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RESEARCH

Refining Used Lubricant Oils with Different Concentration Levels of Sulfuric Acid and Nonylphenol

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A B S T R A C T

The purpose of this research was to evaluate the refining of used lubricating oils (ULOs), and their possible use as drilling fluids. 17 treatments were evaluated and sulfuric acid and nonylphenol were used as reagents at concentrations of 0.12, 0.24 and 0.36 g/mL and temperatures of 40, 60, 80 and 100 °C. AT80 and AT100 ULOs treated at 80 and 100 °C without reagents, presented an average density of 0.84 g/cm^{-3}, a viscosity of 76.3 and 75.3 cP, an electrical stability of 1,731.3 and 1,394.6 V and a flash point of 183 and 190 °C as higher. The ST40C1 and ST40C2 treatments, added with reagents, showed similar results to AT80 and AT100 in the evaluated variables, but they are more expensive treatments. According to the results, it is concluded that the refined ULO can be a substitute for the oil used in the formulation of oil-based drilling fluids.

1. INTRODUCTION

The amount of used lubricating oils (ULOs) has been growing exponentially worldwide, with the automotive sector as one of the leading ULO sources. ULOs are generated at gas stations, garages, mechanic workshops, car dealership showrooms, and other retail companies such as, truck fleets, transportation, and construction along with public transportation and airports [1].

Nowadays, researchers are focused on quantifying the adverse environmental effects of ULOs [2]. It is of particular concern that, the

products of ULO burning generate environmental pollutants that over time, also pollute other places than those close to the ULO generation sites [3]. The combustion of ULOs release heavy metals, and harmful chemical compounds, such as, polynuclear aromatic hydrocarbons, benzene, solvents, polychlorinated biphenyl (PCB) and polycyclic aromatic hydrocarbons (PAH) [4].

In Mexico, the negative environmental effects of the ULOs are of great concern, which has led government agencies in charge of granting authorizations and monitoring their proper management, the SEMARNAT (Secretariat of Environment and Natural Resources) and the PROFEPA (Federal Agency of Environmental Protection) are in charge of estabilishing new tools and guidelines to determine the number of the ULO generators and the volume generated every year [5]. Knowing where the ULO sources are located is a key step towards adopting strategies or the ULOs management.

Globally, it is estimated that between 25 to 28 million tons of ULOs are produced annually, and a portion of these are released into the environment [6]. At this time, this has been one of the best examples of how technological innovation through the refining industry has contributed to the well-being and development of society [7].

Refining of ULOs has been used as part of the integrated management of oily waste generated in the different sectors of the industry. In the last decades, these wastes were treated to take advantage of fuels and lubricants within the automotive and industrial sector [8, 9, 10, 11]. A relevant issue is that ULOs contain high percentages of different additive remnants, which improve viscosity indexes when treated by refining. This property means that they present greater advantages than other used oils, since the added value is greater [12].

ULOs refining techniques consist of eliminating the impurities and the different pollutants that are mixed with automotive combustion, such as the high content of heavy metals (Zn, Cu, Ni, Pb, Cr, Cd, among others); therefore, refining restores most of the oils original properties [6].

In physicochemical terms, the viscosity, pour point, specific gravity and flash point are considered the most important parameters in the treatment processes and quality control of ULOs. For example, in pyrolysis and cracking reactions, it has been observed that oils with lower °API tend to present lower yields compared to lighter oils [13,14]. One of the novel processes that include these parameters is the refining technique, in addition to the fact that it presents greater advantages than other processes in which there is a low impact and environmental risk, great energy savings, and low health risks [15].

The management indicators in the world show a gigantic trend towards the recycling and reusing of ULOs. This has given rise to the stiff competition between recycling by regeneration to produce base oil and reprocessing for energy recovery [16]. There are different refining techniques, but the classic and usual acid-clay treatment has been replaced by the solvent extraction technique, which after use it is recovered for recycling by distillation. This refining process has been used, for example, by the Alexandria Petroleum Company (A.P.C) in Egypt, using a KTI re-refining technology [17].

