African Journal of Pure and Applied Chemistry Vol. 5(7), pp. 194-197, July 2011
Available online at http://www.academicjournals.org/AJPAC
ISSN 1996 - 0840 ©2011 Academic Journals

Full Length Research Paper

Classification of lubricants using neutron slowing-down technique

K. F. Akpan[1], M. Y. Onimisi[1] and S. A. Jonah[2]*

[1]Department of Physics, Nigerian Defence Academy, P. M. B. 2109, Kaduna, Kaduna State, Nigeria.
[2]Centre for Energy Research and Training, Ahmadu Bello University, P. M. B. 1014, Zaria, Kaduna State, Nigeria.

Accepted 17 May, 2011

The neutron slowing down technique employing a facility based on a 1 Ci ^{241}Am-Be neutron source base and ^{3}He neutron detector has been used to classify lubricants. The facility utilizes the fast neutron slowing down technique in which the intensity of reflected thermalized neutrons is proportional to the hydrogen content of the sample exposed to it. A popular market product of different grades was analyzed in this work. Using 600 cm^{3} samples, the standard hydrocarbons calibration line plotted was used to determine hydrogen contents in 5 grades of lubricants. Results show that the total hydrogen weight percent in these various grades of lubricants range between 10 to 12% and increases as the quality decreases.

Key words: Neutron slowing down, total H content, lubricants.

INTRODUCTION

Lubricants are among the most important products derived from crude oil. Lubricating oils are essential for the proper functioning of nearly every machine, equipment and instrument. But the term, “Lubricating” oil, actually describes only one of its five essential functions, namely: lubricant, coolant, carrier, protectant, and sealant (Berker, 1995).

As a lubricant, the oil keeps parts of the machine from touching each other, and reduces friction in moving parts like bearings. As a coolant, oil carries away the heat of friction and gas compression to keep the machine at a moderate temperature. As a carrier, oil removes wear particles and keeps them and other particles in suspension, minimizing damage. The particles are eventually removed by filtration or oil changes. As a protectant, the oil protects the machine from corrosion. As a sealant, the oil improves efficiency, by creating a deep ultimate vacuum in devices like vacuum pumps.

Various compounds, known as additives in general are used to improve the properties of lubricating oils. Among such properties is the oxidation stability, viscosity index, pour point, rust inhibition and foaming.

In Nigeria, a wide variety of lubricants are available. They are branded or better still “sealed oils” available in standard 4 L gallons and those of unbranded status which are readily available in various road junctions and points, and normally sold by direct measurement. The present method for the classification of lubricants in Nigeria is based on wet chemical protocols, which is destructive and entails heating up to 1000°C and may last for several hours.

Lubricants are by-products of crude oil and are therefore composed of hydrogen and carbon as major elements. Because of their intrinsic properties, neutrons are known to act like a ´lens´ and are useful for providing information at microscopic and macroscopic levels in a wide variety of fields, especially in matrices containing hydrogen. Buczko et al. (1975) developed the neutron reflection method for determination of bitumen content in asphalt concrete. Using a sample of approximately 100 cm^{3}, they completed the test in 20 min, as compared to a wet chemical analysis of 1.5 to 10 h. Subsequently, Al-Jobori et al. (1978) used a similar method to determine the hydrogen content in crude oil from different sources. Each measurement was performed for only 5 min with a high degree of repeatability and at the same time reducing the usual high capital demand in equipment. Subsequently, the method based on different experimental arrangements has been used by other workers (Akaho et al., 2001; 2002; Csikai and Doczi, 2007; Jonah and Umar

*Corresponding author. E-mail: jonahsa2001@yahoo.com.

