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# Evaluation of physico-chemical properties of re-refined lubricating oils obtained from fabricated packed bed reactor

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#### ABSTRACT

This study aimed at fabricating a packed bed reactor (PBR) and evaluating its performance on improving physico-chemical properties of used lubricating oils. The sorbent used was a composite of bentonite, limestone, diatomite, guartz, and wood charcoal in the same ratio by mass. Samples of used lubricating oils from two brands (Mobil Super SAE 20W-50 and Total Quartz 5000 SAE 20W-50) were run through the fabricated PBR. The mean values of the analyzed physico-chemical parameters (kinematic viscosity, viscosity index, flash point, pour point, acid number, base number, iodine value, ash content, density, and refractive index) of the exuded (treated) oils were compared with those of used and virgin oils of the same brand. Apart from the iodine value, the mean values obtained for the treated oil samples are close to those of virgin oil samples. The *t*-test results indicated that most of the parameters showed significant difference between their mean values in the used oils indicating distinct properties and hence, good treatment effects, while a good number showed no significant difference between their mean values in the treated Mobil and treated Total oils, indicating similar properties and that the treatment had similar effects on the two brands of oils. Also, various results of cross plots had  $R^2$ values greater than 0.96, indicating high linear relationships of the impact of the treatment on the various oil samples. The overall results established the high effectiveness of the fabricated PBR for the treatment of used lubricating oils.

#### **KEYWORDS**

automobile engine; lubricating oil; PBR; physico-chemical parameter; sorbent

## 1. Introduction

Lubricating oil used in automobile engines accumulates large volume of rubbles, such as metals, sulphur, water, dirt, combustion products, viz ash and carbon residue from engine wear. As a result of the existence of degraded additives and the aforementioned contaminants, used motor oil disposal can be more environmentally detrimental than crude oil pollution. The amount of lubricating oils annually generated worldwide is colossal and is increasing due to increase in automobiles. The large amount of waste engine oils has significant socio-economic and environmental impacts on the society. If discharged onto the land, water or even burnt as a low grade fuel, it may cause serious environmental pollution because harmful metals and other persistent organic and inorganic pollutants are discarded into the environment (Reis and Jeronimo, 1988). These additives and contaminants may cause both short-term and long-term adverse health-effects. Since the base oil does not degrade, used lubricating oils (ULOs) have the prospective to be recycled safely and

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productively thereby saving energy, time, and evading environmental degradation (Boughton and Horvath 2004).

Even though some of the previous conventional procedures of recycling ULO gave high quality product with high output but they necessitated high-cost; usage of toxic materials such as sulphuric acid; wastage of solvents; production of huge magnitude of pollutants; incompetence to treat modern multi-grade oils and the strain in confiscating asphaltic impurities and unsafe as the case may be (Rincon, Canizares, and Garcia 2005; Shakirullah et al. 2006; Ihsan, Talal, and Sardasht 2013; Adebiyi et al. 2014).

The application of the packed-bed reactor using locally available diatomaceous materials employed in this work would be a safe and economical purification method for ULOs. This approach is similar to the principle of chromatography separation.

## 2. Experimental

#### 2.1. The fabricated packed bed reactor

The experimental setup consisted of a reservoir which housed the pre-heated used oil, a tap, a reciprocating pump (powered by 1 horse power geared motor), a crank connected to the motor, piston enclosed in a sleeve, pressure gauge, the packed-bed, and a collecting vessel. The column was formulated to contain about 10 L quantity of the sorbent, while the wall thickness was defined to withstand the required pressure. The packed bed reactor (PBR) was constructed at the Central Technological Laboratory and Workshop (CTLW), Obafemi Awolowo University, Ile-Ife, Nigeria.

