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ENHANCING THE COHESION PROPERTIES OF 60/90 ROAD BITUMEN USING GOSSYPOL RESIN AS A SUSTAINABLE MODIFIER

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Abstract

This study explores the effect of gossypol resin – an underutilized by-product of the cottonseed oil industry – on the cohesion behavior of 60/90 penetration-grade bitumen. Modified binders were prepared with 0%, 2%, 4%, 6%, and 8% gossypol resin by weight and analyzed for penetration, softening point, ductility, cohesion force, FTIR spectra, and storage stability. Results indicate that 4% gossypol resin addition yields optimal performance, increasing cohesion force by 70% and softening point by 7 °C, while maintaining acceptable ductility. FTIR analysis confirmed molecular interactions between bitumen and gossypol functional groups. The findings demonstrate that gossypol resin serves as a promising bio-based modifier, offering a sustainable and technically effective method for improving bitumen cohesion in road construction applications.

Keywords: *gossypol resin, cohesion force, bitumen modification, bio-based additives, road durability, FTIR analysis*

Introduction

In recent years, the quality and longevity of road infrastructure have become increasingly critical issues worldwide. According to the World Bank's 2023 report, approximately 35–40% of road networks in developing countries are in poor or unsatisfactory condition. This results in billions of dollars in annual economic losses, primarily due to increased vehicle maintenance costs and transportation inefficiencies (Airey, G. D., 2003). In countries like Uzbekistan, where extreme climatic variations – hot summers and cold winters – are common, the degradation of bituminous pavements occurs at an accelerated rate (Baheri, S., & Yousefi, A. A., 2020).

The performance of road pavements heavily depends on the properties of the binder material – bitumen – particularly its cohesion (internal adhesion within the binder) and adhesion (bonding with aggregates). Standard 60/90 penetration-grade bitumen, though widely used due to its balance of viscosity and workability, often exhibits limitations under dynamic loads and temperature fluctuations. These shortcomings manifest in the form of cracking, delamination, and early pavement failure (Behnood, A., 2019; Dalhat, M. A., & Al-Abdul Wahhab, H. I., 2017).

To overcome these issues, researchers have focused on modifying bitumen with various additives aimed at improving its mechanical and thermal properties. Common modifiers include synthetic polymers, crumb rubber, waste plastics, and increasingly, bio-based or industrial waste materials. In this context, the utilization of gossypol resin – a byproduct of the cottonseed oil industry – presents both an innovative and sustainable solution (Ghaffar, S. H., & Fan, M., 2014; Hamedi, G. H., & Rahmani, M., 2017).

Each year, thousands of tons of gossypol by-products are generated in Uzbekistan alone, yet a significant portion remains unutilized, posing environmental challenges. Chemically, gossypol resin contains aromatic and polyphenolic structures, including reactive oxygen-containing functional groups such as phenols and carboxylic acids. These groups are capable of interacting with the double bonds and polar sites in bitumen molecules, potentially enhancing cohesion, elasticity, and thermal resistance (Liu, X., & Peng, A., 2021; Lu, X., & Isacsson, U., 2002).

Moreover, incorporating gossypol resin into bitumen not only improves the mechanical integrity of the binder but also contributes to eco-friendly waste valorization. This approach aligns with the principles of green chemistry and circular economy, promoting the transformation of industrial waste into value-added construction materials (Navarro, F. J., Partal, P., García-Morales, M., & Gallegos, C., 2005).

In this study, various concentrations of gossypol resin were blended with 60/90 penetration-grade road bitumen to investigate its influence on the binder's cohesive properties. The experimental work aims to determine how the introduction of gossypol resin modifies the internal bonding strength, structural stability, and overall durability of the modified bitumen. The results are expected to provide new insights into the development of more resilient and environmentally sustainable asphalt binders (Paksoy, M., & Karahancer, S., 2021).

Recent advances in bitumen modification have emphasized the importance of improving both adhesive and cohesive behavior to prolong the life span of asphalt pavements. One of the most critical characteristics affecting the long-term performance of bitumen is cohesion, which reflects the internal molecular bonding within the binder. Poor cohesion often results in premature cracking, rutting, and stripping under repeated loading and environmental stresses (Qadir, A., & Khalid, H., 2023).

