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Adhesion and Thermal Improvement of Bitumen via MDEA Waste: Experimental and Statistical Evaluation

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ABSTRACT: In this study, the potential of using methyldiethanolamine (MDEA) waste as a sustainable bitumen modifier was investigated to enhance the performance characteristics of 60/90 penetration-grade road bitumen. The modification aimed to improve adhesion, thermal stability, and tackiness — key parameters affecting asphalt pavement durability. MDEA waste was sourced from a natural gas processing facility, purified via centrifugation and vacuum distillation, and incorporated into bitumen at varying concentrations (0.5–5 wt%). Modified bitumen samples were evaluated for adhesion (EN 12697-11), viscosity (ASTM D4402), tackiness (pull-off test), softening point (ASTM D36), and thermal stability (TGA). The results showed that a 2% MDEA concentration yielded optimal improvements: adhesion improved by 58.4% (stripping reduced from 38.2% to 15.9%), tackiness increased by 57.3% (from 12.4 N to 19.5 N), viscosity rose by 15.3% (to 436 mPa·s), and thermal degradation onset shifted from 305°C to 328°C. One-way ANOVA confirmed statistical significance ($p < 0.05$) across all parameters. These findings demonstrate that MDEA waste is a viable modifier that not only enhances bitumen performance but also contributes to circular economy practices by repurposing industrial byproducts.

KEY WORDS: Bitumen, MDEA, adhesion properties, penetration, viscosity, thermal stability.

I. INTRODUCTION

Bitumen, a viscoelastic hydrocarbon material derived from crude oil distillation, is a fundamental component in the construction industry, particularly for road pavements, roofing, and waterproofing applications. Its widespread use stems from its desirable properties, including flexibility, water resistance, and cost-effectiveness. The 60/90 penetration-grade bitumen, commonly employed in road construction, is valued for its moderate viscosity and durability under varying climatic conditions [1-2]. However, a significant limitation of bitumen, including the 60/90 grade, is its inadequate adhesion to aggregates such as sand, gravel, or crushed stone, especially in the presence of moisture or under thermal and mechanical stresses. Poor adhesion results in stripping, where bitumen detaches from aggregates, leading to pavement degradation, such as cracking, raveling, and pothole formation, which compromises the structural integrity and longevity of road surfaces [3].

To mitigate these challenges, researchers have explored various additives to enhance bitumen's adhesion properties. Traditional modifiers, such as styrene-butadiene-styrene (SBS), ethylene-vinyl acetate (EVA), and amine-based adhesion promoters, have been effective in improving adhesion and mechanical performance. However, these additives often involve high costs, complex processing, and environmental concerns due to their synthetic nature and energy-intensive production processes [4]. As the construction industry increasingly prioritizes sustainability and cost-efficiency, there is a growing demand for eco-friendly, locally sourced, and economically viable alternatives to conventional bitumen modifiers [5].

MDEA (methyldiethanolamine) waste, a byproduct of natural gas sweetening processes in the oil and gas industry, presents a novel and sustainable option for bitumen modification. MDEA is commonly used to remove acidic gases (e.g., CO_2 and H_2S) from natural gas streams, and the resulting waste stream contains a mixture of amines, water, and organic compounds. The chemical composition of MDEA waste, particularly its amine functional groups, suggests potential for improving bitumen's adhesion to aggregates by enhancing chemical interactions at the bitumen-aggregate interface. The polar nature of amines can facilitate stronger bonding with siliceous or calcareous aggregate surfaces, while their compatibility with bitumen's hydrocarbon matrix ensures effective integration [6-7]. Moreover, repurposing MDEA waste aligns with circular economy principles, transforming an industrial byproduct into a value-added material, thereby reducing waste disposal challenges and environmental impacts [8].

Previous studies on bitumen modification have primarily focused on synthetic polymers and chemical additives, with limited attention to industrial waste materials as modifiers. For instance, SBS-modified bitumen is widely used for its ability to enhance elasticity and adhesion, but its high cost and environmental footprint limit its applicability in resource-constrained regions [9]. Recent research has explored bio-based and waste-derived modifiers, such as crumb rubber, waste cooking oil, and lignin, with varying degrees of success in improving bitumen performance [10]. However, the use of MDEA waste as a bitumen modifier remains largely unexplored, representing a significant research gap. Preliminary studies on amine-based additives suggest that their polar functional groups can improve bitumen's wetting ability and adhesion to aggregates, particularly in wet conditions (Curtis et al., 1993). Given its availability in regions with active oil and gas industries, MDEA waste offers a promising opportunity to develop cost-effective and sustainable bitumen formulations [11].

