# TREATMENT OF THE OIL OF *LALLEMANTIA IBERICA* WITH ACTIVATED ADSORBENTS

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*In this paper a simple, laboratory-scale procedure for the treatment of crude Lallemantia iberica oil, using activated adsorbents, is described. The purification involved the use of a bleaching clay and of aluminium oxide (alumina). For the assessment of the purification process the oil samples have been characterised before and after treatment, using proton nuclear magnetic resonance spectroscopy, gas-chromatography, UV spectroscopy for colorimetry, as well as by determination of some characteristics like acid value and iodine number.*

**Keywords**: *Lallemantia iberica* oil, bleaching clay, alumina, acid value, iodine number, purification

## 1. Introduction

In an era of unstable petroleum prices, global warming and critical waste management problems, switching from fossil raw materials to renewable resources can make a significant contribution to sustainable development.

Vegetable oils can potentially replace petrochemicals, since starting chemicals, monomers and polymers can come directly from these resources.

An interesting candidate in this respect appears to be the oil obtained from the seeds of *Lallemantia iberica*. This oil has remarkable characteristics due to its high degree of unsaturation, coming from an unusually high content of alpha-linolenic acid. With an iodine number in the range of 190-205, it exceeds by far the commercial linseed oil (iodine number between 170-185). *Lallemantia* oil is not yet produced on an industrial scale but has already attracted considerable attention due to its potential as oleochemical raw material.

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As with any crude vegetable oil, the use in synthesis or as cooking oil requires some previous purification steps, in order to remove unwanted components like free fatty acids, phospholipids, carotenoids, chlorophylls, peroxides etc. [1]. Some of these impurities may affect negatively the chemical behaviour, the colour, taste, odour or stability in time. The selected method used must avoid damaging the oils themselves or their beneficial minor components. Another important factor in this choice is the destination of the purified oil, whether edible or technical [1-2].

The materials generally used in the adsorption process are mineral clays which have high adsorptive properties [2-8]. There are techniques which aim to improve these qualities, like the acid activation of clay with sulfuric or hydrochloric acid [2-3, 5-6, 8].

Recently, in order to avoid problems arising when using acid-activated clays, like a residual acid effect on the treated oil and the formation of soap during neutralization, *Akinwande et al.* investigated the activation of clays with bases [3,4]. In the present paper alumina was also used as an adsorbent and in order to increase its efficiency in lowering the acid value a base activation was attempted.

We describe here a simple, laboratory-scale procedure for the treatment of crude *Lallemantia iberica* oil, using activated adsorbents, with the aim of making it suitable for a non-edible application, namely functionalisation and subsequent production of thermosetting resins.

## 2. Experimental

### *2.1. Materials*

*Lallemantia iberica* oil (LALO) obtained by a cold-pressing process, was acquired from PTG Deutschland, Flurstedt, Germany.

The F-160 clay (former Engelhard) was purchased from Engelhard, now EP Engineered Clays, and the aluminium oxide (granular – 4-8 mesh) from Sigma-Aldrich. The aluminium oxide was milled before used.

Some of the solvents and reagents were obtained from Sigma-Aldrich (ethanol, diethyl ether, sodium hydroxide, phenolphthalein indicator, chloroform, bromine), other from Fisher Chemical (acetic acid glacial, potassium iodide), Fluka (iodine), Chimreactiv (sodium thiosulphate solution 0,1 mol/L). All products were used as received, without any purification.

### *2.2. Instrumentation*

***Gas Chromatography–Mass Spectroscopy Analysis (GC-MS)***

Gas-chromatograms of the fatty acid methyl esters (FAME) mixtures were recorded on an Agilent Technologies 6890 N instrument with flame ionization detector. Separation into components was made on a capillary column especially

designed for the FAME analysis (Supelco SPTM 2560: 100 m length, 0.25 mm inner diameter, 0.2 μm film thickness). Fatty acids identification was made by comparing the retention time for each peak with a commercially available standard, with a mixture of 37 fatty acid methyl esters provided by Supelco ™.

***Nuclear Magnetic Resonance Analysis ($^1$H-NMR)***

$^1$H nuclear magnetic resonance ($^1$H-NMR) spectra were recorded on a Bruker Advance III Ultrashield Plus 500 MHz spectrometer, operating at 11.74 Tesla, corresponding to the resonance frequency of 500.13 MHz for the $^1$H nucleus, equipped with a direct detection four nuclei probe head and field gradients on z axis. The chemical shifts are reported in ppm, using the TMS as internal standard.

