



## **SPENT OIL MANAGEMENT AND ITS RECYCLING BY HYDROTREATING CATALYST FOR OIL WELL DRILLING FLUID**

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### **ABSTRACT**

Used oils are lubricating oils, which have become unsuitable for their proposed use. They may be recycled through the use of rerefining process to achieve useful products. Environmentally-conscious design of processes and products is increasingly viewed as an integral strategy in the sustainable development of new refining and chemical processes. This study compares the lubricating oil before and after treated oil with rerefining processes. A laboratory setup of dehydration, extraction and hydrotreating processes were established to rerefine used oil collected from different sources. A comparison of its product characteristics with untreated oil characteristics is introduced, the results showed that pour point increased from -21°C for used oil to -6°C for treated oil. Sulfur content was found about 3124.9 ppm for used oil and 90.4 for treated oil. Also, viscosity (at 70°C, cst.) and density (at 15°C, g/cm<sup>3</sup>) decreased from 30.73 and 0.897 for used oil to 9.44 and 0.855 for treated oil. This study concentrates on the investigation of energy and environmental benefits for used oil pertaining to its reuse through: (i) recovering the heating value of used oils in a combustion process and (ii) rerefining of used oil to produce fresh lube oil products. Also, the recycling used oil has been evaluated as a continuous phase in synthetic based mud instead of synthetic hydrocarbons for oil well drilling fluids. The rheological, filtration properties and electrical stability of the synthetic mud, which formulated with the recycle used oil, were studied and compared to the synthetic mud formulated with pure synthetic hydrocarbons (MR). The thermal stability of the mud were also tested and compared to the MR.

**Key words:** Spent oil management, Recycling, Hydrotreating catalyst, Oil, Drilling fluid.

### **INTRODUCTION**

Rerefining of used lube oil is an economically attractive recycling method in terms of resources conservation and environment protection. It allows processing of hazardous material in a safe and effective way to recover a high quality base oil product. This results in

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a strong economic incentive for rerefining considering lube oil price. Rerefining can produce base oils or VGO distillate that is a suitable feedstock to FCC or HDC refinery units.

Used lube oil is generally a mixture of different types and grades of used lube oils, coming from motor crankcases and industry users. Used lube oils made up of a multitude of small individual batches collected at garages, maintenance shops, transportation companies, industries and depends on local situation, seasonal consumption, handling practice at the collection source and collecting system organization.

Used oil recycling is a procedure that involves reprocessing used motor oil so that it can be used again. Motor oil can be recycled indefinitely because the lubricant does not wear out. Recycled oil is cleaned of contaminants such as dirt, water, used additives, and fuel. Used oil may also contain toxic substances such as lead, benzene, zinc, and cadmium. Recycling saves oil and helps the environment<sup>1-3</sup>.

Therefore, the oil quality gradually decreases to a level that the used oil should be replaced by a new one<sup>4</sup>. Disposing the used oil pollutes environment to a great extent<sup>5</sup>. Due to the increasing necessity of environmental protection and the more and more strict environmental legislation, the disposal and recycling of waste oils become very important<sup>6</sup>. As a result, the used oil needs proper management to make it a valuable product<sup>6</sup>. The recycling of used oil can be accomplished with different methods, which are the reprocessing, reclamation and rerefining<sup>2,7,8</sup>. The most important methods for the recycling of used lubricating oils are rerefining and reclamation<sup>7</sup>. The processes of rerefining of used oil depend greatly on the nature of the oil base stock and on the nature and amount of contaminants in the lubricant resulting from operations. Used oil has been rerefined using many techniques such as chemical (acid/clay) treatment<sup>2,9</sup>, physical treatment by distillation, thin film evaporation and solvent extraction<sup>10</sup>.

Since the chemical treatment creates environmental problems, therefore, solvent extraction was proposed as an alternate method<sup>11</sup>. The solvent extraction treatment has received considerable attention in recent years, because it overcomes the problems associated with acid sludge produced from chemical treatment<sup>1</sup>. Solvent could be recovered by distillation<sup>12</sup>. Recently, used oil has been rerefined by vacuum distillation with chemical treatment or clay treatment, also, vacuum distillation with chemical treatment and hydrofinishing<sup>2,13</sup>.

In the last decades, the hydrogenation and hydrocracking technologies modern catalysts have been developed, which are able to hydrogenate feeds with high concentration of contaminations (e.g. high metal content refining residues, used engine oils). These

catalysts are applied in high amount in so called guard reactor before hydrogenation and/or hydrocracking of residues due to their large pore size<sup>14</sup>. The Co-Mo/Al<sub>2</sub>O<sub>3</sub> catalyst can produced the product oils, which had the best ASTM color, acidity, and viscosity index. Ni-Mo/Al<sub>2</sub>O<sub>3</sub> catalyst produced the product oils, which had the lowest sulfur content while NiW/Al<sub>2</sub>O<sub>3</sub> catalyst produced the product oils that had the best viscosity and flash point<sup>15</sup>.

Drilling fluids or drilling muds are essential and a key component of the rotary drilling process used to drill for oil and gas on land and in offshore environment. The most important functions of drilling fluids are to transport cutting to the surface, to balance subsurface, cool, lubricate and support part of the weight drill bit and drill pipe<sup>16-18</sup>. Although the high toxicity of oil-based mud, it is used when drilling deep wells due to its high performance and high thermal stability than water based-mud, also using of both water-based mud and oil-based mud causes toxicological and ecological effects in the marine environments when drilling in offshore, so the drilling industry has developed several types of synthetic based-muds (SBMs) to combine the technical advantage of oil-based mud (OBMs) with the lower toxicity, environmental impact and quality of water-based mud (WBMs)<sup>19-21</sup>. Synthetic based-mud has been used to simultaneously improve ecological effect in the marine environment<sup>22</sup>. The synthetic base fluid are a drilling fluid where the base fluid consists of non-water soluble organic compounds and where neither the base fluid nor the additives are of petroleum origin so they are environmentally friends and have a high biodegradability and low toxicity<sup>23-25</sup>.

Salt brine usually is dispersed in the synthetic phase to form an emulsion, the other ingredients of an SBF include emulsifiers barite, clays, lignite and lime. Synthetic base fluids classified into four general categories, synthetic (hydrocarbon, ethers, esters, acetyls)<sup>26-28</sup>.

The novel environmentally-conscious design of an invert mud system, which has excellent toxicity, health and safety aspects and is biodegradable. Based on a synthetic material as the continuous phase, the mud system gives the same high performance as that of material or oil based systems.

Thus a non-toxic drilling fluid (drilling mud), for use in rotary-type well drilling operations, comprises a non-toxic based-oil consisting essentially of saturated hydrocarbons, which in the base. The branched chain paraffins are used as the base-oil for formulating the synthetic-base drilling fluid.

