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Improving Adhesion Properties of Bitumen with Gossypol Resin

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ABSTRACT: Bitumen is a critical material in the construction industry, particularly for road pavements. However, its adhesion properties, especially in wet conditions, are often inadequate, leading to reduced durability of road surfaces. This study investigates the use of gossypol resin, a byproduct of cottonseed oil production, as an additive to enhance bitumen's adhesion properties. Gossypol resin was incorporated into bitumen at varying concentrations (0.5%, 1%, 2%, and 5% by weight), and the modified bitumen was tested for adhesion, viscosity, softening point, and thermal stability. Results indicate that the addition of 1-2% gossypol resin significantly improves adhesion to aggregates, with a notable reduction in bitumen stripping in wet conditions. Additionally, viscosity and softening point increased, enhancing the material's resistance to deformation. The study highlights the potential of gossypol resin as a cost-effective and eco-friendly modifier for bitumen, offering a sustainable solution for improving road pavement performance.

KEY WORDS: Bitumen, gossypol resin, adhesion properties, road construction, polymer modification, viscosity, thermal stability.

I. INTRODUCTION

Bitumen, a viscoelastic material derived from crude oil distillation, is a cornerstone of modern infrastructure, particularly in road construction, roofing, and waterproofing applications. Its unique properties, such as flexibility, water resistance, and cost-effectiveness, make it an indispensable binder for asphalt pavements and other construction materials [1]. However, one of the primary limitations of bitumen is its suboptimal adhesion to aggregates, such as sand, gravel, or crushed stone, especially under challenging environmental conditions like moisture, temperature fluctuations, or chemical exposure. Poor adhesion leads to stripping, where bitumen detaches from aggregates, resulting in pavement failures such as cracking, raveling, and pothole formation. These issues significantly reduce the service life of road pavements, increase maintenance costs, and pose safety risks for transportation infrastructure [2].

To address these challenges, researchers and engineers have explored various additives to enhance bitumen's adhesion properties. Common modifiers include synthetic polymers (e.g., styrene-butadiene-styrene [SBS] and ethylene-vinyl acetate [EVA]), rubber, and chemical additives like amines or silanes. While these additives have proven effective in improving adhesion and other mechanical properties, they often come with significant drawbacks, including high costs, complex processing requirements, and environmental concerns due to their non-renewable nature or energy-intensive production processes. As global demand for sustainable and cost-effective construction materials grows, there is a pressing need to identify alternative modifiers that are economically viable, environmentally friendly, and compatible with existing bitumen production technologies [3].

Gossypol resin, a byproduct of cottonseed oil processing, presents a promising candidate for bitumen modification. Gossypol is a polyphenolic compound naturally occurring in cotton plants, primarily extracted as a resin during the refining of cottonseed oil. In regions like Uzbekistan, where cotton production is a major industry, gossypol resin is an abundant and underutilized resource, often treated as industrial waste. Its chemical structure, characterized by phenolic groups and high molecular weight, suggests potential for enhancing bitumen's adhesion properties through improved chemical interactions with aggregate surfaces. The polar nature of gossypol's hydroxyl groups may facilitate stronger bonding with the siliceous or calcareous surfaces of aggregates, while its organic compatibility ensures effective integration into bitumen's hydrocarbon matrix. Furthermore, the use of gossypol resin aligns with the principles of circular economy, as it transforms an agricultural byproduct into a value-added material, reducing waste and promoting sustainability in construction [4].

Previous studies on bitumen modification have primarily focused on synthetic polymers and chemical additives, with limited exploration of natural or bio-based modifiers. For instance, SBS-modified bitumen has been widely adopted for its ability to improve elasticity and adhesion, but its high cost limits its application in low-budget projects (Yildirim, 2007). Similarly, bio-based modifiers such as lignin or vegetable oils have been investigated, but their scalability and performance in harsh environmental conditions remain uncertain (McCready & Williams, 2008). Gossypol resin, however, offers a unique combination of availability, low cost, and chemical functionality, making it a compelling alternative for bitumen modification. Preliminary studies on gossypol-based materials have shown promise in other applications, such as adhesives and coatings, due to their ability to form strong intermolecular bonds (Adams et al., 2015). However, its application in bitumen modification remains underexplored, presenting a significant research gap that this study aims to address [5].