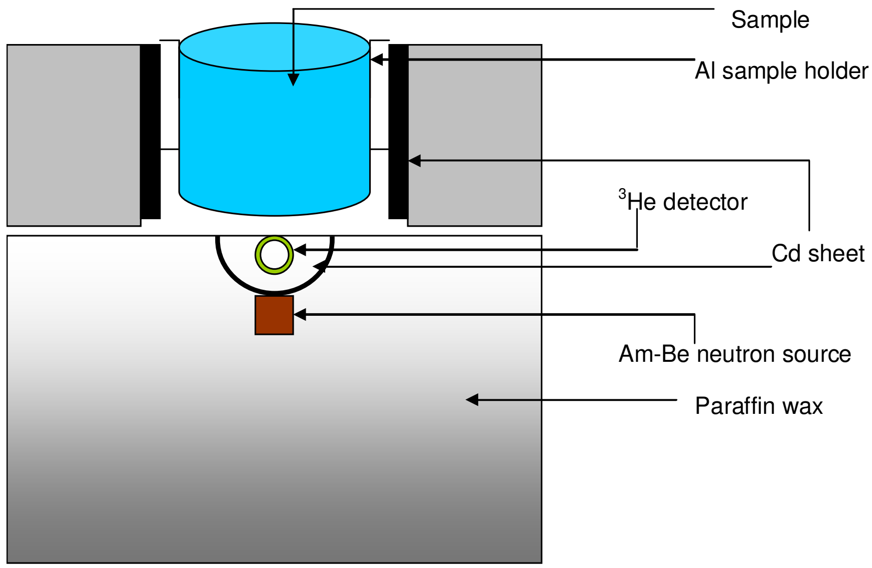

Figure 1. The experimental facility for the classification of lubricants by neutron slowing down method.

2004; Jonah et al., 1999, 2011). In this work, a set-up utilizing a 1 Ci ^{241}Am-Be neutron source and a ^{3}He neutron detector has been used to classify different grades of a popular market product of lubricants used in Nigeria.

MATERIALS AND METHODS

The experimental set-up is shown in Figure 1 and consists of a source holder housing a 1 Ci ^{241}Am-Be neutron source and ^{3}He neutron detector. The source holder and the detector are embedded in a thick cylindrical paraffin block, which serves as a moderator and provides personnel shielding against radiation hazards. The sample holder is a cylindrical aluminum container of 10 cm diameter and 15 cm height, which can accommodate samples of up to 600 cm^3 in volume. This volume corresponds to a sample height of 8 cm in a sample holder of 10 cm, which has been experimentally validated as the saturation height for the experimental setup. The cylindrical surface of sample holder is shielded with 1 mm thick Cd sheet to prevent thermal neutrons from reaching the sample. The Am-Be neutron source is also shielded with 1 mm thick Cd sheet and as can be seen, there is no Cd sheet between the side of sample holder facing the detector and the detector to enhance the signal-to-noise ratio. The source, sample holder and detector are embedded in a container filled with paraffin wax in a reproducible geometry. The ^{3}He detector working at a bias voltage of 1.8 kV is connected via an amplifier to a counter/timer module. The neutron counts reaching the detector are recorded in time intervals of 10 s and the average count rate are obtained from 10 measurements. The neutron count rate reaching the detector depends on the weight percent of total H content in the sample according to the expression given in Equation 1.

$$\eta = \frac{I - I_0}{\rho . I_0} \qquad (1)$$

Where, η = Reflection coefficient, $I =$ Counts with sample, $I_0 =$ Counts without sample, $\rho =$ Physical density of sample.

According to Equation 1, the neutron reflection coefficient η, is the normalized count rate of neutrons reaching the detector and is proportional to the total H weight percent of sample as given in Equation 2.

$$\eta \propto H(wt\%) \qquad (2)$$

In order to determine the total H (wt %) of the lubricants, hydrocarbon standards of well-known total H (wt %) were used to establish a calibration line. The parameters of the hydrocarbon standards used in this work are presented in Table 1. A plot of the neutron reflection parameter η, as function of the total H (wt %) is shown in Figure 2. The Calibration line obtained can be represented by the expression given by Equation 3.

$$\eta = mH(wt\%) + C \qquad (3)$$

Where, m = Slope of the plot, C = Intercept, H (wt %) = Hydrogen weight percent.

The same experimental procedure was used to determine the neutron reflection parameter, η of the five grades of lubricants. The total H (wt%) for each of the samples was deduced from the expression given by Equation 3 and are presented in Table 2. The lubricants Lub-01, Lub-02 and Lub-03 are high grade sealed lubricants in the order of decreasing quality as specified by the manufacturer. Lub-04 is an un-sealed base oil from the same manufacturer purchased from the road side and Lub-05 is a fresh Lub-01, which has been used in a vehicle.