A principal reason for selecting saturated hydrocarbons as base-oils for the drilling fluid is that these paraffin not only have the needed physical characteristics (including low viscosity, low melting points, high flash points, low pour points but are widely recognized as

being nontoxic). The environment regulations can be expected to increasingly limit the use of natural petroleum base oils, such as diesel oil derivatives, most of which contains substantial concentrations of aromatics at least some n-olefins, and which are, therefore considered to be at least somewhat toxic.

The major aim of this study was rerefined Egyptian local crankcase used lubricating oil to generate base oil by two techniques. The first was by solvent extraction followed by hydrotreating, which studies the effects of temperature on the yield of the refined oil. The hydrotreating was carried out by using Co/Mo catalyst based on alumina support. Tests carried out on the used/recycled oil include: flash point, pour point, and other characteristics.

In this study, the use of recycling saturated hydrocarbons instead of pure hydrocarbons for formulating synthetic-based mud for lower cost and economically practical. One aspect of the SBM system, which is its ability to incorporate recent improvements in invert emulsion technology. The system is compatible with low-shear-ratio rheological modified. These elevated low shear viscosities have been shown to be important for whole cleaning in large diameters, and high angle holes.

## EXPERIMENTAL

### Properties of engine oil

The rerefining of Egyptian local used oil was investigated. This used lubricating oil was collected from different places and governorates in Egypt. The specification of used oil was evaluated according to ASTM standard tests and listed in Table 1.

**(i) Viscosity:** Viscosity analysis shows the presence of different type of contamination in used engine oil. The products of oxidation and polymerization (these products can be both dissolved and suspended) in the oil cause the increase the viscosity of used engine oil, while a decrease in the viscosity of engine oil shows the fuel contamination<sup>29</sup>.

**(ii) Flash point:** Flash point is the lowest temperature at which the vapors in air will burn momentarily, if ignited by flame or spark. A decrease in flash point indicates contamination by dilution of lubricating oils with unburned fuel. Increasing of flash point indicates evaporation of the light components from the lubricating oil<sup>30</sup>.

**(iii) Pour point:** Pour point may be defined as the lowest temperature, at which the oil will stop to flow. Lubricating oil having low pour point shows its good quality. It is very important to users of lubricants in low temperature environment<sup>31</sup>.

**Table 1: The comparison between specifications of rerefined and used lube oils**

Parameter	ASTM	Used oil	Treated oil
Pour point (°C)	D97	-21	-6
Flash point (°C)	D92	87	160
Color	D1500	8	2.5
Viscosity at 40°C, cst.	D445	79.82	21.62
Viscosity at 70°C, cst.	D445	30.73	9.44
Sulfur content, ppm	D4294	3124.9	90.4
Density, 15°C (g/cm <sup>3</sup> )	D1298	0.897	0.855

### The major steps in the rerefining processes

#### Dehydration

The first step of the rerefining involved dehydration of the used oil to remove water. The dehydrated oil (feed oil) was collected and sent to the next steps for further treatment.

#### Solvent extraction

The next steps involved the solvent extraction followed with clay treatment. This study used methyl ethyl ketone (MEK) as solvent for treatment instead of butyl alcohol and isopropyl alcohol. This choice was due to the difficulties in recovery of this solvent again that results in great lose in solvent, which leads to high cost, in addition, the ease of recovery, low boiling point and low cost of the MEK. The solvent was of analytical grade. The dehydrated oil was subjected to solvent extraction at 25°C and atmospheric pressure. The mechanical starrier was used for extraction and temperature was controlled with the help of thermostatic bath. Dehydrated oil and solvent were stirred for one hour to ensure adequate mixing, and then subjected to sufficient settling. The solvent was recovered by distillation, as the MEK boiling point is 80°C, to reuse it again. To determine optimum conditions solvent to oil ratio and settling time were varied.

#### Clay treatment

At the clay treatment step, 100 mL of extracted oil mixed with a measured amount of the clay (20% w/w). The mixture was performed at the room temperature on a magnetic stirrer with constant stirring for one hour. The treated oil was separated by settling and then filtrated to remove clay and other solids.

## Hydrotreating

Hydrotreating of extracted oil took place by feeding the sample into a trickle bed reactor, which serves for laboratory study of heterogeneous catalytic processes under pressure occurring in liquid or gaseous phase. The reactor contains catalyst bed of Co/Mo that supported by alumina base. Commercial grade Co-Mo catalyst was provided from Albemarle Catalysts Company (KF 841). The process take place at the following operating conditions, temperature: 300, 325, 350 and 375°C, pressure: 50 Kg/cm<sup>2</sup>, LHSV: 0.7 h<sup>-1</sup> and hydrogen to oil ratio: 300 L/L.

The unit was first flushed with nitrogen to remove air, after loading the catalyst and diluent, the catalytic bed was in situ sulfided with a sulfiding feed containing a 2 wt.% DMDS in heavy gas oil under the following conditions: pressure = 50 bar; a hydrogento oil ratio = 400 L L<sup>-1</sup>; reaction temperature = 350°C; and liquid hourly space velocity (LHSV) = 1.5 h<sup>-1</sup>. Presulfiding was continued for 12 h under the stated conditions to ensure complete catalyst presulfiding.

## Mud formulations and performance

Like invert emulsion systems, the formulation of a SBM drilling fluids uses emulsifiers and wetting agent along with products for viscosity and fluid loss control. As expected, adjustments in surfactant package from these used for diesel and mineral oils are required to attain and maintain stable mud properties, when using a base fluid such as synthetic hydrocarbon. Some conventional products, such as organophilic clay and amine treated lignites (Table 2).

**Table 2: A basic formulation for an 70/30 s/w ratio SBM**

Concentrations	Components
0.58	Saturated hydrocarbonbase fluid (bbl)
5	Emulsifier (ppb)
2	Wetting agent (ppb)
3	Organophilic clay (ppb)
6	Lime (ppb)
0.276	CaCl <sub>2</sub> brine (25%) (bbl)
166	Barite (ppl)

## Preparation of synthetic based mud

According to API; OCMA specification<sup>32,33</sup>, in suitable containers all chemical additives were added during stirring of the mixer in the following order :

- (i) Synthetic hydrocarbons (pure and/or recycle) was placed in a suitable containers with stirring in a mixer.
- (ii) Emulsifier was added.
- (iii) Geltan (organophilic clay) was added gradually as viscofiers and gelling agent.
- (iv) Duratant (organophilic surfactant) was added as filter loss reducer wetting agent for water- based solvent.
- (v) Sodalime was added gradually.
- (vi) EZ-mud blended supplementary emulsifier and wetting agent for oil wetting and emulsification.

