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ANALYSIS OF FACTORS AFFECTING THE EFFICIENCY OF *JATROPHA CURCAS* OIL AS AN ASPHALTENE STABILISER

Tomás Darío Marín Velásquez[a,*], Dany Day Josefina Arriojas Tocuyo[b]

[a] *Universidad de Oriente, Petroleum Engineering Department, Av. Universitaria, Maturín 6201, Monagas, Venezuela*

[b] *Petróleos de Venezuela, Data Analysis Management, Campo Rojo, Punta de Mata 6217, Monagas, Venezuela*

[*] corresponding author: tmarin@gmx.es

Abstract. The effect of temperature and applied dose on the efficiency of *Jatropha curcas* seed oil as an asphaltene stabiliser was studied. Two crude oil samples (light and medium) were used. *J. curcas* oil was subjected to heating at 100, 150 and 170 °C for 24 h with an unheated sample (25 °C) and applied at doses of 2, 4, 6, and 8 µL in 10 ml of sample. The asphaltene instability index (AII) was determined as the ratio between the amount in ml of n-heptane to flocculate the asphaltenes and the amount in ml of xylene to disperse the flocs. The experimental design was Taguchi factorial with a response surface for one response variable (AII) and two experimental factors (the applied dose and heating temperature). For light crude oil, the optimum conditions were 8 µL and T = 127 °C with an 85.3 % efficiency and for medium crude oil, 2 µL and T = 25 °C with a 94.3 % efficiency. The efficiency of *J. curcas* oil and the influence of the type of crude oil on the results obtained were demonstrated.

Keywords: Stability, asphaltenes, flocculation, dispersion, *Jatropha curcas*.

1. Introduction

Asphaltenes are a heavy fraction of petroleum that shows a capacity for self-association and aggregate formation, a phenomenon that can occur in any of the different stages of production and processing, due to variations in pressure, temperature, composition, and shear, among others [1]. Although the definition of asphaltenes has been the subject of discussion over the years, most researchers agree in defining them as the heavy organic components present in crude oil that are soluble in toluene and insoluble in heptane/pentane [2].

According to the colloidal theory, asphaltenes are surrounded by molecules of a similar structure, called resins, which interact with asphaltenes to improve their solubility in aliphatic media and stabilise in crude oil [3]. The aforementioned authors also consider that the asphaltenes-crude oil system is in a thermodynamically unstable state in many cases, which causes crude oils to be produced where the asphaltenes are unstable, that is when changes occur in the system conditions, they tend to separate from the liquid phase producing the phenomenon known as asphaltene precipitation. The stability of asphaltenes depends largely on the structure of the asphaltenes and their interaction with the rest of the oil components. However, the detailed molecular composition of asphaltenes remains unknown in many cases, which is because crude oil is made up of millions of different organic molecules and asphaltenes are the most complex of all [4].

Unstable asphaltenes form aggregates that precipitate and deposit in pipelines and process equipment, causing plugging and loss of productivity, which has generated a wide field of study and research on asphaltene stability and the mechanisms governing it [5–9]. The determination of stability not only leads to defining the tendency of crude oil to produce asphaltene precipitation but also lays the foundation for the application of methods to prevent the phenomenon [10]. The use of asphaltene stabilising chemicals is the most widely used method for the prevention of asphaltene precipitation due to its effectiveness and low cost [11].

The study of the efficiency of asphaltene stabilising chemical compounds is of vital importance for the oil industry, which has led to investigating chemical compounds, such as ethoxylated nonylphenol and hexadecyl-trimethylammonium bromide, which have shown positive performance [11], likewise, the use of solvents such as toluene as a stabiliser have been studied for its effect as an asphaltene solvent [12]. Alkylphenols have also been studied as asphaltene stabilisers, also demonstrating positive effects [13]. The use of aromatic polyisobutylene as an asphaltene stabiliser has also been reported, with equally positive effects [14].

