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Development and Statistical Evaluation of Bio-Based Demulsifiers from Mahogany, Neem, and Calabash Oils for Crude Oil Emulsion Breaking

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Abstract. *The growing demand for environmentally sustainable chemicals in crude oil processing has intensified interest in green demulsifiers as alternatives to conventional petrochemical surfactants, which are often toxic, costly, and poorly biodegradable. This study investigates the synthesis, performance evaluation, and statistical validation of bio-based demulsifiers derived from oils of Khaya spp. (mahogany), Azadirachta indica (neem), and Crescentia cujete (calabash) for crude oil–water separation. The formulations were prepared through controlled thermal blending of lipophilic and hydrophilic components using camphor, paraffin oil, cassava starch, and liquid soap as intermediates and binders. Their efficiencies were evaluated using standardized bottle tests, with a commercial demulsifier from Nigerian National Petroleum Company Limited serving as the reference. The results showed demulsification efficiencies of 96.6%, 93.7%, and 92.6% for the mahogany-, neem-, and calabash-based formulations, respectively, compared with 97.0% for the commercial product. Statistical analysis confirmed significant differences among treatments (ANOVA: $F = 69.83$, $p = 4.44 \times 10^{-6}$), while Tukey's post hoc test revealed no significant difference between the mahogany-based demulsifier and the commercial control ($p > 0.05$), indicating comparable performance. The mahogany formulation also exhibited the highest reproducibility (SD = 0.20), reflecting stable interfacial activity and consistent separation efficiency. Overall, these findings demonstrate that plant-derived oils are promising renewable feedstocks for eco-friendly demulsifiers, supporting cleaner petroleum-processing practices and circular bioeconomy principles by using biodegradable, sustainable industrial materials. The replacement of persistent petrochemical surfactants with biodegradable bio-based demulsifiers can significantly reduce marine pollution from oil-processing effluents while promoting sustainable resource utilization within a circular bioeconomy framework.*

Keywords: *Bio-Based Demulsifier; Mahogany Oil; Neem Oil; Calabash Oil; Statistical Validation*

1. Introduction

Vegetable oils and their derivatives are increasingly being explored as eco-friendly demulsifiers for separating crude oil-water emulsions. Unlike conventional petrochemical surfactants, which are often expensive, poorly biodegradable, and environmentally hazardous (1),(2) vegetable oils provide a renewable, biodegradable, and non-toxic alternative (3),(4). Derived from triglyceride-rich natural resources (5), these oils exhibit amphiphilic properties that enable interfacial activity, making them suitable substitutes for synthetic surfactants in diverse industrial applications (6), including petroleum processing (7). Their molecular architecture, long-chain fatty acids bonded to glycerol backbones, also allows

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chemical modification to tailor surface-active properties for targeted applications such as demulsification, corrosion inhibition, and lubrication (8),(9),(10). Furthermore, vegetable oils are abundantly available from agricultural feedstocks, including seeds, kernels, leaves, and other lignocellulosic residues, ensuring a sustainable and cost-effective supply (11),(12). Their use reduces dependence on fossil-based chemicals while adding value to agricultural by-products and biomass waste (13),(14),(15). The adoption of vegetable oil–based demulsifiers aligns with green chemistry principles by minimizing ecological impact, improving biodegradability, and supporting circular bioeconomy strategies (16),(17), thereby contributing to SDG 12: Responsible Consumption and Production. In addition, replacing persistent petrochemical surfactants with biodegradable bio-based demulsifiers can significantly reduce marine pollution from petroleum-processing effluents while advancing circular bioeconomy principles through the sustainable utilization of renewable biological resources.

