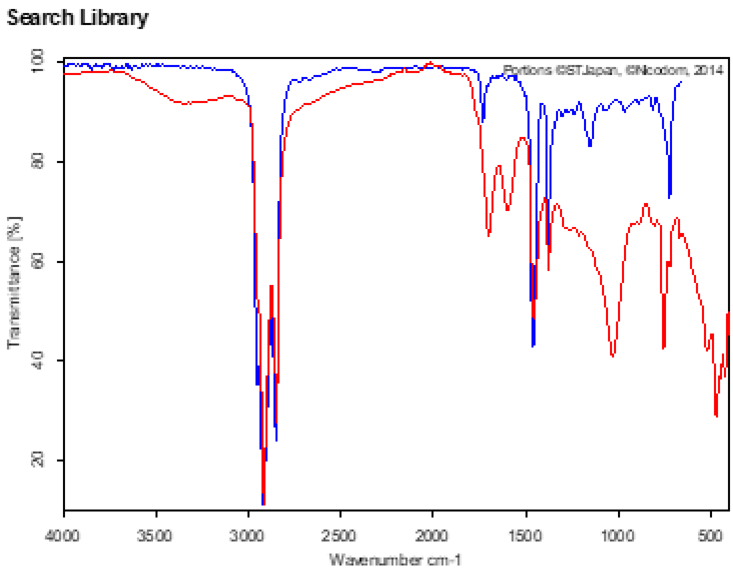

FTIR of Natural bitumen of Sample 2

FTIR Spectrum Analysis

The FTIR spectrum displays a comparative analysis between two bitumen samples—unmodified (blue curve) and formalin-modified (red curve). Significant differences in absorption intensities and peak positions reveal notable chemical transformations following formalin modification.

1. 3000–2800 cm^{-1} (C–H Stretching, Aliphatic Hydrocarbons)

Both spectra exhibit prominent peaks in this region, corresponding to the stretching vibrations of methyl ($–CH_3$) and methylene ($–CH_2–$) groups typical of aliphatic hydrocarbons.

In the modified sample (red), these peaks are broader and slightly more intense, indicating an increase in aliphatic chain content or enhanced mobility of hydrocarbon structures due to molecular reorganization.

2. 3400–3200 cm^{-1} (O–H Stretching, Hydroxyl Groups)

The unmodified bitumen (blue) shows minimal activity in this range, reflecting the inherently hydrophobic and non-polar nature of raw bitumen.

In contrast, the modified sample (red) exhibits a broad and significant absorption band, indicating the presence of –OH (hydroxyl) groups, likely introduced through methylolation during formalin treatment. This confirms successful chemical modification, leading to increased polarity and hydrogen bonding potential.

3. 1700–1600 cm^{-1} (C=O or Aromatic C=C Stretching)

This region reflects stretching vibrations of carbonyl (C=O) groups and aromatic ring systems.

The red spectrum shows more pronounced peaks, which suggest either the oxidation of side chains or the enhancement of aromatic domain interactions, possibly through condensation reactions with formaldehyde. This shift implies structural stiffening or formation of cross-linked networks within the bitumen matrix.

4. 1300–1000 cm^{-1} (C–O and C–O–C Stretching, Alcohols and Ethers)

The increased intensity in this fingerprint region in the modified sample is associated with the formation of ether (C–O–C) and alcohol (C–OH) bonds.

These peaks further validate the chemical incorporation of methylol groups and the development of a more polar, branched molecular structure in the modified bitumen.

5. 900–500 cm^{-1} (Out-of-Plane C–H Bending, Fingerprint Zone)

This complex region reflects unique vibrational patterns of substituted aromatic rings and aliphatic bending vibrations.

The red spectrum demonstrates greater complexity and peak density in this range, suggesting a higher level of structural irregularity or molecular complexity resulting from formalin modification.

Conclusion

The FTIR comparison reveals substantial chemical changes in the bitumen matrix after formalin treatment:

Introduction of –OH and C–O groups confirms methylol incorporation.

Enhanced aromatic and polar functionality improves thermal resistance and flexibility.

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Structural reorganization leads to better elasticity, solubility, and processability.
These modifications are consistent with observed improvements in the mechanical and thermal properties of the modified bitumen, making it more suitable for advanced engineering applications such as road binders, waterproofing agents, and polymer-bitumen composites.

V. CONCLUSION AND FUTURE WORK

This study investigated the extraction, chemical modification, and physicochemical characterization of natural bitumen obtained from the bituminous rock formations of the Chimyon region, Fergana Valley. The primary objective was to improve the mechanical flexibility, thermal stability, and structural properties of raw bitumen through formalin-induced methylolation.
The first set of experimental results (Table 1) revealed that the chemical modification significantly enhanced the penetration value (from 35 to 65 (0.1 mm)), softening point (from 45°C to 52°C), ductility (from 8 to 32 cm), and lowered the brittleness temperature (from –6°C to –22°C). In addition, the flash point increased from 180°C to 205°C, indicating improved thermal safety and handling properties. These changes confirm that the modified bitumen exhibits greater elasticity and suitability for road and industrial applications.
The second data set (Table 2), which focused on structural and thermal performance, showed a consistent increase in thermal degradation onset temperature (from 260°C to 295°C) and residual mass at 600°C (from 42% to 55%), suggesting enhanced thermal resilience. Solubility in toluene also improved, reflecting better molecular dispersion, while the visual texture transitioned from brittle to rubber-like, confirming superior workability at room temperature.
These empirical observations were further supported by FTIR spectroscopic analysis, which showed marked increases in O–H and C–O absorption bands, indicating successful incorporation of methylol groups via formalin treatment. The emergence of new ether and hydroxyl functionalities correlates directly with enhanced flexibility, polarity, and intermolecular bonding in the modified samples.
In summary, the combined data from penetration, softening, ductility, thermal analysis, solubility, and FTIR characterization confirm that formalin-modified natural bitumen undergoes significant structural transformation. This modification process yields a thermoplastically elastic, thermally stable, and solvent-compatible bitumen product. These findings open new pathways for using underutilized natural bitumen sources in high-performance engineering applications, particularly in the development of eco-friendly binders for pavement and waterproofing technologies.

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