The use of refined ULOs has focused more on the production of alternative fuels [18], which are often used by companies that are dedicated to the production of cement [4], biodiesel [12], and mixtures of asphalt with properties and better yields [19]. They are used in the automotive sector as lower quality oil, using them as fillers in old model cars and heavy machinery, marine oil, gear oil, processing oil and industrial oil [20].

Recently, the increasing efficiency in machines also increases the demand for higher quality lubricants [21]. The petroleum well drilling uses many additives that provide satisfactory drilling fluid performance. However, these materials have been found to be hazardous, either to the workforce operating on the site or to the environment [22]. Thus, this sector offers opportunities for new uses of ULOs, since the oil industry has ventured into diesel substitutes, for instance, mixing vegetable oil and biodiesel produced pyrolytic oil, [23, 24, 25].

The motivation of this study stands on the technical and economic feasibility of refining used lubricating oils and, the opportunity to use them as an alternative to formulate oil-based drilling fluids. Only a few studies have been

reported on this topic. A study conducted in Nigeria [23] used lubricating oils which were refined and used ULO to produce petroleum-based drilling fluids. The authors showed not very encouraging results for this activity as they seemed toxic to the environment. In Mexico, the amount of drilling fluids used in the oil industry is unknown. However, the oil industry, currently generates one of the highest shares of economic income in the country, there are no studies on the use of refined ULOs in the preparation of oil-based drilling fluids. The objective of this research was to apply different ULO refining treatments with sulfuric acid and nonylphenol and temperatures at different levels and evaluate the properties of interest for oil-well drilling.

2. MATERIALS AND METHODS

The oils that were evaluated and used in this study were collected in different automotive service stores of Villahermosa, state of Tabasco, Mexico. From all sites, a composite sample was prepared in Intermediate Bulk Container tanks (IBC), because, generally the mechanic shops collect their used oils in tanks and these are collected by companies that in turn sell this to others that use them as fuel for industrial boilers or as asphalt additives. From 98% sulfuric acid and 10 M, nonylphenol was used as reagents. A randomized block experimental design (RBD) with a factorial arrangement was established. The factors studied were: mixture of sulfuric acid/nonylphenol (5:1 ratio) to four levels, temperature in four levels and three repetitions, as described in Table 1. Untreated ULO was used a control.

The parameters to be evaluated were established based on the oil quality requirements for oil-based drilling cuttings. Such properties are: oil, water and solids content that are tested by using the API RP 13B2 method (% by mass), density (g/cm^3) by the method ASTM-D854 viscosity (cp), electrical stability (V), point of inflammation by the method ASTM D93-2000 (°C), and only for the best treatment resulting from those evaluated in this study, SARA fractionation (Saturates, Aromatics, Resins and Asphaltenes) by the method ASTM D2007-2011, using equipment model Agilent Zorbax XDB-C18 Serio 1200. Table 1 shows the description of the treatments for the refining of the ULOs evaluated. The proportions of the mixture of sulfuric acid (H_2SO_4) and nonylphenol ($C_{15}H_{24}O$) (reagent/ULO ratio) were: 0.12, 0.24 and 0.36 g/mL of ULOs. The temperatures evaluated were: 40, 60, 80 and 100 °C.

Table 1. Description of the treatments for the refining of the ULOs evaluated.

TR	C	ULOs (mL)	SA (g)	N (g)	RR/ ULOs (g/ mL)	T (°C)
Control		1000	0	0	0	25
AT40	C0	1000	0	0	0	40
AT60		1000	0	0	0	60
AT80		1000	0	0	0	80
AT100		1000	0	0	0	100
ST40C1	C1	1000	100	20	0.12	40
ST60C1		1000	100	20	0.12	60
ST80C1		1000	100	20	0.12	80
ST100C1		1000	100	20	0.12	100
ST40C2	C2	1000	200	40	0.24	40
ST60C2		1000	200	40	0.24	60
ST80C2		1000	200	40	0.24	80
ST100C2		1000	200	40	0.24	100
ST40C3	C3	1000	300	60	0.36	40
ST60C3		1000	300	60	0.36	60
ST80C3		1000	300	60	0.36	80
ST100C3		1000	300	60	0.36	100

