

## Used motor oil treatment: Turning waste oil into valuable products

R. Abu-Ellella<sup>1</sup>, M.E. Ossman<sup>1,2</sup>, R. Farouq<sup>1\*</sup>, M. Abd-Elfatah<sup>1</sup>

<sup>1</sup>Petrochemical Engineering Department, Pharos University, Alexandria, Egypt

<sup>2</sup>Informatic Research Institute (IRI), City for Scientific Research and Technology Applications (CSAT), BorgElarab, Alexandria, Egypt

### Abstract

This paper investigates the treatment of waste engine oils using four different acids (sulphuric acid, phosphoric acid, acetic acid and formic acid) followed by treatment with clay earth. The effect of these treatments on the properties of the treated used oil was studied. Tests include: specific gravity, flash point, kinematic viscosity, FTIR, total acid number and total base number. The results showed that the formic acid-clay treatment improve the flash point of the used motor oil and made it comparable with fresh motor oil and the sulphuric acid, acetic acid and formic acid followed by clay treatment improved the kinematic viscosity of the oil while the treatment with phosphoric acid-clay has no improvement action on the used oil kinematic viscosity and flash point. The FTIR analysis was used to determine the TAN and TBN and the results showed that the treatments of used motor oil with acetic acid-clay and formic acid-clay has higher TBN values than the TBN value of used engine oil.

**Key words:** Used engine oil, motor oil, acid/clay treatment, recycling, waste oil recovery

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\*Corresponding Author, e-mail: [rania\\_farouq29@yahoo.com](mailto:rania_farouq29@yahoo.com), Tel:0201002706756

### 1. Introduction

Motor oil is used for lubricating various internal combustion engines. During normal use, many impurities such as dirt, metal scrapings, water and chemicals can get mixed in the oil, so the oil will be ineffective for further application [1]. Oil does not wear out; it just gets dirty after use. As a result, the associated impact to the environment is huge when there is uncontrolled dumping and land filling of used oil in the environment. On the other hand used oil is considered a valuable resource and a vital source of energy [2]. Following the proper treatment to remove contaminants, used oil can be re-refined into base lube oil, reprocessed as fuel oil, or used as feedstock to produce petroleum-based products or other commercially valuable products via different processes [3]. Recycling used lubricants is not an altogether new idea, there are many recycling processes in use nowadays include; Solvent extraction [4- 8], Solvent extraction followed by adsorption [9-10], Desludging and adsorption processes [11]. Different adsorbent materials were studied in refining waste lubricating oil [12], such as oil adsorbent egg shale powder, date palm kernel powder and acid activated date palm kernel powder [13]. Acid-clay has been used as a recycling method for used engine oil for a long time [14- 18]. The most popular physical methods include distillation [19]. By comparing between four methods of recycling of used lubrication oils (acid/clay treatment, distillation/clay, acid treatment and activated

charcoal/clay treatment methods); it was shown from the tests that the best results obtained using the acid/clay treatment [20-21].

The objective of this research is focused mostly on treating used motor oil so the product can be used as a fuel, thereby reducing environmental pollution. For this purpose four different acids (sulphuric acid, phosphoric acid, acetic acid and formic acid) were used in the treatment of waste engine oil followed by clay treatment.

### 2. Experimental Work

#### 2.1. Material used

All chemicals used in the experiments are analytic reagent grade. Waste lubricant oils were gathered at collecting stations in the city of Alexandria (Egypt). The samples were homogenized and transferred to an appropriate tank, stored away from light at room temperature for several days to allow large suspended particles to settle under gravity. The acids used for treatment were sulphuric acid (97.99%), phosphoric acid (less than 85%), acetic acid (99%) and formic acid (85%). Industrially produced bleaching earth was used for bleaching, and potassium hydroxide (92%) for neutralization.

## 2.2. Methods:

### 2.2.1. Acid Treatment

300 ml of used engine oil was measured in a 500 ml beaker. Also, 30 ml of acids (sulphuric acid, acetic acid, phosphoric acid, formic acid) was measured in a separate 50 ml beaker. The regulator hot plate was switched on and the measured base oil was placed on top. The temperature of the base oil (used engine oil) was maintained at 40-45°C. At this temperature the (sulphuric acid, acetic acid, phosphoric acid, formic acid) were introduced into the used engine oil simultaneously with stirring of the mixture for 10 minutes.

### 2.2.2. Sedimentation /Decantation

At the end of the acid treatment step, the acidic oil was allowed to settle 24 hours to form sediment at the bottom of the beaker. After this period, the acidic-oil was properly sedimented and was decanted into Another 500 ml beaker using piece of cloth while the residue (acidic sludge) at the bottom of the beaker was discarded.

### 2.2.3. Clay Activation

200g of clay (after dirt, sand and stone have been removed) was made into slurry with distilled water of about 80cm<sup>3</sup>. 50-60cm<sup>3</sup> of acid in 0.35 mole/cm<sup>3</sup> concentration was added to the slurry made. The slurry was transported into aluminum pan and left for one hour at temperature between 90-100°C. After the time duration, the mixture was washed with distilled water in order to remove any excess acid. The pH of washing water was monitored until it was found to be neutral. The washed clay mixture was dried in an oven for one hour and grounded into powdery form.

### 2.2.4. Bleaching

The acidic oil in the beaker was then subjected to bleaching. The oil was placed on a regulator hot plate and the temperature was maintained at a temperature of 110°C. 10 wt % of activated bleaching earth was introduced into the oil and the mixture was continuously stirred for 15 minutes. At the end of the bleaching step, the bleached oil was neutralized.