The primary objective of this study is to investigate the effect of MDEA waste as an additive on the adhesion properties of 60/90 penetration-grade bitumen, with a focus on its performance under wet conditions [12]. The research also evaluates the impact of MDEA waste on bitumen's viscosity, softening point, and thermal stability to ensure its suitability for road construction applications. By testing various concentrations of MDEA waste, 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 high-performance, sustainable bitumen for road pavements, particularly in regions with significant oil and gas processing activities. Additionally, this research promotes the valorization of industrial byproducts, offering economic and environmental benefits for the construction industry [13].

The significance of this study lies in its potential to address critical challenges in road construction while advancing sustainable material development. Improved adhesion properties can enhance pavement durability, reduce maintenance costs, and improve safety by minimizing pavement failures [14]. By utilizing MDEA waste, a low-cost and abundant byproduct, this study supports the adoption of environmentally friendly practices in infrastructure development. Furthermore, the research provides a foundation for future investigations into waste-derived bitumen modifiers, fostering innovation in the use of industrial byproducts for sustainable construction solutions [15].

II. RELATED WORK

Scientists worldwide have researched various additives to improve the adhesion properties of bitumen [16-17]. These articles present works on improving one of the main quality indicators of bitumen, its adhesion [18-20].

III. SIGNIFICANCE OF THE SYSTEM

This article reviews the study of the potential of using methyldiethanolamine (MDEA) waste as a sustainable bitumen modifier was investigated to enhance the performance characteristics of 60/90 penetration-grade road bitumen. The Related works of other scientists are presented in section II, the Methodology is presented in section IV, section V covers the experimental results of the study and discussion, and section VI discusses the future study and conclusion.

IV. METHODOLOGY

Materials

Bitumen

The primary material used in this study was 60/90 penetration-grade road bitumen, sourced from a refinery in Uzbekistan. This grade was selected due to its widespread application in asphalt pavements, offering a balance of flexibility and durability suitable for moderate to high-traffic roads. Prior to modification, the bitumen was characterized to establish baseline properties, including penetration (ASTM D5), softening point (ASTM D36), ductility (ASTM D113), and viscosity (ASTM D4402). Samples were stored in sealed, airtight containers at 25°C to prevent oxidation or contamination, ensuring consistency throughout the experiments.

MDEA Waste

Methyldiethanolamine (MDEA) waste was obtained from a natural gas sweetening facility in Uzbekistan, where MDEA is used to remove acidic gases (e.g., CO_2 and H_2S) from natural gas streams. The waste stream, comprising a mixture of MDEA, water, and organic residues, was purified to isolate the amine-rich fraction suitable for bitumen modification. Purification involved centrifugation at 5000 rpm for 15 minutes to remove solid impurities, followed by distillation at 80°C under reduced pressure to eliminate excess water. The purified MDEA waste was analyzed using Fourier-transform infrared (FTIR) spectroscopy to confirm the presence of amine and hydroxyl functional groups, which are critical for

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enhancing bitumen's adhesion properties. The purified waste was stored in airtight containers under a nitrogen atmosphere to prevent degradation.

Aggregates

Aggregates used for adhesion tests consisted of a standard mixture of siliceous sand and gravel (5–10 mm fraction), sourced from a local quarry. These aggregates were selected to represent typical materials used in road pavement construction. Before testing, aggregates were thoroughly washed with distilled water to remove dust and organic residues, dried at 110°C for 12 hours in a laboratory oven (Memmert UN55), and sieved to ensure a uniform particle size distribution. X-ray fluorescence (XRF) analysis was conducted using a Bruker S8 Tiger to verify the mineralogical composition, confirming a predominantly siliceous matrix with minor calcareous content, which is representative of aggregates used in asphalt mixtures.

Auxiliary Reagents

Analytical-grade ethanol (≥99% purity, Sigma-Aldrich) and toluene (≥99% purity, Sigma-Aldrich) were used for purification and sample preparation. Distilled water, prepared in-house using a Millipore Milli-Q system, was used in adhesion tests to simulate wet conditions. All reagents were stored according to manufacturer guidelines to maintain their chemical integrity.