***UV-VIS Spectrophotometer***

The apparatus used was a Thermo Scientific Evolution 220 UV-Visible Spectrophotometer, with 1.0 nm resolution and double-beam configuration.

### *2.3. Analytical methods*

***Determination of the fatty acids profile*** was carried out by gas-chromatography, by analyzing the mixture of FAME obtained through transesterification (previous conversion of the triglycerides into the corresponding more volatile methyl esters – FAME).

***The iodine number (index)*** was analyzed in three ways: by computation based on GC data, by computation from $^1$H-NMR data [9] and, for comparison, by titration based on AOAC Hanus method [10], involving the use of a solution of iodine monobromide in acetic acid. The sample is treated with excess reagent and the remaining iodine is titrated with a sodium thiosulphate solution.

***The acid value and free fatty acid of the oil*** was determined by titration following AOAC methods [4, 11]. A known amount of oil was placed in a 250 ml conical flask with 50 ml of ethanol-ether solution 1/1 (V/V) neutralized just before used with 0.1 M sodium hydroxide. The mixture was shaked until the substance is completely dissolved, then the solution was titrated with sodium hydroxide until a pink coloration can be observed which persists for 30 s.

The acid value and free fatty acid were then calculated following the relations below:

$$\textbf{\textit{Acid value (AV) = (V x 5.61) / M}}$$

$$\textbf{\textit{\% Free fatty acid (FFA) = 0.503 x acid value (AV)}}$$

Where, ***V*** is the volume of 0.1 M sodium hydroxide used in ml and ***M*** is the mass in g of oil sample.

To ***evaluate the coloured components***, the absorbance of the crude oil was measured by UV-visible spectroscopy. The absorption maxima of extracted

pigments depend on the type of solvent [12]. So, in order to analyze the crude oil, two solvents were used: acetone and n-hexane. In case of acetone, a quantity of 0.5 g of oil was diluted in 5 ml of solvent, and the absorbance of the sample was determined at the maximum absorption wavelength using acetone as reference. For n-hexane, a diluted solution of *Lallemantia* oil:solvent, 1:4, was used. The treated samples of the oil were diluted in n-hexane (1:4, oil:hexane).

### *2.4. Adsorptive treatment*

***The treatment method*** used for the oil was based on the adsorption technique. A known amount of oil (LALO) – 20 g – was placed in a conical flask; the amount of adsorbent, representing 10%, respectively 20% (weight) based on the amount of oil, was added to the flask; the mixture was then stirred and slightly warmed, max. 30-35°C, during 20 min (in order to avoid possible polymerisation of the highly unsaturated oil). The flask was then stored in the dark for 3 hours. Next, the stirring and warming (same conditions) were repeated for 20 minutes. In the end, after 5-6 hours left in the dark for decantation, the content of the flask was filtered through Isolab quantitative filter paper 110 mm.

***Activation of alumina with sodium hydroxide*** solution (NaOH) was done according to the following procedure: 20 g of aluminium oxide were immersed in 20 ml 5% sodium hydroxide solution, the mixture was stirred and heated on a magnetic stirrer at max. 45°C for 30 minutes. It was then filtered (using a folded filter). And then dried for 30 minutes at 200-210°C. After cooling it was used to treat *Lallemantia* oil in a duplicate experiment (1 – with 10% adsorbent, 2 – with 20 % adsorbent). The rest of the treatment was performed following the same steps and conditions as previously described.

## 3. Results and discussion

### *3.1. Characterisation of crude Lallemantia iberica oil*

Chromatograms obtained from GC-MS were recorded in triplicate, the results presented being their mean values. The identification of methyl esters of fatty acids was done by comparing the retention times (RT) of each chromatographic peak with the retention times of a commercial standard mix – Supelco 37 FAME Mix.

Determination of the concentration of each component was made based on the peak's integrals identified, also considering the specific response factors of the detector. Table 1 shows the fatty acid profile of the *Lallemantia iberica* oil, determined in this way.