### 2.2.5. Neutralization

The bleached oil was neutralized to adjust the pH of the oil to neutrality. At this step, 4 wt% of the oil of Hydrated lime was introduced into the bleached oil by taken into consideration the pH of the bleached oil at a given point in time. The bleached oil was neutralized with a continuous manual stirring for 10 minutes. At the end of the bleaching and neutralization steps, the oil was allowed to sediment in the beaker for 24 hours and was decanted into the beaker, while the residue at the bottom of beaker was discarded.

### 2.2.6. Sedimentation /Decantation

During this stage, the oil was allowed to sediment in the beaker for 24 hours and was decanted into another beaker, while the residue at the bottom of the beaker was discarded.

### 2.2.7. Filtration

The sedimented oil was finally filtered using a filter cloth and the filtrate was collected in a filtration flask and was observed to be clear while, the residue (filter cake) was discarded.

## 3. Results & discussion

### 3.1. Specific Gravity

From ordinary theory, we know that density of a substance is equal to the mass of a substance divided by the volume of the substance, that is:

$$\text{density}(\rho) = \frac{\text{mass}(m)}{\text{volume}(V)} \quad (1)$$

Specific gravity is the ratio of the density of the material to density of the equal volume of water. The temperature at which the density has been measured must be known because density changes as temperature changes. Specific gravity is influenced by the chemical composition of the oil. An increase in the amount of aromatic compounds in the oil results in an increase in the specific gravity, while an increase in the saturated compounds results in a decrease in the specific gravity. Used engine oils specific gravity increases with the presence of increasing amounts of solids in the used engine oil. Fig. 1 shows the results for the used and fresh motor oils are 0.96 and 0.869 [15] respectively, while those treated oils obtained by the acid treatments methods were: 0.915 for treating with formic acid, 0.93 for phosphoric acid treatment, 0.92 for sulphuric acid treatment and 0.915 for acetic acid treatment. Fig. 1 shows that the treatment of used oil with formic or acetic acid improve the specific gravity of the used motor oil and make it comparable with specific gravity of fresh oil. While Fig. 2 shows the results for the used and fresh motor oils are 0.96 and 0.869 respectively, while those treated oils obtained by the acid/clay treatment methods were: 0.91 for treating with formic acid, 0.925 for phosphoric acid treatment, 0.91 for sulphuric acid treatment and 0.90 for acetic acid treatment. Fig. 2 shows that the treatment of used oil with sulphuric acid /clay, formic acid/clay and acetic acid/clay improve the specific gravity of the used motor oil and make it comparable with specific gravity of fresh oil.

Fig. 3 shows comparisons between acid treatment and acid/clay treatment methods with respect to specific gravity and the results indicated that for acid treatment only, the selectivity sequence can be given as indicate by the following order to sulphuric acid > formic acid & acetic acid > phosphoric acid while for the acid - clay treatment the selectivity sequence is indicate by the following order to acetic acid - clay > sulphuric acid - clay & formic acid - clay > phosphoric acid - clay.

### 3.2. Flash Point

The flash point of engine oil is the lowest temperature to which the oil must be heated under specified conditions to give off sufficient vapor to form a mixture with air that can be ignited spontaneously by a specified flame. A substantially low flash point of engine oil is a reliable indicator that the oil has become contaminated with volatile products such as gasoline. The flash point increases with increasing molecular mass of the oil [22]. The flash points of samples were analyzed by open cup flash point apparatus by ASTM D97. The flash point of fresh motor oil is 232 °C, for used engine oil flash point is 178°C, formic acid treatment method flash point is 230°C, sulphuric acid treatment 210°C, phosphoric acid treatment method flash point is 200°C and acetic acid treatment flash point is 218°C as shown in Fig. 4. Lowering the value of flash point of used

engine oil is because of presence of light fuels [23]. The flash point of the fresh motor oil is 232 °C because it contains many different additives which contribute to improving its flash point. In contrast, the flash point of the measured used engine oil is 178 °C. This decrease in flash point is a result of contamination with fuel and oxidation products [22]. On one hand, Fig. 4 clearly shows that the flash point of the used oil is highly affected by adding formic acid followed by clay treatment (220 °C) and acetic acid followed by clay treatment (218 °C). On the other hand it is slightly affected by adding phosphoric acid followed by clay treatment (200 °C). The flash point of the used oil treated with formic acid/clay (220 °C) is giving comparable results to the fresh motor oil.

### 3.3. Kinematic Viscosities

Viscosity is a state function of temperature, pressure and density. There is an inverse relationship between viscosity and temperature, when the temperature of the engine oil decreases the viscosity increases and vice versa. Viscosity testing can indicate the presence of contamination in used engine oil. The oxidized and polymerized products dissolved and suspended in the oil may cause an increase of the oil viscosity, while decreases in the viscosity of engine oils indicate fuel contamination. Oxidation of motor oils during use in an engine environment

produces corrosive oxidized products, deposits, and varnishes which lead to an increase in the viscosity [24]. The kinematic viscosity of the samples was measured by the falling ball viscometer and the viscosity can be calculated by the following equation.

$$\mu = gD_p^2(\rho_p - \rho)t_p / 18L \quad (2)$$

Where  $t_p$  is the time required for a sphere to fall a distance,  $L$ ,  $\rho_p$  is the density of the solid sphere,  $\rho$  is the density of the fluid,  $D_p$  is the diameter of the solid sphere and  $g$  is the gravitational acceleration (9.8 m/s<sup>2</sup>).

Kinematic viscosity of fresh motor oil (100 °F) is 82.2 cst [20].

