



Investigating the Potential of Wild Yam (*Dioscorea villosa*) Plant Extract for Asphaltene Dispersion in Crude Oil System

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ABSTRACT:

Asphaltene is a component that has been known to cause major problems when crude oil is extracted and transported from the reservoir. Asphaltenes molecules in crude oil possess the phenomenon of aggregation and therefore causes rigidity and hinders flow of crude oil. This study therefore investigated the use of saponin as plant-based surfactants in the treatment of asphaltenes contained in petroleum crude during production of crude oil. Solvent extraction technique was adopted in extraction of saponin from wide yam using 500ml flask placed on a heating mantle. The dispersion of asphaltene was optimized with a response surface methodology via central composite design of experiment taking surfactant dosage 50 – 500mg/g, dispersion temperature 30 – 60°C and dispersion time 20 – 60 minutes as factors. Results obtained revealed wide yam ethanolic extract contain a high amount of saponin and asphaltene containing crude is very high in viscosity 179.74cP and acid number 1.69mgKOH/g. The statistical optimization yielded a significant model with an F-value of 35.35 and P-values of all factors less than 0.0500. The Predicted R^2 of 0.8326 was in reasonable agreement with the Adjusted R^2 of 0.9421. Therefore, response surface methodology with central composite design of experiment effectively optimized the dissolution of asphaltene having optimum condition for each factor factors were reaction temperature 45.7°C, time 39.8 minutes, surfactant dosage 246.6mg/g.

Keywords: Asphaltene, Saponin, Natural surfactant, Crude oil, Dispersion.

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INTRODUCTION

Asphaltenes are complex, high-molecular-weight compounds found in crude oil, mainly made up of carbon and hydrogen, along with varying amounts of nitrogen, oxygen, and sulfur [1]. Their thermodynamic behavior is quite intricate, showing considerable variability across different oil reservoirs and often lacking a clear molecular weight [2]. While asphaltenes dissolve well in aromatic solvents, they don't mix with normal alkanes, which is why these alkanes are used to precipitate them from crude oil [3]. Generally, the concentration of asphaltenes rises with the heavy oil content, making oil production and refining more challenging. Additionally, asphaltenes can disrupt emulsions



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and change surface wettability, which can significantly impact enhanced oil recovery efforts [4; 5; 6]. Effectively managing asphaltene stability is essential for reducing operational costs and ensuring efficiency during oil extraction and transportation [7]. This study aims to investigate the potential of saponins, natural surfactants derived from wild yams, to improve asphaltene dispersion in crude oil. The goals include extracting and characterizing saponin, assessing its effectiveness in dispersing high asphaltene content, and optimizing this process. By utilizing natural plant extracts, this research could offer cost-effective solutions to tackle asphaltene-related issues in the petroleum industry.

MATERIALS AND METHODS

The investigation focused on wild yams (*Dioscorea villosa*), using it as the key plant extract and main raw material. Table 1 provides a detailed overview of the essential equipment used throughout this study, highlighting their specific roles and contributions to the research process.

Table 1. Equipment Used

Apparatus/Equipment	Description/Model	Uses
Soxhlet extractor	500ml jinotec glass	Used for dewaxing
Digital Weighing Balance	JOANLAB FA2003S Guandong China	Used for measuring the required gram of wild yam (<i>DIOSCOREA VILLOSA</i>) as highlighted from the software for optimization
Magnetic stirrer	MS300 Jinotec Instrument China	Used for mixing the methanol and the grounded wild yam (<i>DIOSCOREA VILLOSA</i>).
Oven	Vacutherm VT 6025 Air-Dry Vacuum Oven. Thermo Scientific NJ, USA	Used in drying of the extract.
Viscosity	NDJ-5S Viscometer and a density bottle. Anton Paar, UK	For determination of asphaltene containing crude oil.

Sample Collection and Preparation

The wild yams used in this study (as shown in Figure 1) were freshly harvested from the forests around the premises of the Federal University of Petroleum Resources Effurun, Nigeria. To keep the plants as fresh as possible, they were harvested early in the morning and quickly taken to the laboratory, arriving before noon the same day.



