

## DESULFURIZATION OF LIGHT CYCLE OIL BY EXTRACTION WITH POLAR ORGANIC SOLVENTS

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### Abstract

The desulfurization of light cycle oil containing 2157 ppmw sulfur has been studied using acetonitrile, N-methylpyrrolidone, and N,N-dimethylformamide as extraction solvents. The performance of these polar solvents has been carefully evaluated and compared under different experimental conditions by considering both desulfurization and raffinate recovery. It has been observed that an increase in the solvent/feed ratio from 1.0 to 2.5 decreased the sulfur content and the yield of the refined products. In addition, increasing the number of extraction stages from 1 to 3 had the same effect on the desulfurization process. The results obtained indicated that the degree of sulfur removal using N-methylpyrrolidone and N,N-dimethylformamide is much higher than with acetonitrile. However, the yield values for N-methylpyrrolidone and N,N-dimethylformamide solvents are lower than acetonitrile. The three-stage extraction experiments carried out at 50°C with a solvent/feed ratio equal to 2.5 produced the raffinates with minimum sulfur contents of 1007, 543 and 575 ppmw using acetonitrile, N-methylpyrrolidone, and N,N-dimethylformamide, respectively. The comparison of the properties of these raffinates with the commercial standard of automotive diesel fuel (NA 8110) showed a significant improvement in the quality of the feed in terms of sulfur content, density, distillation profile, cetane index, and color, particularly after extraction with N-methylpyrrolidone and N,N-dimethylformamide.

**Keywords:** Extractive desulfurization; Light cycle oil; Acetonitrile; N-methylpyrrolidone; N,N-dimethylformamide; Diesel fuel.

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## 1. Introduction

According to a recent projection by the Organization of the Petroleum Exporting Countries (OPEC), middle distillates (gasoil/diesel) demand is expected to increase by 2.9 mb/d during the period 2017–2040 [1]. Diesel is extensively used as fuel both in highway transportation vehicles (e.g., cars, buses, and trucks) and non highway transportation systems (e.g. locomotives, marine vessels, farm equipment, etc.) [2]. Diesel fuel is formed from straight run diesel, light cycle oil from the fluid catalytic cracking (FCC) unit, hydrocracker diesel, and coker diesel [3]. The sulfur content of diesel fuel has been cut down to ultra low levels (10–15 ppm) by environmental regulation in many countries with the aim of reducing diesel engine's harmful emissions and improving air quality [2]. In order to meet diesel fuel specifications, catalytic hydrotreating process is the conventional method used in petroleum refineries for desulfurization and for upgrading the quality of diesel fractions [4–6].

Light cycle oil (LCO) is a poor diesel fuel blending component due to its poor engine ignition performance and its high sulfur content [7]. LCO produced by the FCC units contains up to 2.5 wt % sulfur, it is characterized by a very low cetane number (slightly above 20), a high density and a high content of aromatics [3]. A significant portion of the sulfur in LCO is found in alkyl-dibenzothiophenes, which are relatively difficult to desulfurize by hydrotreating [7–8]. Similar results have been reported in many studies [9–11]. Hydrotreating of LCO for deep sulfur removal

requires relatively high pressure and significant hydrogen consumption but provides only a limited improvement in cetane number, total aromatics content and density [7]. In addition, this process has very high capital and operating costs [12-13]. The operating cost will be more in ultra low sulfur diesel (below 15 ppm) units compared to that of conventional low sulfur diesel (500 ppm) producing units [2]. For economic reasons, numerous research studies have been carried out during the last years on the development of alternative routes for removing sulfur from LCO streams, such as oxidative desulfurization [14-18], biodesulfurization [19,20], adsorptive desulfurization [21-23] and extractive desulfurization by organic solvents [12-13,24-26] or ionic liquids [27-28]. Some authors have also studied the combination of these alternative methods with the hydrotreating process [14,19-20].

