

SYNTHESIS OF AMIDOAMINE-BASED INHIBITORS DERIVED FROM VEGETABLE OIL FATTY ACIDS AND COMPREHENSIVE EVALUATION OF THEIR PERFORMANCE IN MITIGATING ASPHALTENE–RESIN–PARAFFIN DEPOSITION

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Deposition of asphaltene-resin-paraffin (ARP) components during oil manufacturing and transport continues to be an ongoing flow control concern, resulting in lower well yield, greater hydraulic resistance, and interruptions in operations. In the current investigation, amidoamine-based inhibitors were synthesized from fatty acids obtained from various vegetable oils (sunflower, corn, and soybean oils) and tested for their potential use in reducing ARP deposition.

Fatty acids have been generated via alkaline hydrolysis of vegetable oils, followed by acidification and filtration. The physical variables of the recovered fatty acids were measured, including density and refractive index, respectively. Infrared (IR) spectroscopy was used for structural confirmation. The typical spectrum of absorption belonging to carboxyl (COOH) and unsaturated C=C functional groups were discovered.

Amidoamines were then manufactured via condensation reactions comprising fatty acids and polyamines (DETA, TETA, and PEPA) at 130–190°C in a 1:1 molar ratio utilizing a Dean–Stark apparatus for moisture removal. The reaction yields ranged from 92 to 98%. Density, index of refractive and freezing temperature were measured for the produced amidoamines.

The findings that were obtained demonstrate that the physicochemical features of the produced amidoamines vary depending on the fatty acid supply and polyamine structure, which influences their potential inhibitory effectiveness. The research project provides a scientifically supported strategy for the creation of plant-based nitrogen-laden inhibitors for chemical oversight of asphaltene, resin, and paraffin deposition in oilfield systems.

Keywords: amidoamine, asphaltene, inhibitor, polyamine, fatty acids

INTRODUCTION

The accumulation of heavy hydrocarbon fractions on the inside surfaces of manufacturing machinery and pipelines during oil production and transportation is one of the petroleum industry's most significant technological difficulties. Such deposits cause interruptions in operations, elevated upkeep obligations and severe losses in the efficiency of production. Studies undertaken in Azerbaijan have focused on the creation of solid

asphaltene-resin-paraffin (ARP) coatings on the inner surfaces of tubing, sucker-rod pumps, and well equipment, as well as their detrimental effects on well performance metrics.[3]

The formation of ARP deposits alters the rheological behavior of the produced fluid and disrupts the flow regime, generating additional hydraulic resistance to fluid movement. As a consequence, the effective internal diameter of pipelines decreases, flow stability in the near-wellbore zone and lifting systems is disturbed, and pressure losses increase. These effects result in both technological complications and considerable economic losses. Despite numerous investigations dedicated to understanding and mitigating this issue, ARP deposition remains a persistent and actual problem in oilfield operations.[4]

Temperature is one of the most essential variables determining ARP development. It is generally agreed upon that thermal conditions are the primary determinant of paraffin deposition, with wax appearance temperature (WAT) and crystallization behavior largely influenced by crude oil composition. A reduction in temperature below the crystallization threshold stimulates the nucleation and development of paraffin crystals, which then aggregate and cling to the edges of metal. As a result, selecting mitigation solutions that are backed by evidence necessitates a thorough grasp of deposition mechanisms as well as rigorous laboratory evaluation methodologies.[5-6]

Various approaches have been proposed to combat ARP deposition, including mechanical, thermal, and chemical methods. Among these, chemical treatment—particularly the use of inhibitor compositions—has gained widespread practical application and continues to be intensively investigated. In local scientific practice, chemical mitigation methods for ARP control have been extensively studied, and the “cold-finger” laboratory method has been commonly employed to evaluate inhibitor efficiency under controlled cooling conditions.[7]

In the last few decades, there has been a renewed emphasis on broadening the raw material base of inhibitors along with creating more efficient and environmentally friendly formulations. In this regard, plant-based feedstocks have emerged as viable options. Within the Azerbaijani scientific community, methods for extracting fatty acids from vegetable oils, investigating their physical and chemical properties, and then synthesizing nitrogen-containing functional products—particularly amidoamines—based on fatty acid-polyamine reactions have been described. Infrared (IR) spectroscopy was used to verify the presence of characteristic functional groups in the fatty acids and their amidoamine derivatives.