Figure 1. Freshly Harvested Wild Yams (*Dioscorea villosa*)

Once in the laboratory, the fresh yam was prepared by peeling and cutting it into small, manageable pieces. These pieces were then sun-dried for two days (see Figure 2) to significantly lower their



moisture content. After that, an oven-drying process at 65°C for 6 hours was carried out until a constant weight was reached, following the method outlined by Yunita et al. [8].



Figure 2. Sun Dried Peeled Wild Yams (*Dioscorea villosa*)

Subsequently, the dried yam was then ground using a mechanical grinder and carefully sieved to achieve a smooth consistency with a 250µm sieve as seen in Figure 3. The finely processed yam powder was packed securely in a nylon bag, wrapped up, and stored in an airtight container to maintain its quality until it was needed for the study.



Figure 3. Granulated Wild Yam Sample before Dewaxing

The chemicals and reagents used in this study were carefully sourced from a mix of internationally certified producers and local analytical chemical suppliers, ensuring they met the highest standards. This included Methanol (99.8% pure) and Ethanol (99.7% pure) from JHD in Shatou, Guangdong, China; Phenolphthalein Indicator from Kermel Chemicals Reagent Company Ltd in Tianjin, China; and N-Hexane (37% pure analytical grade) from Loba-Chemie in India. We also obtained distilled and deionized water from the LUCO Chemical Engineering Laboratory at Benin City, Nigeria.



The experimental procedure featured a two-stage solvent extraction process:

- i. Saponin extraction and
- ii. Optimization of Asphaltene Dispersion.

For the saponin extraction, we used a reflux technique. A dewaxed plant sample was weighed and placed into a round-bottom flask with a specific volume of ethanol, then agitated vigorously for 120 minutes before being refluxed at a consistent temperature for one to 6 hours. After that, the extract as seen in Figure 4 was filtered through a Buchner funnel, and the filtrate was concentrated by slow evaporation at 65 °C until we obtained a dry solid extract, which was then weighed and stored for phytochemical screening. Equation (1) was used to determine the percentage yield.

$$\text{Yield (\%)} = \frac{\text{mass of extract}}{\text{mass of sample}} \quad (1)$$



Figure 4. Wild Yam Ethanolic Extract

Experiment Design for Asphaltene Dispersion

The procedure was carried out according to the experimental design (Table 2) obtained from statistical software (DESIGN EXPERT 13.0.1). Twenty (20) experimental runs were obtained from varying extraction solvent amount, extraction time and extraction temperature. A three-factor Central Composite Design (CCD) was employed for experimental design. The responses obtained from the CCD were optimized using response surface methodology. Factors to be optimized were coded at 3 levels (see Table 3) which gave range for the surfactant dosage (50 – 500mg/g), temperature (30 – 60°C), Time (20 – 60minutes). The depression in viscosity was chosen as the response for process optimization using Response Surface Methodology (RSM). The experimental design carried out made up to 20 runs. Experimental observations from the asphaltene depression process were analyzed and fitted according to a quadratic design model as a second order polynomial equation. Analysis of variance (ANOVA) and response surface plots were generated from the statistical Design Expert software. The optimized value of the independent variables for optimum response was determined using numerical optimization.

Table 2. Design Build Information

Design	Information
File Version	13.0.1.0
Study Type	Response Surface
Design Type	Central Composite
Design Model	Quadratic
Build Time (ms)	65.00
Subtype	Randomized



Table 3. Design Factors and Ranges

Factor Name	Units	Minimum	Coded Low	Mean	Coded High	Maximum
A: Surfactant dosage	mg/g	50.00	-1 ↔ 141.21	275.00	+1 ↔ 408.79	500.00
B: Temperature	°C	30.00	-1 ↔ 36.08	45.00	+1 ↔ 53.92	60.00
C: Time	minutes	20.00	-1 ↔ 28.11	40.00	+1 ↔ 51.89	60.00

