

## Upgrade of Low Sulfur Light Cycle Oil with Acetonitrile as Extraction Solvent

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**Abstract:** The increased efficiency of compression ignition engine compared to spark ignition engine has resulted into increased share of diesel engines for light and heavy duty vehicles in the European Union. Diesel fuel produced from hydrotreatment of straight run gasoil is inadequate to meet the market demand. Cracked gasoils from catalytic cracking processes, like light cycle oil (LCO) from FCC have very poor ignition quality characteristics (low cetane number), high density, high aromatic content, and high sulfur content and they are not proper for use as automotive diesel fuel. Solvent extraction is a well-known process in the petroleum industry, and has been in use for many years for the improvement of lubricating oils. This technique can be applied for the extraction of aromatic hydrocarbons from LCO. Acetonitrile, a low boiling point polar solvent was used for the extraction of aromatics. Acetonitrile was mixed with LCO in solvent to feed ratios from 1 up to 2,5 by volume. Results showed that as solvent to feed ratio was increasing, the raffinate yield was decreasing (57,0 to 26,5%), but the quality of the raffinate was continuously improving. For example, aromatics content was reduced from 69,2% to 45,6% m/m, the cetane index was increased from 24,3 to 40,0, and the sulfur content was reduced from 0,47% to 0,31% m/m. Although the properties of the raffinate are not adequate in order to be used as diesel fuel, this improvement can be seen as a first step for the upgrade of a fuel that could possibly give some more feedstock for the production of automotive diesel fuel, or a blending component for marine distillate fuel.

**Keywords:** Diesel fuel, light cycle oil, solvent extraction

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### I. Introduction

Despite the introduction of biofuels in the transport sector the continuous increase of automotive diesel consumption, especially in the European Union, demands new sources of diesel fuel. The increasing demand of diesel fuel drives for the investigation of alternative methods and new feedstocks in order to cover the current diesel demand. Petroleum derived diesel fuel will play a dominant role in the transport sector for the next few decades. The characteristics of these petroleum derived diesel fuels have significantly changed in the last two decades due to the more stringent environmental regulations set by governments and international authorities. The more stringent environmental specifications for exhaust emissions from diesel engines (Euro 5 and Euro 6 emission limits) need cleaner fuels in order to comply. These cleaner diesel fuels are ultra low sulfur diesel fuels (ULSD) having sulfur content below 10 mg/kg. It is also desirable that these fuels have low aromatic, low polyaromatic content, and high cetane numbers. These fuels are produced by severe hydrotreatment of atmospheric gasoil, or by severe hydrocracking of vacuum gasoil. [1] Another significant demand for low sulfur fuel comes from the recent IMO regulation for low sulfur fuels in the marine sector from 1/1/2020. A large amount of the required low sulfur fuel is expected to be covered by distillate fuels. [2]

Diesel engine (also referred to as compression ignition engine) is the powertrain of choice for a wide range of applications, varying from light duty passenger cars to enormous marine propulsion engines and from small home diesel generators to high power remote electrical stations. In comparison to the spark ignition engine, the compression ignition engine is more robust, less sensitive on fuel quality, which means that it can be fueled with a wide range of fuel qualities, and can achieve higher thermal efficiencies. The higher thermal efficiency results into lower carbon dioxide (CO<sub>2</sub>) emissions. These advantages however are often overshadowed by its exhaust emissions, mainly nitrogen oxides (NO<sub>x</sub>) and particulate matter (PM), for which diesel engines are notorious [3-5]

Light cycle oil (LCO), is a side product of the fractionation of fuel catalytic cracking (FCC) reactor effluent. The distillation characteristics of LCO are within the diesel range (180 – 360 °C), but it is a poor diesel

fuel blending component due to its poor engine ignition performance which is caused by the high aromatics content and the high sulfur content. [6] Beyond middle distillate blending, LCO has also historically been used as a viscosity cutter into heavy fuel oil for viscosity adjustment. [7-9] In the overall context of a high conversion, clean-fuels refinery, light cycle oil is an issue, both in terms of product blending and product-value maximization. In addition to the use of conventional hydrotreating, high pressure hydrocracking units have historically been used to crack LCO into naphtha and lighter products. These units are relatively high in capital cost, consume large quantities of hydrogen, and the naphtha product requires reforming before blending to gasoline. [10] The properties of LCO depend on FCC feed, and operating severity varies depending upon the market. Cracked products such as LCO and coker distillates have a considerably lower cetane value compared to straight run distillates derived from most of the world's crude sources. The cetane number of LCO is in the range 15 – 25, compared to 40 – 60 for the straight run distillates produced from the same crude. North American refiners operate their FCC units at high severity to maximize gasoline production with LCO yield of less than 20%, whereas European refiners operate a lower severity for greater LCO production. [11, 12]