## 2.2. Materials and their preparation

Used and unused lubricating oil samples of two brands (Mobil Super SAE 20W-50 and Total Quartz 5000 SAE 20W-50) were used in the study. The unused (virgin) oil samples of the two brands were purchased from standard lubricant stations in Ile-Ife, Nigeria, and they were introduced at the time of routine services into five selected cars for each brand. The oils were drained after 12 weeks and retained as the ULO samples. The sorbent materials employed consisted of a blend of bentonite, limestone, diatomite, quartz, and wood charcoal in the same ratio by mass. The sorbent materials were prepared at the Federal Institute for Industrial Research, Oshodi (FIIRO), Lagos, Nigeria. Slurry of the sorbent mixture was made and fired at 900°C to produce a cake, which was then ground and screened to produce uniform material of narrow particle-size distribution (1.0-1.5 mm). The particle-size distribution of the ground material was estimated using two sieves of 1.0 and 1.5 mm pore sizes. The ground sorbent was activated by soaking in 6 M H<sub>2</sub>SO<sub>4</sub> at 60°C for 6 hr. This was followed by filtration and then drying in a Muffle furnace at 250°C for 4 hr. The used oil samples were allowed to stand still for two weeks to give way for sedimentation. The top part of each was decanted into stainless pan and heated in open air at about 115°C for 2 hr to drive out trapped moisture and some volatile constituents and to reduce its viscosity so that flowing through the packed bed can be enhanced. The hot oil was filtered into the reservoir after which the tap was opened. Five sets of used oil samples from each of the two brands were run through the PBR. The physico-chemical properties of the exuded oils were determined and the results were compared with those of used (untreated) and unused (virgin) oils of the same and other brands.

## 2.3. Measurement of the physico-chemical parameters

The qualities of the re-refined, virgin, and the ULOs were assessed in terms of the chemical and the physical parameters. The chemical parameters were defined by the total acid number, the total base number, iodine value, and ash content of the oils; while the physical parameters were described by the density, kinematic viscosity, pour point, and flash point. 1714 👄 A. F. ADEYEMI ET AL.

#### 2.3.1. Total acid number

Total acid number measurement was carried out using the titrimetric method of (ASTM 2004). The acid value was calculated from the expression below.

TAN 
$$(\text{mg of KOH/g}) = \frac{(T_{\text{S}} - T_{\text{B}}) \times 0.25 \text{ M} \times 56.10}{\text{weight of oil sample (g)}}$$
 (1)

where  $T_S$  and  $T_B$  = Titre values (mL) for sample and blank titrations.

#### 2.3.2. Total base number

Total base number determination was done using titrimetric technique of (ASTM 2004). The base number was calculated from the expression below:

TBN (mg of KOH/g) = 
$$\frac{(T_{\rm S} - T_{\rm B}) \times 0.1 \,\mathrm{M} \times 56.10}{\mathrm{weight of oil sample (g)}}$$
 (2)

 $T_{\rm S}$  and  $T_{\rm B}$  = Titre values (mL) for sample and blank titrations.

#### 2.3.3. Iodine value

Iodine value was determined using titrimetric procedure and was calculated using the expression below (AOAC 1990):

I.V. 
$$\left(\frac{\text{g of } I_2}{100 \text{ g oil}}\right) = \frac{(T_B - T_S) \times 126.9 \times 0.1 \text{ M}}{\text{weight of oil sample (g)}}$$
 (3)

 $T_{\rm S}$  and  $T_{\rm B}$  = Titre values (mL) for sample and blank titrations.

#### 2.3.4. Density

The density of the oil samples was determined using density bottle at 29 °C and was calculated as below (ASTM 2004).

Density = 
$$\frac{\text{mass of oil (g)}}{\text{volume of oil (mL)}} = \frac{(W_2 - W_1) \text{ g}}{32 \text{ mL}}$$
 (4)

## 2.3.5. Refractive index

The refractive index of the oil samples was determined using Abbe refractometer with code number 44–501 and serial number AX 00474 from Bellingham Stanley Limited, United Kingdom (ASTM 2004).

#### 2.3.6. Viscosity

The kinematic viscosity at 40 and 100°C, and the viscosity index were determined using a viscometer of model PSL ASTM-IP 2009772 with calibration constant of 0.09827 from England. The viscosities of the oil samples were determined using the following equations (ASTM 2004):

Kinematic viscosity (K.V) = calibration constant  $\times$  mean time of flow of oil

$$= 0.09827 \text{ mean time of flow of oil}$$
(5)

where

$$N = (\log H n \log U) \log Y; \quad U = K.V \text{ of the oil at } 40^{\circ}C \text{ (cSt)}$$
(7)

Y = K.V of the oil at  $100^{\circ}C$  (cSt)

H = kinematic viscosity at 40°C of an oil of 100 viscosity index having the same kinematic viscosity at 100°C as the oil whose viscosity index is to be calculated mm<sup>2</sup>/s (cSt).

## 2.3.7. Flash point

Cleveland open cup apparatus method was employed in the measurement of the flash point. The minimum temperature at which the vapor caught fire was taken as its flash point (ASTM 2004).

## 2.3.8. Pour point

This was measured using the procedure of ASTM (2004). The highest temperature at which the oil sample seizes to flow was taken as its pour point.

