

Production of Virgin Lubricating Oil from Used Engine Using Solvent Extraction and Adsorption Process

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Abstract: Modern lubricating oil is made of base stock or base oil blended with a few ppm of chemical additives according to its grade and specific duty. During usage, lubricating oils undergo changes termed degradation and contamination, which render them ineffective for further application.

This research investigated the effect of two different solvent mixtures A=(xylene+butanol+methanol) and B=(xylene+butanol+isopropanol) followed by bleaching on activated alumina as adsorbent material to produce lubricating oil.

To achieve this objective, firstly used oil was allowed to settle for (1hr), after being centrifuged at 1500rpm for 30 minutes then exposed to filtration on Buncher funnel with vacuum pump to remove impurities.

The results showed that all ratio (1:1,1:3 and 1:5) of solvent mixture (A) is the best performance with the maximum percent sludge removal. The characterization and physico-chemical properties showed that the refined oil from solvent mixture (A) followed by treatment with activated alumina have good properties with comparing to the standard specification of new lubricating oil .

Keywords: Used engine oil, Refining process, solvent extraction and adsorption technique.

1. INTRODUCTION

Lubricating oils are widely used in industries to reduce friction and wear by interposing a thin film of oil between metallic surfaces. [1]

When lubricating oils are used in service, they help to protect rubbing surfaces and promote easier motion of connected parts. In the process, they serve as a medium to remove high build up of temperature on the moving surfaces. Further build up of temperature degrades the lubricating oils, thus leading to reduction in properties such as: viscosity, specific gravity, etc. Dirt and metal parts worn out from the surfaces are deposited into the lubricating oils. With increased time of usage, the lubricating oil loses its lubricating properties as a result of over-reduction of desired properties, and thus must be evacuated and a fresh one replaced. With the large amount of engine oils used, the disposal of lubricating oils has now become a major problem [2].

Used engine oil is a high pollutant material that requires responsible management. Waste engine oil may cause damage to the environment when dumped into the ground or into water streams including sewers. This may result in groundwater and soil contamination [3].

Used oil has been re-refined using many techniques such as chemical (acid/clay) treatment [4,7] physical treatment by distillation and thin film evaporation and solvent extraction [8]. Furthermore, process waste lube oil can be converted into pyrolysis oil [9]. These techniques have different yield and product qualities, construction and operational cost. Since the

chemical treatment creates environmental problems, therefore, solvent extraction was proposed as an alternate method [10].

In this work, the solvent extraction treatment followed by adsorption has received considerable attention in recent years; because it overcomes the problems associated with acid sludge produced from chemical treatment [11]

Solvent extraction is a simple process; used oil and solvent mixtures are mixed in appropriate proportions to assure complete miscibility of the base oil in the solvent. The extracting solvent should also reject the additives and carbonaceous impurities normally found in used oils. These impurities flocculate and settle under gravity action. The solvent is then recovered by distillation for recycle purpose [12]. The application of an adsorption process using adsorbent material such as activated alumina was investigated.

2. EXPERIMENTAL

Pre-treatment process of used oil sample

1. sample collection and Material used in the study :

Used engine oil (Total Quartz 9000) was collected from oil service station in EPRI. All the chemical reagents (purchased from Merck Co., Aldrich and Fluka chemical Co.) were of analytical grade and used without further purification (Xylene, 1-butanol, methanol and iso propanol, cyclohexane and alumina).

2. purification of used oil sample:

The procedure for the purification of the used engine oil consists of centrifugation and filtration of the oil before subjecting to treatment. The used engine oil was left to settle for one hour after being centrifuged at 1500 rpm for 30 minutes then exposed to filtration to remove impurities such as metal chips, sand and dust particles. This was done using a Buchner funnel with a vacuum pump.

Refining processes of used oil sample:

1-Solvent extraction process:

- Two extracting solvent mixtures, A = (xylene + 1-Butanol + Methanol) and B = (xylene + 1-Butanol + 2-propanol) with ratio (1:1:1) respectively, were used. Each one of the solvent mixtures (A and B) are treated individually with used engine oil sample at different ratios of oil: solvent mixture (1:1, 1:3 and 1:5 vol.) for each solvent mixture (A and B).
- The used engine oil sample with solvent mixtures were mixed. Then, the mixture was refluxed using glass condenser with stirred at 300 rpm for 2 hours and at temperature (70°C). This step was carefully conducted to ensure no oil loss in the flask with proper mixing and appropriate vessel system. The final mixture was left for 48 hrs. to settle under gravity action. The heavy black sludge is observed at the bottom of the flask after settling. The solvent-oil mixture was separated from the sludge by filtration.
- The solvent mixture is recovered by atmospheric distillation at a temperature of 130°C and refined oil obtained.