Extractive desulfurization of diesel feedstocks has received increasing attention in recent years. This is because, the sulfur extraction technologies with selective solvents could reduce the cost of desulfurization substantially since they do not demand hydrogen, and are carried out at atmospheric pressure and relatively low temperature. Perspectives on the integration of this method in oil refineries have been seriously examined by Kumar *et al.* [29]. The application of organic solvents (conventional) in petroleum refining and industrial chemistry is well established, leading to their production at large scale and easy availability at reasonable cost in open market [29]. Ionic liquids are more expensive in comparison to common organic solvents; thus, the use of organic solvents is still more attractive than that of ionic liquids in extraction processes [30]. Various organic solvents, namely acetonitrile (ACN) [12-13,26], N-methylpyrrolidone (NMP) [24], and N, N-dimethylformamide (DMF) [25], have been employed as extractants for sulfur compounds present in different LCO fractions. There is no study in the literature that compares the extractive desulfurization efficiency of these three solvents (ACN, NMP, and DMF) for the same LCO stream.

In the present work, ACN, NMP, and DMF were used separately as extraction solvents for the desulfurization of LCO produced by the Adrar refinery in Algeria. First, the effect of increasing the solvent to feed ratio was studied using a single-stage mixer-settler batch apparatus. The efficiency of the extraction experiments was evaluated by determining the yield of refined products, the sulfur content of the raffinate and the degree of sulfur removal. Further multi-stage extraction experiments were performed in crosscurrent with each solvent; the raffinates obtained after three extraction stages were analyzed to determine their quality as potential components of automotive diesel fuel.

## 2. Experimental

### 2.1. Materials

The extraction experiments were carried out using LCO feed from the residue fluid catalytic cracking (RFCC) unit of the Adrar refinery. LCO leaves the main fractionator section of the RFCC unit at 50 °C, the temperature at which all the extraction experiments were performed. The total sulfur content of this feedstock is 2157 ppmw, which appears slightly lower than conventional LCO. ACN (99.5 %, Panreac Quimica), NMP (99 %, Sigma-Aldrich), and DMF (99.5 %, Reachim) were used as received without any pretreatment. To increase the extraction selectivity and the raffinate yield, 5.0 wt % of water was added to each solvent. Anhydrous calcium chloride (Merck) was used to remove traces of water present in the raffinates.

### 2.2. Apparatus and procedures

*Single-stage extraction.* 60 g of LCO with an appropriate amount of solvent were charged in a jacketed glass mixer-settler provided with a mechanical stirrer. The extraction temperature was maintained at 50°C within  $\pm 0.5^\circ\text{C}$  with the help of a thermostatic bath (Figure 1). The same extraction system was employed in our previous study for the purification of a hydrotreated gasoil fraction [31]. The mixture was stirred vigorously for 30 min, which was sufficient for establishing equilibrium. After a settling time of 30 min, the oil-rich phase (raffinate phase) and solvent-rich phase (extract phase) were separated. The raffinate phase contains mainly the less polar hydrocarbons with low cohesion to the polar solvent [12]. The

extract phase contains the more polar compounds (sulfur, nitrogen and aromatic compounds) that have better cohesion with the solvent. The small amount of solvent present in the raffinate phase was removed by washing several times with distilled water. The water-washed raffinate phase was dried for 24 h over calcium chloride before being analyzed. The resulting anhydrous solution termed as raffinate was used for the calculation of the yield.

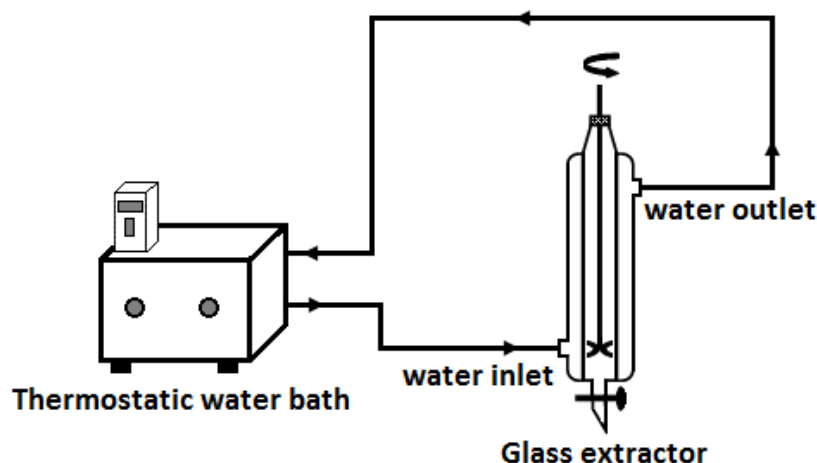


Figure 1. Schematic diagram of the experimental setup

**Multistage crosscurrent extraction.** Crosscurrent extraction consists of a series of one-stage extractions such that the raffinate phase from the first stage is placed in contact with fresh solvent in the second stage and so on until a raffinate with the desired purity is obtained [32]. Figure 2 illustrates the principle of this method of extraction. Each extraction stage of the series was carried out in the same apparatus as the single-stage experiments and under the same operating conditions. The amount of solvent corresponding to the solvent/feed ratio was divided equitably between the extraction stages. The raffinate phase from the last stage was further treated in the same way as in the single-stage extractions. The multistage experiments were done in batch mode using 60 g of LCO, except for the experiments in three-stage where the amount of treated LCO was 450 g in order to ensure that the recovered quantity of raffinate should be adequate to perform all the analyses.