*Table 1*

**Fatty acids profile for Lallemantia iberica oil**

| *Lallemantia iberica (LALO)* | | | |
|---|---|---|---|
| ***Peak*** | ***Fatty Acid*** | ***Number of C atoms and double bonds*** | ***Mean*** |
| 1 | Palmitic | C16-0 | **6.635** |
| 2 | Palmitoleic (9 cis Hexadecenoic) | C16-1 | **0.173** |
| 3 | Stearic | C18-0 | **1.714** |
| 4 | cis-9-Oleic | C18-1 | **13.313** |
| 5 | Linoleic | C18-2 | **12.980** |
| 6 | cis-11-Eicosanoic | C20-1 | **0.641** |
| 7 | Linolenic | C18-3 | **64.367** |
| 8 | cis-11,14,17-Eicosatrienoic | C20-3 | **0.177** |

From the above data it appears that the main component of the oil is the highly unsaturated linolenic acid (64%), which determines the remarkable properties of the oil.

Fig. 1 shows the $^{1}$H-NMR spectrum of *Lallematia iberica* oil (LALO); signal assignments and chemical shifts are shown in Table 2.

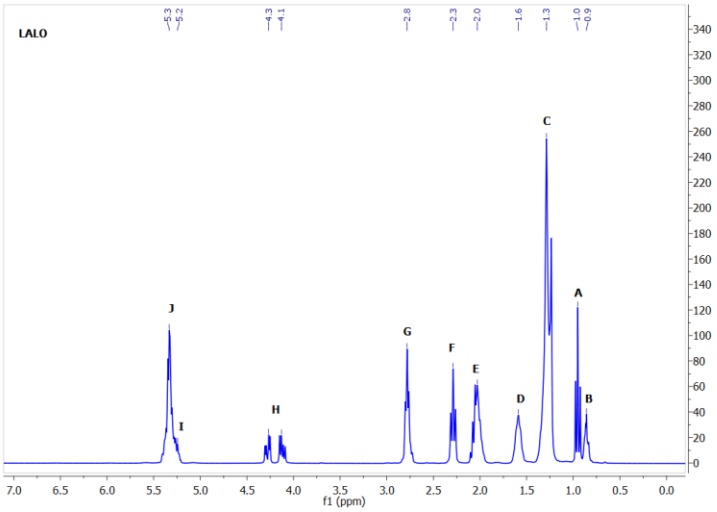


Fig. 1. $^{1}$H-NMR spectrum of Lallemantia iberica oil (LALO).

*Table 2*

**Signal assignments and chemical shifts in $^{1}$H-NMR spectrum of Lallemantia iberica oil (LALO)**

| ***Signal*** | ***δ*** | ***Integral*** | ***Proton*** | ***Compound*** |
|---|---|---|---|---|

| | *(ppm)* | | | |
|---|---|---|---|---|
| **A** | **0.95** | **4.97** | -CH=CH-CH2-C***H***3 | Linolenic acid |
| **B** | **0.85** | **3.00** | -CH2-CH2-CH2-C***H***3 | All alkyl chains, except for linolenic |
| **C** | **1.20** | **31.63** | -(C***H***2)$_n$- | All alkyl chains |
| **D** | **1.60** | **5.32** | -C***H***2-CH2-COOH | All alkyl chains |
| **E** | **2.02** | **9.36** | -C***H***2-CH=CH- | Allylic protons (all unsaturated fatty acids) |
| **F** | **2.20** | **5.22** | -C***H***2-COOH | All acyl chains |
| **G** | **2.76** | **7.11** | -CH=CH-C***H***2-CH=CH- | *bis*-allylic protons (linoleic and linolenic acid) |
| **H** | **4.19** | **3.31** | -C***H***2OCOR | Glycerol (α position) |
| **I** | **5.15** | | -C***H***OCOR | Glycerol (β position) |
| **J** | **5.29** | **12.72** | -C***H***=C***H***- | All unsaturated fatty acids |

The iodine number expresses the degree of unsaturation of fats. Vegetable oils, depending on the iodine index, fall into three categories: "***Drying oils"*** (flax, hemp, poppy, walnut, etc.) having a high iodine index, usually higher than 120; "***Semi-drying oils"*** (rapeseed, cotton etc.), with values ranging between 100 to 120; "***Non-drying oils"*** (almonds, olives, etc.) with index below 90.

The iodine index for *Lallemantia iberica* oil varies between 185 and 205, which classifies it in the ***drying oil*** category, like the linseed oil.