The sulfur content in average light cycle oils can range from 0,2 to 2,5% m/m. A detailed sulfur speciation of LCO shows that a significant portion of the sulfur is found in alkyldibenzothiophenes (DBT), which are relatively difficult to desulfurize by hydrotreating. [6-8] The aromatics content of LCO from FCC units in a normal gasoline-oriented operation can be as high as 70% m/m. The organic nitrogen is almost entirely composed of non-basic aromatic compounds, such as carbazoles, with a concentration range of 100-800mg/kg. [13] The components of LCO boil in the diesel range with a 95% point of 360 °C or higher, representing thermally stable cracked hydrocarbons that are not further reacted in the FCC process. Over 70% of the aromatic hydrocarbons present in LCO have two rings, while the remainder is typically evenly split between single ring and 3-plus ring aromatics. Two and 3+ ring aromatics combust poorly in the diesel engine. They have very low cetane values and are the root cause of the low blending quality of LCO. The upgrade of LCO with severe hydrotreating and hydrocracking processes is under investigation, and gives satisfactorily results, but these processes have very high capital and operating costs. [10, 14]

Solvent extraction is a liquid – liquid extraction process for separating components in solution by their distribution between two immiscible liquid phases. A primary extraction solvent is used to extract one of the solutes from a mixture (similar to stripping in distillation), and a wash solvent is used to scrub the extract free from the second solute (similar to rectification in distillation). Solvent extraction is used primarily when distillation is impractical or too costly to use. A typical example is the extraction of aromatic fractions from lubricating oil base stocks, in order to improve their viscosity index. In this case, the undesirable aromatic compounds have similar distillation characteristics with the desirable paraffinic compounds, so they cannot be removed by distillation. Specific polar compounds like phenol, furfural and n-methyl pyrrolidone (NMP) have been used in such processes. [15, 16] The solvent extraction has been used also in the case of LCO, in an attempt to separate different types of hydrocarbons. The results showed that acetonitrile was more efficient extraction solvent in comparison with methanol for the separation of aromatics from non aromatic compounds. [17, 18] The quality of the extraction products (raffinate) was improved with increasing solvent/feed ratio, but the amount of the raffinate was reduced. [19]

This paper describes the use of the solvent extraction process, as an alternative method for the upgrade of LCO. The main task was the removal of polyaromatic compounds and the production of a product with improved characteristics, compared to the original LCO, and the evaluation of this product as possible component for the production of diesel fuel.

## **II. Materials and methods**

**Base Fuel:** In order to investigate the upgrade of low quality middle distillates by solvent extraction, a batch of light cycle oil from Motor Oil Hellas, AghiiTheodori in Greece as base fuel. The base fuel was analyzed regarding basic characteristics like density, viscosity, distillation characteristics, sulfur content and aromatic hydrocarbons content. All test methods were carried out according to the official ISO and EN methods described in EN 590 standard, even though LCO does not satisfy the requirements of this standard. Sulfur content was measured according to the method described in the ISO 8217 standard for marine fuels. The properties of the base fuel are given in Table 1. It is obvious that the LCO has very high aromatic hydrocarbon content (69,2% m/m), with very high polyaromatic hydrocarbons content (di-, tri+), that amounts to 52,2 m/m%. It is also a relatively high sulfur content fuel, since it contains 0,47 % m/m, well above the specification limit for automotive diesel 10 mg/kg. The ignition quality is very poor with the cetane index calculated 24,3 and the derived cetane number measured 16,2.

**Solvent:** The solvent used for the extraction was acetonitrile. Acetonitrile is a polar solvent (containing the polar cyanide group) with low boiling point. Before proceeding to the tests, acetonitrile was checked in a simple qualitative test in separation funnel with the LCO, and it was found to separate two phases, indication that it

could be used as extraction solvent. The main properties of acetonitrile are given in Table 2. Acetonitrile was provided by Acros Chemicals.

The main advantages of acetonitrile as extraction solvent for this process are:

- It separates the LCO feed into two phases in a variety of feed to solvent ratios.
- It has low boiling point and can be easily removed from the raffinate and extract phases with flash distillation.
- It is fully miscible with water, therefore traces of solvent after the flash distillation can be removed by simple water wash.