The use of synthetic chemical compounds as asphaltene stabilisers generates expenses and environmental risks that have led to the search for alternatives, among which are vegetable oils, such as coconut oil, sweet almond oil, andiroba oil, and sandalwood oil, which have shown a certain degree of efficiency in asphaltene stabilisation [15]. Also, coconut oil was evaluated with positive results indicating that such oil can achieve efficiencies even higher than those of synthetic commercial products [16, 17]. Another vegetable oil that has been investigated is that of *Jatropha curcas* [18], with results showing that it can be applied

Property	crude oil A	crude oil B	Standard
API Gravity	30.8	25.5	ASTM D287
Viscosity [cP to 40 °C]	5.6	32.7	ASTM D2196
Asphaltenes [%]	1.5	6.7	ASTM D6560
Water and Sediment [%]	0.5	0.5	ASTM D4007
Viscosity-Gravity Constant (VGC)	0.878	0.854	ASTM D2501

Table 1. Properties of the crude oil samples.

as an asphaltene stabiliser. Similarly, oils, such as turnip, rosemary, sesame, chamomile, and olive oils, have been evaluated, which have shown asphaltene stabilising efficacy [19], and hazelnut and walnut oils, also with positive results as asphaltene stabilisers [20].

The objective of the present research was to determine the effect of *J. curcas* oil heating and the applied dosage on asphaltene stability in two crude oil samples, to achieve a better understanding of the parameters that may influence the performance of this vegetable oil as an asphaltene stabiliser, as an alternative for oil treatment.

2. Materials and methods

2.1. crude oil samples

Two crude oil samples, which were donated by personnel from the production management of *Petróleos de Venezuela (PDVSA)* and came from the producing fields of *El Furrial* and *Punta de Mata* in the north of the *Monagas* State, *Venezuela*, were used. The properties of the crude oil samples are detailed in Table 1.

2.2. *Jatropha curcas* oil

The seeds of *J. curcas* were collected in the town of El Furrial in Monagas State, Venezuela. Mature fruits were collected when the drupe capsule had a dark brown or black coloration. The seeds were transferred to the hydrocarbon processing laboratory of the Universidad de Oriente Monagas Nucleus, Venezuela, and the seeds were manually extracted, the shell was removed and dried in the sun for 4 days.

The seeds were crushed using a laboratory mixer and the oil was extracted by the solid-liquid extraction procedure, using a Soxhlet extraction equipment, with n-hexane as the extraction solvent. The extract was concentrated in a rotary evaporator and stored in a glass vial, according to the procedure established in previous research [18]. The extraction was performed at a ratio of 70 g of seeds per 250 ml of n-hexane and an extraction time of four hours. Several extractions were performed until 100 ml of oil was obtained. The oil was divided into four parts of 20 ml each, stored in glass bottles, and numbered consecutively from one to four. The oil one was not subjected to heating and was kept at laboratory temperature (25 °C), the other oils were subjected to heating in a laboratory oven at different temperatures for 24 hours, as shown in Table 2.

Oil sample	Heating temperature [°C]
1	25
2	100
3	150
4	170

Table 2. Heat treatment of *J. curcas* oil samples.

Temperatures were set at the researcher's discretion, taking into account previous research and the capacity of the laboratory equipment. Each heated oil sample was allowed to cool to laboratory temperature (25 °C) and then characterized by standardized density [21] and viscosity tests [22].

2.3. Asphaltenes Instability Index (AII) calculation

The Asphaltenes Instability Index (AII) was determined for the crude oil samples, defined for the research as the ratio between the amount in millilitres of n-heptane needed to obtain asphaltene aggregates visible under an optical microscope (asphaltene flocculation onset – FO) and the amount in millilitres of xylene needed to redissolve the asphaltene aggregates visible under an optical microscope or dispersion point (DP), according to Equation 1.

$$AII = \frac{DP}{FO}, \tag{1}$$

where

AII = Asphaltene instability index [ml],

FO = Flocculation Onset [ml],

DP = Dispersion point [ml].

The asphaltene instability index measures the amount of dispersant (in this case xylene) used to stabilise the asphaltenes in millilitres per millilitre of flocculants used to form the aggregates (n-heptane). The higher the AII value, the more unstable the asphaltenes are. The procedure performed to determine the original AII of the crude oil samples is shown in Figure 1.

Figure 2 shows examples of microphotographs of asphaltene aggregates obtained by the addition of n-heptane and dissolved asphaltenes upon the application of xylene.

