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RECYCLING AND CHARACTERIZATION OF SPENT ENGINE OIL USING TWO STAGES: SOLVENT EXTRACTION AND CLAY PERCOLATION

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ABSTRACT

Spent lube oils are continuously generated from factories, vehicles and other machineries. Most spent engine oil contains harmful substances such as heavy metals and indiscriminate disposal of this oil causes environmental pollution. The recycling of spent engine oil has also become important due to its economic benefits. In this study, solvent extraction and clay percolation techniques were used to recycle spent engine oil. The specific gravity, flash point, viscosity, pour point total acid number and bases number were determined. The specific gravity was 0.8252-0.8937, viscosity at 40ºC was from 79.37 to 130.12cSt while at 100ºC was 8.50 to 15.64 cSt. The flash and pour points were 121-214ºC and -14 to -9ºC respectively. The total acid number were 5.21 and 2.21 mgKOH/g for untreated oil, 0.43 and 0.315 mgKOH/g for treated oil while the total based number were 8.42 and 6.30 mgKOH/g for the treated oil. The results suggested that this method can be employed to improve the quality of spent engine oil for industrial applications as well as reducing environmental pollution due to spent engine oil.

Keywords: Clay, pollution, properties, solvent extraction, spent engine oil

INTRODUCTION

Spent lube oil otherwise known as condemned engine oil is any petroleum base or synthetic oil that becomes unsuitable for lubricating purposes after being used for lubrication. It is unfit for lubrication activity due to the loss of its original properties. This loss occurs because of physical contaminants from the fuel combustion, air, oxidation, and additives (Kannan et al, 2014).

Disposal of used lubricating oils on land and water bodies is of great concern due to the impact on the economic and environment through the release of harmful heavy metals and other pollutants. This oil also contains several poisonous substances such as poly-aromatic hydrocarbons and poly-cyclic benzenes that are carcinogenic (Udonne et al. 2016; Hamawand et al. 2013). Heavy metals such as Pb, Zn, Cr, Cd, Co, Mn, Ni, Cu, etc are toxic to the living organism and can impair important biochemical process posing a threat to human health, plant growth and animal life (Abdullahi and Musa, 2023).

The contaminants in spent engine oil are mainly sludge, oil soluble products and lacquer. These are introduced by air, dust, dirt and moisture from the surrounding. Others are metallic particles resulting from the wearing of the engine parts (Ogbeide, 2010).

Lubricant oils have they advantages of reducing and removing heat, corrosion prevention, reducing friction and minimizing wear and tear of various types of machineries. Lubricating oil also help in improving the efficient of equipment and keeping them clean (Dineshet al. 2006).

Many studies have shown that spent engine oil can be regenerated and re-use. The regeneration provides a cleaner, safe and pollution free environment as well conserving the natural resource, thus saving cost (Ugwele et al 2020). Many regeneration methods of spent engine oils have been reported, among which are acid activation, physical and chemical processes, solvent extraction, membrane technology, hydrogenation, catalytic process as well as combine methods such as vaxon, cyclone process (Boadu, et al.2019).

Various extraction methods such as solvent extraction, ionization, re-distillation, etc have been employed for the treatment of spent engine oils. The solvent extraction process can be single or multi-components while the re-distilling technique is employed in most developed countries to and improve the quality of used spent engine oil (Adewola et al. 2019).

Recycling of spent lubricants is getting more attention due to environmental impact and the fear of dwindling of oil reserves globally. Recycled oil is expected to be clean, safe and free from contaminants such as moisture, dust, fuel, etc. (Shaban et al.2015). The adsorption process for the treatment of spent engine oil is based on the adsorbent ability to selectively extract the pollutants like resinous and sulfur containing compounds, unsaturated and polycyclic material, organic residues of sulfuric acid and solvents from oils. Natural, activated and calcined clays are used as adsorbents for the removal of contaminants in waste lubrication oil. Therefore, this research is aimed at treatment of spent engine through solvent extraction and clay percolation techniques and also to investigate the fuel quality of the recycled oil compared with the Society of Automotive Engineers (SAE) Standard. This will curb the menace of environmental pollution as well saving cost.