The demulsifying performance of vegetable oils arises from their triglycerides, fatty acids, and naturally occurring surface-active compounds (18),(19),(20), which adsorb at the oil-water interface, reduce interfacial tension, disrupt stabilizing films of asphaltenes and resins, and facilitate coalescence of water droplets (21),(22). Efficiency is influenced by structural features such as chain length, degree of unsaturation, and functional groups (23),(24),(25). Vegetable oils can be applied directly or chemically modified (e.g., sulfonation, ethoxylation, esterification) to improve solubility and interfacial activity under challenging conditions (26). Notable examples include neem (Azadirachta indica), calabash (Crescentia cujete), mahogany (Khaya spp.), castor, and palm kernel oils (27),(28), which have demonstrated moderate to high demulsification efficiency. The use of these bio-based oils promotes SDG 9: Industry, Innovation and Infrastructure by enabling cleaner industrial practices and SDG 14: Life Below Water by reducing potential contamination from petrochemical surfactants.

Despite their potential, knowledge gaps persist, particularly concerning underutilized non-edible oils like calabash, mahogany, and neem. Few studies systematically compare multiple natural oils with commercial demulsifiers under identical conditions, or employ statistical validation (ANOVA, Tukey post hoc) to assess significant performance differences. Data on reproducibility and consistency, essential for industrial adoption, are also limited. This study addresses these gaps by synthesizing and characterizing demulsifiers from neem, calabash, and mahogany oils and evaluating their efficiency, reproducibility, and statistical performance against a commercial control, providing evidence for locally sourced, sustainable alternatives to petrochemical demulsifiers. Another limitation of earlier investigations is the lack of detailed reproducibility and consistency data, which are essential for assessing industrial feasibility. Many studies rely primarily on qualitative observations or single-parameter evaluations without integrating quantitative efficiency metrics, statistical significance, and benchmarking against industry standards. Therefore, this study addresses these limitations by synthesizing demulsifiers from neem, calabash, and mahogany oils and evaluating their demulsification efficiencies, reproducibility, and statistical performance relative to a commercial demulsifier supplied by Nigerian National Petroleum Company Limited.

The primary goal of this study is to develop and evaluate environmentally friendly demulsifiers derived from locally available vegetable oils-Mahogany (Khaya spp.), Neem (Azadirachta indica), and Calabash (Crescentia cujete) for their effectiveness in breaking crude oil-water emulsions as sustainable alternatives to conventional petrochemical surfactants. To achieve this, bio-based demulsifiers is synthesized through controlled thermal blending of lipophilic and hydrophilic components using camphor, paraffin oil, cassava starch, and liquid soap as reactants and binders. The demulsification efficiency of each formulation is evaluated using standardized bottle tests, and their performance are compared with a commercial NNPC demulsifier to assess industrial applicability. Reproducibility and stability were examined by calculating mean efficiency, standard deviation, and coefficient of variation across replicate tests, while statistical analyses, including ANOVA and Tukey's post hoc test, were employed to determine the significance of differences among the formulations.

2. Methods

Oils obtained from Khaya spp. (mahogany), Azadirachta indica (neem), and Crescentia cujete (calabash) were locally sourced and used as the primary feedstocks for demulsifier preparation. Additional materials included analytical-grade camphor powder (lipophilic enhancer), paraffin oil (viscosity modifier and facial activity enhancer), cassava starch (hydrophilic stabilizer), and liquid soap (a binder), all purchased from local suppliers. The chemicals were used as received without additional purification, while distilled water was employed throughout all experimental procedures.

2.1 Preparation of Vegetable Oil–Based Demulsifiers

The synthesis of each demulsifier was performed in three successive stages: development of the lipophilic component, formation of the hydrophilic component, and subsequent binding of both phases to generate a stable amphiphilic system.

2.2 Formation of the lipophilic phase

Approximately 25 g of camphor powder was added to a beaker containing 15 g of the selected vegetable oil (mahogany, neem, or calabash). The mixture was placed on a thermostatically controlled hot plate at 40 °C and gently stirred until the camphor completely dissolved, producing a uniform lipophilic solution.