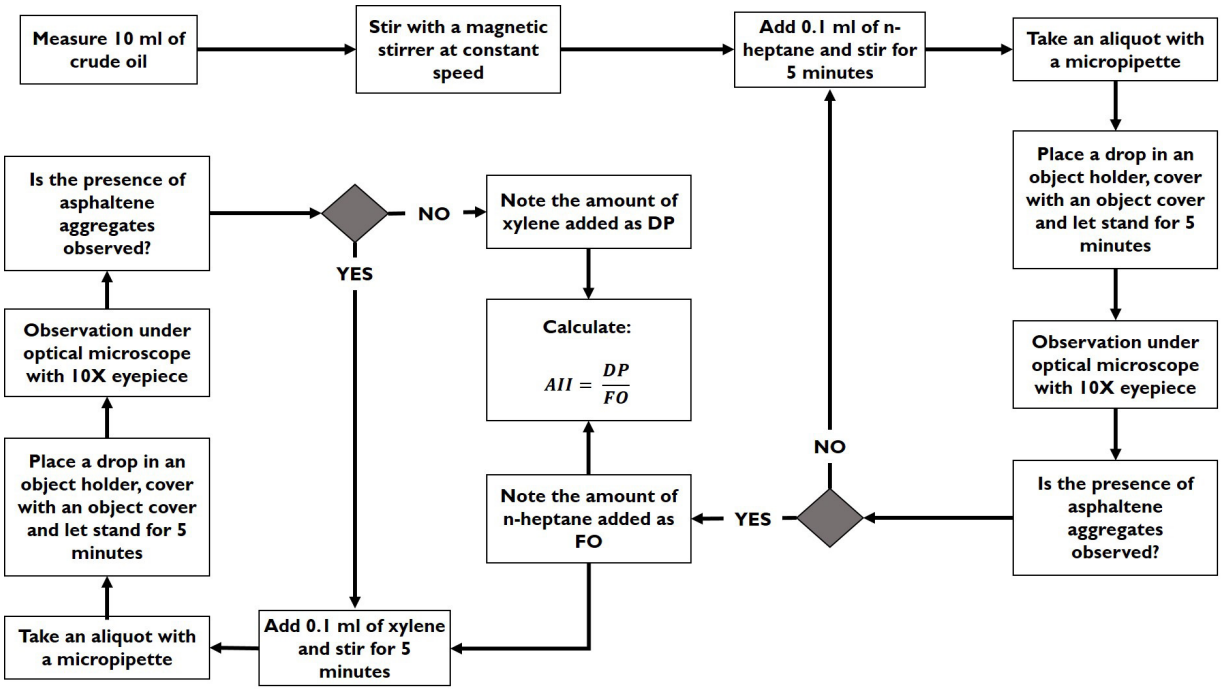

FIGURE 1. Flowchart of the procedure to obtain the AII.

FIGURE 2. Micro-photographs taken for an oil sample. A: Aggregates formed in the FO and B: Asphaltenes solubilized in the DP.

J. curcas oil samples were applied to each crude oil sample in doses of 2, 4, 6, and 8 µL in 10 ml and after mixing for 5 min with magnetic stirring, the AII was determined according to the procedure described in Figure 1. The efficiency of each oil sample at each dose applied was calculated by Equation 2.

$$\%Ef = \frac{AII_{original} - AII_{dosed}}{AII_{dosed}} \times 100, \qquad (2)$$

where

$\%Ef$ = Percentage efficiency of *J. curcas* oil,

$AII_{original}$ = Original instability index of Asphaltenes,

AII_{dosed} = Asphaltene instability index of crude oil dosed with J. curcas oil.

2.3.1. EXPERIMENTAL DESIGN

The experimental design evaluated was factorial, Taguchi type, with one response variable (AII) and two experimental factors (oil heating temperature and applied oil dose). The selected design has 16 runs, with one sample taken in each run. The model allowed estimating the effects of the 2 control factors on the response variable.

The result of the experimental analysis included Analysis of Variance (ANOVA), Pareto plot, main effects plot, response surface, and response optimization. Statistical analyses were performed with the statistical package Statgraphics Centurion XVII.