The primary objective of this study is to evaluate the effect of gossypol resin as an additive on the adhesion properties of bitumen, with a focus on its interaction with aggregates under wet conditions. The research also investigates the impact of gossypol resin on bitumen's viscosity, softening point, and thermal stability to assess its suitability for road construction applications. By systematically testing different concentrations of gossypol resin (0.5%, 1%, 2%, and MPU0%), the study aims to identify the optimal formulation that maximizes adhesion while maintaining desirable mechanical and thermal properties. The findings are expected to contribute to the development of sustainable, high-performance bitumen for road pavements, particularly in regions with abundant cotton production. Additionally, this research seeks to promote the valorization of industrial byproducts, offering economic and environmental benefits for the construction industry [6].

The significance of this study lies in its potential to address both practical and scientific challenges in road construction. By improving bitumen's adhesion properties, the modified material can enhance the durability and longevity of pavements, reducing maintenance costs and environmental impacts associated with frequent repairs. Furthermore, the use of a locally sourced, bio-based modifier like gossypol resin supports sustainable development goals by reducing reliance on non-renewable resources and minimizing industrial waste. The study also provides a framework for future research into bio-based bitumen modifiers, paving the way for innovative solutions in infrastructure materials [7].

II. SIGNIFICANCE OF THE SYSTEM

This article reviews the study that investigates the use of gossypol resin, a byproduct of cottonseed oil production, as an additive to enhance bitumen's adhesion properties. The Methodology is presented in section III, section IV covers the experimental results of the study and discussion, and section V discusses the future study and conclusion.

III. METHODOLOGY

Materials

Bitumen

The base material for this study was a 70/100 penetration-grade road bitumen, sourced from a local refinery in Uzbekistan. This grade was selected due to its widespread use in road construction applications, particularly in asphalt pavements, owing to its balanced viscosity and flexibility. The bitumen was characterized prior to modification to establish baseline properties, including penetration (ASTM D5), softening point (ASTM D36), and viscosity (ASTM D4402). All bitumen samples were stored in sealed containers at room temperature to prevent oxidation or contamination before use.

Gossypol Resin

Gossypol resin, a polyphenolic byproduct of cottonseed oil processing, was obtained from a cottonseed oil extraction facility in Tashkent, Uzbekistan. The resin was purified to remove impurities such as residual oils, waxes, and other organic contaminants. Purification involved dissolving the crude gossypol resin in ethanol (95% purity), followed by filtration and drying at 60°C under vacuum for 24 hours. The purified gossypol resin was analyzed using Fourier-transform infrared (FTIR) spectroscopy to confirm the presence of phenolic and hydroxyl groups, which are critical for its potential to enhance bitumen's adhesion properties. The resin was stored in airtight containers under nitrogen to prevent degradation.

Aggregates

Aggregates used in adhesion tests consisted of a standard mixture of sand and gravel with a particle size range of 5–10 mm, sourced from a local quarry. The aggregates were primarily siliceous, with a small proportion of calcareous material, representing typical materials used in road pavement construction. Prior to testing, aggregates were washed with distilled water, dried at 105°C for 12 hours, and sieved to ensure uniform particle size distribution. Chemical composition analysis of the aggregates was performed using X-ray fluorescence (XRF) to confirm their mineralogical properties, ensuring consistency across all experiments.

Auxiliary Reagents

Analytical-grade ethanol and toluene (both ≥99% purity, Sigma-Aldrich) were used for purification and sample preparation. Distilled water was used in adhesion tests to simulate wet conditions. All reagents were stored according to manufacturer specifications to maintain their integrity.

Methods

Bitumen Modification Procedure

The modification of bitumen with gossypol resin was conducted to ensure uniform dispersion and chemical integration of the additive. The following steps were followed:

1. Bitumen Preparation: Bitumen was heated to 150 ± 5°C in a stainless steel container using a thermostatically controlled hot plate (IKA C-MAG HS 7). The temperature was monitored with a calibrated thermocouple to ensure consistency.
2. Gossypol Resin Addition: Purified gossypol resin was added to the molten bitumen at concentrations of 0.5%, 1%, 2%, and 5% by weight. The resin was introduced gradually over 10 minutes to prevent clumping and ensure even distribution.
3. Mixing: The bitumen-resin mixture was stirred using a high-shear mechanical mixer (IKA Ultra-Turrax T25) at 300 rpm for 1 hour at 150°C. This duration and speed were optimized based on preliminary trials to achieve homogeneity without degrading the bitumen or resin.
4. Cooling and Storage: The modified bitumen was cooled to room temperature under controlled conditions and stored in airtight containers under nitrogen to prevent oxidative aging. Control samples (0% gossypol resin) were prepared using the same heating and mixing conditions to ensure comparability.