2.3 Formation of the hydrophilic phase

While maintaining continuous stirring, 10 g of paraffin oil was introduced into the heated camphor–oil mixture to facilitate blending. Thereafter, 30 g of prepared cassava starch was slowly added to incorporate the hydrophilic fraction of the formulation.

2.4 Binding and homogenization

To stabilize the interaction between the lipophilic and hydrophilic components, 20 g of liquid soap was added as a binding agent. The mixture was then stirred continuously on the heated hot plate at 40 °C for 60 minutes until a homogeneous product was formed. The same synthesis procedure was repeated separately using neem and calabash oils to produce their corresponding demulsifier formulations. Each prepared demulsifier was cooled to ambient temperature and stored in labeled airtight containers before evaluation.

2.5 Demulsification Test

For performance testing, 10 mL of crude oil emulsion was transferred into a graduated glass bottle, followed by the addition of 0.5 mL of the synthesized demulsifier (mahogany-, neem-, or calabash-based). A commercial demulsifier supplied by Nigerian National Petroleum Company Limited was used as the control. The bottles were tightly sealed and shaken vigorously for 2 minutes to ensure adequate mixing, after which they were left undisturbed at room temperature to allow phase separation. The volume of separated water was recorded at specified time intervals until no further separation occurred. The demulsification efficiency (%) was subsequently calculated according to Equation 1.

$$Demulsification\ Efficiency\ (\%) = \frac{V_s}{V_t} \times 100 \qquad (1)$$

where V_s is the volume of separated water, and V_t is the total volume of water initially present in the emulsion.

Each test was conducted in triplicate to ensure reproducibility. The mean values and standard deviations were determined, and statistical analyses such as ANOVA and Tukey's post hoc tests were used to evaluate the significance of performance differences among the demulsifiers.

3. Results and Discussion

The triplicate results of demulsification carried out with three synthetic vegetable oil demulsifiers, Neem oil, Calabash oil, and Mahogany oil, and a commercial demulsifier used as a control are presented in Table 1. The results presented in Table 1 indicate the demulsification efficiency of three synthesized crude oil demulsifiers (Neem, Calabash, and Mahogany) in comparison with a commercial demulsifier (Control, NNPC standard), with each test conducted in triplicate. Figure 1 shows the demulsification efficiency of the three demulsifiers and the NNPC control demulsifier.

Table 1. Demulsification of crude by synthesized and standard demulsifiers

Demulsifiers	**Demulsification Efficiency (%)**		
	1st	**2nd**	**3rd**
Neem	93.2	94.1	93.8
Calabash	92.0	93.4	92.5
Mahogany	96.4	96.8	96.6
Control	96.8	97.2	97.0