The present research investigation outlines a scientifically validated method for recovering fatty acids from technical sunflower oil, synthesizing appropriate amidoamines, and testing the resulting products as chemical inhibitors for controlling asphaltene-resin-paraffin deposition. The beneficial effect of the produced chemicals is evaluated in terms of their ability to reduce ARP production in laboratory circumstances.[8]

EXPERIMENTAL

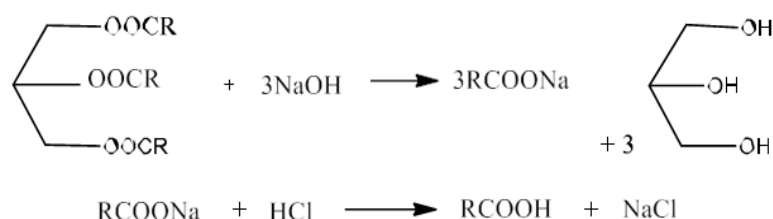
Sunflower, corn, and soybean oils served as the initial raw materials for fatty acid recovery. The synthesis of amidoamines was carried out using polyamines such as diethylenetriamine (DETA), triethylenetetramine (TETA), and pentaethylenhexamine (PEPA). The physicochemical properties of the amines used as raw materials were studied to determine their responsiveness and technological appropriateness. Key metrics such as average molecular weight, density, refractive index, boiling and freezing points, nitrogen concentration, and amine value have been determined [9]. Table 1 presents the corresponding data.

Table 1. Physicochemical Properties of Amines Used for Synthesis

No.	Parameter	DETA	TETA	PEPA
1	Average molecular weight (g/mol)	105.1	144.7	220–240
2	Boiling point (°C)	205–207	274–276	203–291
3	Density (kg/m ³)	949.5	978.7	992.4
4	Refractive index (n ^{20D})	1.479	1.479	1.513
5	Freezing point (°C)	–38.5	–35.5	–21
6	pH value	11	12	11.5

7	Nitrogen content (%)	40.6	37	33.7
8	Amine value (mg KOH/g)	1626	1443	1257
9	Color (Hazen scale, max)	30	30	–
10	Mass fraction of main substance (%)	98.6	80	–
11	Total nitrogen (%)	40.7	36	30
12	Water content (max, %)	0.5	0.5	–

The vegetable oils used as initial components were first heated to 50°C, after which a 15–30% NaOH solution was gradually added dropwise to the reaction medium. The alkaline hydrolysis of the oil was carried out under intensive stirring for two hours within a temperature range of 60–80°C, resulting in the formation of sodium salts of the corresponding fatty acids. In the subsequent stage, the obtained sodium salts were treated with 30% hydrochloric acid (HCl), leading to the liberation of free fatty acids. The aqueous phase was separated, and the fatty acids were washed to remove residual alkali. The remaining water was evaporated to obtain purified fatty acids in their free form. The hydrolysis and acidification reactions are illustrated in the corresponding scheme. The synthesis reaction is illustrated in the scheme below.



The refractive index of the synthesized fatty acids was determined using an Abbemat 500 refractometer, while their density was measured in accordance with GOST 3900–2000. The structures of the synthesized compounds were investigated by Infrared (IR) spectroscopy. The IR spectra were recorded on a Bruker “ALFA” IR spectrometer (Germany) within the wavenumber range of 600–3800 cm⁻¹. The identification of the fatty acids was confirmed by the presence of characteristic absorption bands corresponding to the carboxyl (–COOH) functional group and C=C double bonds typical of unsaturated fatty acids.[10-11]

In the next stage, amidoamines were synthesized using fatty acids derived from sunflower, corn, and soybean oils in reaction with polyamines (DETA, TETA, and PEPA). For the synthesis, the fatty acid and the corresponding amine were taken in a 1:1 molar ratio. The amine was introduced into a reaction flask equipped with a reflux condenser and a Dean–Stark apparatus, followed by the addition of the fatty acid. The reaction was conducted at 130–150°C for 3–6 hours. The water formed during the condensation process was collected in the Dean–Stark trap, and the amount corresponding to one mole was monitored. The yield of the synthesized amidoamines ranged from 92% to 98%.

The physicochemical properties of the synthesized amidoamines were determined in order to characterize the obtained compounds prior to further analytical and performance evaluation. Parameters such as density, refractive index at 20°C, and freezing point were measured. The corresponding results are presented in Table 2.