2-Bleaching of refined oil sample by alumina :

Neutral alumina 200 mesh sieve are activated at 300°C for 24 hours. The refined oil sample after solvent extraction processes are treated with alumina at oil : alumina ratio 1:15 in open glass column 150cm in length and 5cm in diameter. Elution was carried out with cyclohexane and ethanol. The elute was freed of solvent by distillation. The separated oils were dried in a vacuum oven till constant weight and were determined. The alumina used to improve the colour of regenerated oil and the smell which result from oxidation of some components in the oil.

Determination Physico-chemical Properties of used & all re-refined oils

Physico-chemical properties (Density, Kinematic viscosity, Viscosity index, Flash point, Pour Point, Ash content, Carbon residue, Water content, Heavy Metals, Total Acid Number and Sulfur Content) of used oil sample, refined oils obtained from solvent extraction at different ratio and lubricating oil obtained after bleaching with alumina are carried out according to ASTM methods (13).

3. RESULTS AND DISCUSSION

The term "used oil" refers to lubricating oils that are collected after usage in small engines and automotive engines, used oil is a complex mixture of recoverable base oil, polymeric additives, water, light hydrocarbons, metals and carbonaceous particles.

Solvent Extraction process:

In this work different solvent mixtures (A and B) constituted of both polar and non-polar solvents were used. The role of non-polar like xylenes was used due to its ability to dissolve particularly base oil and precipitate other substances present in the used oil to form sludge, while the polar solvents (butanol, methanol, ethanol and isopropanol) behave as efficient settling for used oil due to their good solubility parameter().

The different interaction between the solvent and oil molecules, due to the size of the carbon chain and the solvent molecules. It is found that the efficiency of solvent mixture contains methanol is higher followed by solvent mixture contains isopropanol. This is due to the ability of solvent to dissolve sludge and contaminants from used oil is depended on its solubility. As oil to solvent mixture increase the solvency power increase from 1:1 to 1:3 but by increasing the ratio to 1:5 we found that the removal of sludge decreased and becomes very difficult for separation of sludge and contaminants due to higher miscibility of used oil in solvent mixture, so the ratio of 1:3 was considered to be the better oil to solvent ratio used for treatment.

Comparative study on physico-chemical properties of used, refined oils after solvent Extraction(A&B) and Produced oil after alumina treatment:

The physico-chemical properties of all samples were determined according to the standard method of analysis in ASTM and IP(13,14).

The physical properties of the used oil investigated in this work are represented in Table (1). Due to Oxidation of used oil would result in the formation of volatile component, the oxidized and polymerized products and suspended substances in the oil which leads to decrease the flash points, pour points. Also leading to increase viscosity, Specific gravity, sulfur content and carbon residues of used oil. Water content in used oil is high. Water created in used oil is a result of absorbing moisture directly from the air condensation, heat exchanger, oxidation and it could be mixed with washing water at the collection point water attacks the additives such as oxidation inhibitor, rust inhibitor viscosity improvers and the oil's base stock forming sludge – therefore, the sludge content in used oil is high. Total acid number is high. It has been considered to be an important indicator for engine oil quality.

The physico-chemical properties of the refined oils obtained by recycling of the used oil with two solvent mixtures (A,B) are improved, due to the removal of the impurities compounds. The refined lubricating Oil possesses good pour point (-3°C) because pour point is important when considering oil under cold weather. It can be seen increase in flash points from (102) and viscosity index (VI) at all solvent/oil ratios reach maximum values (256°C) for solvent mixture (A) and (205°C) for solvent mixture (B), at solvent oil ratio 3:1 as given in Tables (1). The difference between them is related to the solubility of the base oil in the two solvent mixtures. The kinematic viscosity, total acid number, sulfur content, water content, ash content and carbon content decreases with increasing solvent/oil ratios. This means that the three solvent mixtures are effective in removing the impurities from used oil. The efficiency of the extraction agents can also be related to the different interaction between the solvent and oil molecule. The optimum improvement in physical properties reaches at solvent/oil ratio 3:1 at which the high reactivity of the two solvent mixtures with the used oil to produce lubricating oil.