Fig. 5 shows a decrease in kinematic viscosity of the used oil; this is due to contamination in the form of sludge in the used oil. Viscosity increase can occur due to oxidation or contamination [25]. Viscosity decrease can be caused by dilution with light fuel. The result of the viscosity test shows that, the used lube oil has lost most of its viscosity due to contamination. However, treatment has restored most of its viscosity. The result shows also that refining using sulphuric acid and filtration method gave the highest viscosity. This can be attributed to the possible conversion of possible contaminants by the acid and removal by the filtration from the motor oil.

**Table 1. Fourier transform infrared (FTIR) spectrometer for used motor oil:**

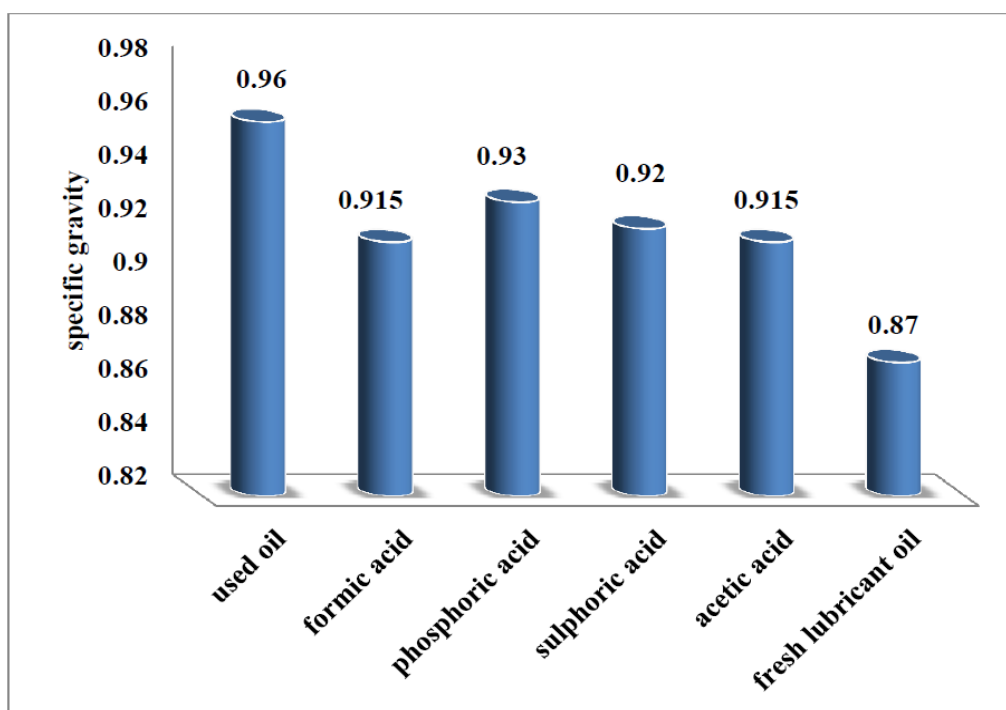
Frequency cm <sup>-1</sup>	Bond	Functional group
725.21	C-H	Alkans
813.46	C-H	Aromatic
884.53	C-H	Aromatic
1033.48	C-O	Carboxylic acid
1158.63	C-O	Carboxylic acid
1374.89	C-H	CH3
1448.16	C-H	Alkan
1603.88	C=C	Aromatic
1743.74	C=O	Carbonyl compounds
2335.07	O-H	Carboxylic Acid
2727.21	H- C=O:CH	Aldehyd
2922.01	C-H	Alkan
3379.41	-OH	water

**Table 2. Fourier transform infrared (FTIR) spectrometer for used motor oil treated with formic acid/clay:**

Frequency $\text{cm}^{-1}$	Bond	Functional group
723.62	C-H	Alkans
812.21	C-H	Aromatic
1156.83	C-O	Carboxylic acid
1375.84	C-H	CH <sub>3</sub>
1460.03	C-H	Alkan
1599.43	C=C	Aromatic
1741.98	C=O	Carbonyl compounds
2335.07	O-H	Carboxylic Acid
2728.56	H-C=O:CH	Aldehyd
2923.95	C-H	Alkan
3396.15	-OH	water

**Table 3. Fourier transform infrared (FTIR) spectrometer for used motor oil treated with acetic acid/clay :**

Frequency $\text{cm}^{-1}$	Bond	Functional group
723.08	C-H	Alkans
812.07	C-H	Aromatic
1153.86	C-O	Carboxylic acid
1375.84	C-H	CH <sub>3</sub>
1460.17	C-H	Alkan
2924.15	C-H	Alkan
3430.09	-OH	water