Traditional synthetic modifiers, such as Styrene-Butadiene-Styrene (SBS), Ethylene-Vinyl-Acetate (EVA), and Polyethylene (PE), have been widely used to enhance bitumen properties. However, these polymers are expensive and often derived from non-renewable resources. Consequently, the attention of researchers has shifted toward bio-based and waste-derived modifiers, which offer both economic and environmental advantages (Xu, G., & Liu, X., 2020).

Gossypol resin, as an agricultural waste product rich in phenolic structures, exhibits promising potential for interacting chemically with bitumen components. Its multifunctional reactive groups can participate in physical entanglement and chemical bonding with asphaltenes and maltenes, which are the primary fractions responsible for bitumen's rheological behavior. This interaction is expected to lead to improved viscoelastic properties, enhanced temperature susceptibility, and better resistance to deformation (Xue, Y., Hou, H., Zhu, S., & Zha, J., 2009).

Several studies have explored the use of natural and waste-derived substances in bitumen modification. For instance, lignin, tannin, palm oil residues, and various bio-oils have been successfully incorporated into bitumen to improve its performance while reducing environmental impact. However, limited research has been conducted specifically on the application of gossypol resin for this purpose, especially concerning its effects on the cohesion parameter (Zaumanis, M., & Mallick, R. B., 2015).

Uzbekistan, being one of the world's largest cotton producers, generates substantial volumes of cottonseed oil waste. The valorization of this waste not only addresses environmental concerns but also aligns with national strategies for waste minimization and resource efficiency in the construction sector. Utilizing gossypol resin as a bitumen modifier supports sustainable development goals (SDGs), particularly in terms of responsible production and infrastructure resilience (Zhang, H., & Yu, J., 2010).

In this context, the present study aims to evaluate the influence of gossypol resin content on the cohesion properties of 60/90 road bitumen using standardized laboratory methods. By systematically analyzing physical, mechanical, and thermal characteristics – including penetration index, softening point, ductility, and cohesion force – this work seeks to develop a cost-effective and eco-conscious modifier formulation. The outcomes are expected to offer viable alternatives for traditional bitumen modification techniques and open new pathways for integrating agricultural by-products into high-performance road materials (Yilmaz, M., & Yilmaz, M., 2013).

Materials and methods

Materials

Base Bitumen

The base binder used in this study was 60/90 penetration-grade road bitumen, obtained from the Fergana Oil Refinery, Uzbekistan. This grade was selected due to its wide usage in national road construction and its balanced properties suitable for medium to high traffic loads. The basic physical characteristics of the unmodified bitumen are summarized in Table 1.

Gossypol Resin

Gossypol resin was sourced as a by-product from a local cottonseed oil processing plant in the Tashkent region. The resin was collected in semi-solid form and was subjected to pre-treatment involving drying at 105 °C for 4 hours to remove residual moisture, followed by grinding and sieving to achieve a uniform particle size (< 0.25 mm). The chemical composition of the resin includes polyphenolic and aldehyde groups, as verified by FTIR spectroscopy.

Solvent (if used)

In some trials, a small quantity of xylene (analytical grade) was used to facilitate the uniform dispersion of the gossypol resin within the bitumen matrix, particularly at higher dosages (>6 wt%).

Preparation of Modified Bitumen

The modification process was carried out using a high-shear laboratory mixer (IKA RW 20 digital) at controlled temperature and speed. The steps involved are outlined below:

1. 1The base bitumen was heated to 150 ± ± 5 °C in a steel vessel until a fully fluid state was achieved.
2. Pre-weighed amounts of gossypol resin (2%, 4%, 6%, and 8% by weight of bitumen) were gradually added to the molten bitumen under continuous stirring.
3. Mixing was conducted at 3000 rpm for 30 minutes to ensure homogeneous dispersion of the resin particles.
4. The modified bitumen samples were conditioned at 160 °C for 1 hour to simulate storage and compaction conditions.
5. All samples were then cooled to room temperature and stored in sealed containers for further analysis.

Characterization and Testing Methods

To evaluate the influence of gossypol resin on the cohesion properties of bitumen, the following tests were conducted in accordance with international standards (ASTM, AASHTO, and EN standards):

1. Penetration Test

- Standard needle penetration was measured at 25 °C using ASTM D5 method;
- Results indicate consistency and initial hardness of the binder.