**Table 1.**Main properties of the LCO

Properties	LCO	Test Method
Density (kg/m <sup>3</sup> , 15 °C)	951,2	EN ISO 12185
Viscosity (mm <sup>2</sup> /s, 40 °C)	3,876	EN ISO 3104
Sulfur (% m/m)	0,47	ISO 8754
AromaticHydrocarbons (% m/m)		EN 12916
Mono	17,0	
Di	44,1	
Tri+	8,1	
Total	69,2	
Distillation (°C)		EN ISO 3405
IBP	193,2	
5%	230,3	
10%	244,9	
50%	294,9	
90%	358,0	
95%	373,1	
FBP	383,0	
Cetane Index	24,3	EN ISO 4264
DerivedCetaneNumber	16,2	ASTM D7170

**Experimental Procedure:** The extraction of LCO with acetonitrile as solvent was carried out in laboratory scale experiments. LCO was mixed with acetonitrile in solvent/oil ratios of 1:1, 1.5:1, 2:1 and 2.5:1 by volume respectively. Each mixture was stirred for 30 minutes in beaker, using magnetic stirrer, at environmental temperature. The mixture of LCO – solvent was transferred in separation funnel, and it was left to settle, where two phases were separated:one phase rich in solvent and one phase rich in oil. The solvent rich phase contains the more polar compounds that have better cohesion with the solvent, contains the more aromatic fraction that is the extract of the process. The oil rich phase, contains mainly the less polar hydrocarbons with low cohesion to the polar solvent, is the raffinate of the extraction process. Both phases, and especially the extract phase, contain significant amount of the solvent, which has to be removed before the analysis of the products. The solvent is removed from the extraction products by simple distillation under atmospheric pressure, using a fractionation column on the top of the distillation flask. The recovery of the solvent is very critical for the economic viability of the extraction process. The amounts of recovered solvent, solvent free raffinate and solvent free extract are recorded and used to check the mass balance of the whole process. The recovered solvent is reused in the next extraction process. The amount of LCO undergone the extraction process in each LCO – solvent ratio was 500 ml. This was done in order to ensure that the recovered amounts of raffinate and solvent in each case should be adequate in order to perform all the analysis tests carried out for the base fuel (density, viscosity, distillation, sulfur content, aromatic hydrocarbons content).

**Table 2.** Physical properties of acetonitrile

Appearance	Clear, colorless liquid
Solubility	Miscible in water
Density (kg/m <sup>3</sup> , 15 °C)	790,0
Boiling Point (°C)	81,6
Melting Point (°C)	-46,0

### III. Results and discussion

The aim of this work was to investigate the possibility to improve the quality of LCO by solvent extraction, which is a simple process, and operates under mild conditions (ambient temperature and pressure). In previous works, acetonitrile has been efficiently used for the separation of specific hydrocarbons from LCO. [17, 19]

**Extraction Yield:**The LCO sample was extracted with acetonitrile in order to produce a better quality middle distillate fuel. The better quality middle distillate fuel is the fuel with the lower aromatic hydrocarbons content,

therefore the raffinate. The yield of raffinate is decreasing as the solvent – oil ratio is increasing. On volumetric basis, the raffinate yield is decreasing from 52,1% to 25,6%v/v as the solvent/oil ratio increases from 1:1 to 2,5:1. On mass basis, the raffinate yield decreases from 49,3% to 23,2% m/m. The results for the yields and raffinates at each solvent/oil ratio are shown in Figure 1.