3. RESULTS AND DISCUSSION

According to the properties of the two crude oil samples shown in Table 1, it is observed that there are differences between the two. Especially in viscosity

Oil sample	Heating temperature [°C]	Density at 25 °C [g/ml]	Viscosity at 25 °C [cP]
1	25	0.917	34.47
2	100	0.919	75.14
3	150	0.922	94.13
4	170	0.910	117.61

Table 3. Shows the results of the density and viscosity properties measured for the *J. curcas* oil samples.

and percentage of asphaltenes. Crude oil B presents properties that characterise it as a heavier and denser fluid.

The VGC is a constant with which the composition of crude oil can be estimated according to the main hydrocarbon groups it contains. Crude oils with CGVs between 0.74 and 0.75 are considered paraffinic, with CGVs between 0.89 and 0.94 as naphthenic, and CGVs between 0.95 and 1.13 are typical of aromatic crudes. VGCs between 0.76 and 0.88 are typical for mixed composition crudes [23]. From the above, both samples are considered to be of a mixed base.

It is observed that the density remains almost constant, since its variations are low, which is corroborated by determining the variation coefficient (VC), whose value was 0.49 %. In contrast, viscosity did show a higher VC value of 38.98 %, which is indicative of a relationship between the oil heating temperature and viscosity.

To analyse the relationship between the two properties and the temperature to which the oil was subjected, a multivariate correlation test was performed, the results of which were that between the temperature and the density, the correlation coefficient $R = -0.1265$ and $p = 0.8394$ show that the relationship is low, negative [24] and not significant ($p > 0.05$) which corroborates the observation made in Table 3. On the contrary, the correlation coefficient between the viscosity and the temperature was $R = 0.9341$ and $p = 0.0201$, indicating a very strong and significant relationship ($p < 0.05$).

The effect of heating on oil viscosity can be accounted for by changes in composition that occur due to alterations in the fatty acid components of the oil due to the exposure to heat and oxygen, resulting in isomerization, polymerization, and oxidation reactions [25, 26]. In addition to the decomposition products mentioned above, heating of fats results in the formation of compounds of relatively high molecular weights [27], which will influence the increase in viscosity.

This behaviour was also reported when analysing the density and viscosity of coconut oil (*Cocos nucifera*) subjected to temperatures between 25 and 200 °C, also observing that the viscosity presented a coefficient of variation greater than 5 %, but without a significant correlation concerning temperature [17]. When comparing the average density of 0.917 g/ml with those reported by other investigations, it is observed that it is consistent with the values of 0.92 g/ml and 0.91 g/ml obtained in previous investigations [18, 28]. Another research reported a density of *J. curcas* oil of 0.938 g/ml, which differs from the one obtained in our work [29]. The differences or similarities in the properties of *J. curcas* oil may vary depending on both climatic and agronomic factors [30], so the results obtained are consistent with those of other investigations.

Run	Dose [µL]	Temperature [°C]	AII	%Ef
1	2	25	1.50	0
2	2	100	0.60	60.0
3	2	150	0.50	66.7
4	2	170	0.77	48.7
5	4	25	1.52	0
6	4	100	0.52	65.3
7	4	150	0.50	66.7
8	4	170	1.06	29.3
9	6	25	1.52	0
10	6	100	0.43	71.3
11	6	150	0.50	66.7
12	6	170	0.80	46.7
13	8	25	1.43	4.7
14	8	100	0.48	68.0
15	8	150	0.24	84.0
16	8	170	0.38	74.7

Table 4. Result to AII and efficiency for crude oil A with an original AII of 1.50.

3.1. Results for crude oil A sample

The data obtained after applying the experimental process with crude oil sample A are shown in Table 4.

The results indicate that the samples of *J. curcas* oil that were subjected to heating showed a promising asphaltene stabilising activity, since the efficiencies between 48.7 and 84.0 % were obtained, while the efficiency of the sample that was not subjected to heating, at a dose of 8 µL, was 4.7 %. On average, the efficiency obtained with a dose of 8 µL was the highest with 75.6 %, so it can be said that the efficiency depends on the dose applied, in addition to the heating temperature. The Taguchi model used was fitted to a quadratic trend, resulting in an ANOVA analysis (Table 5).