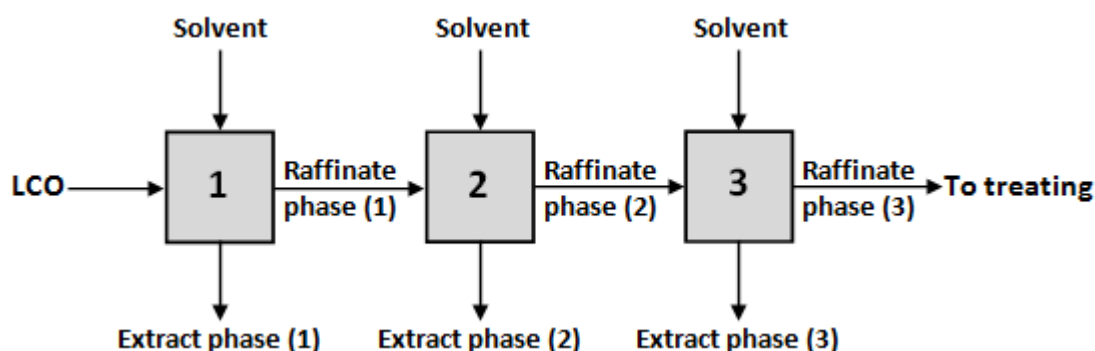


Figure 2. Simplified diagram of three-stage crosscurrent extraction

### 2.3. Methods of analysis

The total sulfur content of LCO and raffinates was measured by the pyro-fluorescence method using an ANTEK 9000NS analyzer, according to ASTM D5453. Density was determined at 15°C with the DMA 48 density meter (Anton Paar), according to ASTM D4052. ASTM D86 method was used for determining the distillation profile of samples. Kinematic viscosity was measured at 20°C with the use of a Cannon-Fenske viscometer according to ASTM D445. Pour

point was determined by an automatic apparatus (ISL CPP 97-2 analyzer), according to ASTM D97. Cetane index was calculated from density and mid-boiling point according to ASTM D976. The color of samples was determined by the ASTM UNION colorimeter, according to ASTM D1500.

## 2.4. Extractive desulfurization process evaluation parameters

The yield of desulfurized LCO is defined as follows:

$$\text{Yield (\%)} = \frac{R}{F} \times 100 \quad (1)$$

where  $R$  is the mass of the raffinate, and  $F$  is the mass of the feed.

The degree of sulfur removal is estimated using the following equation:

$$\text{Degree of sulfur removal (\%)} = \frac{(S_f \times F) - (S_R \times R)}{(S_f \times F)} \times 100 \quad (2)$$

where  $S_f$  and  $S_R$  denote the concentration (ppmw) of sulfur in the feed (LCO) and in the raffinate (desulfurized LCO), respectively.

## 3. Results and discussion

### 3.1. Effect of the solvent to feed mass ratio

The solvent to feed ratio is an important factor in the extraction process, particularly for recirculation aspect. In this study, extractive desulfurization of LCO was performed in single-stage with a solvent to feed ratio ranging from 1.0 to 2.5. The results for the three aqueous solvents (ACN, NMP, and DMF) are given in Table 1.