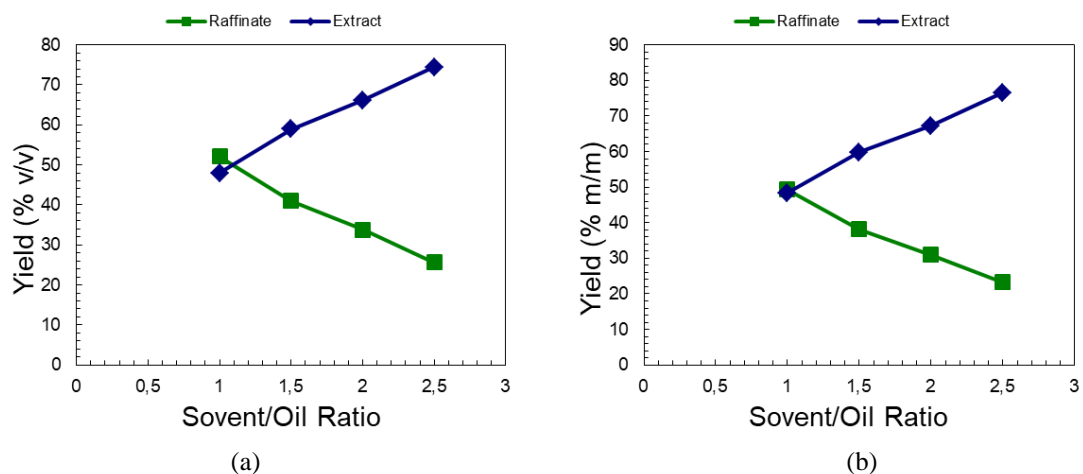


Figure 1. Extraction yield on volumetric (a) and mass (b) basis

**Product Quality:** The evaluation of the quality of the extraction products will be based on the analysis of physicochemical characteristics of these products. The physicochemical properties of the raffinates are given in Table 3 and those of the extracts in Table 4.

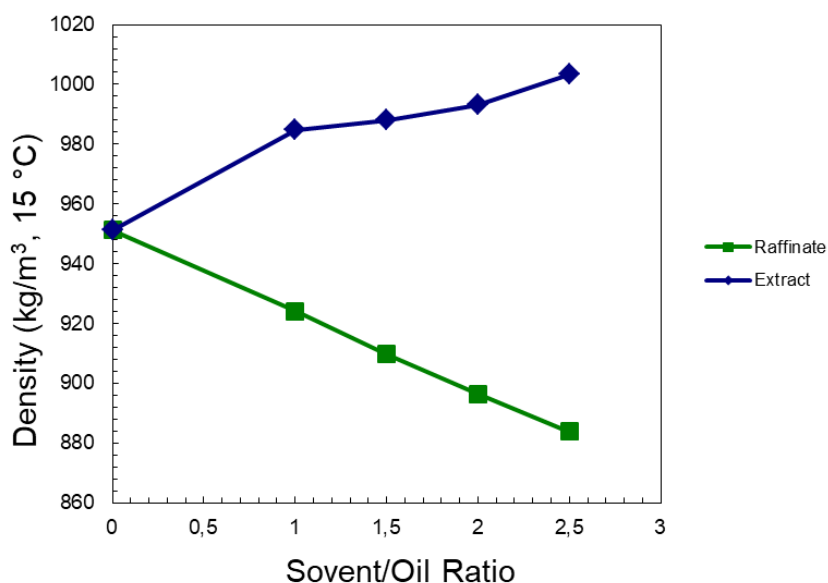


Figure 2. Densities of the extraction products

Density is a significant property, since it controls the amount of fuel that is injected and burned in the combustion chamber. High amount of fuel sprayed in the combustion chamber, results in higher emissions of partially oxidized products. The use of high density fuels, results in higher emissions of particulate matter. [20] Fuel density can affect also NO<sub>x</sub> emissions, since the use of fuels with lower density reduces NO<sub>x</sub> emissions. [21] On the other hand, reduced diesel fuel density increases fuel consumption and reduces power output. [20] Density values were measured according to EN ISO 12185 test method. Density of raffinate decreased as the solvent/oil ratio increased, and density of extract increased, as solvent/oil ratio increased. This behavior is depicted in Figure 2. This reduction in raffinate density is attributed to the reduction of aromatic hydrocarbons in the fuel, since they are removed by the solvent. [17, 19] In all cases, regardless the reduction of density, all the raffinates have densities significantly higher than the specification limits for automotive diesel fuel,

according to EN 590 standard (820 – 845 kg/m<sup>3</sup>). In the case of marine diesel fuel, the raffinates at solvent/oil ratio 2,5:1 meets DMA and DMZ standards (maximum density 890 kg/m<sup>3</sup>).