So we have the mud batches:

**MR:** Synthetic mud (SM), which formulated with pure hydrocarbons,

**MC:** Synthetic mud(SM), which formulated with recycle hydrocarbons,

Rheological properties, high pressure, and high temperature filter loss test, electrical stability, were carried out to each of the two formulated mud batches and the effect of temperature on the rheological properties and thermal stability test were also studied comparing the result with the API specifications

## Rheological properties

Apparent viscosity (AV), plastic viscosity (PV), and yield point (YP) were determined by making a relation between shear rate and shear stress, where the shear rate was taken from the dial reading, which is in degree of a circle.

$$\text{Shear rate, sec}^{-1} = \text{rpm} \times 1.7034$$

$$\text{Apparent viscosity (AV), cp} = \text{Reading at 600 rpm}/2$$

$$\text{Plastic viscosity (PV) cp} = \text{Reading at 600 rpm} - \text{reading at 300 rpm}$$

$$\text{Yield point (YP) lb}/100 \text{ ft}^2 = \text{Reading at 300 rpm} - \text{plastic viscosity}$$

Viscosity of the mud is a function of temperature more than pressure. Commonly, it is necessary to measure viscosity at elevated bottom hole temperature. This is done by using the viscometer cup heater, Chandler Engineering Laboratory Model API viscometer (Chan 35 Model 3500), which is a thermostatic-controlled unit for heating the mud sample directly on a viscometer.

### **Determination of gel strength and thixotropy of a mud**

The gel strength of the mud is a measure of a minimum shearing stress necessary to produce slip-wise movement of fluid. Two readings are generally taken, immediately after agitation of the mud in the cup (10 sec) and after the mud in the cup has been rested for 10 min.

### **High pressure–high temperature filter loss**

The test was carried out by using standard HP-HT filter loss Model 1071C. The experiment was run at (300°F and 500 psi) and the volume of filtrate reading recorded from the graduated cylinder at the end of 2, 5, 10, 15, and 30 min.

### **Electric stability**

A fan apparatus Model 23D was used to indicate the electrical stability of the mud.

### **Thermal stability test**

The thermal stability test of both (MC) and (MR) synthetic-based mud and the rate of deterioration of their filtration and rheological properties under the condition of high temperature, and continuous circulation, the following test was carried out. Samples prepared according to the above procedure and placed in a rolling oven operating at 350°F with continuous circulation for 16 h; samples were then removed and cooled for 20 min in a cold water bath. Samples were then banded in a high speed blender for 5 min and the AV, PV, YP, gel strength for 10 sec, gel strength for 10 min, and fluid loss (FL) were determined.

## **RESULTS AND DISCUSSION**

Used oil is a complex mixture of recoverable base oil, additives, water, light hydrocarbons, metals and carbonaceous particles. A good extraction solvent should dissolve base oil and precipitate other substances to form sludge. This process combines extraction with solvent and hydrotreating by removing the foreign substances by solvent and then improving oil quality by hydrotreating. First, the water and light ends are removed and the used oil is separated through MEK extraction. Then the extracted oil is subjected to hydrotreating for purification purposes.



### **Dehydration step**

Water found in lubricating oil in service depends on where the automobile is being used. In normal operating conditions, traces of water in the lubricant are unavoidable, arising from such sources as leaking oil coolers, engine cooling system leaks and in all types of machinery, from atmospheric condensation. The presence of excessive water contamination will affect the viscosity of the oil and this may give rise to emulsion formation and can also lead to gear tooth and bearing problems. Therefore, the first step of the rerefining of the used oil was dehydration.

### **Extraction step**

The extraction of dehydrated oil by MEK solvent to oil ratio was previously studied<sup>34</sup>. Solvent to oil ratio lower than 5:1 leads to reduce the solvency power and also not to improve the properties of the recycled oil. This means that larger solvent to oil ratio will lead to dissolution of some contaminants forming material, which was considered to be undesirable. As a result of the above mentioned facts, the ratio of 5:1 was considered to be the better solvent to oil ratio used for the treatment of used oil<sup>2</sup>.

### **Hydrotreating step**

By the knowledge, hydrotreating process improves lubricating oils color and color stability and also reduces the level of sulfur and metal compounds. Reduction in metallic content is due to effect of de-metallization bed (alumina). Also, a lot of the aromatics and other unsaturated (which were not eliminated by previous steps) are saturated to an acceptable low level. The hydrotreated oil clearly appeared under a suitable treatment for the removal of color. Color was improved from 8 to approximately 2.5 (Table 1).

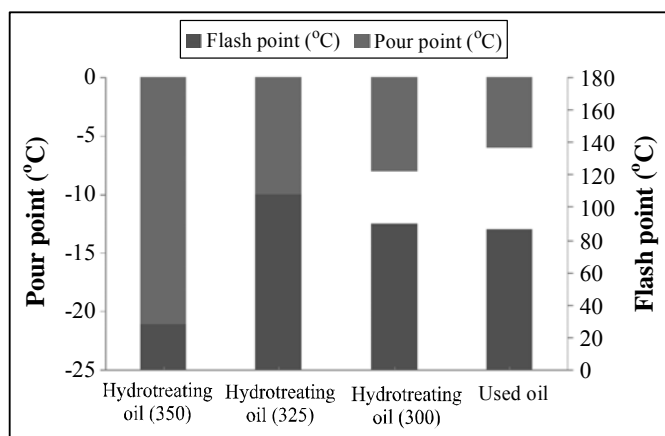
The results of dehydration step, clay treatment, extraction step and hydrotreating step by Co-Mo/Al<sub>2</sub>O<sub>3</sub> catalyst with their respective quality test and discussion of these results are given in Table 1. As shown in Figs. 1-3, the quality of the treated used oil under the tested operating conditions, which revealed that the quality was improved by increasing the reaction temperature from 320-410°C. The properties of treated used oil were improved such as flash point, pour point, viscosity, density and sulfur content<sup>35</sup>.

### **The specifications of rerefined and used lube oils**

#### **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. The flash point of engine oil is an indication of the oil's contamination. 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 is also an aid in establishing the identity of a particular petroleum product. The flash point increases with increasing molecular mass of the oil. Oxidation would result in formation of volatile components which leads to decrease the flash point. Table 1 shows flash point values of different used oil hydrotreated at different reaction temperature. The flash point of the measured used engine oil is 87°C. This decrease in flash point is a result of contamination with fuel and oxidation products, which lead to formation of volatile components and then decrease the flash point<sup>36</sup>. On one hand, the table clearly shows that the flash point of the base oil is highly affected by increase in temperature (Fig. 1)<sup>36</sup>.



**Fig. 1: Effect of refining on pour point and flash point for hydrotreated oil**

### Pour point

The pour point of an engine oil is the lowest temperature at which the oil will remain in a flowing state. Most engine base oils contain waxes and paraffin's that solidify at cold temperatures. Engine oils with high wax and paraffin content will have a higher pour point. Pour point is highly affected by an oil's viscosity, and engine oils with high viscosity are characterized by having high pour points. The pour point of an engine oil is an important variable, especially when starting the engine in cold weather. The oil must have the ability to flow into the oil pump and then pumped to the various part of the engine, even at low temperatures<sup>37</sup>.