RESULTS

Chemical Property of Wild Yam Extract

Figure 5 depicts the FTIR spectrum of phyto-based surfactant conducted to detect the presence of functional groups on the surface of the extracted surfactant. The FT-IR spectra were observed in the 1000 cm^{-1} to -3500 cm^{-1} range. A medium peak was obtained at 3444.57 cm^{-1} and it's attributed to an intermolecular bonded stretching primary amine with a functional group ($-\text{N}-\text{H}$). A strong peak obtained at 1643.82 cm^{-1} was attributed to a monosubstituted stretching alkene with a functional group ($-\text{C}=\text{C}$). The other strong stretching peak observed at 1036.25 cm^{-1} was attributed to anhydrous hydrate sulfoxide with a functional group ($-\text{S}=\text{O}$). As observed, the peaks of the spectrum elucidate the efficient formation of a surfactant with both hydrophobic end $-\text{C}=\text{C}$ and a hydrophilic end $-\text{S}=\text{O}$. These observations support the presence of an active surfactant.

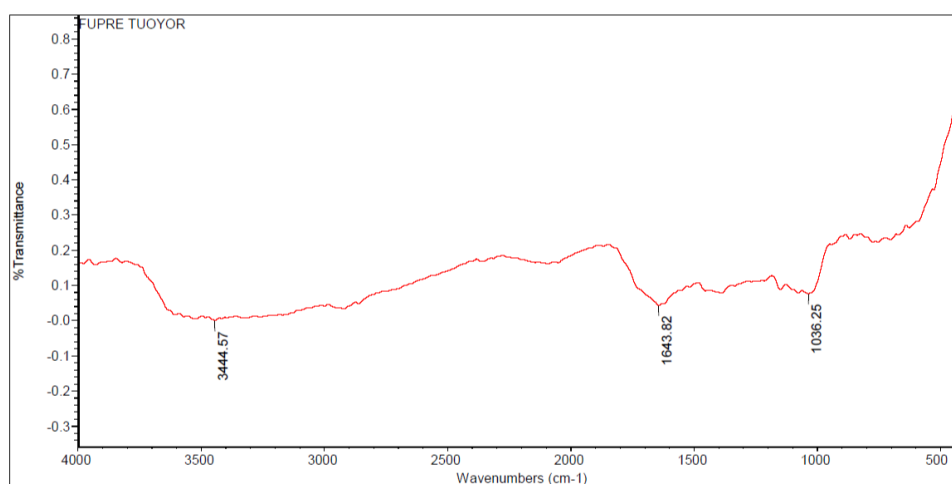


Figure 5. FTIR Spectrum Showing Peaks of Functional Groups Present in Saponin from Wild Yam

Properties of Asphaltene Containing Crude Oil

The density, specific gravity, and viscosity of a crude oil sample determine its cloud point and pour point [8; 9]. Table 4 illustrates the relationship between density and specific gravity. Additionally, since fluid becomes more viscous the denser it is, specific gravity has an impact on viscosity. Viscosity directly affects density, specific gravity, and pour point [10]; the more closely any of these three parameters match the requirements, the more likely it is that a crude oil sample will meet them. A viscosity of 179.74cp is excessive and will impact flow [11].

Table 4. Physiochemical Properties of Asphaltene Crude Oil Sample

Property	Unit	Value
Sp. Gravity at 45°C	-	0.8612
D. viscosity at 30°C	cP	179.74
°API Gravity at 30°C	-	32.81
Sulphur Content	wt%	0.72
Acid content	mg KOH/g	1.69



The API gravity establishes the grade or quality of crude oils [12]. Light crude oil is often defined as having an API gravity of 31 or higher; medium crude oil is defined as having an API gravity of 22 to 31; and heavy crude oil is defined as having an API gravity of 20 or less (Table 4).

Optimization of Asphaltene Depression Process

The efficiencies of the dissolution process evaluated for the asphaltene containing crude. The reflux extraction method adopted yielded saponin for complete asphaltene dispersion and the factors had significant effects on the extraction efficiency. The maximum extract yield was 13.9 % was obtained using 50g of the material.