## 2.3.9. Ash content

This parameter was determined using the procedure of ASTM (2004). The difference in weight of samples before and after ashing gave the ash content of the samples.

## 2.5. Quality assurance and control

All the glassware and sample bottles were cleaned using the procedure of Harrison, Laxen, and Wilson (1981), while blank determinations were also carried out where applicable.

## 3. Results and discussion

## 3.1. The fabricated packed bed reactor

The fabricated PBR is shown in Figure 1. It consists of a reservoir that housed the oil, a tap which controls the flow of oil from the reservoir, a geared motor (1 hp), a crank connected to the motor, a piston enclosed in a sleeve, a pressure gauge, packing column (length: 762 mm; internal diameter: 58 mm; thickness: 2 mm) and a collecting vessel. The packing length was 609.6 mm. The average time taken for first drop oil to exude from the collecting vessels after opening of the tap was 26 min. Darkening of eluting oil was observed at about 5 min later. At this stage, the packing material was already saturated with the oil constituents that have good affinity for it and could no more retain such substances, indicating that the sorption capacity/strength of the system under the operated conditions is within 5 min. It also suggests that improvement on the operating conditions such as pressure (hence, flow rate), packing material's size distribution and volume, and packing length could enhance the sorption effectiveness of the parked bed reactor.

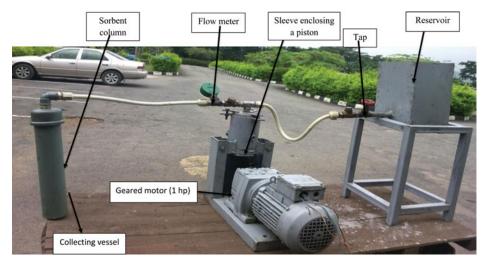


Figure 1. Picture showing the fabricated packed bed reactor employed for the re-refining of the used lubricating oil samples.

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## 3.2. Results of the analyzed physical and chemical parameters

Apart from the iodine values, the values of the parameters including visual color comparison determined for treated oil samples are close to that of virgin oils than used oils. The results are presented in Tables 1 and 2 for the Mobil and the Total oils respectively. The tables also compare the mean values of the results obtained with the available certified (ExxonMobil and Total Quartz) values for the brand (SAE 20W-50).

## 3.2.1. Density

The treated Mobil oils have density values ranging from 0.873 to 0.874 g/cm<sup>3</sup> with mean value of  $0.874 \pm 0.0005$  g/cm<sup>3</sup>, while its used oils have values ranging from 0.878 to 0.883 g/cm<sup>3</sup> with mean value of  $0.881 \pm 0.001$  g/cm<sup>3</sup>. Its virgin oil sample has value of  $0.871 \pm 0.001$  g/cm<sup>3</sup>. The treated Total oils have density values ranging from 0.873 to 0.874 g/cm<sup>3</sup> with mean value of  $0.873 \pm 0.001$  g/cm<sup>3</sup>, while its used oils have values ranging from 0.878 to 0.889 g/cm<sup>3</sup> with mean value of  $0.887 \pm 0.001$  g/cm<sup>3</sup>, while its used oils have values ranging from 0.886 to 0.889 g/cm<sup>3</sup> with mean value of  $0.887 \pm 0.002$  g/cm<sup>3</sup>. Its virgin oil sample has value of  $0.886 \pm 0.001$  g/cm<sup>3</sup>. The high density of the used oil samples is attributed to their relatively high level of impurities. The values for the two brands do not show much difference, indicating similarities in their properties and/or compositions. The slightly higher certified values for the virgin oils (Mobil: 0.890 g/cm<sup>3</sup> at  $27.4^{\circ}$ C; Total: 0.893 g/cm<sup>3</sup> at  $15^{\circ}$ C) than those obtained in this study (Mobil: 0.871 g/cm<sup>3</sup> at  $29^{\circ}$ C; Total: 0.876 g/cm<sup>3</sup> at  $29^{\circ}$ C) may be attributed to the higher temperature ( $29^{\circ}$ C) at which the analyses were carried out in this study, since density decreases with increase in temperature.