Pour point of lube oil may be decreased and increased, depending on the nature of lube oil method of refining. The result chart in Fig. 1 shows that the pour point of treated oil

is  $-6^{\circ}\text{C}$  and used engine oil is  $-21^{\circ}\text{C}$ . Pour point values of refined engine oils by hydrotreating Co-Mo/ $\text{Al}_2\text{O}_3$  catalyst are  $-10^{\circ}\text{C}$ ,  $-8^{\circ}\text{C}$ , and  $-6^{\circ}\text{C}$ , when increasing reaction temperature (Fig. 1)<sup>33</sup>.

### **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 decrease in the viscosity of engine oils indicate fuel contamination. Oxidation of base oils during use in an engine environment produces corrosive oxidized products, deposits, and varnishes, which lead to an increase in the viscosity. Kinematic viscosity for used and treated oil at two temperatures are shown in Table 1, This means that the above methods are effective in removing the oxidized products, deposits, and varnishes from the used oil. This may also mean removing the additives, which have been added to enhance the viscosity, also as shown in Fig. 2 increase in reaction temperature lead to improved viscosity. It could be lower or higher than treated engine oil depending on the nature and type of contaminations<sup>38</sup>.

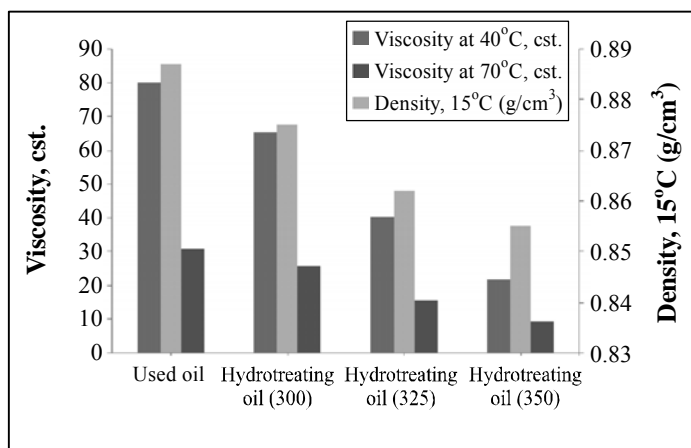
### **Density (Specific gravity)**

The specific gravity of the used lube oil is higher than the refined one from the above results. The results for the treated and used lubricating oils are 0.855 and 0.887, respectively. The specific gravity for the used oil was higher than that for the refined oil. If the used lube oil was contaminated due to fuel dilution and/or water originating from fuel combustion in the engine and accidental contamination by rain, its specific gravity will be lower than that of its fresh lube oil or the rerefined one.

Specific gravity is the ratio of the mass of volume of substance to the mass of the same volume of water and depends on two temperatures, at which the mass of the sample and the water are measured. 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. An approximate correlation exists between the specific gravity, sulfur content, carbon residues, viscosity and nitrogen content<sup>39</sup>. Used engine oils specific gravity increases with the presence of increasing amounts of solids in the used engine oil. One percent of weight of solids in the sample can raise the specific gravity by 0.007<sup>39</sup>. Used engine oil is contaminated with oxidized and condensed products rich in carbon. As shown

in Table 1, treated oil has a specific gravity of 0.855, which is low compared to the used engine oil specific gravity of 0.887. The high value of specific gravity of used engine oil is due to the presence of oxidation products, metals and contamination. Both acids have almost the same effect on the specific gravity when added to the base oil<sup>36</sup>.

The effects of temperature on the specific gravity reduction are shown in Fig. 2; then one can observe that the specific gravity was reduced with the increment of the temperature due to catalytic properties in a wide temperature range. Temperature 300-350°C was the best range to handle the used oil with the Co-Mo/Al<sub>2</sub>O<sub>3</sub> catalyst.

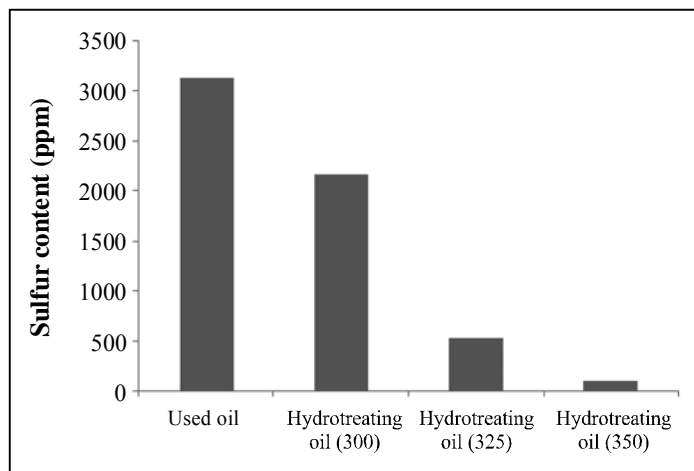


**Fig. 2: Effect of refining on kinematic viscosities and specific gravity for hydrotreated oil**

### Sulphur content

The Table 1 shows the values of sulfur content for the treated lubricating oil and used oil. The sulphur content of used lube oil are high, this is due to the presence of wear caused between moving parts. Sulfur reacts with the metal to form compounds of low melting point that are readily sheared without catastrophic wear. Corrosion in engines is caused by mineral acids formed by the oxidation of sulphur compounds in fuel in internal combustion engines with treated oils; those hydrocarbons that were inherently unstable will have been oxidized during use<sup>40</sup>.

Fig. 3 shows the effect of temperatures on the percent sulfur removal for used lube oil. The percent sulfur removal was found to increase with increase in temperature of the hydrotreating process.



**Fig. 3: Effect of refining on sulphur content for hydrotreated oil**

### **Evaluation of the recycled hydrocarbon (used oil) as a synthetic-based drilling fluids**

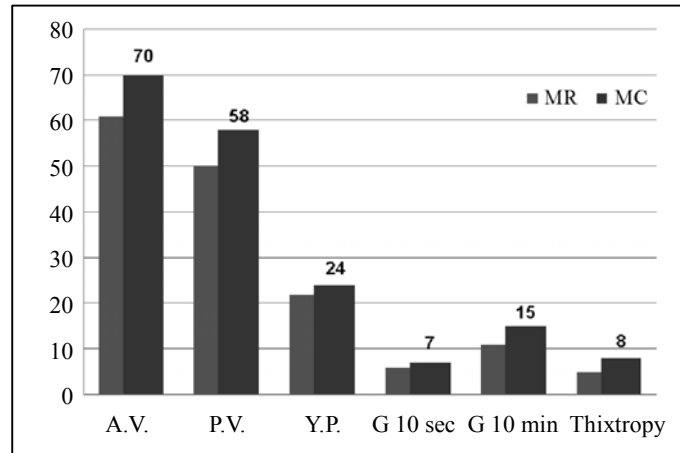
The recycled used oil was evaluated as a synthetic based mud for oil well drilling fluids instead of synthetic hydrocarbons.