Source	Sum of squares	Df	Mean square	F-ratio	*p*-value
A: Dose	0.0479	1	0.0479	2.32	0.1585
B: Temperature	1.8223	1	1.8223	88.42	0.0000
AA	0.1024	1	0.1024	4.97	0.0499
AB	0.0571	1	0.0571	2.77	0.1271
BB	1.1729	1	1.1729	56.92	0.0000
Total error	0.2061	10	0.2061		
Total (corr.)	3.6891	15			

TABLE 5. ANOVA results for crude oil A sample.

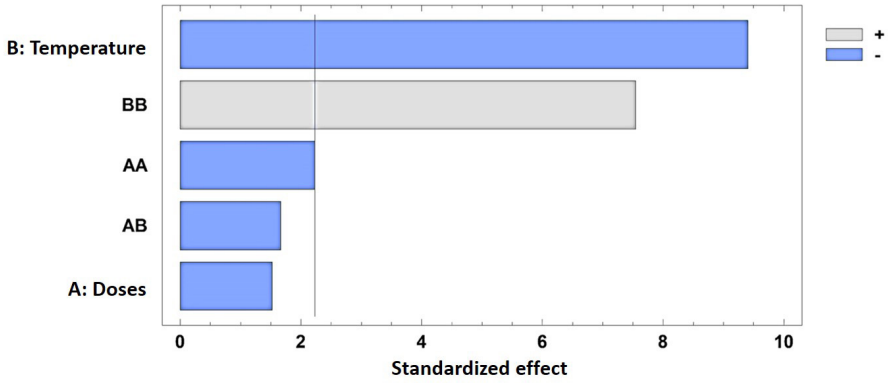

FIGURE 3. Standardized Pareto diagram for AII of crude oil sample A.

Table 5 shows that the factor that had a significant influence (p-value < 0.05) was the temperature at which the *J. curcas* oil was heated. Likewise, in the applied quadratic model, the quadratic interaction of dose (AA) and temperature (BB) also had a significant influence. On the contrary, the applied dose and the interaction between the dose and the temperature did not show a significant influence on IIA values (p-value > 0.05). This result agrees with those obtained by other authors [15, 17, 18]. It is observed that, although the efficiencies varied concerning the doses of oil applied, the differences were not statistically significant with a significance of 5 %, which contrasts with that reported in another research, where *J. curcas* oil was also applied to medium crude oil and the dose applied was significant [18], however, it is agreed that the best dose was that of 8 µL, even with the difference that in the cited research the oil was mixed with diesel oil and not pure as in the present research. For *C. nucifera* oil, it was also obtained that the 8 µL dose was the most efficient [16].

The standardized Pareto diagram (Figure 3) shows that the factor with the most important effect was temperature, followed by the quadratic factor of temperature. This is consistent with the results obtained in the analysis of variance. The effect of temperature is negative, which means that the AII for the crude oil sample varied inversely with temperature. The quadratic factor of temperature was the most important positive effect in the model.

The response optimization of the experimental design to find the dose and temperature values that generate the lowest AII value was performed using a response surface, whose model is shown in Figure 4. The optimal values were found to be a dose of 8 µL and a temperature of approximately 127 °C, with which a minimum AII value of 0.22 is obtained. These results indicate that the estimated maximum efficiency under the experimental conditions will be 85.3 %. This result is superior to that reported when applying *C. nucifera* oil to crude oil with the same API gravity (30.8), which showed a higher maximum efficiency as an asphaltene stabiliser, when heated up to 130 °C, of 78.6 % [17], indicating that *J. curcas* oil can be a more efficient alternative for asphaltene treatment in light crude samples as compared to *C. nucifera* oil.

The quadratic mathematical model used for the response surface presented a fit through the coefficient of determination R^2 of 0.944, which indicates that the model predicts the variability of AII by 94.4 %, representing a good approximation for an optimization.

It is demonstrated for crude oil sample A, that the factor that influences the efficiency of *J. curcas* oil as a stabiliser of asphaltenes is the temperature to which the oil sample is subjected, prior to its application. The effect of the heating temperature of *J. curcas* oil on asphaltenes is mainly due to the compositional change that the oil undergoes due to heating, as reported in previous research [25–27].