TR=Treatment, C=Concentration, SA=Sulfuric acid, N= Nonylphenol, RR= Relation reagent, T= Temperature

A 1000 mL sample of the ULOs was placed in a stainless steel container with valves at the bottom that allowed the distillation residue to be extracted from each sample. The reagents were applied to each of the samples by first adding the (H_2SO_4) and mixing it for 30 min, then nonylphenol ($C_{15}H_{24}O$) was immediately applied, and mixed with a spatula for additional 30 min. Then the mixture was left to rest for a period of 48 h., then the residual was removed from below and finally, the sample was packaged with its respective label. This process was used for the three concentrations at the corresponding temperatures (Fig. 1). Each of the reagents was placed in 250 mL beakers and their mass was determined with a 400 g digital balance, with a precision of 0.01 g. The objective of this step was to determine the proportion of each reagent to be applied to each of the proposed treatments.

Fig. 1. ULO leachates, after the application of sulfuric acid and nonylphenol.

The temperatures to which the samples of used lubricating oils (ULOs) were subjected were: Temperature 1 of 40 °C, Temperature 2 of 60 °C, Temperature 3 of 80 °C and Temperature 4 of 100 °C, control of 25 °C. A time of 30 min was used after the sample reached the indicated temperature. These temperatures were controlled with the knobs of the stove used and, the temperature measurement was made with a thermometer (Fig. 2).

Fig. 2. Stove with knobs to regulate the proposed temperatures with a 250 C mercury thermometer.

2.1 Treatment with temperature and proportion of reagents

The samples with the different proportions (C1= 0.12, C2= 0.24 and C3= 0.36) of sulfuric acid 10 M nonylphenol (See table 1) were processed in the same way at the mentioned temperatures. To increase the temperature, an electric stove with Taurus temperature regulator knobs was used. This temperature was measured with a Taylor brand bimetallic thermometer.

3. RESULTS AND DISCUSSION

3.1. ULOs density

Fig. 3 presents the density results of the ULOs samples treated with the 16 treatments and the control (untreated) sample, equal letters indicate that there are no statistically significant differences among treatments. In the control sample, the density was 0.89 g/cm^3 at 25 °C. It is observed in the ULOs treatments where only temperature was applied that the higher the temperature, the lower the density, except in the AT80 and AT100 treatments in which a similar result (0.84 g/cm^3) was obtained. As expected, the temperature directly influenced the density values. This behavior is related to the °API of the ULO.

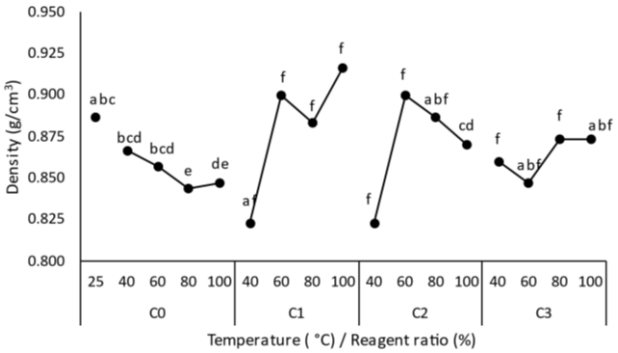

Fig. 3. Behavior of the density of the refined ULOs in each treatment. Equal letters indicate that there are no statistically significant differences.

In the treatments with concentration C1, the general trend is contrary to the one observed in the treatments with only temperature. That is, the density increased as the temperature increased, starting from 0.82 g/cm^3 in the ST40C1 treatment up to 0.91 g/cm^3 in the sample ST100C1. This represents an increase of 3.00 % (having a Coefficient of Variation (CV) of 2.29%, its interaction was significant) with respect to the density obtained in the control sample. The treatment with C2 concentration generated the lowest density, with a value of 0.82 g/cm^3, for ST40C2 treatment, rising to 0.90 g/cm^3 with the ST60C2 treatment. Starting at 60 °C, the density showed a decreasing trend reaching 0.88 g/cm^3 in the ST80C2 sample and remained unchanged for the ST100C2 treatment (0.87 g/cm^3).