The evaluation includes the study of the following parameters:

- (i) Rheological properties (apparent viscosity, plastic viscosity, yield point and gel strength),
- (ii) High pressure high temperature filtration,
- (iii) Electrical stability,
- (iv) Thermal stability and
- (v) Effect of temperature on the rheological properties

### **Rheological properties of the synthetic-based mud**

Synthetic-based mud was formulated from the recycled hydrocarbon (used oil) synthetic oil-water (S/W 70/30): Fig. 4 illustrates the measured values of rheological properties for both synthetic mud formulated from pure synthetic hydrocarbon (MR) and synthetic mud formulated from recycled hydrocarbon (MC) the mud formulation was 70/30 s/w ratio (Table 2).



**Fig. 4: Rheological properties of the synthetic mud (MC) compared to the (MR) mud**

### Viscosity

Apparent viscosity (AV), plastic viscosity (pv), and yield point (yp)): The measured values are illustrated in Fig. 5 and showed that the (AV), (pv) and (yp) for the mud formulation (M.C) were 70 (cp), 58 (cp) and 24 lb/100 ft<sup>2</sup>, which is greater than the viscosity and yield point for (MR) 61 (cp), 50 (cp) and 22 lb/100 ft<sup>2</sup> and compatible with API specifications.

### Gel strength

The gel strength and thixtrophy for both (MR) and (MC) were also illustrated in Fig. 6, the result of gel strength showed that G10 sec for (MC) was 7 lb/100 ft<sup>2</sup>, which is greater than the gel strength 10 sec for (MR) 6 lb/100 ft<sup>2</sup>.

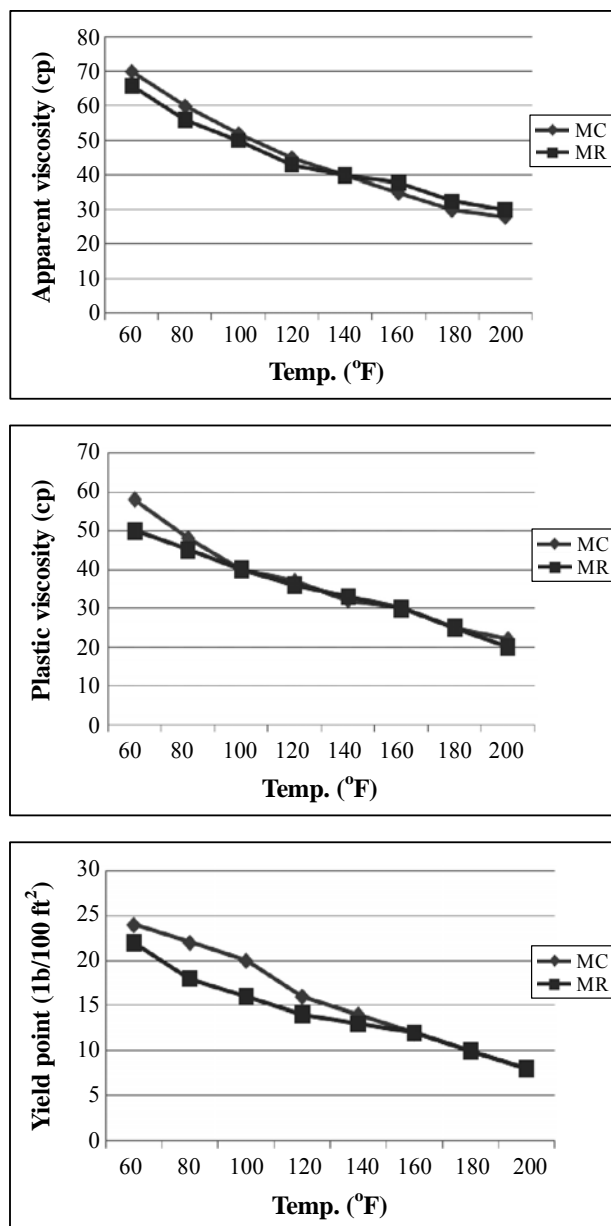
Gel 10 min for (MC) was 15 lb/100 ft<sup>2</sup> and for (MR) was 11 lb/100 ft<sup>2</sup>.

Thixtrophy for (MC) was 8 lb/100 ft<sup>2</sup>, which is greater than thixtrophy for (MR) 5 lb/100 ft<sup>2</sup>. The study of rheological properties indicated that the mud formulated from (MC) is more stable than the (MR) mud.

### Effect of temperature on rheological properties

Rheological properties varied with temperature for synthetic-based mud (MC) formulated with used oil comparing to the synthetic-based mud, formulated with pure synthetic hydrocarbon (MR) Fig. 6 shows the decreases of AV from 70 CP to 28 CP with increasing temperature from 60°F to 200°F, respectively for (MC) mud, and for (MR) mud

decreases from 66 CP to 30 CP. Pv decreases from 58CP to 22 CP for (MC) while it decreases from 50 CP to 20 CP for (MR) and the yield point decreases from 24 lb/100 ft<sup>2</sup> to 8 lb/100 ft<sup>2</sup> for (MC) and decrease from 22 lb/100 ft<sup>2</sup> to 8 lb/100 ft<sup>2</sup> for (MR).



**Fig. 5: Rheological properties-temperature relationship of (MC), compared to (MR)**

### Effect of temperature on gel strength and thixotropy

Fig. 6 illustrates the gel strength and thixotropy of synthetic based mud formulated with recycled used oil (MC) and the gel strength for synthetic based mud formulated with pure synthetic hydrocarbon (MR) as the temperature was raised from 60° to 200°F. The results indicated that :

For (MC) mud: G10 sec decreased from 7 lb /100 ft<sup>2</sup> to 1 lb /100 ft<sup>2</sup>.

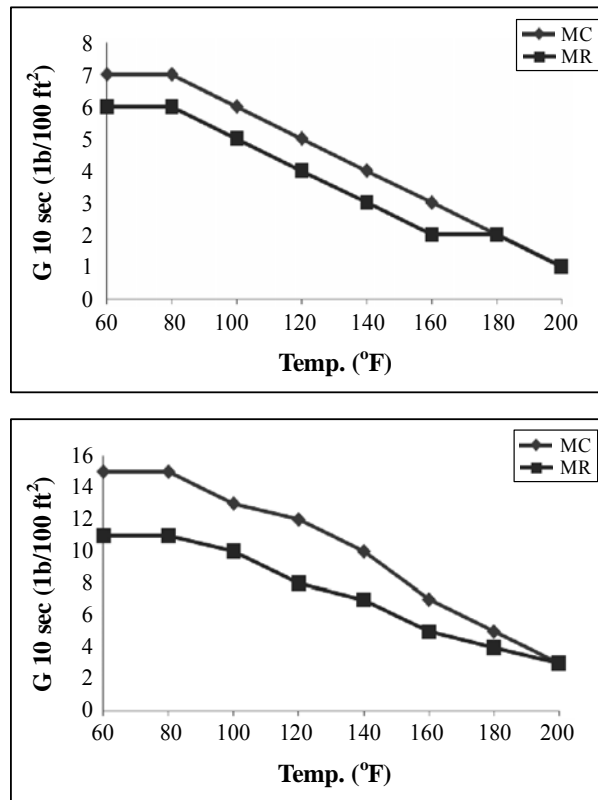
G10 min decreased from 15 lb /100 ft<sup>2</sup> to 3 lb /100 ft<sup>2</sup>

Thixotropy: Decreased from 8 lb /100 ft<sup>2</sup> to 2 lb /100 ft<sup>2</sup>.