Tabel 3 shows the iodine index values (g $I_2$/100 g oil) for the *Lallemantia iberica* oil determined with three different methods:

*Table 3*

**Iodine Values of Lallemantia iberica oil (LALO) determined with three different methods**

| | ***Determination Method for Iodine Index*** | ***Values*** | ***Iodine Values*** | ***Average*** | ***STDEV=SD (Standard Deviation)*** |
|---|---|---|---|---|---|
| 1 | Chemometric determination - based on GC data | 203.22 g I2/100 g | 203.22 | | |
| 2 | Chemometric determination - based on RMN data | 199.13 g I2/100 g | 199.13 | | |
| 3 | Titration - AOAC Hanus Method | 193.84 g I2/100 g | 193.84 | | |
| | | | ***596.1900*** | ***198.7300*** | ***4.7028*** |

The standard deviation computed for these three values is less than 5% (4.7%) which proved that the number are consistent with each other and confirm the classification of the studied *Lallemantia* oil as ***drying oil***.

The saponification index of *Lallematia iberica* oil was also calculated chemometrically [9], the value being **196 mg KOH/ g oil**; the saponification index of oils represents the necessary amount (in mg) of KOH for the saponification of 1 g of oil.

The acid value (AV) is a common parameter in the specification of fats and oils. It is defined as the weight of KOH in mg necessary to neutralize the free organic acids present in 1g of oil. The AV for the crude LALO, determined by titration following AOAC methods, is **5.24 mg KOH/g oil**. Therefore, AV measures the content of free fatty acids (FFA) in the fat or oil. In our case the value of FFA is **2.64%**.

The UV-vis spectrum of crude *Lallemantia* oil was carried out over the entire wavelength range (200nm to 800nm) and shows that the highest absorbance occurs, in case of acetone as a solvent, at 324.8nm (value 0.227) (Fig. 2.a), and in the case of n-hexane in the range between 300 and 360nm, with a maximum at 301nm (value 2.4) (Fig. 2.b). Both spectral bands are in near ultraviolet (NUV) (300 – 400nm). This can be explained by the fact that radiation absorption from one region in the spectrum gives rise to a complementary color associated with another region [1]. Thus, our solution absorbs complementary to its pale-yellow color in the near UV spectral area (300-400 nm).

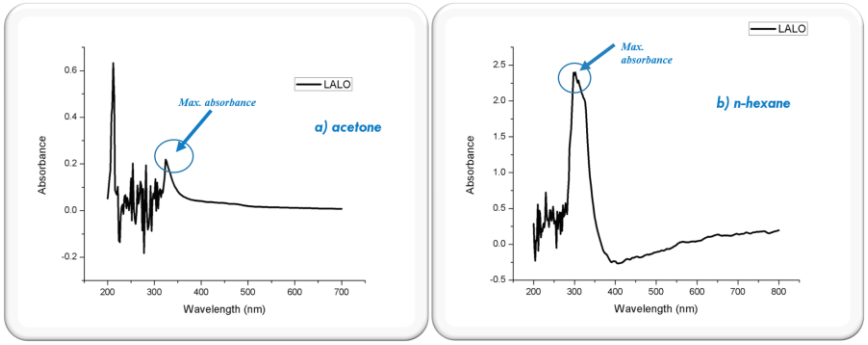


Fig. 2. UV-vis spectrum of crude Lallemantia iberica oil (LALO); solvent used: a) acetone; b) n-hexane

### *3.2. Treatment of Lallemantia iberica oil*

In this study we analyze comparatively ***a)*** two types of adsorbents, ***b)*** two different rations oil:adsorbent, and ***c)*** cold and warm adsorbent treatments.

Five experiments where conducted, each one in duplicate, varying the amount of adsorbent (10% and 20% wt of oil).

The two types of adsorbents that have been used are: Engelhard grade F-160 (experiments 1 and 2) and alumina (experiments 3, 4 and 5). The alumina was milled before use.

The process of adsorption depends, primarily, on the properties of the adsorbent. When choosing the adsorbent, compromises must be made between efficient removal of color, or other unwanted components and the preservation of some good, beneficial components [7]. Engelhard grade F-160 [8] is suitable for chlorophyll and carotenoid removal in vegetable oils, while alumina was used before [13] with better results for improvement of the color without adverse effect on oil properties and under prevailing atmospheric conditions.