Most of the treatments did not show significant differences with respect to the control ($P>0.05$). The same letters in the figures indicate that there were no statistically significant differences

between each of the treatments evaluated. The ULOs that were only subjected to temperatures of 80 and 100 °C did present a significant difference with respect to the control (P>0.05). Similar studies obtained a density of 0.87 g/cm^3 and from 0.87 to 0.91 g/cm^3 [26,27,28] which are important results, since these values of density, are an indication that they treatment processes have removed contaminants from the ULOs. The previous results exceed those found in this study which was 0.84 g/cm^3, but it is slightly above the value of commercial diesel which has a density of 0.83 g/cm^3 [29] and is used to prepare oil-based drilling fluids [14].

3.2. ULOs viscosity

The viscosity results from the ULO samples treated with the 16 treatments, and they are shown in Fig. 4. The control sample showed a value of 101 cP. All the results of the treatments evaluated in the C0 were below those presented in the control, in the order of 75.3 cP to 96 cP.

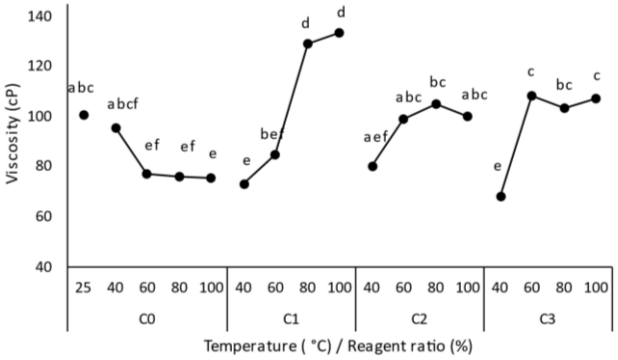

Fig. 4. Behavior of the viscosity of the refined oil in the different treatments.

The viscosity in the treatments with the C1 concentration increased as the temperature increased after the treatment. Starting from an initial value of 73.3 cP, and reaching a maximum of 133.3 cP in the ST100C1 sample, which represents an increase of 59.4% compared to the value presented by the control sample. The treatments with C2 and C3 have a similar trend with respect to the increase of temperature, after being treated, except that they reach a maximum between 105 and 108 cP after 60 °C. Although the ST80C1 and ST100C1 treatments showed significant differences (P<0.05) compared to the control samples, their viscosity value was lower, suggesting that this property is favorable for the suggested use of ULO.

According to the multiple range contrast, only the AT60, AT80, AT100, ST40C1 and ST40C3 treatments presented a statistically significant difference with respect to the control samples (P>0.05) with viscosity values lower than these. Having a Coefficient of Variation (CV) of 10.27%, its interaction was significant. These treatments showed the most favorable viscosity values in the ULOs refining.

Viscosity values lower than those of the control were found in treatments AT60, AT80, AT100, and also the most favorable for the use suggested in this study. Treatments AT80, AT100 and ST40C1 presented values from 75 cP to 96 cP, which were below the ones that were found by [20] with 118 cP. Also [30] reported higher values (153 cP to 219 cP) than ours. But the viscosity values found in our research exceed [27], the obtained values between 40.3 cP to 41.2 cP. The possible explanation is that they (27) used a filter medium and in our study it was not used.

3.3. ULOs flash point

Fig. 5 shows the flash point results of the ULOs samples treated with the 16 treatments and the control sample. According to the ANOVA, there are significant differences for the flash point (P>0.05). Having a Coefficient of Variation (CV) of 5.77%, obtaining a non-significant interaction.