The formation of oxides from the fatty acids of *J. curcas* oil by heating can induce the creation of

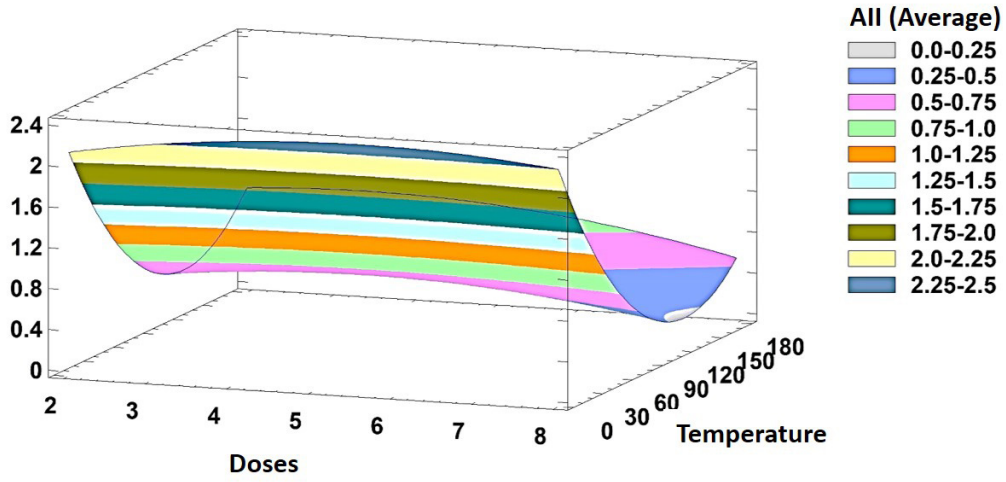

Figure 4. Estimated response surface for AII of crude oil sample A.

a surfactant layer that acts as a stabilising agent, so it is to be expected that the oil samples subjected to heating have had a higher efficiency. This indicates that the temperature to which the oil is subjected can create a more efficient surfactant system, since it has been demonstrated that the quality of the surfactant used is fundamental for achieving the stability of the asphaltenes in crude oil when a chemical treatment is applied, due to the fact that the interaction between the asphaltenes and the surfactant molecules is promoted [31].

3.2. Results for crude oil B sample

The data obtained after applying the experimental process with crude oil sample B are shown in Table 6. In the case of crude oil sample B, it was observed that the efficiencies obtained when applying *J. curcas* oil subjected to heating ranged from 17.1 to 89.3 %. The only dose at which a moderate efficiency values were obtained at all temperatures was 2 µL, which, on average, showed an efficiency of 46.9 %. At the 4 µL dose, efficiency was obtained at temperatures of 25, 100, and 170 °C, and at 150 °C, efficiency was not obtained. The higher doses (6 and 8 µL) only showed potential with the *J. curcas* oil samples heated to 170 °C. The Taguchi model used was fitted to a quadratic trend, resulting in an ANOVA analysis (Table 7).

As can be seen in Table 7, the effect of the factors on the AII of crude oil sample B differs from that obtained in crude oil sample A. In this case, the applied dose significantly influenced the AII values (p-value < 0.05) with a non-significant effect of temperature (p-value > 0.05). Looking at the interactions defined in the quadratic model only the interaction between the two factors (AB) is significant within the model (p-value < 0.05).

The influence of the dose applied coincides with that reported when applying *J. curcas* oil to a medium crude oil [18], which shows that the dose was significant even though the types of crude oil used in both investigations were different in composition.

Run	Dose [µL]	Temperature [°C]	AII	%Ef
1	2	25	0.36	74.3
2	2	100	1.16	17.1
3	2	150	1.10	21.4
4	2	170	0.35	75.0
5	4	25	0.37	73.6
6	4	100	0.88	37.1
7	4	150	1.83	0
8	4	170	0.96	31.4
9	6	25	1.91	0
10	6	100	1.54	0
11	6	150	1.78	0
12	6	170	0.74	47.1
13	8	25	1.95	0
14	8	100	1.71	0
15	8	150	1.40	0
16	8	170	0.15	89.3

Table 6. Result to AII and efficiency for crude oil B with an original AII of 1.40.

The analysis of the Pareto diagram shown in Figure 5 shows that the dose factor not only has the greatest influence on AII but also has a positive influence, while the interaction between the dose and the temperature is the factor with the greatest negative influence. It is also important to highlight that the other factors have a negative influence on the AII, although they were not significant, which may have an important effect on the behaviour of the graphical trend of the response surface method.