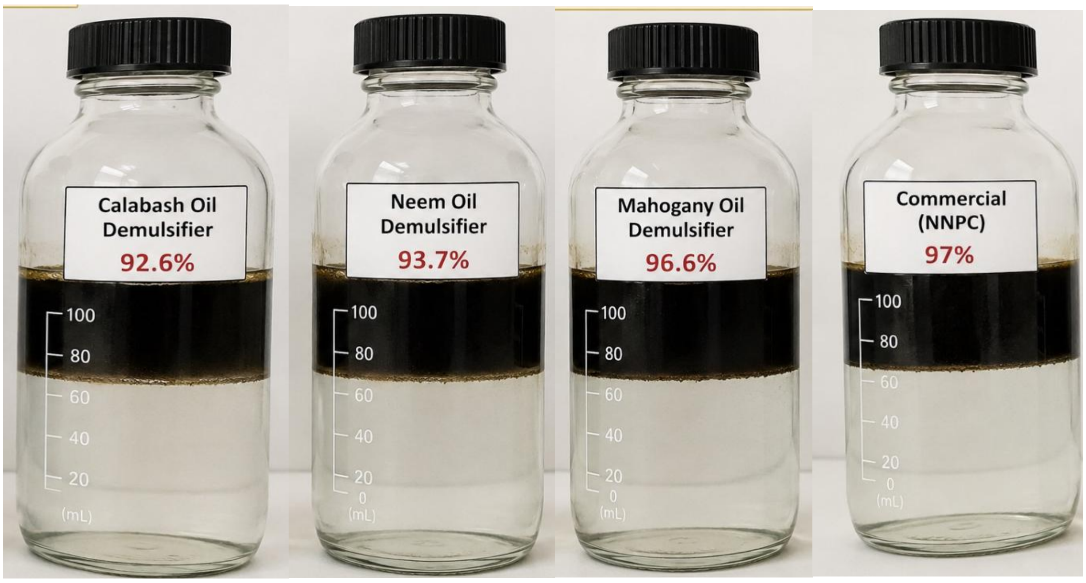

Figure 1. Demulsification of crude oil/water emulsion

3.1. Neem-Based Demulsifier

Neem showed efficiencies of 93.2%, 94.1%, and 93.8%, with an overall consistent performance. The values demonstrate that Neem is an effective demulsifier, achieving results above 93%, though slightly lower than Mahogany and the commercial control. This suggests Neem-derived products possess significant interfacial activity due to their natural surfactant content (triglycerides, fatty acids, and saponins).

3.2. Calabash-Based Demulsifier

Calabash exhibited efficiencies of 92.0%, 93.4%, and 92.5%, making it the lowest-performing of the synthesized demulsifiers. While still effective (>90%), its efficiency falls short of Neem and Mahogany, indicating weaker interfacial disruption or lower solubility compared to the others. This performance, however, demonstrates potential for further optimization through formulation or blending.

3.3. Mahogany-Based Demulsifier

Mahogany gave efficiencies of 96.4%, 96.8%, and 96.6%, closely matching the commercial control. This consistency shows excellent potential as a viable green alternative to petroleum-based demulsifiers. The high efficiency may be attributed to the chemical composition of Mahogany extracts, which likely contain stronger surface-active molecules capable of breaking crude oil-water emulsions effectively.

3.4. Control (NNPC Commercial Demulsifier)

The control exhibited efficiencies of 96.8%, 97.2%, and 97.0%, which represent the benchmark for comparison. Mahogany's performance is nearly identical to this standard, while Neem and Calabash show slightly lower but still appreciable values. Mahogany ≈ Control (NNPC standard): Mahogany demonstrated demulsification efficiency almost indistinguishable from the commercial product, suggesting strong suitability for industrial application. Neem (93-94%) > Calabash (92-93%): Neem outperformed Calabash, indicating that the type of biomass feedstock significantly influences demulsification potential. All synthesized demulsifiers achieved efficiencies above 92%, confirming their promise as eco-friendly and renewable alternatives to synthetic chemical demulsifiers currently in use. The findings highlight that Mahogany is the most promising bio-derived demulsifier, achieving near-parity with the NNPC commercial standard. Neem also performed well, while Calabash, though slightly less efficient, still demonstrated acceptable activity. These results support the feasibility of developing biomass-based demulsifiers as sustainable, cost-effective, and environmentally benign replacements for conventional petrochemical demulsifiers in crude oil processing.

3.5. Reproducibility

The mean demulsification for Neem demulsifier is 93.7%, and its standard deviation is 0.46, as presented in Table 2. This is good productivity. The mean and standard deviation for demulsification by calabash oil were 92.6% and 0.71, respectively. This is a slightly higher variation, but still reproducible. The mean and standard deviation of demulsification by Mahogany oil were 96.6% and 0.2, respectively. This is a very high reproducibility. And finally, the mean and reproducibility of the standard demulsifier's demulsification were 97.0% and 0.2, respectively. Like in Mahogany oil, it has very high reproducibility.