ANOVA for Asphaltene Viscosity Depression

The Model F-value of 35.35 implies the model is significant (Table 5). There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC, A², B², C² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The Lack of Fit F-value of 1.97 implies the Lack of Fit is not significant relative to the pure error. There is a 23.72% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good.

Table 5. The ANOVA for Asphaltene Dispersion

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	8052.28	9	894.70	35.35	< 0.0001	significant
A-Surfactant dosage	1224.30	1	1224.30	48.38	< 0.0001	
B-Temperature	848.44	1	848.44	33.52	0.0002	
C-Time	126.38	1	126.38	4.99	0.0495	
AB	1084.62	1	1084.62	42.86	< 0.0001	
AC	665.94	1	665.94	26.31	0.0004	
BC	60.45	1	60.45	2.39	0.1533	
A ²	2450.14	1	2450.14	96.81	< 0.0001	
B ²	1405.24	1	1405.24	55.52	< 0.0001	
C ²	928.23	1	928.23	36.68	0.0001	
Residual	253.08	10	25.31			
Lack of Fit	167.88	5	33.58	1.97	0.2372	not significant
Pure Error	85.20	5	17.04			
Cor Total	8305.36	19				

Model Fit Statistics

The Predicted R² of 0.8326 is in reasonable agreement with the adjusted R² of 0.9421 (Table 6); i.e. the difference is less than 0.2. Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. A ratio of 21.041 indicates an adequate signal. This model can be used to navigate the design space.

Table 6. The Fit Statistics

Regression	Value
R ²	0.9695
Adjusted R ²	0.9421
Predicted R ²	0.8326
Adequate Precision	21.0408
Standard Deviation	5.03
Mean	37.33
Coefficient of Variance %	13.47



Final Equation in Terms of Coded Factors

The Equation (2) gives prediction in terms of coded factors and can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

$$\text{Dynamic viscosity (cP)} = 16.21 + 9.47A - 7.88B - 3.04C - 11.64AB - 9.12AC + 2.75BC + 13.04A^2 + 9.87B^2 + 8.03C^2 \quad (2)$$

Model Diagnostics of Parity Plot

The relationship between the predicted and actual values of experimentally generated dynamic viscosities are shown in Figure 6 from the central composite experimental design. It is observed from the plots that the actual dynamic viscosity at various surfactant dosages is distributed near the predicted dynamic viscosity. This is an indication that there is high correlation with $R^2 = 0.9695$. This also suggests that the actual experimental data are well fit with the predicting models and convincingly is a good estimate of the actual dynamic viscosity of disperse asphaltene.