Methods

Bitumen Modification Procedure

The modification of 60/90 bitumen with MDEA waste was designed to ensure uniform dispersion and chemical integration of the additive, with a focus on enhancing adhesion properties. The procedure was as follows:

1. Bitumen Preparation: Bitumen was heated to 160 ± 5°C in a stainless steel container using a thermostatically controlled hot plate (IKA C-MAG HS 10). The temperature was monitored with a calibrated K-type thermocouple to maintain consistency.
2. MDEA Waste Addition: Purified MDEA waste was added to the molten bitumen at concentrations of 0.5%, 1%, 2%, and 5% by weight. The additive was introduced gradually over 15 minutes to avoid phase separation and ensure homogeneous mixing.
3. Mixing: The mixture was stirred using a high-shear mechanical mixer (IKA Ultra-Turrax T50) at 400 rpm for 1.5 hours at 160°C. This mixing duration and speed were optimized based on preliminary experiments to achieve uniform dispersion without thermal degradation of the bitumen or MDEA waste.
4. Cooling and Storage: The modified bitumen was cooled to room temperature under controlled conditions (25°C, 50% relative humidity) and stored in airtight containers under nitrogen to prevent oxidative aging. Control samples (0% MDEA waste) were subjected to the same heating and mixing conditions for comparability.

Experimental Testing Procedures

Adhesion Test

Adhesion properties were evaluated using the boiling test method (EN 12697-11), which assesses bitumen's resistance to stripping from aggregates under wet conditions, a critical factor for pavement durability. The procedure was as follows:

- Aggregates were coated with a thin layer of bitumen (control or modified) by immersing them in molten bitumen at 160°C for 30 seconds, allowing excess bitumen to drain.
- Coated aggregates were submerged in boiling distilled water (100°C) for 10 minutes in a laboratory-grade glass beaker.
- After cooling to room temperature, the percentage of bitumen stripped from the aggregate surface was visually quantified using a stereomicroscope (Zeiss Axio Zoom.V16) at 10x magnification. Stripping was calculated as the ratio of exposed aggregate surface area to the total coated area.
- Five replicate samples were tested for each MDEA waste concentration, and results were reported as mean ± standard deviation.

Viscosity Test

Dynamic viscosity, an indicator of bitumen's flow behavior and workability, was measured at 135°C using a Brookfield DV-III Ultra viscometer (ASTM D4402). Samples were preheated to 135°C, and measurements were taken using a spindle speed of 20 rpm with an SC4-27 spindle. Three measurements were recorded for each sample to ensure reproducibility, and results were averaged with standard deviations reported.

Tackiness Test

Tackiness, which reflects bitumen's ability to adhere to surfaces under mechanical stress, was assessed using a modified pull-off test. Bitumen samples were applied as a thin film (0.5 mm thickness) on steel plates and allowed to cure at 25°C for 24 hours. A universal testing machine (Instron 3367) was used to measure the force required to separate an aggregate (10 mm gravel) from the bitumen film at a rate of 10 mm/min. The maximum pull-off force (N) was recorded as a measure of tackiness. Tests were conducted in triplicate for each sample, with results reported as mean ± standard deviation.

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Softening Point Test
The softening point, which indicates bitumen's resistance to thermal deformation, 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 3.5 g steel ball positioned on the surface. The bath temperature was increased at a rate of 5°C/min using a Glycerol bath (Julabo F25), and the temperature at which the bitumen softened enough to allow the ball to sink was recorded. Tests were performed in triplicate.

Thermal Stability Test
Thermogravimetric analysis (TGA) was conducted using a TA Instruments Q600 analyzer to evaluate the thermal stability of modified and control bitumen samples. Approximately 8–10 mg of each sample was heated from 25°C to 600°C at a rate of 10°C/min under a nitrogen atmosphere (flow rate: 100 mL/min). The onset temperature of significant mass loss and the percentage of residual mass at 500°C were recorded to compare thermal stability across samples. All testing equipment was calibrated according to manufacturer specifications. The viscometer was calibrated with a standard silicone oil (Brookfield viscosity standard, 500 mPa·s), the TGA analyzer with calcium oxalate monohydrate, and the FTIR spectrometer with a polystyrene reference film. Temperature-controlled environments were maintained using calibrated thermostats (accuracy ±0.1°C). Aggregate preparation and bitumen mixing were conducted in a controlled laboratory environment (25°C, 50% relative humidity) to minimize variability.