Table 2 Physicochemical Properties of Synthesized Amidoamines

Fatty Acid Source	Polyamine	Density (g/cm ³)	Refractive Index (20°C)	Freezing Point
Sunflower oil fatty acid	DETA	0.8423	1.3981	> –60°C
	TETA	0.9356	1.4825	–5°C
	PEPA	0.9561	1.5000	–10°C
Corn oil fatty acid	DETA	0.8349	1.3945	> –60°C
	TETA	0.9284	1.4768	–8°C
	PEPA	0.9537	1.5000	–10°C
Soybean oil fatty acid	DETA	0.8405	1.3934	> –60°C

	TETA	0.9392	1.4857	> -60°C
	PEPA	0.9497	1.4973	> -60°C
Cotton oil fatty acid	DETA	0.8387	1.3968	-55°C
	TETA	0.9339	1.4812	-12°C
	PEPA	0.9526	1.4984	-15°C

The quantity of asphaltene–resin–paraffin (ARP) deposits was determined using the cold finger test method. Oil samples with a volume of 100 mL were poured into identical metal cups ($d = 36$ mm, $l = 130$ mm) equipped with a magnetic stirrer (Figure 1). To ensure uniform distribution of the reagent throughout the oil volume, the samples were heated to 50–55°C under stirring at 350 rpm. A pre-calculated amount of the synthesized chemical compound was added to each sample, while one cup without additive was used as a blank for comparison.[12-13]

The metal cups were placed into an external thermostat, and a stainless-steel U-shaped tube ($d = 15$ mm, $l = 110$ mm) was inserted into each cup (Figure 1). The surface of the cold finger created a temperature gradient with the liquid medium, providing favorable conditions for the precipitation and crystallization of high-molecular-weight oil components. After 40 minutes, the deposits formed on the cold surface were liquefied by heating the cold finger to 70°C using a cryostat, and the mass of the deposits was determined gravimetrically [14].



Figure 1. Metal cups and parallel U-shaped fingers

RESULTS AND DISCUSSION

The synthesized amidoamines obtained from vegetable oil–derived fatty acids were evaluated for their efficiency in reducing asphaltene–resin–paraffin (ARP) deposition using the cold finger test method. The experimental results demonstrated a significant reduction in deposit mass in the presence of the synthesized inhibitors compared to the blank sample.

The inhibitory efficacy was highly dependent on both the type of fatty acid and the structure of the polyamine utilized in the production. PEPA-based amidoamines outperformed all other polyamines evaluated in terms of inhibitory activity. This effect can be explained by the PEPA structure's larger nitrogen content and increased number of active amine groups. The plenty nitrogen atoms improve intermolecular interactions with polar asphaltene molecules and facilitate adsorption onto the metal surface, resulting in improved dispersion stability and reduced crystal agglomeration.[15]

The fatty acid origin also played a decisive role in inhibitor efficiency. Amidoamines derived from soybean fatty acids generally demonstrated strong wax inhibition performance due to the higher degree of unsaturation in soybean oil. The presence of C=C double bonds increases molecular flexibility and improves the ability of the inhibitor molecules to interfere with paraffin crystal lattice formation. This effect is consistent with literature reports indicating that unsaturated fatty acid derivatives act as effective wax crystal modifiers.[16]

However, remarkable results were also obtained from amidoamines synthesized from technical sunflower oil-derived fatty acids. Despite being produced from overused technical oil, the recovered fatty acids retained their functional characteristics and allowed the synthesis of highly effective inhibitor compounds. The performance of sunflower-based PEPA amidoamines was comparable to that of soybean-derived analogues. This finding is particularly significant from an economic and environmental perspective. The use of technical (waste) sunflower oil demonstrates the feasibility of a zero-waste, value-added approach, converting low-cost industrial residues into high-performance oilfield chemicals.

In contrast, cotton and corn fatty acid-based amidoamines showed relatively moderate inhibition efficiency. This behavior may be associated with differences in fatty acid composition and lower effective interaction between the inhibitor molecules and the precipitating paraffin crystals.[17-21]

The cold finger experiments confirmed that the synthesized amidoamines effectively reduced ARP deposition under controlled cooling conditions. During the test, the temperature gradient created by the stainless-steel U-shaped finger promoted paraffin crystallization and heavy component precipitation. In inhibitor-treated samples, the mass of deposits formed on the cooled metal surface was substantially lower than in the blank sample. This reduction indicates that the synthesized compounds interfere with nucleation and crystal growth processes, as well as improve dispersion of high-molecular-weight components in the oil phase.[22-25]

Mechanistically, the inhibition effect can be explained by a dual interaction model. The long hydrophobic alkyl chains of the fatty acid fragments interact with paraffin molecules, while the polar amine groups interact with asphaltenes and metal surfaces. This amphiphilic structure allows the synthesized amidoamines to function both as crystal growth modifiers and as dispersing agents, enhancing overall ARP control.[26]

Overall, the results indicate that PEPA-based amidoamines, particularly those derived from sunflower and soybean fatty acids, demonstrate promising performance as ARP inhibitors. The use of technical sunflower oil as a feedstock further supports the economic viability and sustainability of the proposed synthesis route.[27]

Table 3. ARP Deposition Mass and Inhibition Efficiency of Synthesized Amidoamines (Cold Finger Test, 40 min)