#### 3.2.2. Viscosity and viscosity index

The treated Mobil oils have values ranging from 165.36 to 168.65 with mean value of 166.522  $\pm$  1.433 at 40°C and from 19.55 to 19.68 with mean value of 19.625  $\pm$  0.065 at 100°C. The indexes ranged from 134 to 138 with mean value of 136  $\pm$  1.5811. Their used oils had K.V values ranging from 175.48 to 178.64 with mean value of 177.672  $\pm$  1.270 at 40°C and from 20.74 to 20.87 with mean value of 20.808  $\pm$  0.055 at 100°C. Their indexes ranged from 137 to 140 with mean value of 138.20  $\pm$  1.304. The corresponding values for the virgin oil sample (162.43, 18.44, and 127) agreed well with that certified values from ExxonMobil (162, 18.1, and 124) thus affirmed the accuracy of the analysis. Similar trends are observed with the results obtained for the Total oils. A higher V.I indicates a smaller decrease in K.V (and higher thermal stability) with increasing temperature of the lubricant (Singh and Gulati 1987). Thus, the used oil samples would have smallest effect with change in temperature followed by the treated oils. The high K.V of the used oils is an indication they contained higher viscous substances than the treated oils. The results justified that some of the very viscous materials in the used oils have been removed by the PBR.

#### 3.2.3. Flash point (F.P)

The used oils have the lowest mean flash points (Mobil: 188°C; Total: 192°C) while the virgin oils have the highest mean values (Mobil: 209°C; Total: 252°C) among the three set of oils. The low F.P values of the used oils indicate the presence of volatile products and contaminants in the lubricating oils. The appreciably higher certified value for the virgin oil by ExxonMobil (209°C) than observed in this study (200°C) could be linked to different methods of determination. The higher values observed for the treated oils than the used oils as well as the closeness of the values to the virgin oils and certified values show that some volatile constituents of the used oils have been removed by the preheating treatment of the used oils and/or the PBR.

## 3.2.4. Pour point (P.P)

The used oils had relatively high P.P values (Mobil:  $-2.4 \pm 0.548^{\circ}$ C; Total:  $-3 \pm 0.707^{\circ}$ C) while the virgin oils had very low values (Mobil:  $<-15^{\circ}$ C; Total:  $<-15^{\circ}$ C). The treated oil samples had mean value higher than the virgin but lower than that of used oils (Mobil:  $-7.4 \pm 0.89$ ; Total:  $-6.4 \pm 0.89$ ). These results show that the treated oils would flow into the oil pump and into the various engine parts at low temperatures better than the used oils.