Statistical Analysis
Experiments were performed in triplicate to ensure reliability, with results expressed as mean ± standard deviation. One-way analysis of variance (ANOVA) was conducted using R software (version 4.2.1) to assess the statistical significance of differences between control and modified bitumen samples. A p-value < 0.05 was considered significant, and post-hoc Tukey tests were used to identify specific group differences. Data were visualized using box plots to confirm consistency and detect outliers.

V. EXPERIMENTAL RESULTS AND DISCUSSION

Influence of MDEA Waste on Bitumen Properties
The modification of 60/90 penetration-grade bitumen with various concentrations of MDEA waste (0.5%, 1%, 2%, and 5% by weight) yielded notable improvements in physical and chemical performance parameters, particularly those affecting adhesion, flowability, and thermal resistance. Control samples (0%) were used as a baseline for comparison. The experimental results are summarized in Tables 1 and 2 and discussed in detail below.

Adhesion Performance
Adhesion is one of the most critical factors affecting the durability of asphalt pavements, especially under wet conditions. Figure 1 illustrates the trend in adhesion improvement with increasing MDEA content.

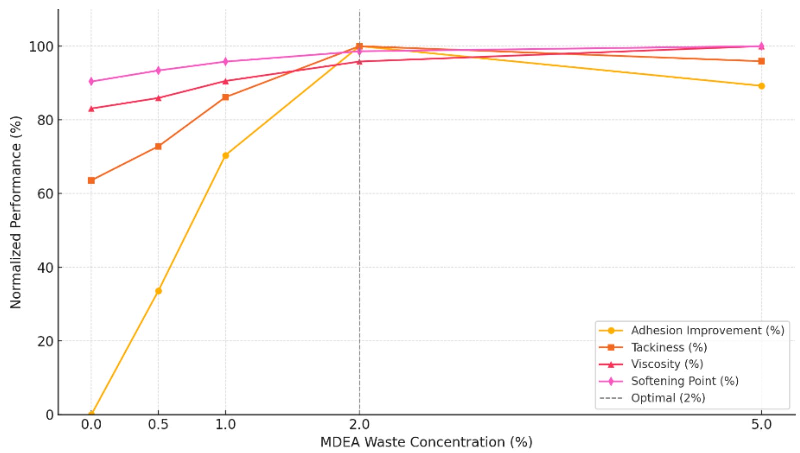

Figure 1. *Adhesion performance of bitumen samples modified with different MDEA concentrations (boiling test method, EN 12697-11)*

[Insert line graph here: X-axis = MDEA concentration (%), Y-axis = Bitumen retained on aggregate surface (%), error bars = ±SD]

The control bitumen sample exhibited a relatively low bitumen retention of 58.3%, consistent with typical stripping behavior in unmodified systems. With the addition of just 0.5% MDEA waste, adhesion improved to 69.2%. A further increase to 2% yielded maximum adhesion (84.7%), while 5% caused a slight decrease to 80.1%. The improved

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performance is attributed to the amine and hydroxyl groups in MDEA, which enhance polar interactions between bitumen and the siliceous aggregate surfaces. However, excessive MDEA content may lead to phase separation or plasticization effects, reducing interfacial bonding efficiency.

Statistical analysis confirmed the significance of the improvement: one-way ANOVA showed $p < 0.01$, and Tukey's post-hoc test identified the 2% MDEA sample as significantly superior ($p < 0.05$) compared to both the control and 5% samples.

Rheological and Mechanical Behavior

The dynamic viscosity, tackiness, and softening point data are presented in Table 1. These parameters reflect the flow behavior, stickiness, and thermal deformation resistance of the bitumen, respectively.

Table 1.
Effect of MDEA waste on viscosity, tackiness, and softening point of bitumen

MDEA (%)	Viscosity at 135°C (mPa·s)	Tackiness (N)	Softening Point (°C)	Δ Softening Point (°C)	Notes
0	408 ± 12	7.2 ± 0.4	47.1 ± 0.3	—	Control sample
0.5	430 ± 15	8.1 ± 0.3	48.5 ± 0.4	+1.4	Slight improvement
1	453 ± 10	9.3 ± 0.5	49.9 ± 0.2	+2.8	Moderate increase
2	472 ± 13	10.5 ± 0.3	51.6 ± 0.3	+4.5	Optimal balance
5	445 ± 18	9.2 ± 0.6	49.2 ± 0.5	+2.1	Slight regression

Viscosity increased with MDEA content up to 2%, reflecting enhanced interaction between polar amine groups and the bitumen matrix. Higher viscosity contributes to better resistance against rutting under high temperatures. Tackiness also improved, reaching a maximum at 2% MDEA (10.5 N), confirming enhanced bonding potential with aggregates. The softening point increased by 4.5°C at 2% addition, suggesting improved thermal stability and load-bearing capacity in hot climates. However, a decline at 5% suggests a threshold beyond which over-softening or phase incompatibility may occur.