MATERIALS AND METHODS

Sample Collection and Preparation

The samples of used lubricating oil were collected from a vehicle repair station at Mechanic Village, Uyo, Akwa Ibom State. The kaolin clay sample was gotten from Edikor Eyibia, Udung Uko Local Government Area, Akwa Ibom State, Nigeria.

The spent oil samples were allowed to first settle in a separating funnel for 24 h at room temperature then followed by decantation to remove fine particles. The used oil was dehydrated by heating in an oven to remove moisture. The clay sample was pulverized and sieved using a 75 µm. The clay slurry was prepared by adding 80cm^3 of distilled water to 200 g of the pulverized kaolin. The slurry was then poured into an aluminum pan at room temperature for 1 h and then oven dried at 110°C for 24 h. The dried clay was crushed and sieved with a 75µm sieve size.

Solvent Extraction and Clay Percolation

Solvent extraction was performed on the two spent oil samples, M_1 and M_2 using butanol, n-Hexane and potassium hydroxide. 300ml of each oil sample was mixed with the solvent containing a mixture of 70% butanol and 30% n-hexane at a ratio 5:1. 9g of KOH was added to the oil-solvent mixture, stirred vigorously for 30 minutes and heated for 30 minutes at a constant temperature of 60ºC to remove light ends hydrocarbon. The mixture was poured into a separating funnel and allowed to settle in for 24 h and thereafter the sludge was removed. The samples were heated again in a sand bath at 70ºC and 120ºC to evaporate n-hexane and butanol respectively.

Clay percolation was conducted using a glass column of length and diameter 35 cm and 0.06 cm respectively. The column was packed with 10 g of clay placed in the middle of the glass column. The bed depth measured as 0.5 cm was supported at top and bottom with a layer of cotton wool. The oil was poured from the top of the glass column and allowed to pass through the clay bed. A schematic diagram of the experimental set-up used for solvent extraction and clay percolation is shown in Figure 1.

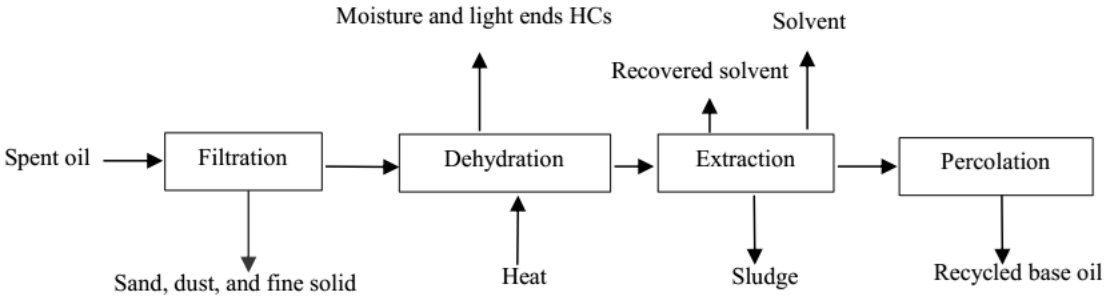

Figure 1: A schematic diagram of the experimental setup used for solvent extraction and clay percolation.

Flash Point

The flash point test was conducted using an open-cup method according to ASTM D-92. 20ml of the oil sample was poured into a cup, heated and stirred continuously at constant temperature. Ignition source was brought to the oil surface at an interval of one minute to check the temperature which a flash appears on the surface of the sample. The temperature at which flash (smoke) appeared was measured with thermometer as the flash point.

Pour Point

20ml of the oil sample was measured into an enclosed container fitted with a thermometer that can read a negative temperature up to -30ºC. It was chilled and, checked at intervals of 5 min until the oil stopped flowing (ASTM D-97). The temperature at which the oil had difficulty flowing was recorded as the pour point.

Specific Gravity

The specific gravity is the ratio of the density of a material to the density of an equal volume of water and it was determined using a density bottle (pycnometer) according to method prescribed by ASTM D-1298. The specific gravity of the oil sample was calculated from the ratio of the density of oil to that of water using equation 1

$$\text{Specific gravity} = \frac{\text{density of oil sample}}{\text{density of water}} \quad (1)$$

Viscosity

The viscosity was determined using an Ostwald viscometer. Oil samples were heated to a temperature between 40 and 100 ºC before pouring into a viscometer. The flow time of the oil was recorded when the oil reached the lower and upper marks on the viscometer (ASTM D-445).