For (MR) mud: G10 sec decreased from 6 lb /100 ft<sup>2</sup> to 1 lb /100 ft<sup>2</sup>.

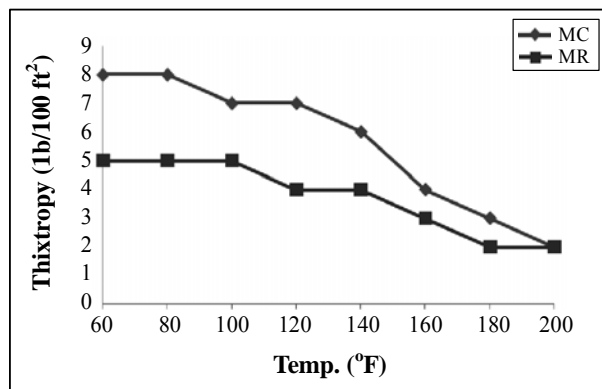
G10 min decreased from 11 lb /100 ft<sup>2</sup> to 3 lb /100 ft<sup>2</sup>

Thixotropy: Decreased from 5 lb /100 ft<sup>2</sup> to 2 lb /100 ft<sup>2</sup>.



Cont...

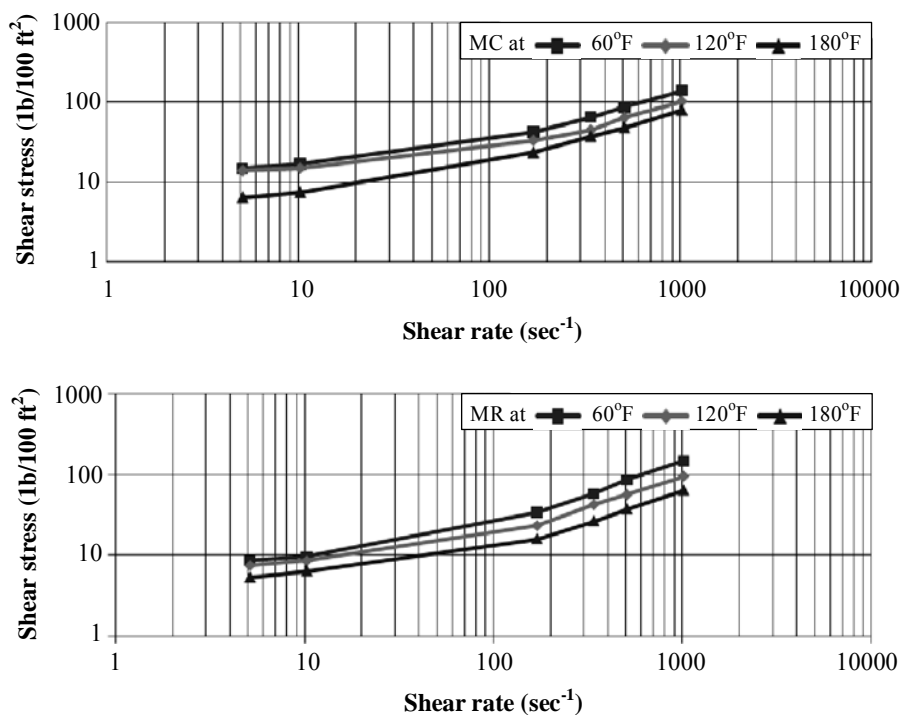




**Fig. 6: Gel strength and thixtropy-temperature relationship for (MC) compared to (MR) mud**

**Shear rate-shear stress relationsh**

Shear rate-shear stress relationship at different temperature 60°, 120°, 180°F were studied and illustrated in Fig. 7.



**Fig. 7: Shear rate-shear stress relationship of MC and MR at 60°, 120° and 180°F**

The value of shear stress decreased as shear rate decreased for synthetic based mud formulated with recycled used oil (MC)

At 60° F: The value of shear stress decreases from (149 to 5.3) as shear rate decreases from 1020 sec<sup>-1</sup> to 5.1 sec<sup>-1</sup>.

At 120° F: The value of shear stress decreases from (96 to 7.4) as shear rate decreases from 1020 sec<sup>-1</sup> to 5.1 sec<sup>-1</sup>.

At 180° F: The value of shear stress decreases from (64 to 5.3) as shear rate decreases from 1020 to 5.1. These results are similar to the result obtained in case of the synthetic based mud formulated with pure synthetic hydrocarbon (MR) at the same temperature.

### Effective viscosity

A log-log graph paper for plotting viscosity for the mud v/s the shear rate is represented graphically in Fig. 8. Vertical lines show the rpm equivalents of shear rates in sec., drilling fluids are usual pseudo plastic, i. e. shear thinning fluids.

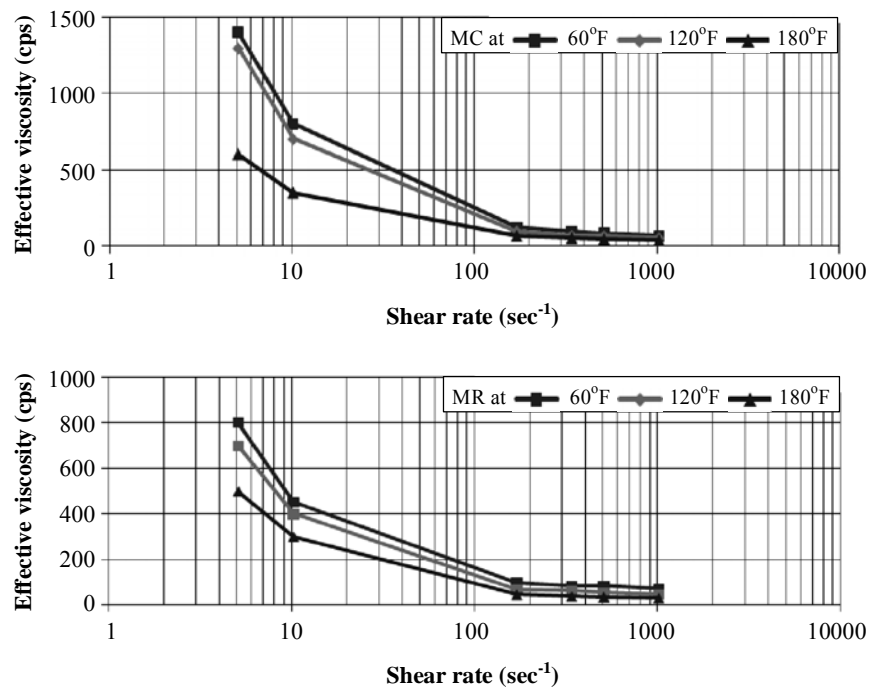


Fig. 8: Effective viscosity of MC and MR at 60°, 120° and 180°F

Tests results illustrated that effective viscosity of the mud MC decreases with increasing shear rate. This results is similar to MR mud formulated with pure synthetic hydrocarbon .