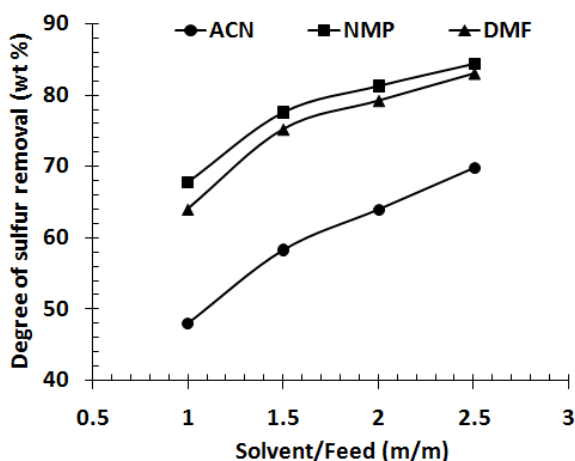


Figure 3. Effect of the solvent to feed ratio on the degree of sulfur removal (single-stage extraction)

It was observed that the increase of the solvent/feed ratio improved LCO quality in terms of lowering sulfur content, but at the same time, led to a reduction of raffinate yield. Similar observations have been reported by Kumar *et al.* [24] in their study concerning the desulfurization of LCO with NMP. As an example, it can be observed from Table 1 for NMP that an increase in the solvent/feed ratio from 1 to 2.5 resulted in a decrease in the sulfur content from 1248 to 807 ppmw and a decrease in the raffinate recovery from 55.6 to 41.7 wt %. Figure 3 indicates that the use of a high solvent/LCO ratio enhances the degree of sulfur removal. It is clear that the gradient for change in the degree of sulfur removal increases in the order: NMP < DMF < ACN.

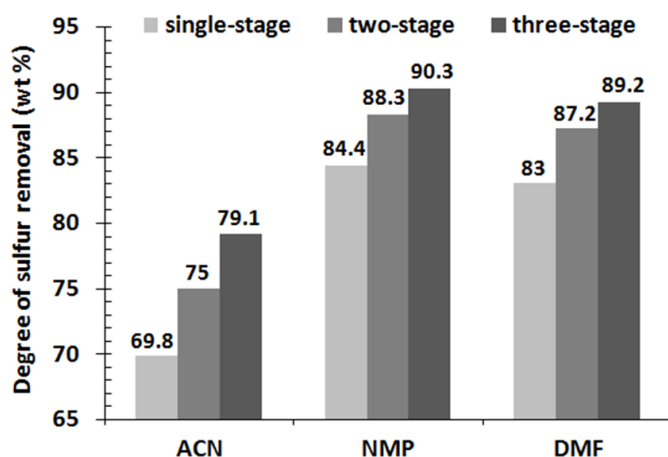
Table 1. Effect of the solvent to feed ratio on the yield and sulfur content of the raffinates (single-stage extraction)

Solvent/Feed (m/m)	Yield (wt %)			Sulfur (ppmw)		
	ACN	NMP	DMF	ACN	NMP	DMF
1.0	66.8	55.6	59.2	1678	1248	1310
1.5	59.5	47.3	49.7	1512	1021	1075
2.0	54.6	44.2	46.5	1422	913	967
2.5	48.5	41.7	43.4	1345	807	846

As shown in Table 1 and Figure 3, the lowest sulfur content of the raffinate corresponding to the highest degree of sulfur removal was obtained for each solvent with a solvent/LCO ratio equal to 2.5. Considering this, the solvent/feed value of 2.5 seems to be the best for use in extractive desulfurization of LCO.

### 3.2. Effect of the number of extraction stages

It is known that for the same quantity of solvent, the multistage extraction allows a better separation than the extraction in a single-stage. The multistage extraction experiments were all performed at 50°C with a solvent/feed ratio equal to 2.5. The results are presented in Table 2. The increase in the number of extraction stages resulted in a decrease in the sulfur content of the raffinates as well as a decrease in yields. As an example, it can be observed for NMP that an increase in the number of extraction stages from 1 to 2 led to a decrease in the sulfur concentration from 807 to 632 ppmw and a decrease in the raffinate recovery from 41.7 to 39.8 wt %.



Minimum sulfur contents of 1007, 543, and 575 ppmw were achieved after three extraction stages with ACN, NMP, and DMF, respectively. The three-stage crosscurrent extraction has significantly improved the degree of sulfur removal (Figure 4); 90.3 wt % of sulfur was extracted in this experiment using NMP as the solvent, but the raffinate yield (38.5 wt %) was rather low. Figure 4 shows that the gradient for change in the degree of sulfur removal follows the order of NMP < DMF < ACN.