**Fig. 1.** Specific gravity of fresh oil, used oil and treated oils obtained by the acids treatment methods

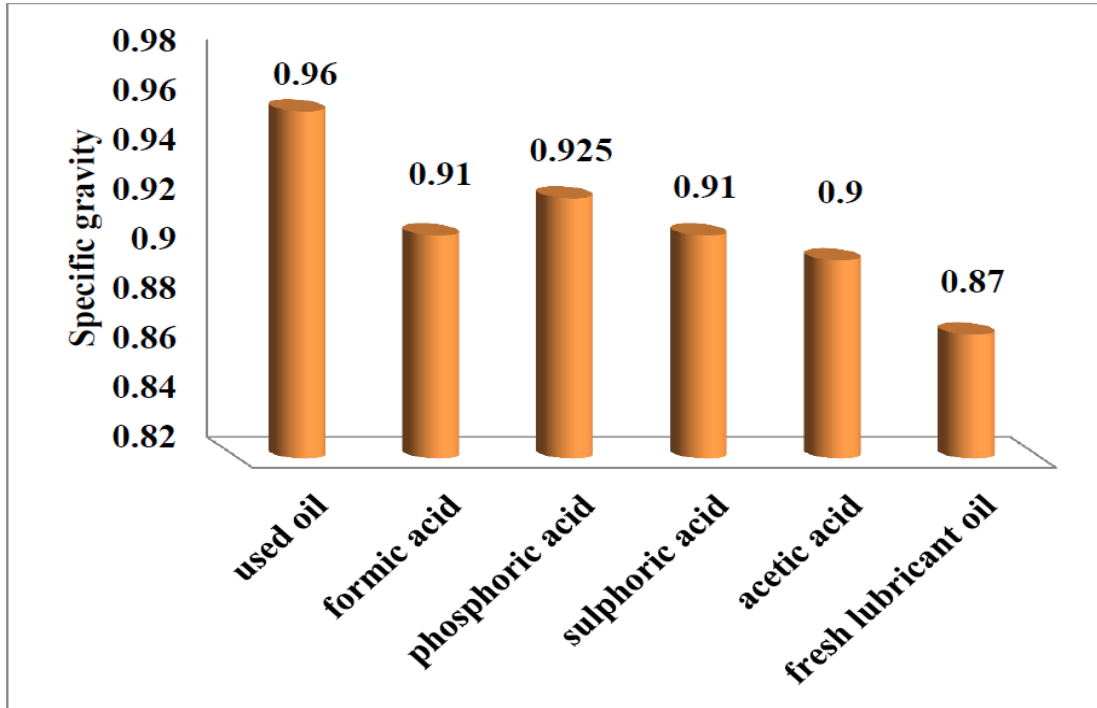


Fig. 2. Specific gravity of fresh oil, used oil and treated oils obtained by the acid-clay treatment methods.

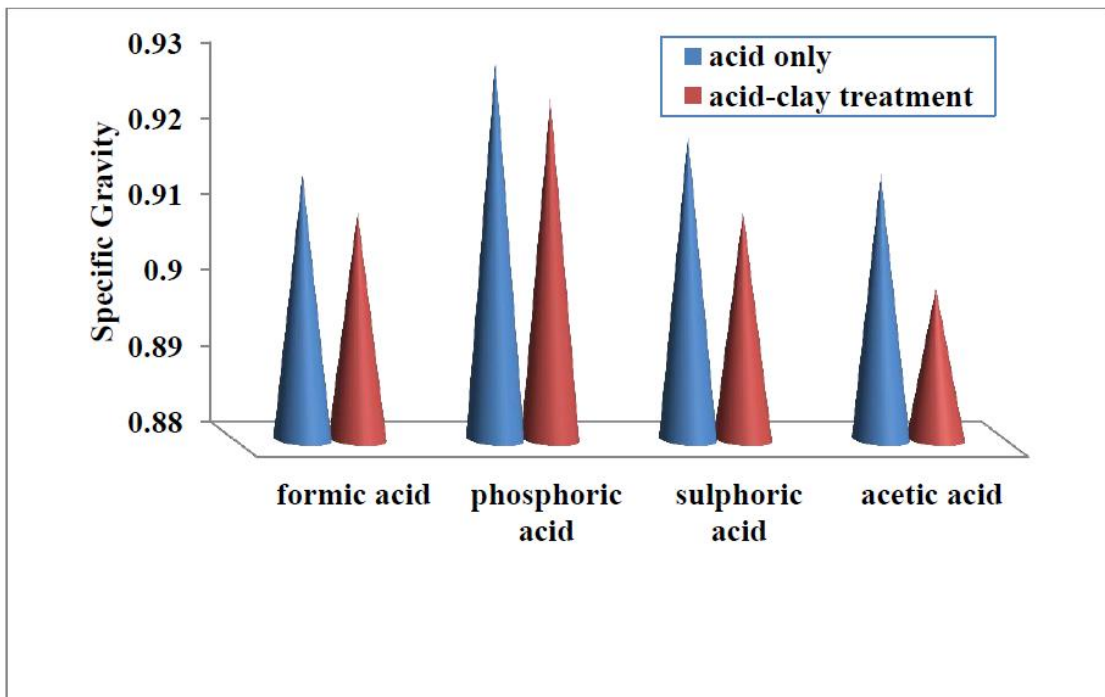


Fig. 3. Comparisons between acid treatment and acid/clay treatment methods with respect to specific gravity