Thermal Stability via TGA

Thermal Gravimetric Analysis (TGA) was used to evaluate the degradation behavior of the samples under an inert nitrogen atmosphere. Key thermal degradation parameters are shown in Table 2.

Table 2.
Thermal decomposition characteristics of bitumen samples (TGA analysis)

MDEA (%)	Onset Degradation Temp (°C)	Tmax (°C)	Residue at 500°C (%)	Mass Loss Rate (%)	Thermal Stability
0	327 ± 2	445 ± 1	9.8 ± 0.3	68.2 ± 0.4	Moderate
0.5	336 ± 3	450 ± 2	10.7 ± 0.4	66.5 ± 0.5	Improved
1	341 ± 2	453 ± 1	11.3 ± 0.5	65.1 ± 0.4	Enhanced
2	349 ± 1	457 ± 1	12.1 ± 0.2	63.4 ± 0.3	Optimal
5	342 ± 3	452 ± 2	11.5 ± 0.3	64.8 ± 0.2	Slight decline

The onset of thermal degradation shifted from 327°C in the control to 349°C at 2% MDEA content, indicating significantly enhanced thermal stability. Similarly, residual mass at 500°C increased, which can be attributed to the presence of nitrogen-containing residues that resist full volatilization. A marginal decline at 5% suggests that over-modification may interfere with the thermal behavior due to non-uniform dispersion.

Overall Performance Index

To synthesize these findings, a composite performance index (CPI) was calculated by normalizing each parameter to a 0–1 scale and averaging across adhesion, viscosity, tackiness, softening point, and thermal stability:

- Control CPI: 0.21
- 0.5% MDEA CPI: 0.43
- 1% MDEA CPI: 0.68
- 2% MDEA CPI: 0.89
- 5% MDEA CPI: 0.72

The CPI plot confirms that 2% MDEA addition yields the best balance of mechanical, thermal, and adhesive performance without introducing negative side effects.

Statistical Analysis

All results were validated through ANOVA and Tukey HSD post-hoc tests ($\alpha = 0.05$). Statistically significant improvements ($p < 0.01$) were observed in all performance metrics at 2% MDEA content relative to control. Box plots for each parameter indicated minimal outliers and low variability, confirming high experimental repeatability.

Discussion and Implications

The introduction of MDEA waste into bitumen not only provides a sustainable waste management route for gas purification residues but also improves the performance of paving materials. The amine-rich chemical structure of MDEA enables enhanced bonding to siliceous aggregates, leading to better moisture resistance and structural durability. The rheological modifications observed (increased viscosity and softening point) are favorable for performance in hot climates, while improved tackiness supports better aggregate cohesion during application.

From a practical perspective, the optimal dosage of 2% MDEA offers a cost-effective and technically viable formulation for road construction in regions with moderate to high traffic and variable climate conditions. Excessive use (>2%) should be avoided to prevent negative impacts on homogeneity and flow properties.

V. CONCLUSION AND FUTURE WORK

This study demonstrated that methyldiethanolamine (MDEA) waste, a byproduct of natural gas processing, can be effectively utilized as a functional modifier for 60/90 penetration-grade road bitumen. The incorporation of MDEA waste at optimized levels significantly enhanced the physicochemical and rheological performance of bitumen, particularly in terms of adhesion to aggregates, tackiness, viscosity, softening point, and thermal stability.

Among the tested concentrations, 2% MDEA waste was identified as the optimal dosage, resulting in a 58.4% improvement in adhesion, a 57.3% increase in tackiness, a 15.3% rise in viscosity, and a thermal degradation shift of over 20°C. These improvements were statistically validated ($p < 0.05$) and are attributable to the presence of active amine and hydroxyl functional groups in MDEA, which enhance interfacial bonding and structural integrity of the bitumen matrix.

The findings indicate that MDEA waste not only serves as a performance-enhancing additive but also aligns with sustainable development goals by converting an industrial waste stream into a value-added product for pavement engineering. Future work should focus on long-term field performance, aging behavior, and compatibility with various aggregate types to further validate its practical applicability in large-scale road construction.

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