Figure 4. Effect of the number of extraction stages on the degree of sulfur removal for solvent to feed ratio of 2.5

Table 2. Effect of the number of extraction stages on the yield and sulfur content of the raffinates (solvent/feed = 2.5)

Number of stages	Yield (wt %)			Sulfur (ppmw)		
	ACN	NMP	DMF	ACN	NMP	DMF
1	48.5	41.7	43.4	1345	807	846
2	46.0	39.8	41.9	1171	632	658
3	44.8	38.5	40.6	1007	543	575

### 3.3. Extractive desulfurization efficiency of polar solvents

In this work, the extractive desulfurization experiments were performed under the same experimental conditions for each polar solvent. The results (Tables 1 and 2) clearly indicate that the sulfur content of the raffinate and the yield of desulfurized LCO strongly depend on the type of solvent used, these two parameters decrease in the order: ACN > DMF > NMP. The degree of sulfur removal using NMP and DMF is much higher than with ACN (Figures 3 and 4). However, the yield values for NMP and DMF solvents are lower than ACN. This indicates that there is a trade-off between the degree of sulfur removal and the yield value. From an economic point of view, maximum sulfur removal with a maximum yield value is obviously desirable.

In summary, the extractive desulfurization efficiencies of NMP and DMF are comparable, but ACN is much less efficient. Mokhtar *et al.* [33] reported similar results in their study about the extractive desulfurization of Malaysian diesel fuel.

### 3.4. Product quality

The main properties of the feed and the raffinates produced during the three-stage cross-current extractions were determined using the ASTM test methods. The obtained results and the specifications of the standard NA 8110 are presented in Table 3. The evaluation of the quality of the products will be based on a comparison of their characteristics with the commercial standard of automotive diesel fuel in Algeria (NA 8110).

Table 3. Main properties of the feed and the raffinates produced during the three-stage crosscurrent extractions

Properties	Test method	LCO	Raffinates			NA 8110 Standard
			ACN	NMP	DMF	
Sulfur (ppmw)	ASTM D5453	2157	1007	543	575	0.15* max
Density at 15°C (kg/L)	ASTM D4052	0.926	0.879	0.842	0.851	0.810-0.860
Distillation (°C)						
Initial boiling point		178	173	171	170	-
10 vol %		214	208	202	205	-
50 vol %	ASTM D86	256	252	247	249	-
65 vol %		269	266	261	263	250 min
90 vol %		306	301	300	298	350 max
Final boiling point		326	319	317	318	390 max
Viscosity at 20°C (cSt)	ASTM D445	5.94	6.03	6.09	6.07	9 max
Pour point (°C)	ASTM D97	- 33	- 27	- 24	- 24	- 12 max** - 7 max***
Cetane index	ASTM D976	24.5	34.9	45.0	42.6	48 min
ASTM Color	ASTM D1500	< 2	< 1.5	< 1	< 1	2.5 max

\* in wt %; \*\* from 01/11 to 31/03; \*\*\* from 01/04 to 31/10

The sulfur present in diesel fuel is a major source of air pollution. During combustion in the diesel engines, the sulfur compounds burn to form harmful sulfur oxides (SO<sub>x</sub>) and sulfate particulates. In addition, total particulate matter (PM) emissions are proportional to the amount of sulfur in diesel fuel. Traces of sulfur present in the diesel fuels also poison the oxidation catalysts in the emission control system and reduce their effectiveness for the oxidation of harmful carbon monoxide, hydrocarbons, and volatile organic matter [2]. The solvent extraction process significantly reduced the sulfur content of the raffinates compared to the feed (Table 3). The sulfur content of the refined products, unlike that of the LCO, is well below the limit required by standard NA 8110 (0.15 wt %).

Density is an important property since it controls the injection time and the amount of diesel fuel that is delivered into the cylinder [34]. This is why the NA 8110 standard limits the diesel fuel density between 0.810 and 0.860 kg/l. The use of diesel fuel with a different density would have a significant impact on fuel consumption and engine power. Table 3 indicates a decrease in the density of the raffinates compared to that of the feed, which can be explained by the extraction of the heavy aromatic hydrocarbons present in LCO during the desulfurization processes with polar solvents. The raffinates produced by extraction with NMP and DMF have densities in accordance with the specification limits for automotive diesel fuel in Algeria.



The results of the distillation profile of LCO and the refined products presented in Table 3 coincide with the specifications required by the standard NA 8110. No significant differences could be seen between the base fuel and the raffinates. However, after the desulfurization process, a decrease of 50 vol % and 90 vol % points were observed, which will allow reducing the particulate emissions from vehicles [34].