### Filtration properties

Table 3 reveals that the Hp-HT filter loss at 250°F, 300 psifor both synthetic-based mud (MC) formulated from recycled used oil and (MR) synthetic based mud formulated from pure synthetic hydrocarbon. From the filtration results, one can conclude the filter loss of (MC) mud is less than (MR) mud and this result indicated the stability of (MC) mud and each of them both compatible with API specification

**Table 3: High pressure-high temperature filter loss**

Mud formulation	Filter loss (mL)
(MC)	2
(MR)	3.5

### Electrical stability

The break down voltage in (mV) was measured for both (MC) mud that formulated from recycle used oil and (MR) mud, which was formulated from pure synthetic hydrocarbon. Table 4 illustrates that the value of electrical stability (mV) of (MC) mud is higher than (MR) mud and it decreased, when temperature increased from 60° to 200°F for both (MC) and (MR) muds.

**Table 4: Electrical stability of synthetic-based mud**

Temp. °F	MC (mV)	MR (mV)
60 °f	1200	1050
200 °f	950	800

### Thermal stability

The result of thermal stability of synthetic-based mud, which formulated from the recycled hydrocarbon (MC) before and after aging for 16 hour at 35°F. High hydrostatic pressure and continuous circulation represented on Table 5 and compared to the (MR) mud shared that (MC) mud is a thermally stable the (MR) mud.

**Table 5: Thermal stability of synthetic-based**

Mud	Aging	A.V. (c.p)	P.V. (c.p)	Y.P. (1b/ 100 ft <sup>2</sup> )	G10 sec. (1b/ 100 ft <sup>2</sup> )	G10sec. (1b/ 100 ft <sup>2</sup> )	FL (mL)
MC	0	105	89	32	7	15	2
	16	80	65	28	6	13	4
MR	0	88	76	23	6	11	3.5
	16	70	58	16	5	10	6

### Application of the power law model to the rotary viscometer data

The flow behavior index (n) and consistency index (K) values are calculated according to the power law model Herschel-Buckley model and is given by<sup>41,42</sup> :

$$\tau = \tau_0 + k\dot{\gamma}^n$$

$\tau$  = Shear stress,

$\tau_0$  = Yield stress or yield point,

K = The consistency factor (viscosity in case of a Newtonian or Bingham fluid)

$\dot{\gamma}$  = Share rate

n = The power of low exponent (rate index) or flow behavior.

Table 6 illustrated the flow behavior index (n) and the consistency index (k) of MC and MR.

**Table 6: The flow behavior index (n) and consistency index (K)**

Mud type	Dial reading at 600 rpm	Dial reading at 300 rpm	Flow behavior index (n) dimensionless	Consistency index (K) lb.sec <sup>n</sup> /ft <sup>2</sup>
MC	140	82	0.771	1.669
MR	122	72	0.61	1.136

## CONCLUSION

This study was carried out on Egyptian local crankcase used lubricating oil collected from different places and governorates in Egypt. The two rerefining processes were studied, namely: solvent extraction and hydrotreating. The most excellent quality of the base oil

rerefinery by solvent extraction was obtained using solvent to oil ratio 5:1 with settling time 24 hrs, while, it can be carried out at ambient temperature with cheap and low boiling point solvent MEK. There is high difference in quality between the rerefine base oil using solvent extraction and hydrotreating process with the used oil. With the increase in reaction temperature, the properties of the rerefine oil were enhanced. The above method causes favorite changes in the rerefined oil product, which are compatible with the coming specifications. All required technical and environmental specifications of the rerefined oil product are met by this method product, and also it can have the extra economic benefit of using hydrotreating catalysts. Moreover, almost no harmful or useless byproduct hydrocarbon is produced via this method.

The recycled used oil was evaluated as a continuous phase in the formulation of synthetic-based mud instead of the pure synthetic hydrocarbon for oil well drilling fluids. The experimental work and evaluation of the recycled oil showed a good results that meet API specification and have high performance than the commercial synthetic hydrocarbon – mud that used for this purpose (MR).

Rheological, filtration, and electrical stability properties performed a superior results compared to (MR).

Thermal stability of the mud formulated from the recycled oil was higher than that of the mud formulated with the pure synthetic hydrocarbon.

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### **REFERENCES**

1. H. A. Durrani, M. I. Panhwar and R. A. Kazi, Re-refining of Waste Lubricating Oil by Solvent Extraction, *J. Eng. Tech.*, **30(2)**, 237-246 (2011).
2. A. Kamal and F. Khan, Effect of Extraction and Adsorption on Re-Refining of Used Lubricating Oil, *J. Oil Gas Sci. Technol.*, **64(2)**, 191-197 (2009).
3. W. Manasomboonphan and S. Junyapoon, Production of Liquid Fuels from Waste Lube Oils used by Pyrolysis Process, 2<sup>nd</sup> International Conference on Biomedical Engineering and Technology, IPCBEE **Vol. 34**, IACSIT Press, Singapore (2012).

4. J. Baladincz, L. Szabo, G. Nagy and J. Hancsok, Possibilities for Processing for Used Lubricating Oils – Part 1, *MOL Sci. Mag.*, **3**, 81-86 (2008).
5. H. Bridjanian and M. Sattarin, Modern Recovery Methods in Used Oil Re-refining, *J. Petroleum Coal*, **48(1)**, 40-43 (2006).
6. F. B. Hani and H. Al-Wedyan, Regeneration of Base-Oil from Waste-Oil Under Different Conditions and Variables, *Afr. J. Biotechnol.*, **10(7)**, 1150-1153 (2011).
7. H. A. Durrani, M. I. Panhwar and R. A. Kazi, Determining an Efficient Solvent Extraction Parameters for Re-refining of Waste Lubricating Oils, *J. Eng. Tech.*, **31(2)**, 265-270 (2012).
8. P. N. Josiah and S. S. Ikiensikimama, Effect of Desludging and Absorption Ratios on Recovery of Low Pour Fuel Oil (LPFO) from Spent Engine Oil, *J. Chem. Eng. Res. Bull.*, **14**, 24-28 (2010).
9. N. M. Abdel-Jabbar, E. A. H. Al Zubaidy and M. Mehrvar, Waste Lubricating Oil Treatment by Adsorption Process using Different Adsorbents, *World Acad. Sci., J. Eng. Technol.*, **62**, 9-12 (2010).
10. S. S. Lam, A. D. Russell, C. L. Lee and H. A. Chase, Production of Hydrogen and Light Hydrocarbons as a Potential Gaseous Fuel from Microwave-Heated Pyrolysis of Waste Automotive Engine Oil, *Int. J. Hydrog. Energy*, **37**, 5011-5021 (2012).
11. S. O. Ogbeide, An Investigation to the Recycling of Spent Engine Oil, *J. Eng. Sci. Technol. Rev.*, **3(1)**, 32-35 (2010).
12. A. Hamad, E. Al-Zubaidy and M. E. Fayed, Used Lubricating Oil Recycling using Hydrocarbon Solvents, *J. Environ. Manage.*, **74**, 153-159 (2005).
13. S. Jhanani and K. Joseph, Used Oil Generation and Management in the Automotive Industries, *Int. J. Environ. Sci.*, **2(2)**, 638-648 (2011).
14. J. Lukic, A. Orlovic, M. Spiteller, J. Lovanovic and D. Skala, Re-refining of Waste Mineral Insulating Oil by Extraction with N-methyl-2-pyrrolidone, *J. Sep. Purif. Technol.*, **51**, 150-156 (2006).
15. A. Kaewsaiyoy, P. Reubroycheroen, T. Vitidsant and S. Damronglerd, Hydrocracking of Used Lubricating Oil on Ni-Mo/Al<sub>2</sub>O<sub>3</sub> and HZSM-5 Catalysts, Bangkok: Chulalongkorn University (2003) p. 137.
16. R. L. Brazzel, Multi-component Drilling Fluid Additive and Drilling Fluid System Incorporating the Additive, USA Patent 7 635 667 December 22 (2009).