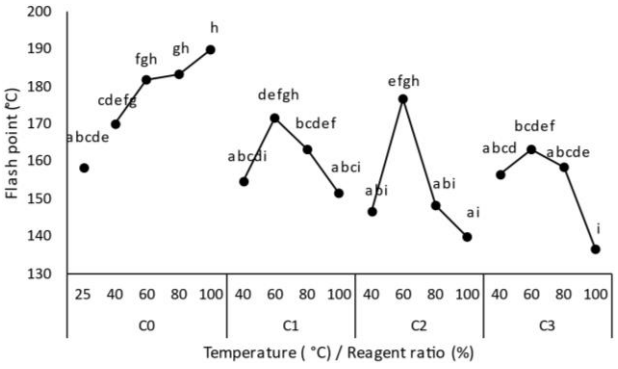

Fig. 5. Behavior of the flash point of the refined oil in the different treatments.

The results of the flash point (the degree at which the oil ignites) in the control ULOs was 158.3 °C. The ULOs that were subjected to temperature alone showed an increase as the temperature increased (i.e 170 °C in AT40 to 190 °C in AT100). The maximum value reached in the AT100 treatment was 20% higher than the value

obtained in the control. For the treatments with C1 there was a slight increase between ST40C1 and ST60C1 of 17°C in the flash point, at a higher treatment temperature the behavior was the opposite, decreasing 20 °C between ST60C1 and ST100C1. A similar behavior was obtained in the treatments with C2, although the drop in the flash point was greater in ST100C2, which resulted in a temperature of 140 °C, which represents a decrease of 37 °C with respect to the highest value reached by the ST60C2.

The concentration of reagents C3 did not show increase of the flash point either. The values varied between 163 °C with the treatment that generated the highest value ST60C3 up to 137 °C in the treatment with the lowest flammability temperature ST100C3.

According to the multiple range contrast, only the AT60, AT80 and AT100 treatments presented statistically significant differences with respect to the flash point values of the control samples (P>0.05) and with values higher than 158.33 °C.

The AT60, AT80 and AT100 treatments presented values of flash point at 181.67 °C to 190 °C, which were above those obtained in the control sample. These results were below those obtained by [27], which obtained a flash point at 210 °C compared to its control that was at 185 °C. While [29] it was found at 225 °C below its control that was 230 °C; and [31] with 190.67 °C to 220.40 °C lower than the control that was 224.50 °C. This finding suggests that when the ULO is refined, a part of the hydrocarbon fractions that are present in new lubricanting oils are burned resulting in an increase of the flash point. In a previous study [32] it is mentioned that the flash point of diesel is 72 °C and biodiesel at is 160 °C, although [29] it says that commercial diesel can also ignite at 58.5 °C.

3.4. Electrical stability

In Fig. 6 it can be seen that the electrical stability in the control sample had a value of 887.33 V, which was low compared to the data presented in this study in the refined ULOs, whose treatment consisted only of applying different temperatures, this result is favorable for the suggested use in this investigation. A considerable increase is observed from 80 °C with a maximum value of 1731 V. According to the ANOVA (data not showed) performed, there are statistically significant differences between the electrical stability results of the different treatments (P<0.05). Having a Coefficient of Variation (CV) of 26.70%, its interaction was significant.

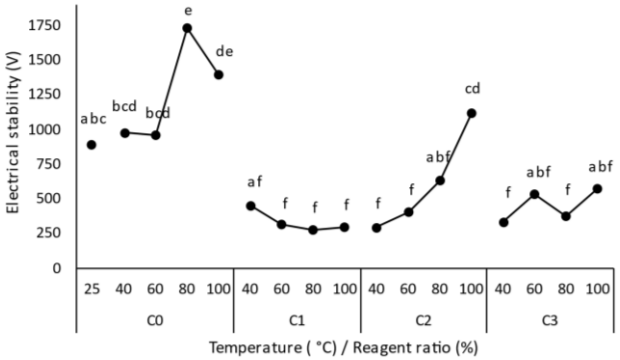

Fig. 6. Behavior of the electrical stability of the refined oil in the different treatments.