| ParameterRangeMean $\pm$ SDMean $\pm$ SDMean $\pm$ SDDensity (29°C) (mg/L)0.873 - 0.8740.873 \pm 0.0010.873 \pm 0.0010.871 \pm 0.0020.871 \pm 0.001Density (29°C) (mg/L)0.873 - 0.8740.873 \pm 0.0010.375 480 - 178.640177.672 \pm 1.270165.243 0 \pm 0.011K.V40°C (cst)19.550 - 168.65019.65.52 \pm 1.603175.480 - 178.640177.672 \pm 1.27016.5.240 \pm 0.002Viscosity index133.790 - 137.860135.948 \pm 1.597137.000 - 140.000138.005 \pm 1.306127.120 \pm 0.002Pour point (°C)201.000 - 197.000138.800 \pm 1.643187.000 - 189.000137.120 \pm 0.002127.120 \pm 0.002Pour point (°C)201.000 - 16.000)-7.400 \pm 0.894(-2.000) - (-3.000)-2.400 \pm 0.548 $\sim -15.000$ Acid no. (mg KOH/g Oil)1.681 - 18241.767 \pm 0.0601.3155 - 3.4883.255 \pm 0.1260.381 \pm 0.002Acid no. (mg KOH/g Oil)1.681 - 18241.767 \pm 0.0601.3155 - 3.4883.255 \pm 0.1260.381 \pm 0.002Aci content (%)1.176 - 1.1871.182 \pm 0.0061.675 - 1.4841.5758 \pm 0.0020.3988 \pm 0.001Refractive index1.483 - 1.4841.485 + 0.0011.485 \pm 0.0011.482 \pm 0.001Refractive index1.483 - 1.4841.485 + 0.0011.485 \pm 0.0011.482 \pm 0.001 |   | Treated oils        | oils                | Used oils           | ils                 | Virgin oil          | ExxonMobil      |
|---|---|---------------------|---------------------|---------------------|---------------------|---------------------|-----------------|
| $ \begin{array}{cccccccccccccccccccccccccccccccccccc$   | Parameter                                     | Range               | $Mean\pmSD$         | Range               | $Mean\pmSD$         | $Mean\pmSD$         | certified value |
| $ \begin{array}{cccccccccccccccccccccccccccccccccccc$   | Density (29°C) (mg/L)                         | 0.873-0.874         | $0.873 \pm 0.001$   | 0.878-0.883         | $0.881 \pm 0.002$   | $0.871 \pm 0.001$   | 0.890 (27.4°C)  |
| $ \begin{array}{cccccccccccccccccccccccccccccccccccc$   | K.V 40°C (cSt)                                | 165.360-168.650     | $166.522 \pm 1.603$ | 175.480–178.640     | $177.672 \pm 1.270$ | $162.430 \pm 0.011$ | 162.000         |
| $ \begin{array}{cccccccccccccccccccccccccccccccccccc$   | 100°C (cSt)                                   | 19.550–19.680       | $19.626 \pm 0.065$  | 20.740-20.87        | $20.808 \pm 0.055$  | $18.440 \pm 0.002$  | 18.100          |
| $ \begin{array}{cccccccccccccccccccccccccccccccccccc$   | Viscosity index                               | 133.790–137.860     | $135.948 \pm 1.597$ | 137.000–140.000     | $138.050 \pm 1.306$ | $127.120 \pm 0.002$ | 124.000         |
| $ \begin{array}{c ccccccccccccccccccccccccccccccccccc$  | Flash point (°C)                              | 201.000-197.000     | $198.800 \pm 1.643$ | 187.000–189.000     | $188.000 \pm 0.707$ | $209.000 \pm 0.112$ | 200.000         |
| 2.805-3.086 2.861 ± 0.125 3.185-3.488 3.265 ± 0.126   1.681-1.824 1.767 ± 0.060 1.683-1.895 1.789 ± 0.082   45.684-48.222 46.306 ± 1.099 43.146-47.848 45.558 ± 1.669 3   1.176-1.187 1.182 ± 0.004 1.675-1.680 1.678 ± 0.002 3   1.483-1.484 1.484 ± 0.001 1.485-1.487 1.485 ± 0.001 0   | Pour point (°C)                               | (-8.000) - (-6.000) | $-7.400 \pm 0.894$  | (-2.000) - (-3.000) | $-2.400 \pm 0.548$  | < 15.000            | -27.000         |
| 1.681-1.824 1.767 ± 0.060 1.683-1.895 1.789 ± 0.082   45.684-48.222 46.306 ± 1.099 43.146-47.848 45.558 ± 1.669 3   1.176-1.187 1.182 ± 0.004 1.675-1.680 1.678 ± 0.002 0 1   1.483-1.484 1.484 ± 0.001 1.485 ± 0.001 0.01 0.002 0  | Acid no. (mg KOH/g Oil)                       | 2.805–3.086         | $2.861 \pm 0.125$   | 3.185–3.488         | $3.265 \pm 0.126$   | $0.842\pm0.002$     | N.A             |
| 45.684-48.222 46.306 ± 1.099 43.146-47.848 45.558 ± 1.669 3   1.176-1.187 1.182 ± 0.004 1.675-1.680 1.678 ± 0.002 0   1.483-1.484 1.484 ± 0.001 1.485 ± 0.001 0.001 0   | Base no. (mg KOH/g Oil)                       | 1.681–1.824         | $1.767 \pm 0.060$   | 1.683–1.895         | $1.789\pm0.082$     | $6.381 \pm 0.003$   | 8.200           |
| 1.176-1.187 1.182 ± 0.004 1.675-1.680 1.678 ± 0.002 0   1.483-1.484 1.484 ± 0.001 1.485 ± 0.001 0 0   | lodine value (g of l <sub>2</sub> /100 g Oil) | 45.684-48.222       | $46.306 \pm 1.099$  | 43.146-47.848       | $45.558 \pm 1.669$  | $38.070 \pm 0.004$  | N.A             |
| $1.483 - 1.484 = 1.484 \pm 0.001$ $1.485 - 1.487 = 1.485 \pm 0.001$ $1$   | Ash content (%)                               | 1.176–1.187         | $1.182\pm0.004$     | 1.675–1.680         | $1.678\pm0.002$     | $0.988\pm0.001$     | N.A             |
|   | Refractive index                              | 1.483–1.484         | $1.484\pm0.001$     | 1.485–1.487         | $1.485\pm0.001$     | $1.482\pm0.001$     | N.A             |

Table 1. Results of the analyzed parameters of the Mobil super oil samples and comparison with certified values.