17. M. Melbouci and A. C. Sau, Water-based Drilling Fluids, U.S.A. Patent 7 384 892, June 10 (2008).
18. S. Pål, Drilling Fluids Engineering, 1<sup>st</sup> Ed., Pålskalle & Ventu Publishing APS (2011) pp. 120-126.
19. M. M. Dardir and M. I. Abdou, Ether-based Muds Show Promise for Replacing Some Oil-based Muds, J. Pet. Sci. Technol., **31**, 2335-2347 (2013).
20. M. M. Dardir and A. A. Hafiz, Ester Amide as an Environmentally Friendly Synthetic-based Drilling Fluids, J. Am. Sci., **9(6)**, 133-142 (2013).
21. M. M. Dardir, S. Ibrahim, M. Soliman, S. D. Desouky and A. A. Hafiz, Preparation and Evaluation of Some Esteramides as Synthetic Based Drilling Fluids, EGYPT, J. Pet., **23**, 35-43 (2014).
22. C. Aldea, Bruton Jr; W. R. Dobbs and A. L. Klein, SPE Drilling and Completion, **20(1)**, 37-43 (2005).
23. A. Deutch, A. Marten and H. Lieske, Investigations on Heterogeneously Catalysed Condensations of Glycerol to Cyclic Acetals, J. Catal., **245(2)**, 428-435 (2007).
24. S. B. Hamed and M. Belhadri, Rheological Properties of Biopolymers Drilling Fluids, J. Pet. Sci. Eng., **67(3-4)**, 84-90 (2009).
25. J. Dieffenbayghur, R. Dupre and G. Authement, SPE-65277 IADC Drilling Conference Proceedings P (2005) pp. 809-821.
26. K. C. S. Sigamshetty, Authement Jr. and J. Gervais, IADC-SPE-Drilling Conference, 801-821 (2004).
27. M. Sumrow, Rising Drilling-Waste Costs Promote Novel Technologies, Approaches Oil and Gas- J., **100(52)**, 43-48 (2002).
28. P. Coussot, F. Bertrand and B. Herzhaft, J. Oil Gas Sci. Technol., **59(1)**, 23-29 (2004).
29. M. A. Scapina, C. Duarteb, M. H. O. Sampab and I. M. Satoa, Recycling of the used Automotive Lubricating Oil by Ionizing Radiation Process, Radiat. Phys. Chem., **76(11-12)**, 1899-1902 (2007).
30. J. Rincon, Regeneration of used Lubricant Oil by Polar Solvent Extraction, Ind. Eng. Chem. Res., **44(12)**, 4373-4379 (2005).
31. R. Abro, X. Chen, K. Harijan, Z. A. Dhakan and M. Ammar, A Comparative Study of Recycling of used Engine Oil using Extraction by Composite Solvent, Single Solvent, and Acid Treatment Methods, Hindawi Publishing Corporation ISRN Chem. Engg., **(2013)** Article ID 952589 (2013).

32. American Petroleum Institute API RP 13B-1, Standard Practice for Field Testing Water-based Drilling Fluids, Recommended Practice, 2<sup>nd</sup> Edition, Washington D.C., 17-19 (1997).
33. Oil Companies Materials Association (OCMA), Specification No. DFCP-4, Drilling Fluid Materials, The Institute of Petroleum, London, 5-9 (1983).
34. E. A. Emam and A. M. Shoaib, Re-Refining of used Lube Oil, I- By Solvent Extraction and Vacuum Distillation Followed by Hydrotreating, *J. Pet. Coal*, **55**(3), 186, 179-187 (2013).
35. S. A. Sadeek, H. S. Ahmed, E. A. ElShamy, H. A. El Sayed and A. A. Abd El Rahman, Hydrotreating of Waste Lube Oil by Rejuvenated Spent Hydrotreating Catalyst, *Egypt. J. Pet.*, **23**, 53-60 (2014).
36. I. Hamawand, T. Yusaf and S. Rafat, Recycling of Waste Engine Oils using a New Washing Agent, *J. Energies*, **6**, 1023-1049 (2013).
37. M. R. Riazi and T. E. Daubert, Predicting Flash and Pour Points, *J. Hydrocarb. Proc.*, **66**, 81-83 (1987).
38. R. M. Diaz, M. I. Bernardo, A. M. Fernandez and M. B. Folgueras, Prediction of the Viscosity of Lubricating Oil Blends at Any Temperature, *J. Fuel*, **75**, 574-578 (1996).
39. Forsthoffer and W. E. Lube, Seal and Control Oil System Best Practices, in Forsthoffer's Best Practice Handbook for Rotating Machinery, 1<sup>st</sup> Ed., Elsevier: Oxford, UK (2011) pp. 347-468.
40. J. Rincón, P. Cañizares and M. T. García, Regeneration of used Lubricant Oil by Polar Solvent Extraction, *J. Ind. Eng. Chem. Res.*, **44**(12), 4373-4379 (2005).
41. M. J. Davison, S. Clary, A. Saases, M. Allouche, D. Bodline and A. V. Nguyen, Rheology of Various Drilling Fluid Systems under Deep Water Drilling Conditions and the Importance of Accurate Predictions of Down Hole Fluid Hydraulics SPE Paper (56632), SPE Annual Technical Conference and Exhibition Held in Houston, Texas, 3-6 (1999).
42. J. Hermoso, F. Martinez-Boza and C. Gallegas, Influence of Viscosity Modifier Nature and Concentration in the Viscous Flow Behavior of Oil-Based Drilling Fluids at High Pressure, *J. Appl. Clay Sci.*, **87**, 14-21 (2014).

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