The first and the third experiments consisted in treatment of the LALO with cold F-160 (first) and cold alumina (for the third).

In the experiments two and four, treatment of oil was done with calcined and cooled adsorbent, exp. 2 with F-160 and exp. 4 with alumina; the adsorbent was previously dried at 200-210°C for 30 min and cooled.

In the last experiment (exp. 5) the alumina was activated with sodium hydroxide 5% solution and then calcinated at 200-210°C for 30 min and allowed to cool down.

Table 4 summarizes the five duplicate experiments (1 – with 10% adsorbent, 2 – with 20 % adsorbent) for *Lallemantia* oil treatment.

*Table 4*

**Summary of the 5 Lallemantia iberica oil (LALO) treatment experiments.**

| *No* | *LALO* | *LALO quantity (g)* | *Adsorbent quantity (g)* | *Adsorbent quantity (wt % of oil)* | *Notations* |
|---|---|---|---|---|---|
| 0 | Crude LALO | - | - | - | LALO |
| 1 | Treatment with cold F-160 | 20.00 | 4.00 | 20% | AER-2 |
| | | 20.00 | 2.00 | 10% | AER-1 |
| 2 | Treatment with calcined F-160 | 20.00 | 4.00 | 20% | AEC-2 |
| | | 20.00 | 2.00 | 10% | AEC-1 |
| | | | | | |
| 3 | Treatment with cold alumina | 20.00 | 4.00 | 20% | OAR-2 |
| | | 20.00 | 2.00 | 10% | OAR-1 |
| 4 | Treatment with calcined alumina | 20.00 | 4.00 | 20% | OAC-2 |
| | | 20.00 | 2.00 | 10% | OAC-1 |
| 5 | Treatment with NaOH activated and dried alumina | 20.00 | 4.00 | 20% | OAA-2 |
| | | 20.00 | 2.00 | 10% | OAA-1 |

### *3.3. Oil characterisation after treatment*

In order to assess the performance of treatment methods, each treated sample was then analyzed by determination of major lipid indices: acid value and iodine value.

Table 5 summarizes the figures for acid values, % of free fatty acids which is corelated with acid value, and iodine values of *Lallemantia* oil before and after treatment with the two types of adsorbents.

The acid values data summarized in Table 4 demonstrate that the treatment of LALO oil with NaOH activated alumina leads to superior results to those obtained with the other treatments, the reduction in acidity being the most effective (≈ 96%).

*Table 5*

**Acid values, % of free fatty acid and iodine values of Lallemantia iberica oil (LALO) before and after treatment.**

| *No* | *LALO* | *Adsorbent quantity (wt % of oil)* | *Not.* | *Acid Values (mg KOH)/g oil)* | *Free Fatty Acid (%)* | *Iodine Values (g I/100g oil)* |
|---|---|---|---|---|---|---|
| 0 | Crude LALO | - | LALO | 5.24 | 2.64 | 193.84 |
| 1 | Treatment with cold F-160 | 20% | AER-2 | 4.63 | 2.33 | 198.70 |
| | | 10% | AER-1 | 5.22 | 2.63 | 197.12 |
| 2 | Treatment with calcined F-160 | 20% | AEC-2 | 4.47 | 2.25 | 198.01 |
| | | 10% | AEC-1 | 5.08 | 2.56 | 190.93 |
| | | | | | | |
| 3 | Treatment with cold alumina | 20% | OAR-2 | 2.41 | 1.21 | 194.58 |
| | | 10% | OAR-1 | 3.14 | 1.58 | 189.58 |
| 4 | Treatment with calcined alumina | 20% | OAC-2 | 0.84 | 0.42 | 196.03 |
| | | 10% | OAC-1 | 3.50 | 1.76 | 188.94 |
| 5 | Treatment with NaOH activated and dried alumina | 20% | OAA-2 | 0.39 | 0.20 | 191.62 |
| | | 10% | OAA-1 | 2.04 | 1.03 | 189.91 |

Also, when comparing clay treatment and non-activated alumina treatment, whether the adsorbents were dried or not, the most effective acidity decrease is obtained when treating with alumina.

In the third place, comparing the treatment with cold or calcined adsorbent, only in the clay treatment, better values are obtained if the adsorbent is previously dried.