Table 2. Reproducibility Table of demulsification of the synthesized demulsifiers

Demulsifier	Mean Efficiency (%)	Std. Dev.
Neem	93.7	0.46
Calabash	92.6	0.71
Mahogany	96.6	0.20
Control	97.0	0.20

3.6 ANOVA

Statistical Significance (ANOVA*)* is shown in Table 3, the f-value and p-value are 69.83 and 4.44×10^{-6}, which is less than 0.05 (<0.05). This means there is a highly significant difference among the demulsifiers in terms of demulsification efficiency. All four demulsifiers show good reproducibility, but Mahogany and Control perform best with the highest efficiency and lowest variability. The ANOVA confirms that the observed differences between demulsifiers are statistically significant. The very low p-value (< 0.001) confirms that the type of demulsifier has a highly significant effect on efficiency.

Table 3: Analysis of statistical significance for the demulsification efficiency

Source	Sum of Squares	df	F-value	p-value
Demulsifier	41.55	3	69.83	0.000004
Residual	1.59	8		

The post-hoc comparison results in Table 4 reveal clear statistical differences among the demulsifiers tested. Calabash exhibited significantly lower demulsification efficiency compared with both the Control and Mahogany (mean differences of -4.37 and -3.97, p = 0.001, respectively). This indicates that Calabash is less effective than these two formulations.

Similarly, Calabash did not differ significantly from Neem (mean difference -1.07, $p = 0.053$), suggesting that these two demulsifiers perform at a comparable level.

Table 4. Tukey Post-Hoc Test results

Comparison	Mean Diff	p-value	Lower CI	Upper CI	Significant
Calabash–Control	-4.37	0.001	-5.61	-3.12	Yes
Calabash–Mahogany	-3.97	0.001	-5.21	-2.72	Yes
Calabash–Neem	-1.07	0.053	-2.31	0.17	No
Control–Mahogany	0.40	0.468	-0.85	1.65	No
Control–Neem	3.30	0.001	2.05	4.55	Yes
Mahogany–Neem	2.90	0.001	1.65	4.15	Yes

When comparing the Control with Mahogany, no significant difference was observed (mean difference, 0.40; p = 0.468), indicating that the two are statistically equivalent in efficiency. However, the Control was significantly more effective than Neem (mean difference, 3.30; p = 0.001), while Mahogany also significantly outperformed Neem (mean difference, 2.90; $p = 0.001$), as shown in Table 4. Overall, the results establish a performance hierarchy: Mahogany and Control are the most effective demulsifiers, with no significant difference between them; Neem and Calabash exhibit lower performance, with no significant difference between the two. This suggests that Mahogany is a promising natural alternative comparable to the commercially available Control, whereas Neem and Calabash are relatively less potent.

Painuly *et al.* (28)(Painuly & Anand, 2024) reported high separation efficiency in bottle tests, indicating sunflower oil derivatives can be viable demulsifier feedstocks under optimized conditions. Azuokwu & Eiroboyi (29) reported the formulation of demulsifiers from Nicotiana tabacum (tobacco) seed oil combined with plant extracts/ash, carried out tests, and compared them to commercial demulsifiers. The authors reported promising demulsification performance comparable to some commercial demulsifiers for specific crude oils, highlighting a low-cost local feedstock option. Udourioh *et al.* (30) synthesized soybean oil chemically modified to form biobased demulsifiers (condensation with amines / surfactant-forming reactions) and carried out bottle tests (static) and BS&W measurements, achieving 100%. Soybean-derived demulsifiers (SOSD) reached comparable water-separation efficiencies to coconut-based formulations, with reported good biodegradability and eco-profile. Coconut oil (and coco-betaine derivatives)