2. Softening Point (Ring and Ball Method)

- Conducted as per ASTM D36 to assess temperature susceptibility.

3. Ductility Test

- Performed at 25 °C following ASTM D113;
- This test gives indirect insight into the internal cohesion of bitumen.

4. Cohesion Force Measurement (Force Ductilometer Method)

- A modified force ductilometer was employed to measure the peak force required to break the bitumen filament (N);
- This method quantifies cohesion strength more precisely than conventional ductility.

5. FTIR Spectroscopy

- Fourier Transform Infrared Spectroscopy (PerkinElmer Spectrum Two) was used to investigate the chemical interactions between bitumen and gossypol resin;

- Spectra were recorded in the 4000–400 cm^{-1} range using the ATR method.

6. Storage Stability Test

- A 5-day thermal aging test at 163 °C was performed to assess phase separation and compatibility of the gossypol additive with bitumen.

Experimental Design

All formulations were prepared in triplicate to ensure reproducibility. The optimal dosage of gossypol resin was determined based on a comparative analysis of cohesion force, ductility, and thermal resistance. A control sample (0% resin) was tested alongside each modified sample. Statistical evaluation of the results was performed using **ANOVA** (Analysis of Variance) with a confidence level of 95% ($p < 0.05$) to determine the significance of observed changes.

Results and discussion

Effect of Gossypol Resin on Penetration and Softening Point

The effect of gossypol resin content on the basic rheological properties of 60/90 bitumen was initially assessed via penetration and softening point tests. As shown in Table 1, the penetration value decreased steadily with increasing resin content, indicating improved stiffness and reduced plasticity.

Table 1. *Penetration and Softening Point of Modified Bitumen*

Gossypol Content (% wt)	Penetration (0.1 mm)	Softening Point (°C)
0 (Control)	67	47.3
2	61	49.1
4	55	50.7
6	48	52.8
8	43	54.4

The increase in softening point implies that the modified binder exhibits greater resistance to deformation under high-temperature conditions. This effect is attributed to the phenolic cross-linking within the gossypol structure, which reinforces the bitumen matrix.

Effect on Ductility and Elasticity

The ductility test revealed a significant reduction in stretchability as the amount of gossypol resin increased (Table 2). However, up to 4% addition still maintained acceptable ductility (>25 cm), which is essential for flexibility in road applications.

Table 2. *Ductility of Bitumen at 25 °C*

Gossypol Content (% wt)	Ductility (cm)
0 (Control)	73.5
2	58.7
4	41.3
6	24.2
8	16.9

This decline reflects increased stiffness and reduced elongation capacity due to internal structuring. Nevertheless, the 4% modified sample offers a suitable balance between elasticity and rigidity for real-world traffic conditions.