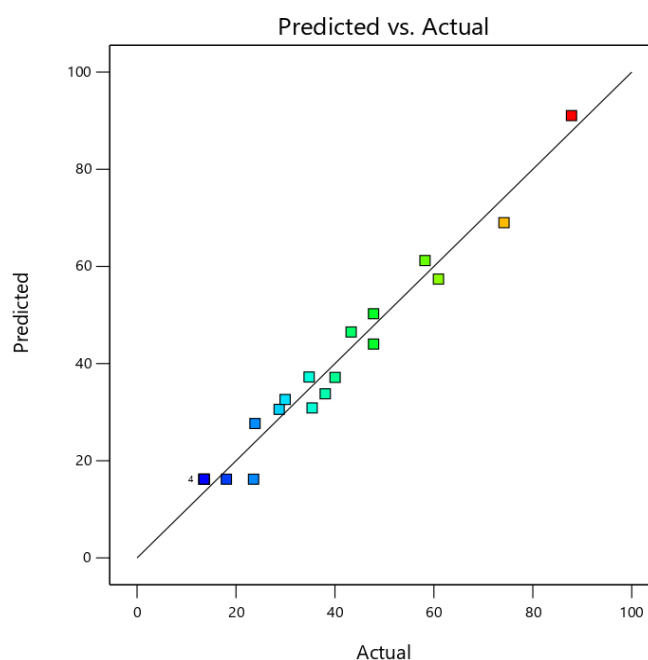


Figure 6. Parity Plot of Predicted Viscosity

Optimization Response Surface Plots of Dynamic Viscosity Reduction

Response surface methodology (RSM) was successfully applied for optimization of dynamic viscosity reduction of asphaltene dispersion by natural surfactant. RSM was illustrated with three-dimensional plots by presenting the effects of two variables on the dynamic viscosity reduction and keeping the other variable constant. The dynamic viscosity reduction in relation to the interactions between surfactant dosage, temperature, and time are shown in Figures 7 and Figure 8 for the optimization results. The plots clearly demonstrate the effect of varying the surfactant dosage on dynamic viscosity reduction at different solid masses. It can be observed that as the surfactant dosage and time increases, the dynamic viscosity also decreases, reaching a peak at a certain point



before increasing. The dispersion time, on the other hand, shows a less significant impact on dynamic viscosity reduction.

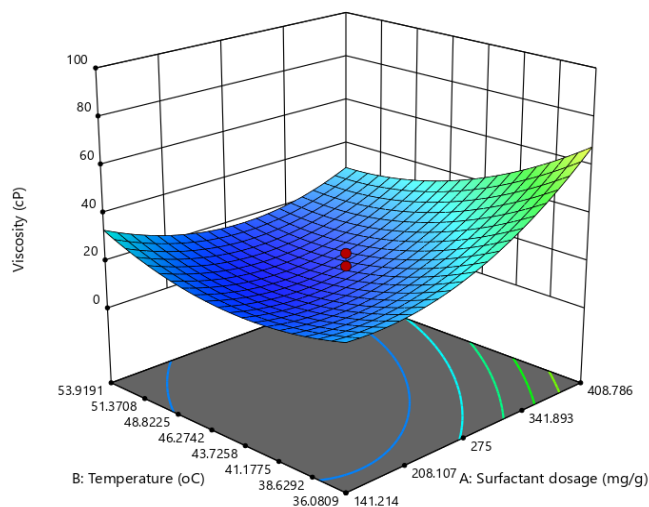


Figure 7. Response Surface Plot of the Interaction of Surfactant Dosage and Solubilization Temperature on Dynamic Viscosity of Dispersed Asphaltene Containing Crude Oil

The effects of these three independent variables (surfactant dosage, time and temperature) on the dynamic viscosity is shown in Figures 7 to 10. Viscosity reduction improved with increasing surfactant dosage and temperature of the dispersion process as shown in Figure 9; when the time was increased from about 28 minutes to 40 minutes at which viscosity reduction 38cP to about 19cP was recorded. However, upon increasing the surfactant dosage and time beyond 40 minutes leads to increase viscosity and therefore reduces wettability.

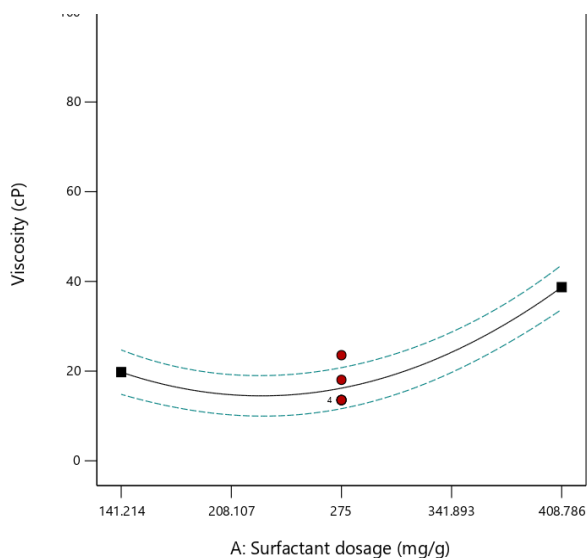


Figure 8. 2-D Response Surface Plot of the Effects of Surfactant on Dynamic Viscosity of Dispersed Asphaltene Containing Crude Oil