Also, the amount of adsorbent (10% or 20%) is significantly influencing the results of treatment, the samples treated with 20% adsorbent presenting the highest decreased of acid value. That, on the other hand, has a negative effect on the yield of the treatment (about 10-15% less). The yield is influenced also by the type of adsorbent: with alumina this is 9-10% larger than with the clay.

In our study, according to the data in Table 4 and comparing with the crude oil iodine index, the iodine values for the treated oil samples varied by ± 4.9 (193.84 ± 4.9), meaning less than 3%. This indicate that the iodine value didn't change too much during the treatment [14]. The color of the oil may be derived from several pigments present in different concentrations, minor or not, which have different chemical natures. Because of that, one single type of processing is not effective in removing all of them [1].

In the present study the amount of pigment removed was evaluated by calculating the bleaching capacity of the adsorbent [2-3], determined with the following equation:

$$\textit{Bleaching capacity} = 100*(A_0 - A)/A_0,$$

where $A_0$ is the absorbance of the crude oil and $A$ is the absorbance of each oil sample, respectively.

A diluted solution of *Lallemantia* oil in n-hexane was prepared for each of the treated samples of the oil (1:4, oil: hexane), and the absorbance of the sample was determined at the maximum absorption wavelength (302 nm).

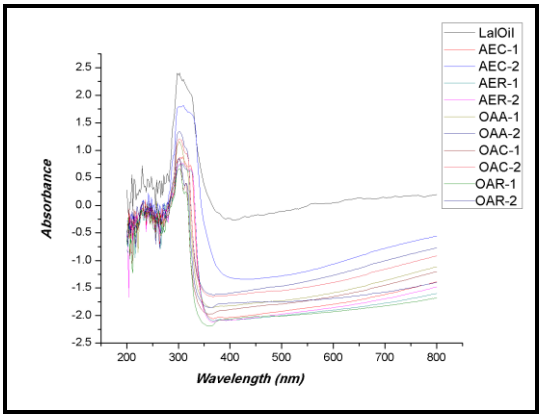


Fig. 3. UV-vis spectrum of treated and crude oil samples of LALO; solvent used – n-hexane

Table 6 and Fig. 3 summarizes values from the UV-vis spectrum: the absorbance of the samples of *Lallemantia* oil before and after treatments.

Fig. 3 shows the drop of absorbance values (the UV-Vis spectra of the treated samples) for the treated samples as compared to the untreated LAL oil spectrum.

*Tabel 6*

**Values from UV-vis spectrum for each LALO sample before and after treatment.**

| *No* | *LALO* | *Adsorbent quantity (wt % of oil)* | *Not.* | *Max. Absorbance (301 nm)* | *Bleaching Capacity (%)* |
|---|---|---|---|---|---|
| 0 | Crude LALO | - | LALO | 2.401 | |
| 1 | Treatment with cold F-160 | 20% | AER-2 | 0.752 | 68.68 |
| | | 10% | AER-1 | 0.731 | 69.55 |
| 2 | Treatment with calcined F-160 | 20% | AEC-2 | 0.785 | 67.31 |
| | | 10% | AEC-1 | 0.867 | 63.89 |
| | | | | | |
| 3 | Treatment with cold alumina | 20% | OAR-2 | 0.948 | 60.52 |
| | | 10% | OAR-1 | 0.963 | 59.89 |
| 4 | Treatment with calcined alumina | 20% | OAC-2 | 1.207 | 49.73 |
| | | 10% | OAC-1 | 1.341 | 44.15 |
| 5 | Treatment with NaOH activated and dried alumina | 20% | OAA-2 | 1.344 | 44.02 |
| | | 10% | OAA-1 | 1.162 | 51.60 |

The data in Table 6 shows that the most efficient treatments for color reduction are those with Engelhard F-160.

## 4. Conclusions

The improved quality of the treated *Lallemantia* oil depends on the nature of the adsorbent used, the preliminary treatment of it and the treatment procedure.

Thus, treatment with the F-160 clay affords better results in the bleaching while the treatment with alumina resulted in decreasing oil acidity in a more efficient way.

The activation of alumina with sodium hydroxide proved to increase the treatment efficiency resulting in acid values dropped almost to zero (0.39 – value of the acid value in case of 20% adsorbent treatment), the reduction of acidity, as expected, is more pronounced if a higher quantity of activated alumina is used.

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