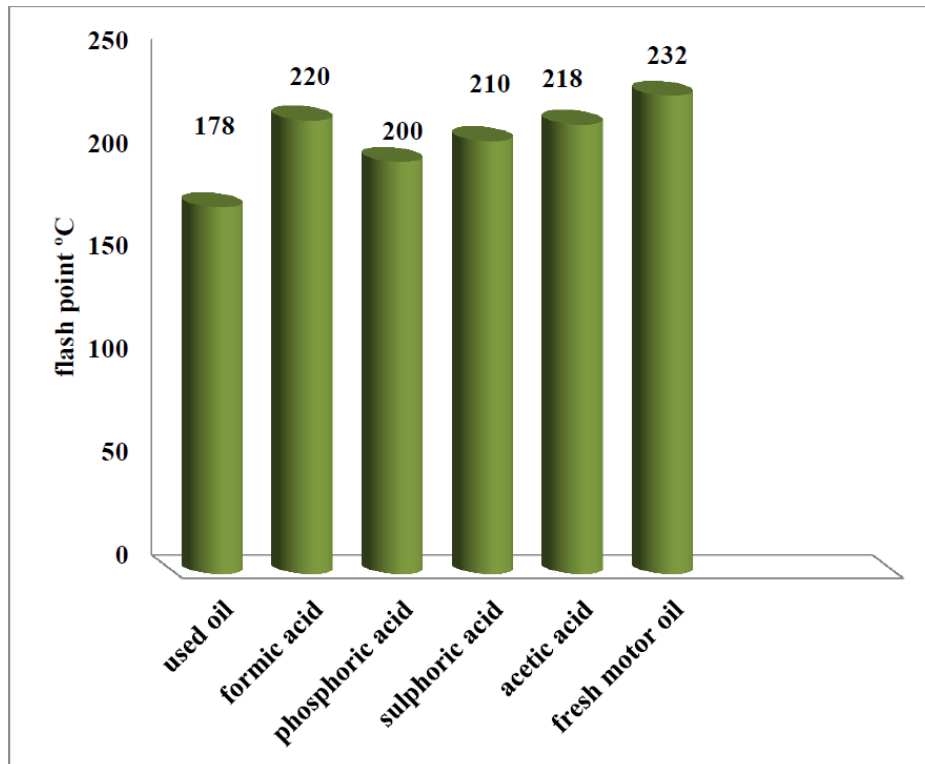


Fig. 4. Flash Point for used, fresh and treated oil samples.

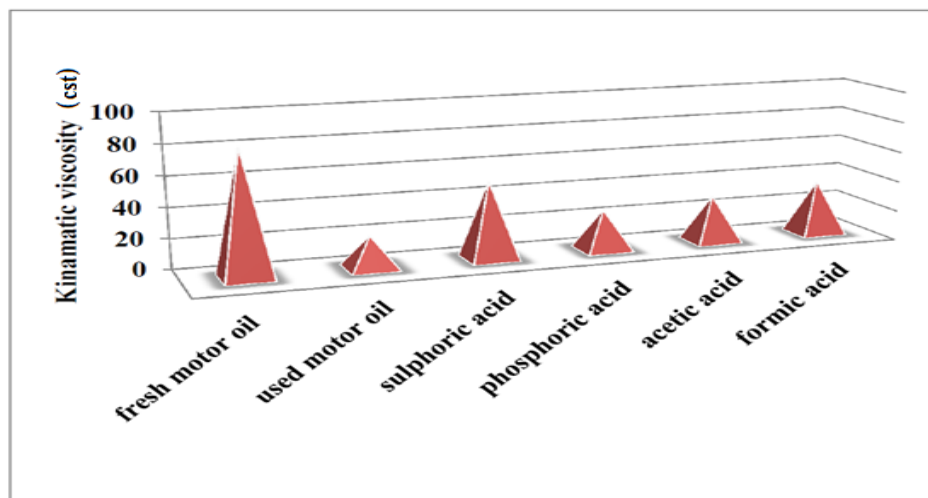


Fig. 5. Kinematic viscosity of fresh motor oil, used motor oil and oil sampled treated with acids.