The viscosity was evaluated using equation 2.

$$n_2 = \frac{\rho_2 t_2}{\rho_1 t_1} * n_1 \quad (2)$$

Where

ρ_1 density of reference material $and\ t_1$ time of reference material. $\rho_2\ and\ t_2$ are density and time it takes the oil to flow respectively. n_2 is oil and n_1 the reference material.

Total Acid Number

Total acid number (TAN) was determined using the procedure specified by ASTMD-974.10g of lubricating oil was dissolved in a mixture of toluene and 2-propanol solution with a small amount of water. The dissolved solution was titrated with 0.1 mol/L/potassium hydroxide 2-propanol solution using titration method. The total acid number was calculated from equation 3.

$$Acid\ value\ \left(\frac{mgKOH}{g}\right) = \frac{vol\ of\ KOH\ (ml)\ *\ Normality\ *\ 56}{weight\ of\ the\ oil} \quad (3)$$

Total Base Number

The total base number (TBN) was determined by acid-base titration using $HClO_4$ as the titrant. 10 g of treated oil sample was weighed and diluted in a mixture of chlorobenzene and acetic acid as described by ASTM D-2896. The solution was titrated to obtain the total base number using equation 4.

$$TBN\ \left(\frac{mgKOH}{g}\right) = \frac{0.1\left(\frac{eq}{L}\right)(V_t - V_b)(ml) * 56.11\left(\frac{g}{mol}\right)}{1 * m_s} \quad (4)$$

Where M_s is the mass of the sample, V_t is volume of the titrant and V_b volume of the sample with blank or zero solvent and $^{eq}/_{L}$ is the equivalent volume.

RESULTS AND DISCUSSION

The spent engine oil was treated using solvent extraction and clay percolation. The untreated and treated oil samples were characterized for fuel properties such as specific gravity, pour point, flash point, total acid number and total base number as presented in Table 1. These parameters evaluated were compared to the standard properties specified by the Society of Automotive Engineers (SAE) in Table 2.

Table 1: Fuel properties of treated and untreated spent engine oil

Properties	Untreated oil		Treated oil	
	M_{U1}	M_{U2}	M_{T1}	M_{T2}
Specific gravity	0.86667	0.89367	0.8252	0.82637
Kinematic viscosity @ 40ºC (cSt)	130.12	128.03	101.49	79.37
Kinematic viscosity @ 100ºC (cSt)	15.63	11.30	11.61	8.50
Flash point (ºC)	121	131	208	214
Pour point (ºC)	- 9	-11	-10	-14
Total acid number (mgKOH/g)	5.21	2.33	0.430	0.305
Total base number (mgKOH/g)	-	-	8.42	6.30

Table 2: Comparison on the properties of the treated oil and Standard lube oil

Properties of treated engine oil			Lube oil specification	
	M_{T1}	M_{T2}	SAE20	SAE30
Specific gravity	0.825	0.827	0.87 - 0.90	0.87 - 0.92
Kinematic viscosity @ 40ºC (cSt)	101.49	79.37	45 - 75	80 – 120
Kinematic viscosity @ 100ºC (cSt)	11.61	8.50	7.5 - 11	10 - 13.5
Flash point (ºC)	208	214	225 - 260	235 – 280
Pour point (ºC)	- 10	- 14	> -15	> -12
Total acid number (mgKOH/g)	0.430	0.305	0.1 - 2	0.1 – 2
Total base number (mgKOH/g)	8.42	6.30	5 - 15	5 – 15

Source: SAE, J300 (2019)

The specific gravity of the untreated oil (Mu_1 and Mu_2) obtained from two different sources were 0.86667 and 0.89367 while for treated oil sample M_{T1} and M_{T2} were 0.8252 and 0.82637 respectively. There was a decrease in the specific gravity after treatment. The reduction in the specific gravity could be attributed to the removal of impurities in the spent engine oil during the treatment process. The presence of impurities in untreated oil contributed to the high specific gravity as many of these contaminated impurities are typically denser than the oil (Musa et al., 2021). The specific gravity of the treated oil is closed to the standard specified for lube oils (SAE20 and SAE30) as presented in Table 2.