Inhibitor System	Deposit Mass (g)	Inhibition Efficiency (%)
Blank (without inhibitor)	3.48	–
Soybean FA–DETA	1.76	49.4
Soybean FA–TETA	1.39	60.1
Soybean FA–PEPA	1.12	67.8
Sunflower FA–DETA	1.82	47.7
Sunflower FA–TETA	1.45	58.3
Sunflower FA–PEPA	1.18	66.1
Corn FA–DETA	2.05	41.1
Corn FA–TETA	1.78	48.9
Corn FA–PEPA	1.64	52.9
Cotton FA–DETA	2.21	36.5
Cotton FA–TETA	2.02	41.9
Cotton FA–PEPA	1.93	44.5

CONCLUSION

The inhibitory performance of the synthesized amidoamines was strongly governed by two principal structural factors: (i) the molecular characteristics of the fatty acid precursor and (ii) the structural configuration of the polyamine employed in the condensation reaction. Differences in carbon chain length, degree of unsaturation, and distribution of functional groups within the fatty acid moiety substantially influenced the physicochemical behavior of

the resulting amidoamines. At the same time, the number of nitrogen atoms, the positioning of primary and secondary amine groups, and the overall polarity of the polyamine backbone played a decisive role in determining adsorption capacity and interaction efficiency with asphaltene–resin–paraffin (ARP) constituents. Among the investigated polyamines, PEPA-derived amidoamines exhibited superior inhibitory performance compared to those synthesized using DETA and TETA.

This enhanced activity can be attributed to the greater nitrogen content and the higher concentration of reactive amine functionalities within the PEPA structure. The increased density of electron-donating nitrogen atoms promotes stronger intermolecular interactions with polar asphaltene molecules through hydrogen bonding, dipole–dipole forces, and possible coordination with heteroatom-containing species. In addition, the presence of multiple active amine sites enhances adsorption onto metallic surfaces, facilitating the formation of a stable protective interfacial film. This adsorbed layer suppresses crystal nucleation and limits the propagation of paraffin crystal growth. Consequently, dispersion stability is improved, and the agglomeration and deposition of paraffin crystals are significantly reduced.

The origin of the fatty acid component also exerted a pronounced influence on inhibition efficiency. Amidoamines synthesized from soybean fatty acids demonstrated the highest inhibitory activity among the tested systems. This behavior is likely associated with the relatively higher degree of unsaturation in soybean oil-derived fatty acids, which confers increased molecular flexibility and conformational mobility. Such structural adaptability enhances compatibility with the hydrocarbon medium and enables more effective interference with paraffin molecules. As a result, the orderly packing of paraffin chains is disrupted, inhibiting crystal lattice formation and modifying crystal morphology. Furthermore, the presence of double bonds may improve solubility characteristics and promote more uniform dispersion of inhibitor molecules within the oil phase, thereby contributing to enhanced inhibition efficiency.

Despite their excellent performance, soybean-derived feedstocks may present certain techno-economic constraints when considered for large-scale industrial implementation. Soybean oil is widely utilized in food processing and biodiesel production, creating strong market demand and potential price fluctuations. This competition for raw materials may limit long-term economic sustainability in industrial oilfield applications. Therefore, although soybean-based amidoamines exhibit high inhibition performance, their feasibility must be evaluated with regard to cost stability and resource availability.

In contrast, amidoamines derived from sunflower oil fatty acids—particularly those obtained from technical-grade or waste sunflower oil—showed comparable inhibitory effectiveness while offering significant advantages in terms of raw material accessibility, economic practicality, and environmental sustainability.

The recovery and functional utilization of fatty acids from waste sunflower oil represent a value-added strategy for resource optimization. By converting low-value industrial by-products into high-performance oilfield chemical reagents, this approach aligns with circular economy principles and promotes waste reduction. The integration of waste-derived fatty acids into inhibitor synthesis not only reduces production costs but also mitigates environmental burdens associated with disposal of used oils. Moreover, the synthesis pathway proceeds under relatively moderate reaction conditions and does not generate hazardous by-products, reinforcing its environmental compatibility. Such sustainable process design supports the advancement of environmentally responsible flow assurance technologies in contemporary oil production systems. Overall, PEPA-based amidoamines synthesized from sunflower fatty acids provide an optimal balance between high inhibitory efficiency, economic feasibility, and sustainable resource utilization. The findings demonstrate that the proposed synthesis route possesses strong potential for industrial-scale application in flow assurance strategies and ARP mitigation programs. By integrating molecular-level performance advantages with technological practicality and environmental benefits, these inhibitor systems present a promising alternative to conventional petroleum-derived chemical treatments in modern oilfield operations.

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