• SD = Standard deviation; K.V = kinematic viscosity; N.A = Not available.

|                                  | Treated oils        | oils                | Used oils           | ils                 | Virgin oil          | Total Quartz    |
|----------------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|-----------------|
| Parameter                        | Range               | $Mean\pmSD$         | Range               | $Mean\pmSD$         | $Mean\pmSD$         | certified value |
| Density (29°C) (mg/L)            | 0.873-0.874         | $0.873 \pm 0.001$   | 0.884-0.889         | $0.887 \pm 0.002$   | $0.876 \pm 0.001$   | 0.893 (15°C)    |
| K.V 40°C (cSt)                   | 171.880–174.220     | $173.148 \pm 0.958$ | 174.620–75.680      | $175.168 \pm 0.492$ | $169.880 \pm 0.013$ | 171.300         |
| 100°C (cSt)                      | 19.480–19.870       | $19.622 \pm 0.154$  | 19.880–20.320       | $20.118 \pm 0.164$  | $18.690 \pm 0.001$  | 18.800          |
| Viscosity Index                  | 133.790–137.860     | $130.092 \pm 1.548$ | 131.517-135.031     | $133.380 \pm 1.303$ | $123.225 \pm 0.003$ | 123.815         |
| Flash point (°C)                 | 206.000–212.000     | $209.800 \pm 2.490$ | 190.000–194.000     | $192.000 \pm 1.871$ | $252.000 \pm 0.101$ | 240.000         |
| Pour point (°C)                  | (-5.000) - (-7.000) | $-6.400\pm0.894$    | (-4.000) - (-2.000) | $-3.000 \pm 0.707$  | <                   | 24.000          |
| Acid no. (mg KOH/g Oil)          | 1.844–2.203         | $2.881 \pm 0.079$   | 3.168–3.488         | $3.424 \pm 0.143$   | $0.420\pm0.004$     | N.A             |
| Base no. (mg KOH/g Oil)          | 44.214-45.262       | $2.044 \pm 0.172$   | 1.848–2.257         | $2.071 \pm 0.191$   | $7.789\pm0.002$     | 10.000          |
| lodine value (g of l2/100 g Oil) | 1.179–1.188         | $44.686 \pm 0.390$  | 43.712-44.834       | $44.168 \pm 0.413$  | $38.070 \pm 0.003$  | N.A             |
| Ash content (%)                  | 1.483–1.485         | $1.185\pm0.004$     | 1.675–1.692         | $1.683 \pm 0.007$   | $0.991 \pm 0.003$   | N.A             |
| Refractive index                 | 1.844–2.203         | $1.484\pm0.001$     | 1.484–1.485         | $1.485\pm0.001$     | $N.483\pm0.001$     | N.A             |
|                                  |                     |                     |                     |                     |                     |                 |

Table 2. Results of the analyzed parameters of the total quartz oil samples and comparison with certified values.

• SD = Standard deviation; KV = kinematic viscosity; N.A = Not available.

#### 3.2.5. Iodine value (I.V)

From Tables 1 and 2, the treated oils had highest mean I.V (g I<sub>2</sub>/100 g oil) (Mobil:  $46.306 \pm 1.099$ ; Total:  $44.686 \pm 0.390$ ), followed by the used oils (Mobil:  $45.558 \pm 1.669$ ; Total:  $44.168 \pm 0.413$ ) and lowest in the virgin oil sample (Mobil:  $38.070 \pm 0.004$ ; Total:  $38.274 \pm 0.003$ ). This implies that the treated oils had highest degree of unsaturation. It could be that more of the saturated compounds were better removed than the saturated compounds by the PBR treatment or that the preheating of the treated oils samples might had resulted in the rearrangements and/or cracking of some components leading to formation of unsaturated compounds. Furthermore, a good proportion of the volatile components of the used oil might be saturated compounds which were removed via vaporization during the preheating treatment leading to higher concentrations of unsaturated fractions in the treated oils.