Only the ST100C2 treatment obtained an average above the control, although the difference with this, is that it is not statistically significant (P>0.05). Only the AT80 and AT100 treatments showed statistically significant differences (P<0.05) and higher values than the control samples.

In the electrical stability results, a considerable increase is observed from 80 °C with a maximum value of 1731 V just by applying the different temperatures. Only the ST100C2 treatment obtained a value slightly above the control. The best electrical stability was reflected in the treatment AT80 with 1731.33 V and AT100 with 1394.67 V, these values represent greater electrical stability than those reported by [13] a refined used lubricating oil with values of 480 V, which are well below those obtained in this study.

3.5. Saturated, aromatic, resins and asphaltenes (SARA)

Fig. 7 shows the behaviour of saturates, aromatics, resins and asphaltenes in the refined oil in the different selected treatments. The amounts on the bars represent the percentage of each one of the fractions.

The objective of this analysis was only exploratory, due to the economic scope of the investigation. The recovery percentage is similar in the three samples. They only vary in the range from 97.36 to 97.53, a difference that is less than 1% of the core recovery value.

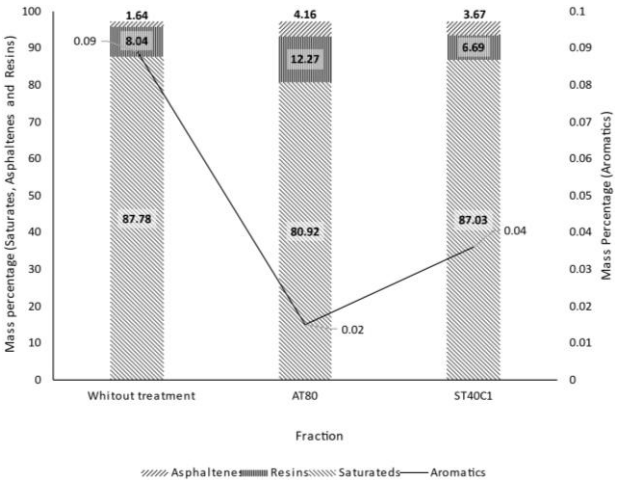

Fig. 7. Behavior of saturates, aromatics, resins and asphaltenes in the refined oil in the different selected treatments. The amounts on the bars represent the percentage of each one of the fractions.

The AT80 treatment presented a reduction of 7.8 % of saturates with respect to the content of the control. The ST40C1 sample had very similar proportions to the control, only a difference of 0.92% with respect to the saturates content, with a marginal increase in the asphaltenes. Considering the results of both treatments (AT80 and ST40C1), it can be seen that the decrease in saturates content corresponds to a proportional increase in asphaltenes.

Regarding the SARA components, in the AT80 and ST40C1 treatments they were found to be saturated in the order of 87.03 to 87.77%, aromatics from 0.03 to 0.08%, resins from 6.68 to 8.03% and asphaltenes from 1.63 to 3.66%, showing a decrease in the saturates content that corresponds to a proportional increase in asphaltenes. The aromatics were not representative in the evaluated treatments. In this study it was shown that the refined ULO contains higher percentages of saturates compared to those found by [33] who evaluated two types of crude oil, finding saturates in a range of 51.36 to 58.42%, aromatics from 20.41 to 38.16%, resins of 7.15 to 20.83% and asphaltenes from 0.34 to 3.33%. Also [34], it was reported that a vegetable oil contains 2.74% saturates, 92.82% aromatics, 4.11% resins and 0.33% asphaltenes; found that an oil extract has 20.56% saturates, 72.82% aromatics, 6.37% resins, and 0.25% asphaltenes [33]. The previous results show that vegetable oils can present a higher % of saturates, aromatics, but lower % of resins and asphaltenes than the refined ULO found in this study.

3.6. Percentage of solids, oil and moisture

Fig. 8 shows the results of % solids, % moisture and % oil of the ULOs samples treated with the 16 treatments and the value of the control sample (untreated).