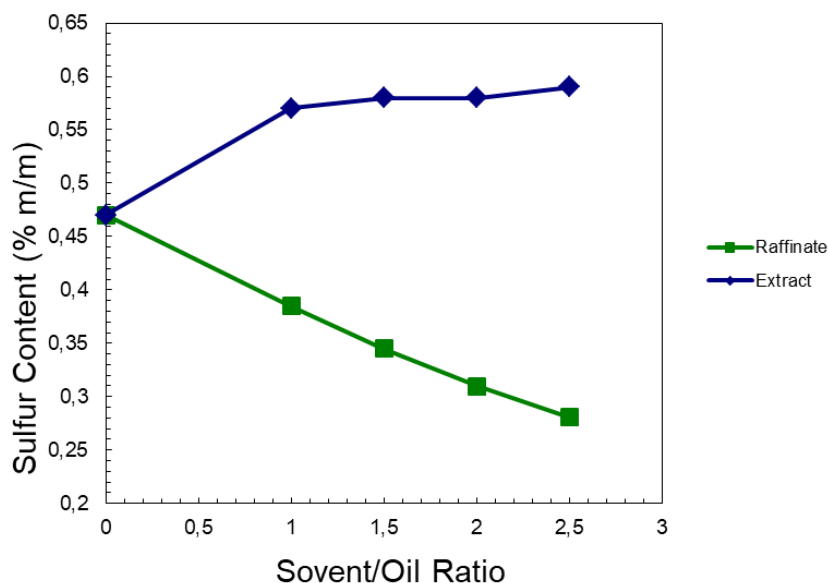
**Table 3.**Main properties of the raffinates

Properties	Solvent/Oil				Test Method
	1:1	1,5:1	2:1	2,5:1	
Yield (% v/v)	52,1	41	33,8	25,6	
Yield (% m/m)	49,3	38,2	31,0	23,2	
Density (kg/m <sup>3</sup> , 15 °C)	951,2	924,0	909,7	896,4	EN ISO 12185
Viscosity (mm <sup>2</sup> /s, 40 °C)	3,876	3,925	3,934	3,934	EN ISO 3104
Sulfur (% m/m)	0,47	0,38	0,35	0,31	ISO 8754
AromaticHydrocarbons (% m/m)					EN 12916
Mono	16,5	16,1	16,4	15,8	
Di	36,7	33,2	29,7	26,9	
Tri+	8,0	6,8	5,8	5,2	
Total	59,4	54,4	49,8	45,6	
Distillation (°C)					EN ISO 3405
IBP	174,4	177,2	170,6	173,4	
5%	215,9	208,2	217,8	215,9	
10%	240,0	236,1	241,0	236,1	
50%	291,8	291,8	289,8	288,8	
90%	352,9	349,8	348,7	347,7	
95%	365,8	362,7	361,7	360,6	
FBP	375,7	370,4	371,5	370,4	
Cetane Index	29,5	32,5	36,8	40,0	EN ISO 4264
DerivedCetaneNumber	26,1	30,6	35,1	38,8	ASTM D7170

**Table 4.**Main properties of the extracts

Properties	Solvent/Oil				Test Method
	1:1	1,5:1	2:1	2,5:1	
Yield (% v/v)	47,9	59	66,2	74,4	
Yield (% m/m)	48,4	59,8	67,3	76,5	
Density (kg/m <sup>3</sup> , 15 °C)	984,7	988,0	993,2	1003,4	EN ISO 12185
Viscosity (mm <sup>2</sup> /s, 40 °C)	3,837	3,808	3,789	3,740	EN ISO 3104
Sulfur (% m/m)	0,47	0,58	0,58	0,59	ISO 8754
AromaticHydrocarbons (% m/m)					EN 12916
Mono	15,2	16,5	17,2	17,8	
Di	48,9	51,2	52,2	53,9	
Tri+	8,2	8,3	8,3	8,4	
Total	74,2	77,9	79,7	82,3	
Distillation (°C)					EN ISO 3405
IBP	174,4	167,8	161,2	169,6	
5%	206,3	203,4	200,6	200,6	
10%	231,2	227,3	223,4	222,5	
50%	287,7	282,6	279,5	275,4	
90%	348,7	346,7	344,6	339,5	
95%	358,6	357,5	356,5	354,4	
FBP	367,3	367,3	366,3	364,2	
Cetane Index	16,7	16,5	15,7	14,3	EN ISO 4264