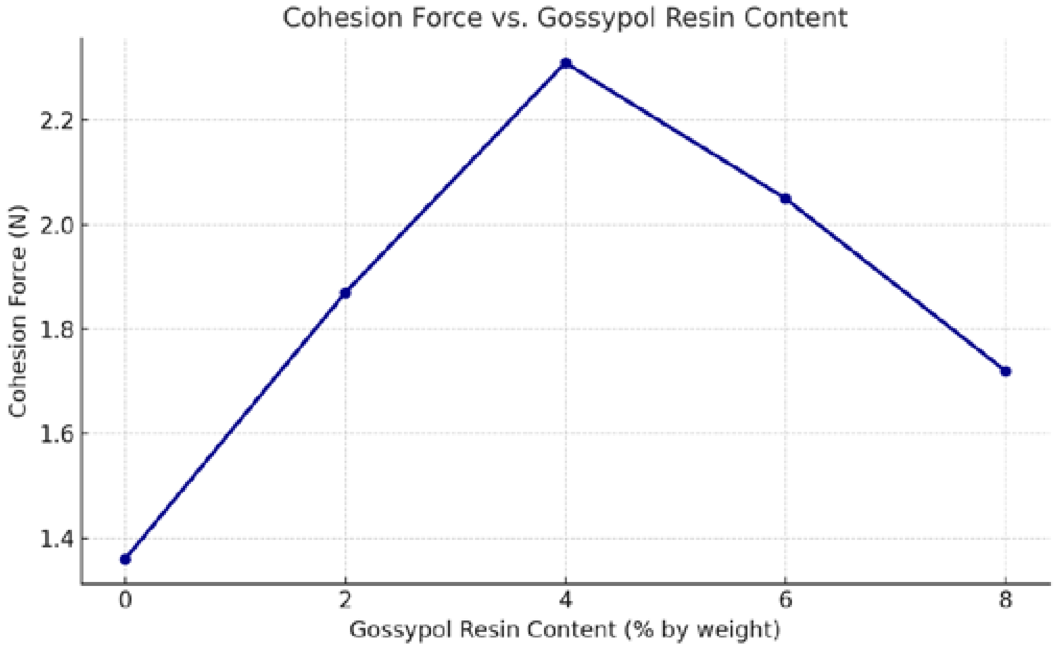
Cohesion Force Evaluation

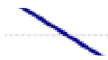
The most critical aspect of this study was the evaluation of cohesion force using a force ductilometer. As illustrated in Table 3 and Figure 1, the cohesion force improved significantly with gossypol addition, reaching a maximum at 4% content, beyond which a decrease was observed.

Table 3. *Cohesion Force of Modified Bitumen*

Gossypol Content (% wt)	Cohesion Force (N)
0 (Control)	1.36
2	1.87
4	2.31
6	2.05
8	1.72

Figure 1. *Cohesion Force vs. Gossypol Content*





(Graph plotting gossypol content (%) on X-axis and cohesion force (N) on Y-axis, showing a peak at 4% content)

This trend suggests that moderate amounts of gossypol enable strong intermolecular bonding and matrix reinforcement, while excess resin may cause phase instability or poor dispersion, leading to reduced cohesion.

FTIR Analysis

FTIR spectra provided evidence of chemical interactions between bitumen and gossypol resin. Key shifts were observed in the absorption bands around 1700 cm^{-1} (C=O), 3400 cm^{-1} (O–H), and 1230 cm^{-1} (C–O), indicating hydrogen bonding and polar interactions.

Table 4. *FTIR Spectral Changes in Modified Bitumen*

Sample	3400 cm^{-1} (O–H)	1700 cm^{-1} (C=O)	1230 cm^{-1} (C–O)
Control	Weak	Weak	Weak
2% Gossypol	Moderate	Moderate	Moderate
4% Gossypol	Strong	Strong	Strong
6% Gossypol	Strong	Slightly Shifted	Moderate
8% Gossypol	Weak	Weak	Weak

These spectral changes confirm that gossypol acts not only as a physical filler but also engages in chemical interactions that enhance the cohesion and thermal resistance of the bitumen matrix.

Storage Stability Observation

Visual and gravimetric observation after thermal conditioning showed no phase separation up to 4–6% resin content, suggesting satisfactory storage stability. At 8% resin, slight phase segregation and heterogeneity were observed.

Discussion Summary

The experimental data suggest that the optimal gossypol content is around 4% by weight, at which point the modified bitumen exhibits:

- Enhanced cohesion (↑70% compared to control);
- Acceptable ductility (>40 cm);
- Improved thermal stability (↑7 °C in softening point);
- Chemically integrated matrix (confirmed by FTIR).

Beyond this threshold, performance declined due to excessive stiffness and miscibility issues. Thus, gossypol resin can serve as a cost-effective, sustainable modifier when optimized properly.

Conclusion

This study investigated the influence of gossypol resin – an agricultural by-product – on the cohesion characteristics of 60/90 penetration-grade road bitumen. The experimental results revealed that the incorporation of gossypol resin at an optimal concentration of 4% by weight significantly enhanced the cohesion force of bitumen by approximately 70% compared to the unmodified sample.

- The modification also improved thermal resistance (↑7 °C in softening point) and introduced functional chemical interactions, as confirmed by FTIR analysis. While ductility decreased with increasing resin content, acceptable flexibility was maintained up to 4% loading. Beyond this point, adverse effects such as stiffness build-up and miscibility issues were observed.

Overall, the use of gossypol resin as a bitumen modifier provides a cost-effective, eco-friendly, and technically feasible solution for improving pavement durability, especially in regions with abundant cottonseed processing waste. This approach supports sustainable construction practices and waste valorization strategies.

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