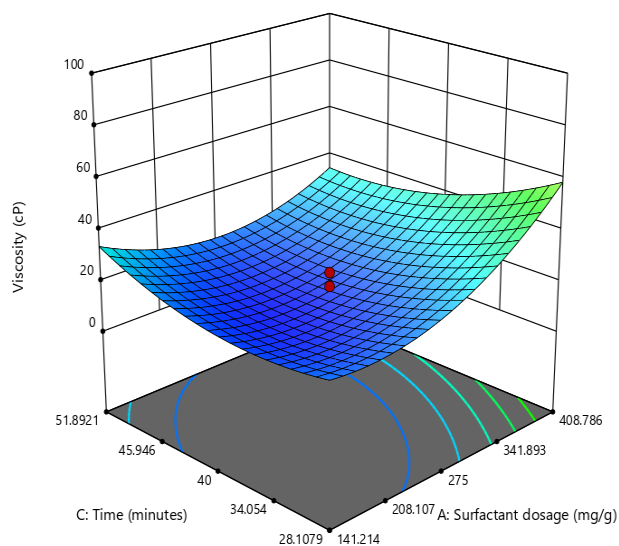


Figure 9. Response Surface Plot of the Interaction of Resident Time and Surfactant Dosage on Dynamic Viscosity of Dispersed Asphaltene Containing Crude Oil



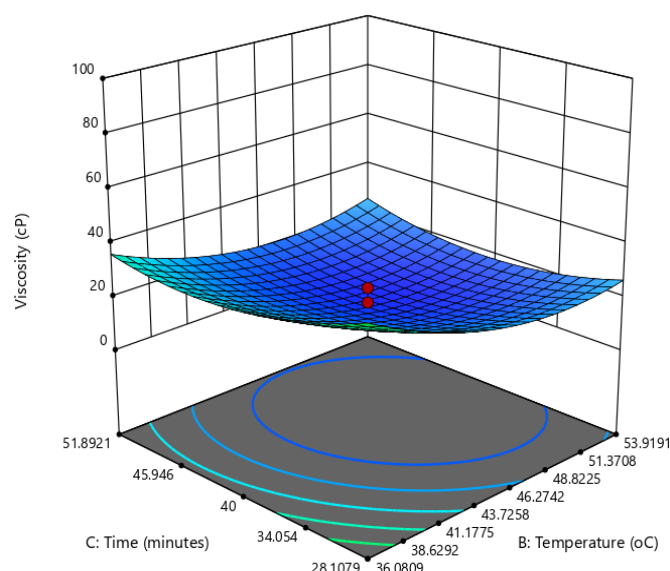


Figure 10. Response Surface Plot of the Interaction of Resident Time and Dissolution Temperature on Dynamic Viscosity of Dispersed Asphaltene Containing Crude Oil

Optimized Results of Experimental Design

The optimum conditions of the input variables using central composite design in a response surface methodology were determined from the numerical optimization. Figure 11 shows the comprehensive optimum condition for the asphaltene depression process. Optimum condition for each factor factors were reaction temperature 45.7°C, time 39.8 minutes, surfactant dosage 246.6mg/g.