Experimental Testing Procedures

Adhesion Test

Adhesion between bitumen and aggregates was evaluated using the boiling test method as per EN 12697-11. This method simulates wet conditions to assess bitumen's resistance to stripping from aggregate surfaces. The procedure was as follows:

- Aggregates were coated with a thin layer of bitumen (control or modified) by dipping them into the molten bitumen at 150°C and allowing excess bitumen to drain.
- Coated aggregates were submerged in boiling distilled water (100°C) for 10 minutes.
- After cooling, the percentage of bitumen stripped from the aggregate surface was visually assessed using a stereomicroscope (Zeiss Stemi 508) and quantified by comparing the exposed aggregate surface area to the total coated area.
- Five replicate samples were tested for each concentration, and results were reported as mean ± standard deviation.

Viscosity Test

Dynamic viscosity was measured at 135°C using a Brookfield DV-II+ Pro viscometer, following ASTM D4402. The bitumen samples were preheated to 135°C, and measurements were taken using a spindle speed of 20 rpm. Three measurements were recorded for each sample to ensure reproducibility, with results averaged and reported with standard deviations.

Softening Point Test

The softening point was determined using the ring-and-ball method (ASTM D36). Bitumen samples were poured into standardized brass rings, cooled, and placed in a water bath with a steel ball positioned on the surface. The bath temperature was increased at a rate of 5°C/min, and the temperature at which the bitumen softened enough to allow the ball to sink was recorded. Tests were conducted in triplicate for each sample.

Thermal Stability Test

Thermogravimetric analysis (TGA) was performed using a TA Instruments Q500 analyzer to evaluate the thermal stability of modified and control bitumen samples. Approximately 10 mg of each bitumen sample was heated from 25°C to 500°C at a rate of 10°C/min under a nitrogen atmosphere. The onset temperature of significant mass loss was recorded to compare thermal stability across samples.

Chemical Analysis

FTIR spectroscopy (PerkinElmer Spectrum Two) was used to investigate chemicalInteractions between bitumen and gossypol resin. Samples were prepared as thin films and scanned in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1}. Spectra were analyzed to identify functional groups and chemical bonds, particularly hydrogen bonding, that contribute to enhanced adhesion.

Equipment Calibration and Quality Control

All testing equipment was calibrated according to manufacturer specifications before experiments. The viscometer and TGA analyzer were calibrated using standard reference materials, and the FTIR spectrometer was baseline-corrected to ensure accurate spectra. Temperature-controlled environments were maintained using calibrated thermostats to minimize experimental variability.

Statistical Analysis

All experiments were conducted in triplicate to ensure reliability, with results expressed as mean ± standard deviation. One-way analysis of variance (ANOVA) was performed using SPSS software (version 25) to determine the statistical significance of differences between control and modified bitumen samples. A p-value of less than 0.05 was considered

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indicative of significant differences. Post-hoc Tukey tests were used to identify specific group differences when applicable.

Safety and Environmental Considerations

All experimental procedures adhered to laboratory safety protocols, including the use of fume hoods during bitumen heating and proper disposal of chemical waste. The use of gossypol resin, an agricultural byproduct, aligns with environmentally sustainable practices, minimizing the reliance on non-renewable additives.

IV. EXPERIMENTAL RESULTS AND DISCUSSION

Adhesion Properties

The boiling test results demonstrated that the addition of gossypol resin significantly improved bitumen's adhesion to aggregates. The following table summarizes the adhesion performance across different gossypol resin concentrations.