Fuel viscosity has an important effect on the fuel pump and injector system. The shape of the fuel spray is affected by viscosity. If this is too high, the fuel will not be properly atomized into the cylinder, which will result in poor combustion, loss of power, and efficiency, with an increase in CO and hydrocarbon emission [34]. The viscosity of the refined products has slightly increased compared to that of the feed, but it remains below the maximum value (9 Cst) required by the standard NA8110 (Table 3).

The pour point is defined as the lowest temperature at which the sample will flow. It indicates how easy or difficult it is to pump the product, especially in cold weather. It also indicates the aromaticity or the paraffinity of the fraction. A lower pour point means that the paraffin content is low [35]. As shown in Table 3, the extractive desulfurization of the LCO led to an increase in its pour point, indicating an increase in its paraffin content. This can be explained by the removal of the aromatic hydrocarbons at the same time as the sulfur compounds during the extraction process. The amount of paraffin extracted is certainly lower because of the low solubility of this type of hydrocarbons in polar solvents. The pour point of the feed and the refined products are below the maximum limits imposed in winter and in summer by the standard NA 8110.

The cetane number is the result of an engine test that compares the ignition delay for a fuel with a blend of two reference fuels. The first is the normal cetane, and the second is heptamethylnonane. Higher cetane numbers indicate that the fuel has a shorter ignition delay. A higher cetane number also results in less CO and unburnt hydrocarbons in the engine emission gases [34]. The cetane index is an empirical estimate of cetane number based on distillation range and density. Table 3 shows that the cetane index of the raffinates has increased compared to that of the feed, giving an indication of the improved ignition quality of the raffinates. This result is due to the extraction of the aromatic hydrocarbons initially contained in the LCO by the polar solvents. Aromatic compounds, in particular multiple ring aromatics, are characterized by a poor cetane number [34]. In all cases, regardless of the increase in the cetane index, all the raffinates have a cetane index lower than the specification limit for automotive diesel fuel.

The ASTM color of LCO and refined products meets the NA 8110 standard (Table 3). It has been observed that the color of the LCO after purification using the polar solvents becomes clearer, indicating the removal of undesirable nitrogen compounds. Nitrogen compounds are naturally present in LCO used as feedstock for diesel fuel production. They are traditionally responsible for color and gum formation [23].

In summary, the extractive desulfurization process using ACN, NMP, and DMF improved the LCO quality in terms of sulfur content, density, distillation profile, cetane index, and color. After treatment, the viscosity and the pour point of the LCO remain within the limits of the standard NA 8110. The products refined with NMP and DMF have a better quality than the raffinate obtained by extraction with ACN. As an example, extraction using NMP increased the cetane index of the LCO from 24.5 to 45.0 (Table 3). Under the same operating conditions, extraction with ACN gave a raffinate with a lower cetane index (34.9).

Karonis *et al.* [12] reported comparable results in their research on upgrading LCO with ACN as extraction solvent. The authors confirmed the removal of undesirable aromatic compounds during the extraction process by appropriate analyzes. Indeed, an increase in aromatics present in diesel fuel causes an increase in smoke, NO<sub>x</sub>, unburnt hydrocarbons, and particulate emissions substantially [30].

#### 4. Conclusions

In the present investigation, an attempt was made to study the extractive desulfurization process of LCO using different polar solvents (ACN, NMP, and DMF). It was found that:

1. For all the three solvents, with an increase in the solvent/feed ratio, the desulfurization of LCO increases, and the recovery of raffinate continuously decreases.
2. For all the three solvents, the desulfurization of LCO increases as the number of extraction stages increases, but the raffinate recovery decreases continuously.
3. The effects of increasing the solvent/feed ratio and the number of extraction stages on the degree of sulfur removal increase in the order: NMP < DMF < ACN.
4. The extractive desulfurization efficiency of polar solvents increases in the following order: ACN < DMF < NMP. However, the yield of desulfurized LCO decreases in the same order. The desulfurization efficiencies of DMF and NMP remain comparable under the same extraction conditions.
5. The extractive desulfurization process allowed a significant improvement in the quality of LCO, in particular by using DMF and NMP as extraction solvents. However, the refined products do not meet the NA 8110 standard and, therefore, cannot be used as such for automotive diesel engines.

As a closing remark, the desulfurization process of LCO by extraction with polar solvents is a promising method for producing a diesel fuel component with better quality than LCO and cleaner for the environment.

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