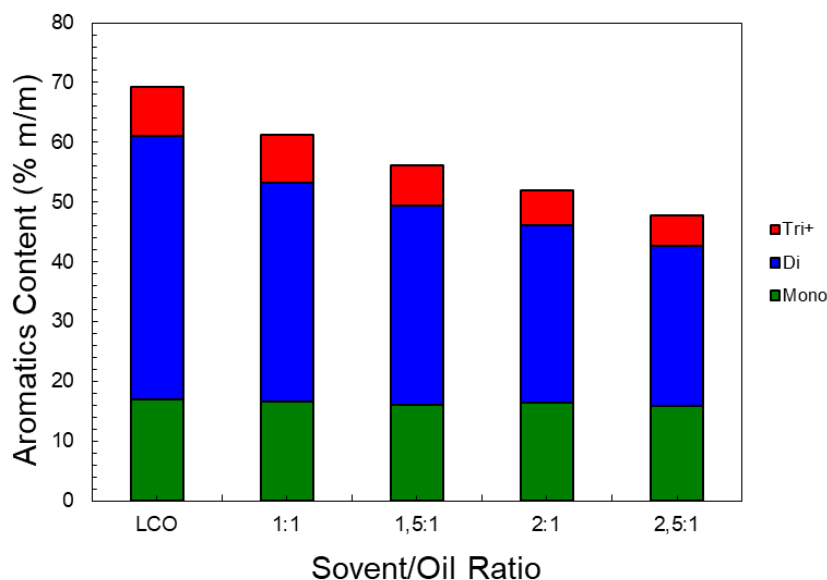
Sulfur content is a critical property affecting particulate matter emissions. Increasing sulfur content of diesel fuel results in higher sulfates emissions that contribute to the total particulate matter emissions. [22-23] Sulfates act also as precursors for the formation of the agglomerates that form particulate matter. Solvent extraction process reduced the sulfur content of the raffinate, compared to the base fuel. Sulfur content was measured according to ISO 8754 test method. The sulfur content decreased in the raffinate and increased in the extract, as the oil ratio increased. The decrease in sulfur content of the raffinate is attributed to the extraction of polar sulfur compounds by the polar solvent. [17, 19] For the same reason, the sulfur content of the extract increased, and was higher than the sulfur content of the base fuel. In all cases, the sulfur content of the raffinate was high, well above the specification limit for automotive diesel fuel. Figure 3 depicts the change in sulfur content for the raffinate and extract fractions, with solvent – oil ratio. In all cases, sulfur content was higher than the IMO specification for low sulfur marine distillates (0,1% m/m), but the products can be used as blending components for low sulfur residual fuel oil.



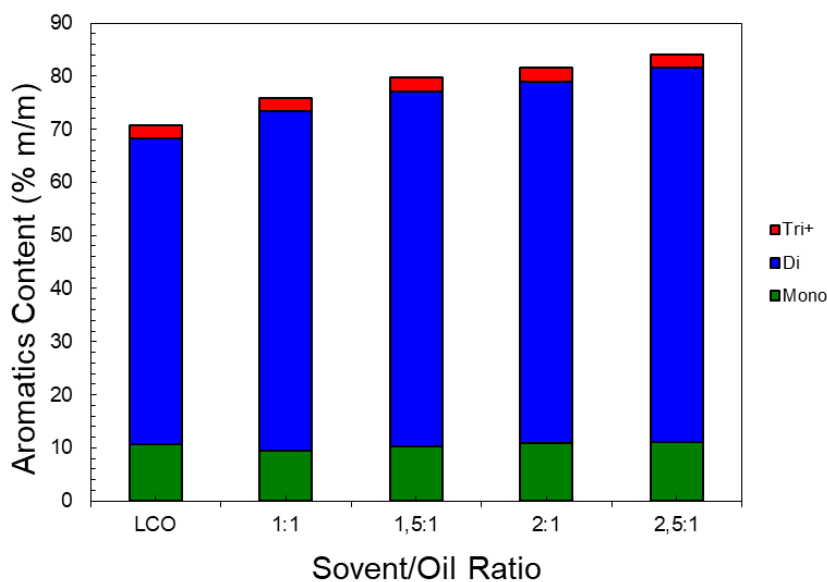
**Figure 3.** Sulfur content of the extraction products

Aromatic content is another critical parameter. The aromatic ring is very stable and does not decompose easily in high temperatures. On the other hand, formation of particulate matter from aromatics takes place by condensation and polymerization via formation of PAH molecules. [24] Instead of total aromatics, polyaromatics content is more critical for the regulation of PAH and particulate matter emissions, since it is strongly correlated to these types of exhaust emissions. [25] The main purpose of solvent extraction was the selective removal of specific compounds, which are fully miscible to the solvent. In this case, acetonitrile was used as polar solvent for the extraction of aromatic hydrocarbons from LCO. Aromatic hydrocarbons content was measured by EN 12916 test method. As shown in Figure 4, the aromatic hydrocarbons concentration in raffinate was reduced as solvent/oil ratio increased. This is in accordance with previous findings where the presence of paraffinic hydrocarbons was higher in the raffinate samples. [17] The decrease was more significant in the case of diaromatics (the majority of aromatic hydrocarbons in LCO). Tri+ aromatics also decreased, while monoaromatics did not significantly change. The case of the extract fraction is shown in Figure 5. The behavior was different, since increasing the solvent/oil ratio, the aromatics content increased, due to the removal of aromatic hydrocarbons with the solvent. Diaromatic hydrocarbons content clearly increased, while mono- and tri+ aromatics increased slightly, as it was found in previous research. [17, 19] In all cases the polyaromatics content of the raffinate samples was well above the limit according to EN 590 standard for automotive diesel (8% m/m). It is noted that there is no specification for aromatics content in ISO 8217 for marine fuels.