## 3.2.6. Total acid number (TAN) and total base number (TBN)

The mean TAN (mg (KOH)/g sample) obtained for the used oils (Mobil:  $3.265 \pm 0.126$ ; Total:  $3.424 \pm 0.143$ ) was higher than those of the virgin oils (Mobil: 0.842; Total: 0.42). This could be 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 (Fox 1991). The mean values for the treated oils (Mobil:  $2.861 \pm 0.125$ ; Total:  $2.881 \pm 0.079$ ) were lower than those of used samples. On the other hand, the mean TBN values (mg (KOH)/g sample) followed the opposite trend, viz virgin (Mobil: 6.181; Total: 7.783); used (Mobil:  $1.789 \pm 0.082$ ; Total:  $2.071 \pm 0.191$ ) and treated (Mobil: 1.767  $\pm$  0.060; Total: 2.044  $\pm$  0.172) oils which connotes depletion of basic components during use and removal of some base components by the treatment. As engine oils are subjected to elevated temperatures, the process of oxidation occurs. Oxidation leads to the formation of organic acids in the lubricating oil. The presence of oxygen, in most engine oils environments, and hydrocarbons that make up the base oil lead to some reactions. This reaction may lead to the formation of carbonyl-containing products. Subsequently, these undergo further oxidation to produce carboxylic acids, which results in an increase in the TAN value. These products tend to be neutralized by basic compounds fortified in the engine oil. In addition, with time and elevated temperature, the oxidation products formed then polymerize leading to precipitation of sludge which decreases the efficiency of engine oil and causes excessive wear (Fox 1991).

#### 3.2.7. Ash content

As expected, generally, the used oils had the highest mineral content that could be the reflection of higher levels of presence of wear metals, foreign particles, and dirt in the used oils than they were in the treated or the virgin oils.

| Parameter         | TM/UM | TT/UT | UM/VM | UT/VT | UM/UT | TM/TT |
|-------------------|-------|-------|-------|-------|-------|-------|
| Deneitre          | (D    | (D    | (D    | (D    | (D    | NCD   |
| Density           | SD    | SD    | SD    | SD    | SD    | NSD   |
| KV @ 40°C         | SD    | SD    | SD    | SD    | SD    | SD    |
| KV @ 100°C        | SD    | SD    | SD    | SD    | SD    | NSD   |
| Viscosity index   | SD    | SD    | SD    | SD    | SD    | SD    |
| Flash point       | SD    | SD    | SD    | SD    | SD    | SD    |
| Pour point        | SD    | SD    | SD    | SD    | SD    | NSD   |
| Total acid number | SD    | NSD   | SD    | SD    | SD    | NSD   |
| Total base number | NSD   | SD    | SD    | SD    | SD    | SD    |
| lodine value      | SD    | SD    | SD    | SD    | SD    | SD    |
| Ash content       | SD    | SD    | SD    | SD    | NSD   | NSD   |
| Refractive index  | NSD   | NSD   | NSD   | NSD   | NSD   | NSD   |

Table 3. Result of *t*-test analysis of the analyzed parameters.

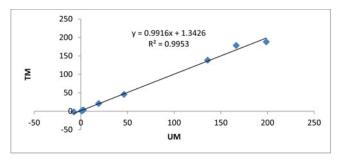
 TM = Treated Mobil; UM = Used Mobil; TM = Treated Mobil; VM = Virgin Mobil; TT = Treated Total; UT = Used Total; VT = Virgin Total; SD = Significant difference; NSD = No significant difference; KV = Kinematic viscosity. 1720 👄 A. F. ADEYEMI ET AL.

#### 3.2.8. Refractive index (R.I)

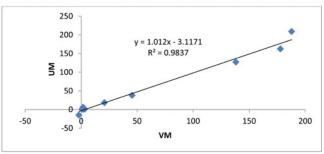
The R.I values of the oil samples were very similar; the mean values for used oils (Mobil: 1.4851; Total: 1.4846) were higher than those of the virgin oils (Mobil: 1.4822; Total: 1.4826). The treatment reduced the R.I of the used oils by a small fraction (Mobil: about 0.081%; Total: about 0.034%). The lower values of the R.I in virgin oils of the two brands indicate the presence of more paraffin material than the other set of samples, while higher values in used oils indicate the presence of more aromatic compounds than both the treated and virgin oils (Riazi and Roomi 2001). The used oils were most likely to have more of additives like polymers, polar organic compounds, organic compounds, metals, copolymers of olefins, and hydrogenated diene styrene copolymers than the other set of samples. These components increase the molecular mass of the base oil and consequently its refractive index.