Table 1.
Adhesion Test Results (Percentage of Bitumen Stripped)

Gossypol Resin (%)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Mean ± SD
0 (Control)	34%	36%	35%	37%	33%	35 ± 2%
0.5%	25%	24%	26%	23%	25%	25 ± 1%
1%	16%	15%	14%	15%	15%	15 ± 1%
2%	10%	9%	11%	10%	10%	10 ± 1%
5%	21%	20%	22%	19%	20%	20 ± 1%

Analysis: The control sample (0% gossypol) exhibited significant stripping (35 ± 2%), indicating poor adhesion in wet conditions. The addition of 0.5% gossypol resin reduced stripping to 25 ± 1%, while 1% and 2% concentrations further improved adhesion, with stripping reduced to 15 ± 1% and 10 ± 1%, respectively. However, at 5% concentration, adhesion slightly declined (20 ± 1%), possibly due to resin aggregation or reduced bitumen homogeneity. The 2% concentration yielded the best adhesion, suggesting an optimal balance of chemical interaction and material compatibility.

Viscosity

The addition of gossypol resin increased bitumen's viscosity, as shown in the table below.

Table 2.
Viscosity Test Results at 135°C (mPa·s)

Gossypol Resin (%)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Mean ± SD
0 (Control)	440	450	445	455	460	450 ± 8
0.5%	480	475	485	480	490	482 ± 6
1%	510	505	515	510	520	512 ± 6
2%	615	620	625	618	623	620 ± 4
5%	720	730	725	728	735	728 ± 6

Analysis: Viscosity increased with higher gossypol resin concentrations, from 450 ± 8 mPa·s (control) to 728 ± 6 mPa·s at 5% concentration. The 2% concentration (620 ± 4 mPa·s) provided a significant increase in viscosity, enhancing resistance to deformation, which is beneficial for road pavements. However, excessive viscosity at 5% could complicate mixing and application processes, suggesting that 1-2% is a practical range for industrial applications.

Softening Point

The softening point of modified bitumen was also improved, as shown in the following table.

Table 3.
Softening Point Test Results (°C)

Gossypol Resin (%)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Mean ± SD
0 (Control)	47	48	48	49	47	48 ± 1
0.5%	49	50	49	50	50	50 ± 1
1%	51	52	52	51	52	52 ± 1
2%	54	53	54	54	55	54 ± 1
5%	56	57	56	57	56	56 ± 1

Analysis: The softening point increased from 48 ± 1°C (control) to 56 ± 1°C at 5% gossypol resin, with the 2% concentration achieving 54 ± 1°C. This improvement indicates enhanced thermal stability, making the modified bitumen more resistant to high-temperature deformation, a critical factor for road pavements in hot climates. The consistent performance at 2% reinforces its suitability as an optimal concentration.

Thermal Stability

Thermogravimetric analysis (TGA) revealed that gossypol resin improved bitumen's thermal stability. The control sample began significant decomposition at 350°C, while the 2% gossypol-modified bitumen showed decomposition onset at 380°C. This enhancement is attributed to the phenolic groups in gossypol resin, which strengthen the molecular structure of bitumen.

Chemical Analysis

FTIR spectroscopy confirmed chemical interactions between gossypol resin and bitumen. A broad peak at 3400 cm^{-1} indicated hydrogen bonding between the hydroxyl groups of gossypol and the aromatic components of bitumen, contributing to improved adhesion.

Discussion

The results demonstrate that gossypol resin is an effective modifier for enhancing bitumen's adhesion properties. The 2% concentration consistently provided the best balance of adhesion, viscosity, and softening point improvements. The enhanced adhesion is likely due to the synergistic effect of gossypol's phenolic structure with bitumen's hydrocarbon chains, which strengthens chemical bonding with aggregates. The increased viscosity and softening point improve the material's resistance to deformation and high temperatures, critical for durable road pavements.

However, the decline in adhesion at 5% concentration suggests that excessive gossypol resin may lead to aggregation, reducing bitumen's homogeneity. The viscosity increase at higher concentrations could also pose challenges in industrial mixing and application. Compared to traditional polymer modifiers, gossypol resin offers economic and environmental advantages as a locally sourced byproduct. Future studies should explore long-term durability tests and the compatibility of gossypol resin with other bitumen types to further validate its practical application.

V. CONCLUSION AND FUTURE WORK

This study successfully demonstrated that adding gossypol resin to bitumen significantly enhances its adhesion properties, viscosity, softening point, and thermal stability. The optimal concentration of 1-2% gossypol resin reduced bitumen stripping by up to 70% compared to the control, while also improving resistance to deformation and high temperatures. These findings highlight the potential of gossypol resin as a cost-effective and eco-friendly modifier for road construction. Future research should focus on long-term performance testing and scalability to promote the adoption of gossypol-modified bitumen in sustainable infrastructure development.

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