The optimization of the AII by response surface was determined considering a quadratic interaction model and the obtained graph can be seen in Figure 6.

Figure 6 shows that the lowest values of AII were obtained for the dose of 2 µL and at the lowest tem-

Source	Sum of squares	Df	Mean square	F-ratio	p.Value
A: Dose	1.4409	1	1.4409	7.10	0.0237
B: Temperature	0.2548	1	0.2548	1.26	0.2886
AA	0.1661	1	0.1661	0.82	0.3869
AB	1.4055	1	1.4055	6.93	0.0251
BB	0.8533	1	0.8533	4.21	0.0674
Total error	2.0290	10	0.2029		
Total (corr.)	5.5348	15			

TABLE 7. ANOVA results for crude oil A sample.

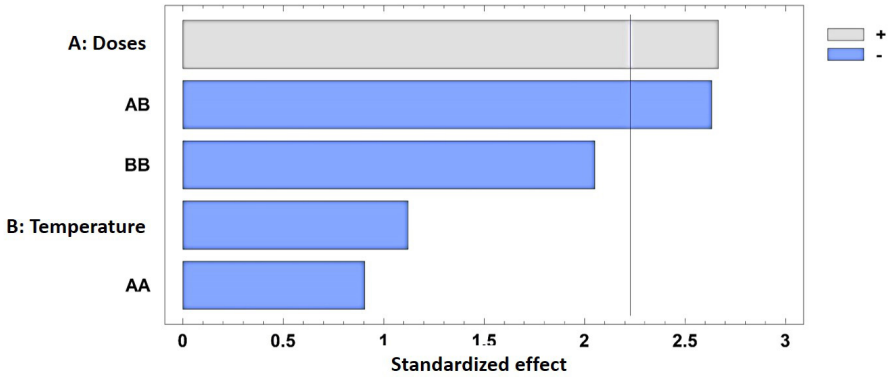

FIGURE 5. Standardized Pareto diagram for AII of crude oil sample B.

perature, which was evidenced when obtaining the dose and temperature that optimise AII, which were 2 µL and 25 °C where the response surface predicts a value of AII = 0.08, being the maximum predicted efficiency of 94.3 %. When comparing the estimated optimum efficiency with the maximum efficiency reported for a *J. curcas* oil heated at 150 °C applied to a medium crude oil sample of 28.8 °API, which was 88.33 % [18], it follows that for the crude oil sample of 25.5 °API, the oil was more efficient, although it should be taken into account that samples with different characteristics were used.

With the above, it is demonstrated that *J. curcas* oil is more efficient for the stabilisation of asphaltenes in the crude oil sample B and without being subjected to heating, as statistically obtained.

The quadratic mathematical model used for the response surface analysis, according to the coefficient of determination $R^2 = 0.633$, predicts, by 63.3 %, the optimal combination of the Dose and Heating Temperature values of *J. curcas* oil for crude oil sample B.

The results showed higher efficiency of *J. curcas* oil as compared to synthetic resins with which values of 55.56 % were obtained [32], however, as was also observed in other research, the stabilisation of asphaltenes does not depend only on the products used, but also on the characteristics of the oils where they are applied.

In the case of crude oil B, the stability behaviour is different from that observed in crude oil A. It is observed that the applied dose has a significant effect, in addition to its interaction with temperature. The lowest doses were those with the best performance, and amongst them, the 2 ml dose was shown to be efficient both for the oil sample not subjected to heating and for the one subjected to 170 °C. From the previous result, it can be said that for this particular crude oil, the composition of its asphaltenes seems to interact better with the oil in its original state (without being subjected to heating) and at lower doses, which was corroborated by the response surface, so the structural changes that the oil underwent when heated do not favour the stability of the asphaltenes in this sample.

The stability trend of asphaltenes, with respect to different chemicals, is, in most cases, not continuous, which is due to the fact that the interactions between asphaltenes and surfactants is dependent on the complexity of the former [33, 34].

According to what was obtained, it can be said that the characteristics of the crude oil and its composition can determine the optimum doses of asphaltene stabiliser product, as well as the most indicated product for its treatment. These results are consistent with those reported for the stability of two samples of medium crude oil after applying *C. nucifera* oil, where it was concluded that the results were dependent on the crude oil sample used [16].