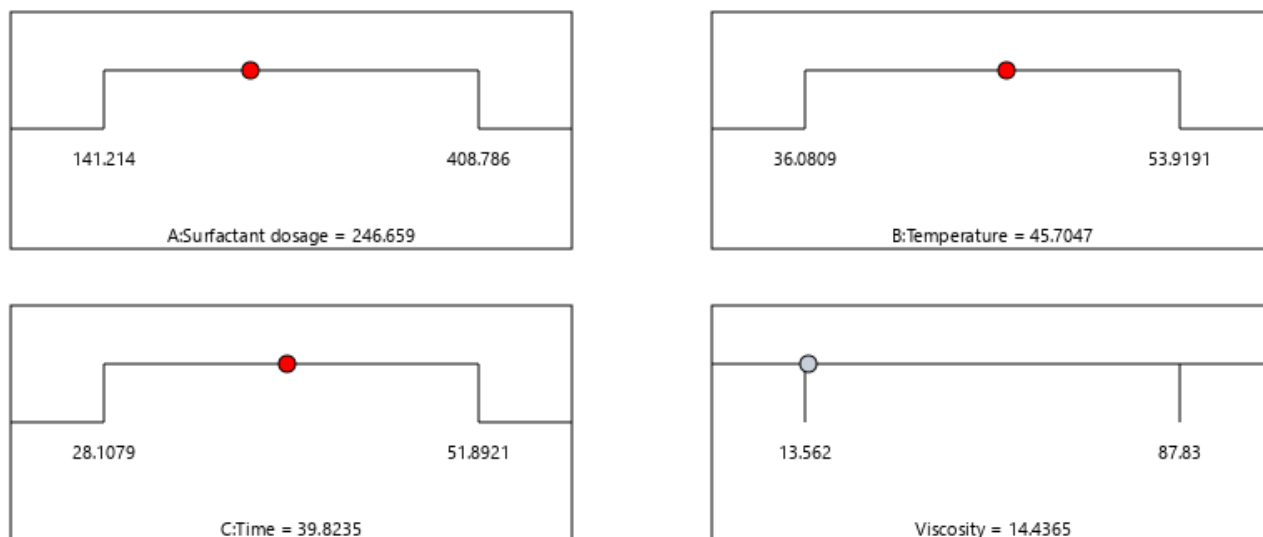


Figure 11. Optimum Results of Resident Time. Temperature and Surfactant Dosage on Dynamic Viscosity of Dispersed Asphaltene Containing Crude Oil

DISCUSSION

This study explored the potential of saponins extracted from wild yams (*Dioscorea villosa*) as natural surfactants for improving asphaltene dispersion in crude oil. As previously highlighted, asphaltenes present significant challenges to oil production and refining processes due to their high molecular



weight and propensity to disrupt emulsions [4; 5; 6]. The insights garnered from this investigation contribute meaningfully to the existing body of literature by providing an environmentally friendly approach to mitigating the issues associated with asphaltene stability. Our findings demonstrated that the saponin extracts from the wild yams sample effectively dispersed high concentrations of asphaltenes, aligning with our hypothesis that natural surfactants could enhance dispersion efficiency. This result corroborates existing knowledge regarding the utility of surfactants in altering the behaviour of asphaltenes and emphasizes the potential of plant-derived compounds as alternatives to synthetic surfactants in line with Al-Hosani et al. [2] and Fakher et al. [7]. The optimization of the dispersion process suggests that saponins may also offer a cost-effective solution to the petroleum industry, addressing not only the technical issues of asphaltene stability but potentially reducing operational expenditures associated with chemical surfactants.

One notable limitation of this study was the focus on a single type of plant extract. While wild yams (*Dioscorea villosa*) showed promise, the variability in saponin content across different plants and environmental conditions could affect the reproducibility of results. The results of this study challenge the notion that only synthetic surfactants can effectively manage asphaltene-related issues and pave the way for further exploration of natural alternatives in the petroleum sector. By integrating saponins into oil recovery processes, the research not only highlights a sustainable option but also enhances existing theories regarding surfactant interactions with complex crude oil components.

CONCLUSION

This study has demonstrated that saponins extracted from wild yams (*Dioscorea villosa*) can effectively improve asphaltene dispersion in crude oil. This finding has implications not only for the petroleum industry but also for other sectors that deal with complex hydrocarbon mixtures. By utilizing natural surfactants, we may be able to reduce reliance on synthetic chemicals, which can be harmful to the environment. This aligns with the growing trend toward sustainable practices in industrial processes.

Future research should explore the broader applications of saponins in various oil field conditions to assess their effectiveness in real-world scenarios. Additionally, investigating other plant species known for their saponin content could reveal new solutions for managing asphaltenes. There is still much to learn about the optimal conditions for saponin extraction and application, as well as understanding potential interactions with other compounds in crude oil. Thus, practical applications could extend beyond oil recovery. Future studies could assess how saponins perform in varying concentrations and their effects on other aspects of crude oil processing.

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CONFLICT OF INTERESTS

No conflict of interest.

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