The viscosity of the untreated oil at 40ºC were 130.12 and 128.03 cSt while for the treated oil were 101.49 and 79.37cSt. Similarly, the kinematic viscosity at 100 ºC were 15.63 and 11.30 cSt for untreated oil while the treated were 11.61 and 8.50 cSt as summarized in Table 1. Viscosity of a substance depends strongly on the temperature as well as pressure and density. It is also a measure used to show the presence of impurities in spent lubricating oil. Low viscosity in used engine oil might be from fuel contamination while high viscosity in spent lubricants could be due to products resulting from oxidation and polymerization dissolved and suspended in the oil (Ahamad et al. 2015). The oil viscosity also decreased after treatment at 40 and 100ºC. The decreased in the viscosity of treated oil could be due to the removal of contaminants in the untreated spent oil which were removed during treatment (Machado et al., 2017). The low viscosity at 100ºC might be due to an increased in shear rate at higher temperature thus, causing the oil molecules to break down and becoming less viscous (Hsieh et al., 2015). The viscosity of the treated oil at 40ºC is within the range (80-120cSt) for standard lube oil of SAE30 type. The viscosity of the treated oil at 100ºC oil was slightly below SAE20 and above SAE30 as shown in Table 2.

Flash point is an important parameter that is used to assess the risk of fire and explosion during the storage and handling of engine oil. It indicates the presence of volatile compounds in the oil. The flash points of the untreated were 121 and 131°C while for the treated engine oil were 208 and 214°C. There is an increase in the flash point in the treated samples and this could be attributed to the removal of impurities from the untreated oil during solvent extraction and clay percolation processes employed for the treatment. The low flash point in spent oil was an indication that the oil was contaminated with substances (Yusuf et al. 2021). The flash point of the treated oil is closed to the standard lube oil (type SAE20) depicted in Table 2.

The pour point is an indicator that shows the temperature at which oil becomes too viscous to flow effectively under the influence of gravity in a lubricating system. The pour points for the spent engine oil were -9 and -11ºC while for treated oil were -10 and, -14ºC. The pour point of the spent oil decreased after treatment and this could be due to the removal impurities mainly wax compounds that causes oil to solidify at low temperatures (Ogbeide et al. 2020). The pour point of the treated oil was very close to the specified SAE30 lube oil value as shown in Table 2.

The total acid number is a parameter that is used to measure the level of acidity in engine oils. The total acid number in the spent oil were 5.21 and 2.33 mgKOH/g while for the treated oil were 0.430 and 0.305 mgKOH/g. The result showed a significant decrease in the total acid number in the treated oil and this could be attributed to the fact that solvent extraction with the clay percolation techniques was effective in the removal of acidic compounds in the spent oil (Kaur, 2019). The total acid number of the treated oil was within the standard specified (0.1-2 mgKOH/g) for lube oil.

A high total base number of 8.42 and 6.30 mgKOH/g were obtained in treated oil samples. These values are within the range specified for general internal combustion engines. Lower total base number could be attributed to the accumulation of acidic by-products of combustion, oxidation, and contamination from the fuel (Tawfik et al., 2021). The total base number was within the range for standard lube oil as presented in Table 2.

CONCLUSION

The study investigated the efficacy of utilizing solvent extraction and clay percolation techniques for treatment of spent engine oil. The fuel properties determined were compared with standard specified properties for lube oil grades SAE20 and SAE30. The results obtained demonstrated the effectiveness of the treatment methods of

solvent extraction and clay percolation employed in removing impurities in the spent engine oil. The specific gravity, pour point, total acid and total base number were within the range for standard specified lube oil. These findings suggest that the utilization of solvent extraction and clay percolation is efficient in treatment of spent oil and suitable for industrial applications.

COMPETING INTERESTS

The authors declare that there are no competing interests.

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