According to previous research, it is accepted that composition influences the stability of asphaltenes in crude oil. Stable crude oil has higher values of the polar fraction, i.e., asphaltenes, resin, and aromatics. Therefore, when different crude oil samples are used in terms of their properties, different asphaltenes

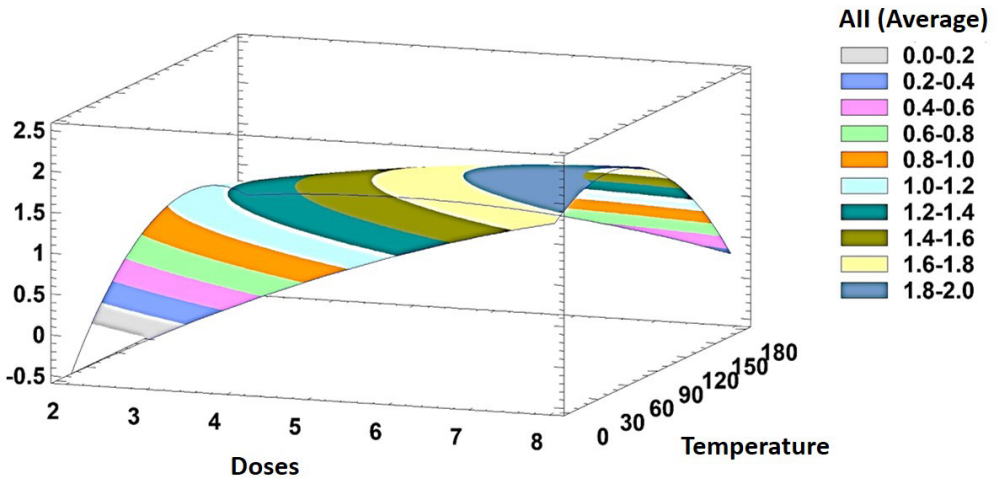

Figure 6. Estimated response surface for AII of crude oil sample B.

stability results and behaviour can be obtained [35], which justifies the differences observed in the present investigation when using two different crude oil samples.

One of the determining factors in the stability of asphaltenes that can influence the efficiency of the stabilising products is the presence of resins, which, in the crude oil, are considered a key factor as molecules that stabilise the colloidal particles of asphaltenes against aggregation, their mechanism of action being a combined repulsion/adhesion process [36, 37]. Likewise, other elements present in the crude oil composition, such as paraffin waxes, water, and organometallic compounds, act as destabilisers of asphaltenes, and also the presence of fine solids in the oil can stabilise asphaltenes [38, 39].

The use of *J. curcas* oil as an asphaltene stabiliser is shown to be feasible according to the results obtained, this is possibly due to its composition, formed by fatty acids, mostly linoleic acid [40, 41] which has been shown to have asphaltene-stabilising properties, as well as other acids such as palmitic acid, which is also present in the composition of the evaluated oil [15]. The polar nature of the fatty acid components of vegetable oils, such as that of *J. curcas*, give them surfactant properties, making them potential asphaltene stabilisers, which can be supported by the following factors: it is of utmost importance to find new substances with a higher solubility, because of the good results obtained with organic acids, taking into account that vegetable oils are mixtures rich in free or glyceride-forming organic acids, also vegetable oils are easy to obtain and cheaper than most of the polymeric dispersants used commercially Also, once the state of conservation of such substances is sufficient, their use could have positive social and economic consequences [19].

4. Conclusion

It is concluded that *J. curcas* oil is a viable alternative for the stabilisation of asphaltenes; however, its maximum efficiency depends on the dose applied, the temperature to which it is subjected in a heating process before its application, and the characteristics of the crude oil in which it is applied.

The compositional changes that occur in *J. curcas* oil when it is heated are a determining factor in its efficiency as a stabiliser of asphaltenes in crude oil, showing that when the oil is heated, a higher stabilisation efficiency is obtained.

The Taguchi factorial experimental design and response surface demonstrated that the optimum dosage and type of oil (based on heating temperature) to be applied to a particular crude oil can be obtained, which can also be applied to the selection of other products used for asphaltene stabilisation at the laboratory level.

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