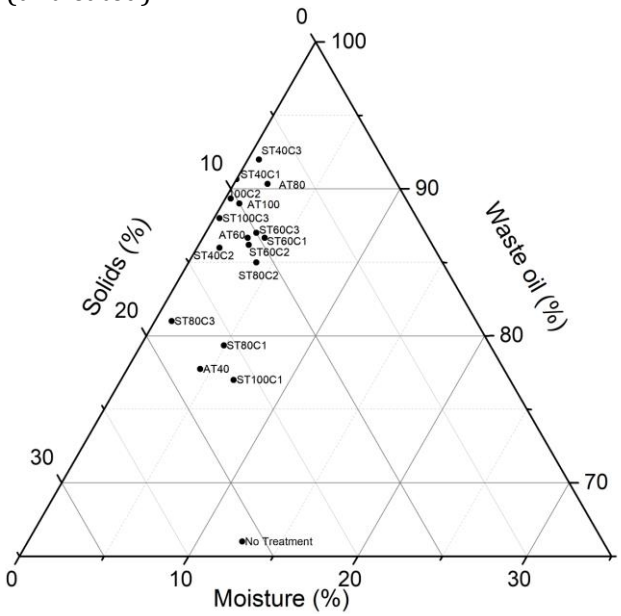

Fig. 8. Behavior of the % solids, % oil and % moisture of the refined oil in the different treatments.

The best treatments were ST40C1, ST40C3 and AT80 that gave solid content ranging between 7.33% and 9.33%. Regarding humidity, values in the range of 0% to 2% were obtained; and finally, the amount of oil was in the order of 90.33% to 92%.

The refining of the ULOs with the different treatments used in this study, improved the evaluated variables (Density, Viscosity, Flash point, electrical stability, percentage of solids and percentage of water). This was observed in the treatments ST40C1, ST40C3 and AT80 in the order of 90.33% to 92% of recovered oil. The results of the variables evaluated are summarised in Tables 2 and 3. It should be noted that AT80 and AT100 treatments meet the most favorable values for most of the evaluated parameters.

Table 2. Summary of results obtained with the most efficient ULOs refining treatments.

Treatment	Density	Viscosity	% oil
Control	0.88 ± 0.01	101.0 ± 0.0	66.0 ± 2.0
AT60	0.85 ± 0.01	77.0 ± 0.0	86.6 ± 4.6
AT80	**0.84** ± **0.01**	76.3 ± 0.5	**90.3** ± 3.5
AT100	**0.84** ± **0.01**	**75.3** ± 0.5	89.0 ± 1.0
ST40C1	**0.82** ± **0.04**	**73.3** ± 2.3	**90.6** ± 2.3
ST40C3	0.86 ± 0.04	**6 8.6** ± 17.6	**92.0** ± 7.8

Table 3. Summary of results obtained with the most efficient ULO refining treatments in flash point and electrical stability.

Treatment	Flash Point	Eléctrical stabiity
Control	158 ± 10.40	887 ± 187.2
AT60	**182** ± 10.41	964 ± 356.8
AT80	**183** ± 20.82	**1731** ± 176.6
AT100	**190** ± 13.23	**1395** ± 422.5
ST40C1	155 ± 22.91	458 ± 109.6
ST40C3	157 ± 7.64	333 ± 56.5

4. CONCLUSIONS

The temperature improved the properties of used lubricating oils (ULOs). Although the addition of the reagents sulfuric acid combined with nonylphenol, in most of the treatments the characteristics of the ULOs improved compared to the control, it was not higher than in the AT80 and AT100 treatments.

The positive effect of temperature treatments is reflected in the decrease in density and viscosity, the increase in the flash point and the electrical stability.

On the other hand, although thermal treatments offer better results, they can modify the composition of the fractions, this would possibly cause losing alkanes that may volatilize at low temperatures.

From a technical-economic approach, the best treatment that meets the requirements of shorter treatment time and lower cost of supplies is AT80.

According to the results obtained, refined ULOs can be considered an alternative in the formulation of oil-based drilling fluids, by applying different mixtures between ULOs and Diesel, reducing the economic and environmental costs as a result of the ability to re-use them.

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