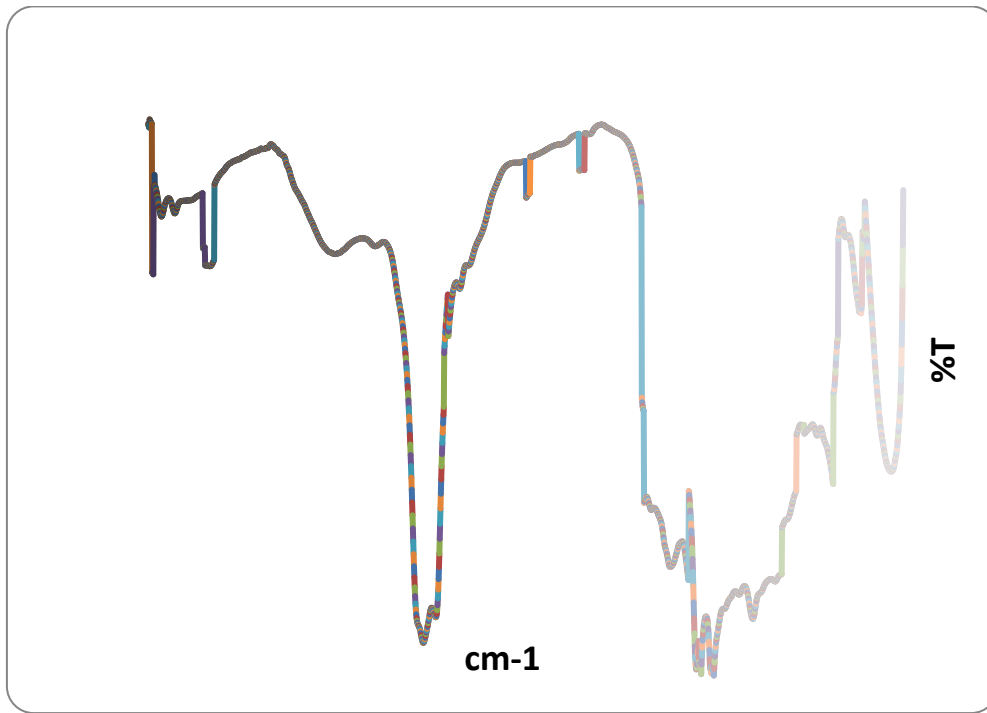


Fig. 6. (FTIR) spectrometer for used motor oil

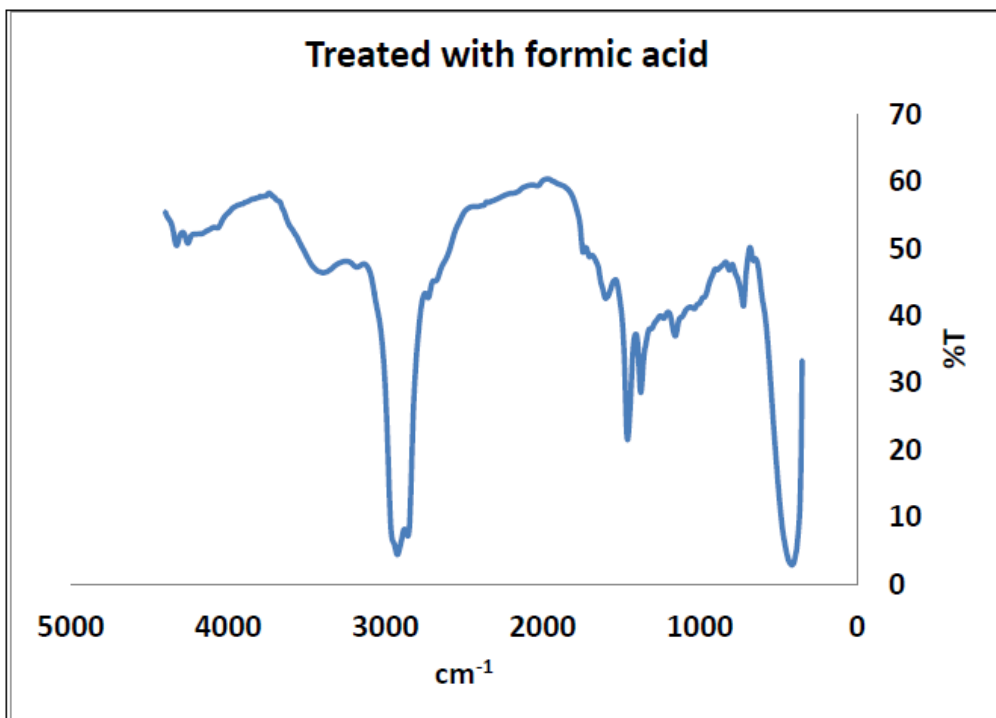


Fig. 7. (FTIR) spectrometer for used motor oil treated with formic acid/clay.