## 3.3. Statistics results

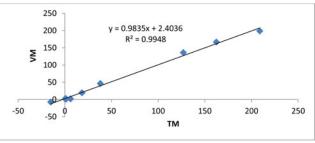
Table 3 shows the results of the *t*-test analyses (at 95% confidence level) done on the values of some of the analyzed physicochemical parameters for the following pairs of the lubricating oil samples: treated Mobil and used Mobil (TM/UM), treated Mobil and treated total (TM/TT), used Mobil and virgin Mobil (UM/VM), used Mobil and used Total (UM/UT), treated Total and used Total (TT/UT), used Total



(a) Treated Mobil oil versus used Mobil oil samples



(b) Used Mobil oil versus virgin Mobil oil samples



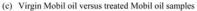
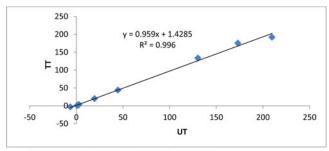
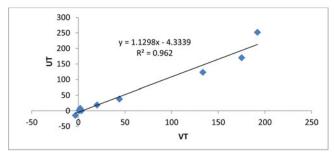


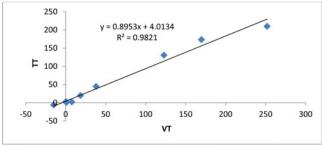
Figure 2. Cross-plots of the various possible pairs of the Mobil oil samples using the mean values of the analyzed parameters as variables.



(a) Treated Total oil versus used Total oil samples



(b) Used Total oil versus virgin Total oil samples



(c) Virgin Total oil versus treated Total oil samples

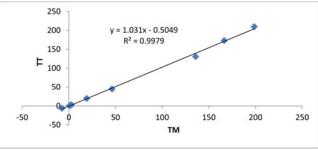
Figure 3. Cross-plots of the various possible pairs of the Total oil samples using the mean values of the analyzed parameters as variables.

and virgin Total (UT/VT). It is observed that there was significant difference between the mean values of each of the analyzed parameters (except R.I) of the used and the virgin oils, irrespective of the brand. This explains that, apart from the R.I, the usage of the oils have strong effect on these properties. It is inferred that there exist significant difference in the mean values of the analyzed physicochemical parameters between TM and UM except for TBN and R.I and also between TT and UT except for TAN and R.I. Similar trend was observed in the comparison of the mean values of these parameters between TT and UT except for TAN and R.I. This shows that the treatment has pronounced impacts on many of the physico-chemical properties of the oil samples. Apart from K.V at 40°C, VI, F.P., TBN, and I.V, there was no significant difference between the mean values of each the analyzed physicochemical parameters in TT and TM sample. This shows that the treated oils have close mean value in some of the analyzed parameters buttressing the effect of the treatment in the used lubricating samples irrespective of the brand. The significant difference observed in a good number of these properties may be attributed to the difference in chemical constitutions of the different brands. This is also affirmed in the comparison between UT and UM where all except the ash content and R.I. differ significantly.

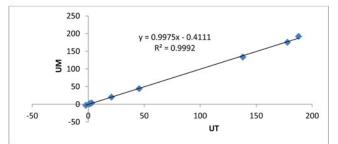
Figures 2, 3, and 4 show the cross-plots of the various possible samples combination for the Mobil, Total, and Mobil-Total oils using the mean values of the Physico-chemical parameters as variables.

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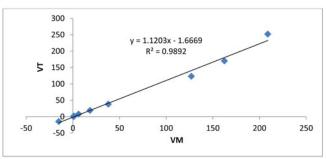
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(a) Treated Total oil versus treated Mobil oil samples



(b) Used Mobil oil versus used Total oil samples



(c) Virgin Total oil versus virgin Mobil oil samples

Figure 4. Cross-plots of the similar sample pairs from the Mobil–Total oil samples using their mean values of the analyzed parameters as variables.

The results indicate that there are statistically significant and very strong positive linear correlations between the various possible pair of samples as their coefficient of correlation ( $R^2$ ) values ranged from 0.9620 to 0.9992, suggesting similar factors influencing their values and similar trend in the variation their values in the oil samples. The high values also suggest that any variation in the conditions of treatment has similar effects on the properties of the treated oils, independent of their brands. Consequently, it is confirmed that the PBR had similar treatment effects on the ULOs from different brands.

## 4. Conclusion

The results obtained showed that the values of most of the analyzed parameters in the treated oils were close to those of virgin oil samples. It is observed that the efficiencies of the PBR in recycling the used oils from both brands were similar. The t-test results confirmed that, with the exception of refractive index, the mean values of the analyzed parameters in the treated oils were significantly differed from those of the used oils. Various results of cross plots had  $R^2$  values greater than 0.96, indicating high linear relationships of the impact of the treatment on the various oil samples. All these give credence to the efficacy of the fabricated PBR for the treatment of ULOs.

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