Udourioh *et al.* (30) reported the direct application of coconut oil and its conversion into derivatives such as coco-betaine and other coconut-oil-based surfactants/demulsifiers, which were evaluated through bottle tests and thermal treatment methods. Several studies achieved water separation efficiencies of approximately 70-80% for crude oil emulsions under optimized conditions, with coco-betaine derivatives exhibiting faster phase separation and superior demulsification performance compared to unmodified coconut oil. Similarly, Wei *et al.* (31) enhanced the interfacial activity of castor oil by functionalizing it through maleation, oligomerization, and reactions with maleic anhydride and related reagents. The resulting modified castor-oil-based demulsifiers showed significant water removal capability, often reported as "maximum water removal" in both bottle and interfacial tension (IFT) tests. Saad *et al.* (32) also synthesized efficient corn-oil-based surfactant/demulsifiers via condensation reactions with diethanolamine or related pathways, achieving up to 98% water removal in bottle tests for certain crude oil emulsions. By comparison, the vegetable oils investigated in the present study, Neem, Calabash, and Mahogany, also demonstrate strong potential as renewable feedstocks for the formulation of crude-oil demulsifiers. These comparative studies are presented in Table 5.

Table 5. Summary of Selected Vegetable Oil-Based Demulsifiers and Their Performance

Study (Year)	**Vegetable Oil Source**	**Modification / Derivative**	**Evaluation Method(s)**	**Key Findings / Performance**
Udourioh *et al.* (30)	Coconut oil	Converted to derivatives such as coco-betaine and other coconut-oil-based surfactants/demulsifiers	Bottle tests and thermal treatment methods	Achieved 70-80% water separation under optimized conditions; coco-betaine derivatives exhibited faster phase separation and superior demulsification efficiency compared to unmodified coconut oil.
Wei et al. (31)	Castor oil	Functionalized via maleation, oligomerization, and reactions with maleic anhydride and related reagents	Bottle tests and interfacial tension (IFT) measurements	Modified castor-oil demulsifiers showed high interfacial activity and substantial water removal, often reported as "maximum water removal."
Saad et al. (32)	Corn oil	Chemically modified through condensation with diethanolamine and related routes	Bottle tests	Produced efficient corn-oil-based surfactant/demulsifiers, achieving up to 98% water removal in certain crude oil emulsions.
Present study	Neem, Calabash, and Mahogany oils	— (Evaluated as potential feedstocks)	Bottle tests (planned/ongoing)	Oils show strong potential as renewable feedstocks for crude-oil demulsifier development.

Conclusions

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This study successfully developed and validated bio-based demulsifiers derived from Mahogany (*Khaya* spp.), Neem (*Azadirachta indica*), and Calabash (*Crescentia cujete*) oils as sustainable alternatives to conventional petrochemical demulsifiers for crude oil–water separation. All formulations exhibited high demulsification efficiencies above 92%, with the Mahogany-based demulsifier achieving the highest efficiency (96.6%), closely comparable to the commercial NNPC control (97.0%). The Mahogany formulation also demonstrated the best reproducibility and interfacial stability, as indicated by its low standard deviation (0.20), while the Neem- and Calabash-based formulations showed effective and stable separation performances of 93.7% and 92.6%, respectively.

Statistical analyses using ANOVA and Tukey's post hoc test confirmed significant differences among the formulations ($p < 0.001$), although the Mahogany-based demulsifier showed no significant difference from the commercial product, indicating comparable performance. These findings demonstrate the strong potential of locally sourced vegetable oils as efficient, biodegradable, and environmentally friendly substitutes for synthetic demulsifiers in crude oil processing.

Beyond their technical effectiveness, the developed bio-based demulsifiers contribute to sustainable industrial practices by reducing dependence on fossil-derived chemicals, promoting renewable resource utilization, and minimizing environmental pollution associated with petrochemical surfactants. Their adoption supports cleaner crude oil processing operations and aligns with circular bioeconomy principles as well as SDG 9 (Industry, Innovation and Infrastructure), SDG 12 (Responsible Consumption and Production), and SDG 14 (Life Below Water).

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Recommendation

Future work should focus on optimizing the molecular design of these natural formulations through mild chemical modification (e.g., sulfonation or ethoxylation) to enhance their solubility, thermal stability, and field-scale applicability under varying crude oil compositions and operating conditions.

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Conflicts of Interest

There is no conflict of interest among the authors as regards to the submission of this article to the Asian Journal of Environmental Research.

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