The distillation profile was used to characterize the difference in the volatility between various types of fuels. It was very difficult to draw clear conclusions regarding the quality of the fuel, using only the distillation characteristics, since all type of hydrocarbons had similar boiling points, which were affected mainly by the carbon atoms in the molecule, and less by the hydrocarbon type. [25] Distillation characteristics along with other fuel properties have been reported to affect exhaust emissions from diesel engines. [21, 26] Distillation characteristics were measured for all samples according to EN ISO 3405 test method. The distillation curves of the raffinates are shown in Figure 6a. For comparison purposes, the distillation curve of the LCO is also shown in the same Figure. No significant differences could be seen between the base fuel and the raffinates. The case of distillation curves of the extracts, compared with the LCO is given in Figure 6b. In this case, the extracts seem to have lower distillation points compared to the base fuel.



**Figure 4.** Aromatics content of raffinate products



**Figure 5.** Aromatics content of extract products

Cetane number is one of the more significant diesel fuel parameters, since it gives the antiknock properties of the diesel fuels. [25, 27] Cetane index is used as a good approximation of the cetane number, in cases where a prototype test engine is not available. [25] Cetane index is calculated from the density, and 3 specific points of the distillation curve: 10%, 50% and 90%, according to EN ISO 4264 test method. Figure 7 depicts the variation of cetane index for the raffinates and the extracts, with the solvent – oil ratio. In the case of raffinates, as solvent – oil ratio increases, the cetane index of the raffinate increases, giving an indication of the improved ignition quality characteristics of the raffinates, compared to the relevant characteristics of the LCO. This improvement in ignition quality is related with the reduced content of aromatics in the raffinate. [17, 19] The cetane index of the best raffinate (solvent/oil 2,5:1) was increased from 24,3 to 40,0, reaching the minimum acceptance limit according to ISO 8217 standard for marine distillate fuels. In the case of the raffinate samples, derived cetane number (DCN) was measured as well, according to ASTM D7170 test method. The results of these measurements are presented also in Figure 7. It is clear that there is a difference in the cetane index and DCN, especially for fuels of lower ignition quality. The cetane index of the extracts is lower in all cases than the cetane index of the LCO. The low cetane index of the extract is expected, since the high aromatic hydrocarbons content of the extracts reduces significantly the ignition quality characteristics of these fuels. [27-28]