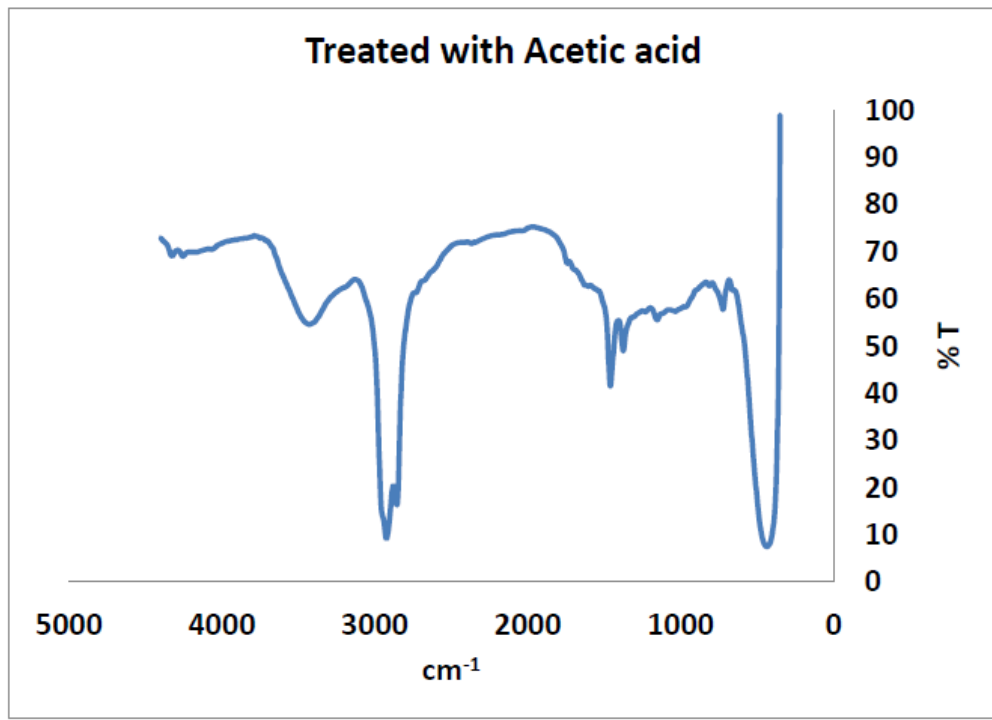


Fig. 8. (FTIR) spectrometer for used motor oil treated with acetic acid/clay

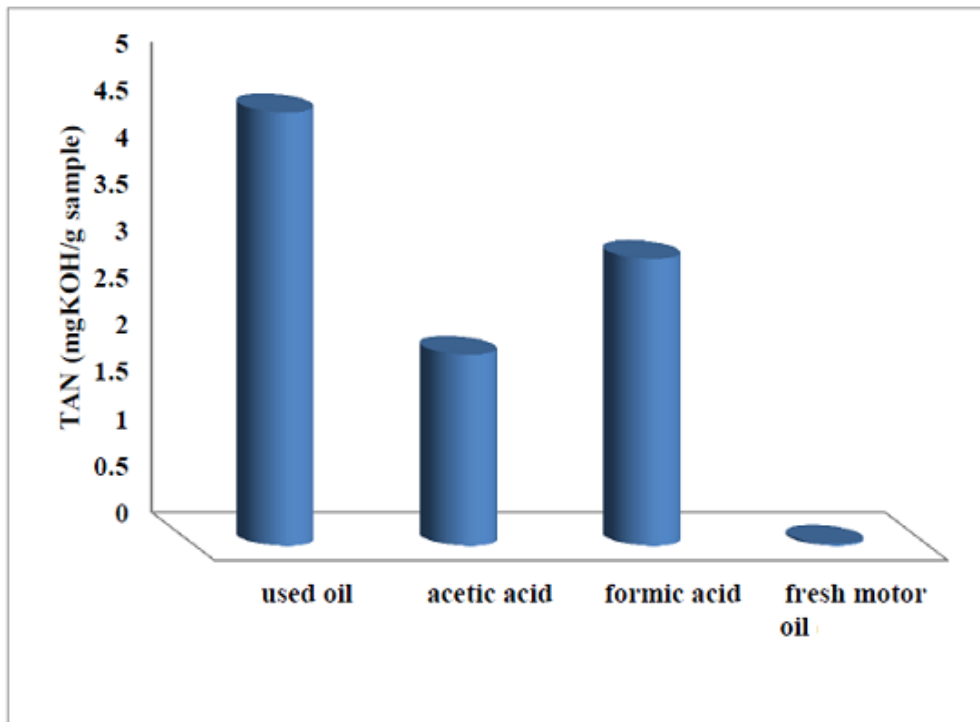


Fig. 9. TAN for used oil, fresh oil and treated oil sample



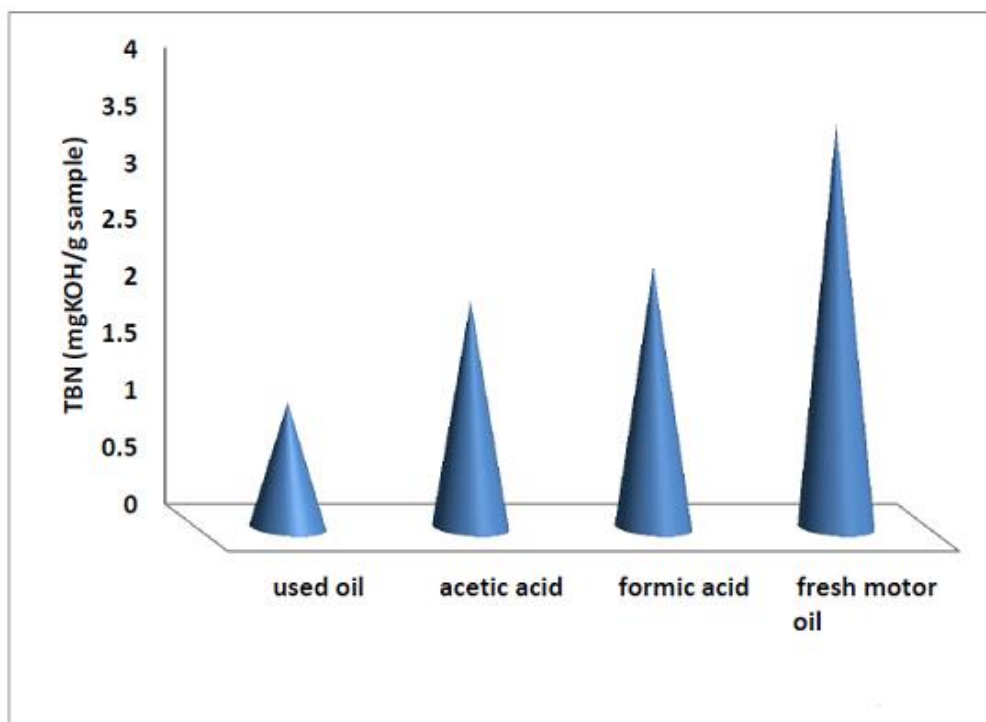


Fig. 10. TBN for used, fresh and treated motor oil

### 3.4. Fourier transform infrared (FTIR) spectrometers:

The Fourier transform infrared (FTIR) spectrometers are considered valuable multifunctional analytical tools for oil condition monitoring because of their ability to provide a composite molecular fingerprint of oil. Each organic constituent in the oil has its own fingerprint, and many key constituents and contaminants (antioxidants, water, soot, etc.) give rise to unique spectral features that can be measured and tracked. This is the basis for the FTIR oil analysis protocols established by the Joint Oil Analysis Program (JOAP), which allow the tracking of a wide range of parameters indicative of oil condition and contamination, ranging from soot to nitration. The objective of condition monitoring is to replace a lubricant only when its functionality is compromised, a practice that is cost-effective for fleets, the military and critical systems. Although the FTIR oil analysis protocols that have been developed provide valuable information for condition monitoring purposes, the results are expressed in qualitative and rather arbitrary terms related to absorbance values of the constituents. The ability to express these results in more quantitative terms is compromised by the impracticality of having to rely on the availability of reference oil relative to which changes can be compared [26]. IR spectra for used engine oil represented in Table 1 and Fig. 6 showed additional peaks at 1033.48, 1158.63 and 2335.07  $\text{cm}^{-1}$  which represent the primary oxidized products at high temperatures. The broad feature centered around 1743.74  $\text{cm}^{-1}$  is due to the presence of carbonyl-containing degradation products of oil. These have been identified as lactones, esters, aldehydes, ketones, carboxylic acids and salts, as well as others. The spectral features around 800  $\text{cm}^{-1}$  (813.46 and 884.53) are examined the aromatic content to indicate if fuel is present. IR spectra indicated that fuel residue is present in the used oil. Nitration products which responsible for the dark color is presented in

1603.88  $\text{cm}^{-1}$ . Water is detected as a broad feature, centered on 3379.41  $\text{cm}^{-1}$  that is caused by the hydroxyl (-OH) group. Soot is detected at 2032.02  $\text{cm}^{-1}$ . Table 2 and Fig. 7 show that the oil produced by the formic acid-clay method still show some oxidized components. There are alkan components at 1460.03  $\text{cm}^{-1}$ , carboxylic acids at 1156.83  $\text{cm}^{-1}$ , aldehyde at 2728.56  $\text{cm}^{-1}$  and carboxylic acid at 2335.07  $\text{cm}^{-1}$ . And also there is no fuel residue presented in the spectra. The Nitration products which is responsible for the dark color is still presented in 1599.43  $\text{cm}^{-1}$ . Table 3 and Fig. 8 show that the oil produced by the acetic acid-clay method has no aldehyde and no carboxylic acid at 2359.56  $\text{cm}^{-1}$ . And also there is no fuel residue presented in the spectra. This method gives the best results because it shows no aldehyde and no carboxylic acid at 2359.56  $\text{cm}^{-1}$ . There are no Nitration products around spectra 1600.00  $\text{cm}^{-1}$ .