The physico-chemical properties of the produced oil obtained by solvent mixture (A) at solvent/oil ratio 3:1 vol. at 70°C after treatment with activated alumina are also represented in Table (1). From the results it is clear that the physical properties fall with the specification of the fresh oil. The viscosity index (132.4), flash point (315°C), pour point (-3°C), total acid number (0.165 mg KOH/g sample). It is found that all organic and inorganic acids are removed.

Table (1): Physico-chemical properties for used, refined oils obtained by extraction method using solvent mixture(A and B) at ratio (1:1,1:3 &1:5) vol. at 70 °C and produced lubricating oil after alumina treatment.

Test	ASTM	Used oil	Refined oil at ratio 1:1 vol.		Refined oil at ratio 1:3 vol.		Refined oil at ratio 1:5 vol.		Produced oil aftertreatment with alumina A at ratio(1:3)
			A	B	A	B	A	B	
Denisty@ 15.56°C	D-4052	0.9077	0.8802	0.8993	0.8610	0.8931	0.8795	0.8948	0.852
Kinamaticviscosity,cSt @40°C @100°C	D-445	192.82 18.82	127.74	157.95	100.97	146.75	117.25	147.61	90.6
			14.79	16.35	13.21	15.89	14.02	15.97	12.4
Viscosity index	D-2270	109.48	117.411	108.699	128.547	112.74	118.988	112.906	131.733
Flash point, °C	D-93	102	224	195	256	205	229	235	315
Pour point, °C	D-97	-15	-6	-15	-3	-9	-6	-15	-3
TAN,mg KOH/g	D-664	2.461	1.14	1.764	0.364	1.242	0.9218	2.13	0.1651
Sulfur content,wt %	D-4294	1.24	1.08	1.19	0.51	0.99	0.91	1.15	Nil
Water content,ppm	D-6304	2405.115	127.97	216.97	50.485	148.75	92.215	151.2	Nil
Ash content,wt %	D-482	1.17	0.791	0.971	0.11	0.34	0.20	0.71	Nil
Carbon residue,wt %	D-189	1.65	0.97	1.24	0.21	0.43	0.34	0.98	Nil
Sludge content,wt %	D-4310	5.8	3.99	4.52	2.01	2.94	2.43	3.85	Nil

Table (2) Physico-chemical properties of Fresh oil according to the Egyptian organization for standardization and quality(EOS).

Test	Used oil	Refined oil after alumina treatment	Fresh oil according to (EOS)
Denisty@15.56°C	0.9077	0.852	0.841
Kinamaticviscosity,cSt @40°C @100°C	192.82	90.6	92.8
	18.82	12.4	12.5
Viscosity index	109.48	131.733	125
Flash point, °C	102	315	268
Pour point, °C	-15	-3	-9
TAN,mg KOH/g	2.461	0.1651	0.1
Sulfur content,wt %	1.24	Nil	Nil
Water content,ppm	2405.115	Nil	Nil
Ash content,wt %	1.17	Nil	0.05
Carbon residue,wt %	1.65	Nil	0.05
Sludge content,wt %	5.8	Nil	Nil
Metal analysis,ppm			
Fe	35.004	Nil	Nil
Cu	7.525	Nil	Nil
Cr	15.973	Nil	Nil
Ni	2.559	Nil	Nil
Pd	3.431	Nil	Nil
Mg	263.18	Nil	Nil
Zn	842.37	Nil	Nil

4. CONCLUSION

The re-refining processes were studied, namely: solvent extraction/adsorption processes. An important experimental measurement of re-refining process is represented by the amount of sludge removed from the used oil, we found that the removal of sludge by using solvent mixture contains methanol (A) is higher as compared with solvent mixture contains isopropanol (B), due to the higher solubility of methanol is $29\text{ J/m}^3)^{1/2}$. The rate of removal of sludge increases with increasing solvent oil ratio. The optimum of solvent oil ratio was found to be 3:1 at 70°C . In all cases examined in this work, the physico-chemical properties of the base oils obtained by recycling of the used oil with two solvent mixtures (A and B) are improved. This is due to the removal of the impurities compounds.

The physico-chemical properties of the base oils obtained by solvent mixture (A) at solvent/oil ratio 3:1 vol. at 70°C after treated with activated alumina fall within the specification of the base virgin oil.

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