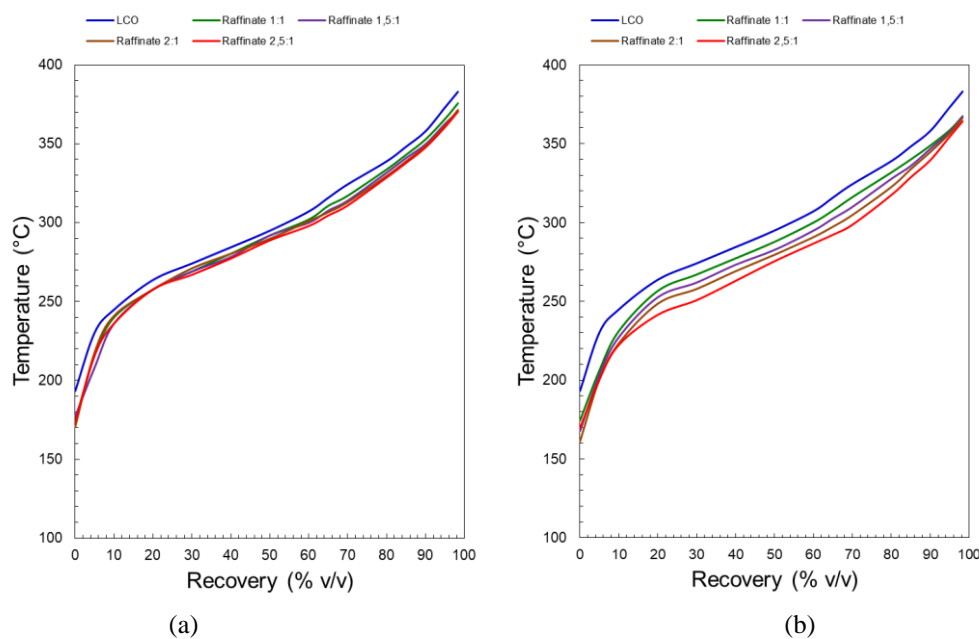


Figure 6. Distillation profiles of raffinate products

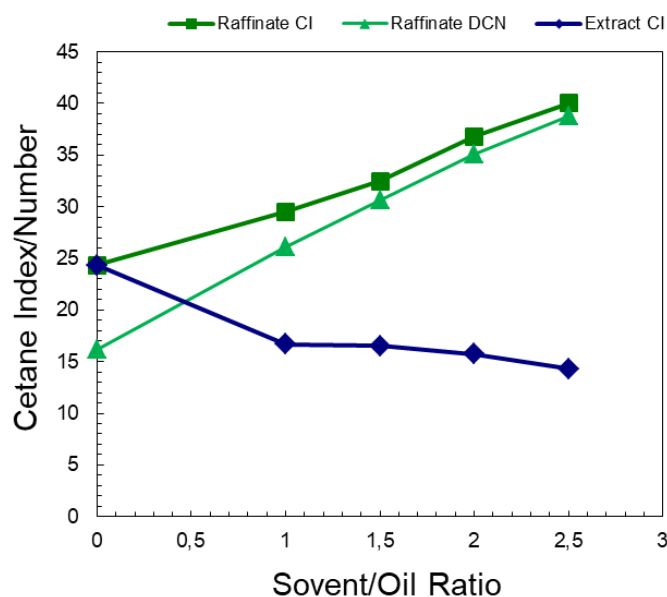


Figure 7. Cetane index and cetane number of extraction products

The above observations indicate that in order to improve the quality of the extraction product for possible use as diesel fuel, a high solvent – oil ratio has to be used, the penalty being the reduced yield of the final raffinate. In all the cases examined, the properties of the raffinate products did not meet the quality standards that apply for automotive diesel, but the properties of the raffinate from high solvent/oil ratio do comply with the standards of ISO 8217 for marine distillate fuels.

#### IV. Conclusions

The aim of this work was to investigate the improvement of the characteristics of LCO with solvent extraction in an attempt to increase the availability of better quality fuel derived from a low quality middle distillate. LCO has boiling characteristics in the diesel fuel range, but due to the very high aromatic hydrocarbons content cannot be used as automotive diesel fuel, or even marine distillate fuel. Extraction of aromatic compounds from LCO may give an extra stream that, if desulfurized, may increase the diesel fuel availability. For this reason, an LCO sample was extracted with acetonitrile, in order to evaluate the quality of the extraction products. The main conclusions can be summarized as follows:

1. The yield on the raffinate was reduced from 49,3% to 23,2% m/m as solvent/oil ratio increased from 1:1 to



2,5:1.

2. The quality of the raffinate improved as the solvent/oil ratio increased. More specifically, as solvent – oil ratio increased from 1:1 to 2,5:1, density decreased from 951,2 to 896,4 kg/m<sup>3</sup>, sulfur content decreased from 0,47% to 0,31% m/m, polyaromatic hydrocarbons content was reduced from 44,7% to 32,1% m/m, cetane index increased from 29,5 to 40,0, and the derived cetane number increased from 26,1 to 38,8.
3. The properties of the raffinate deviated from those currently accepted as automotive fuel. The improved properties of raffinate, compared to the LCO, make easier the inclusion of desulfurized raffinate in the diesel fuel pool. The results were even better if the raffinate was to be considered as marine diesel fuel blending component.
4. The properties of the extract decreased in quality, as the solvent/oil ratio increased. The produced extract had very poor properties in order to be used as diesel blending component, but it can be used as viscosity cutter for the production of heavy fuel oil.

As a closing remark, it can be noted that the results showed that the upgrade of LCO with solvent extraction is a process that can improve the quality characteristics of this low quality feedstock, but the final product of the extraction process (raffinate) is still a low quality fuel and cannot be used as it is for automotive diesel fuel as it is, without further improvement by other processes.

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