### 3.5. Total Acid Number (TAN) and Total Base Number (TBN)

Acid Number (AN) and Base Number (BN) represent two of the most fundamental quantitative measures common to the assessment of lubricant quality for both new and used oils. In simple terms, AN represents acidic constituents in an oil (additives or acids formed by oxidation/fuel combustion) and BN represents basic constituents usually added to neutralize acids accumulating in an oil. These two measurements are commonly used to characterize new and used oils in terms of formulation, oxidative status or reserve alkalinity. The ASTM methods for AN and BN determination are tedious and problematic titrimetric methods, which can be subdivided into colorimetric and potentiometric methods. The colorimetric method is simple but is limited to colorless oils, which prevents most formulated and used oils from being analyzed. The potentiometric methods, of which there are several, are more

versatile overall, as they can handle most oils and can be automated using an auto titration. The various ASTM potentiometric methods differ in their procedural details, and the results can be quite different. One of the main problems with all AN and BN analyses is the variety of endpoints, their interpretation and terminology (i.e., acid number, base number, total acid number, total base number, strong acid number, strong base number, and neutralization number) all expressed as mg KOH/g oil [27]. It has taken several years of research to develop quantitative FTIR based AN and BN methods, but practical and readily implemented methods are now available [28].

### 3.5.1. Determination of TAN in Lubricants

In terms of tracking lubricant degradation, the main components are carboxylic acids formed as a result of oxidation processes, and these are readily observed and measured by FT-IR spectroscopy, the TAN can be calculated as follows [29].

$$\text{TAN} = 1.32 + 5.02 A \quad (3)$$

$$R = 0.999, \text{SD} = 0.068$$

Where: TAN = Total Acid Number mg KOH/g

A = Absorbance at 1565  $\text{cm}^{-1}$

R = Correlation coefficient

SD = Standard deviation.

Fig. (9) shows the TAN for used motor oil, treated with acetic acid-clay and treated with formic acid-clay.

As shown in Fig. 9, the TAN of the used engine oil is measured to be 4.62 mg (KOH)/g (sample) which is much higher than the TAN of the fresh oil [0.02 mg (KOH)/g (sample)]. This is due to the presence of organic, inorganic, heavy metal salts, ammonia slots, resin, water and corrosive materials which result from the oxidation process that occurred at elevated temperatures in the engine [30].

The recycled oil by formic and acetic acid is high in TAN around 3 and 2 mg (KOH)/g (sample) respectively.

### 3.5.2. Determination of TBN in Lubricants

Experiments showed that condition monitoring of performance -enhancing additives in lubricating oil is very important for predictive maintenance and extending equipment life. TBN represents the overall reserve alkalinity contributed to lubricants by additive packages. TBN is determined by spectroscopically measuring the COO-functional group of the salt produced when trifluoroacetic acid (TFA) reacts with basic constituents present in the lubricant. The TBN can be calculated as follows [29]

$$\text{TBN} = -4.35 + 21.44 A \quad (4)$$

$$R = 0.9998, \text{SD} = 0.160$$

Where: TBN = Total Base Number

A = (Absorbance at 1672  $\text{cm}^{-1}$ ) – (Absorbance at 2110  $\text{cm}^{-1}$ )

Fig. 10 shows that used oil's TBN is low and this is due to the depletion of the additive package as a result of the high temperature and the acid effect of water and oxidant product contamination. While the treatments of used motor oil with acetic acid-clay and formic acid-clay has higher TBN values than the TBN value of used engine oil [1.1 mg KOH/g (sample)]. This is due to the presence of a high alkaline additive package that was not depleted and has the ability to

neutralize large quantity of acids and certain salts of heavy metals [31].

## 4. Conclusions

This research shows an effort to treat used oil by acid-clay treatment method using four acids (Phosphoric acid- Sulphuric acid- Acetic acid and formic acid) with clay earth. The results showed that the acetic acid-clay treatment improved the specific gravity of the oil and made it comparable with fresh motor oil while the treatment with phosphoric acid-clay has no improvement action on the used oil. Regarding the flash point it was found that the formic acid-clay treatment improved the flash point of the oil and made it comparable with fresh motor oil while the treatment with phosphoric acid-clay has less improvement action on the flash point. The results also showed that the sulphuric acid, acetic acid and formic acid –clay treatment improved the kinematic viscosity of the oil while the treatment with phosphoric acid-clay has no improvement action on the used oil kinematic viscosity. The FTIR analysis showed that the oil produced by the acetic acid-clay method has no aldehyde, no carboxylic acid and no fuel residue. The oil produced by the formic acid-clay method still showed some oxidized components. There are alkanes with no fuel residue presented in the spectra. It means that treating the oil with acetic acid-clay showed better results than that by formic acid-clay. The FTIR analysis was used to determine the TAN and TBN and the results showed that the treatments of used motor oil with acetic acid-clay and formic acid-clay has higher TBN values than the TBN value of used engine oil. However, while many variables have been studied in this research, there are many others that need investigation such as temperature, pressure, settling time, mixing, and centrifugation speed, type of adsorbent and mixture of these acids with different ratios.

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