RESULTS

As shown in Figure 2, the calibration line indicate that the slope m = 0.079 ± 0.025 cm^{3}g^{-1} H^{-1} (wt %) and intercept C = 0.375 ± 0.277 cm^{3}g^{-1}, which represents the effect of the matrix. These data in combination with the neutron reflection coefficients,η, of the respective lubricants to from high

Table 1. Measured and calculated parameters of hydrocarbon standards.

Sample	Physical density $\rho (g/cm^3)$	Reflection parameter η (cm^3/g)	Hydrogen height H (wt %)
C_6H_6 (Benzene)	0.879	0.99±0.07	7.69
C_7H_8 (Toluene)	0.866	1.13±0.06	8.70
$C_6H_4(CH_3)_2$ (Xylene)	0.864	1.17±0.05	9.43
$(CH_3)_2CO$ (Acetone)	0.792	1.25±0.04	10.34
$CH_3CH(OH)CH_2OH$ (Propylene glycol)	1.044	1.16±0.05	10.53
H_20 (Water)	1.000	1.15±0.06	11.11
C_2H_50H (Ethanol)	0.790	1.33±0.04	13.04
CH_2 (Paraffin oil)	0.88	1.45±0.04	14.29
C_6H_{14} (n – Hexane)	0.66	1.79±0.03	16.28

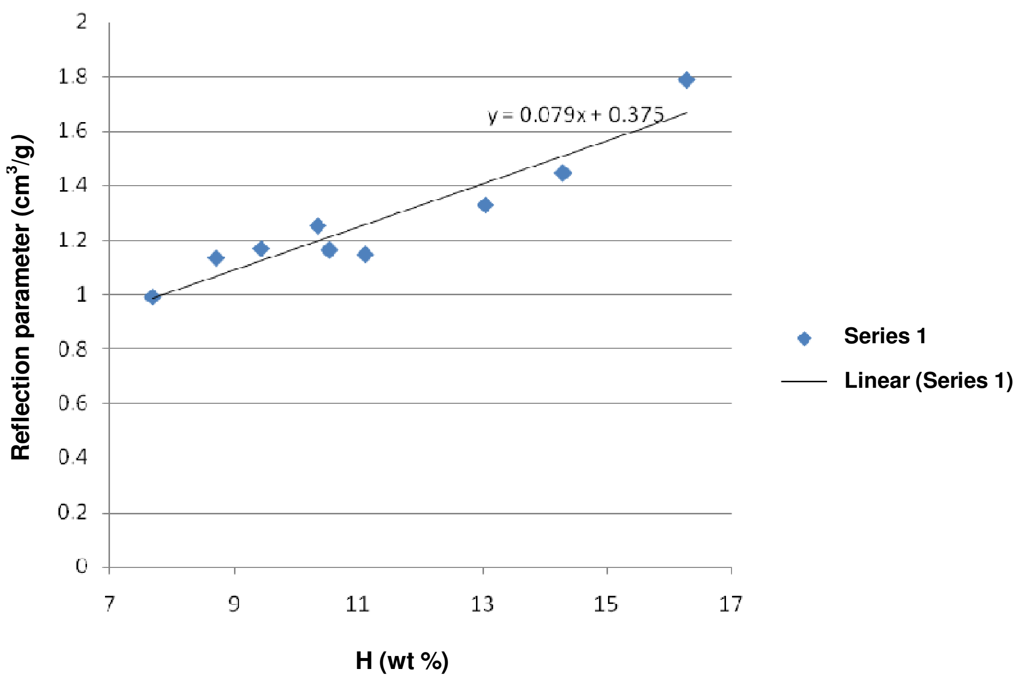

Figure 2. Calibration line for determining hydrogen content of product.

from high grade lubricants to lower grade ones. The H (wt %) of used lubricant was found to be substantially higher than the fresh ones. This can be as a result of thermal degradation and the breakdown of lubricant due to contact with oxidizing agents. As oxidation increases, the oil gets darker and loses its lubricating quality.

DISCUSSION

The study employed neutron slowing-down technique, which is fast and non destructive when compared with other chemical methods. Standard laboratory grade hydrocarbons were used to establish a calibration line for the quantification of total hydrogen content of the lubricants. Initially, the hydrogen weight percent H (wt %) of the standard samples were deduced directly from their chemical formulas. The uncertainties associated with the data of H (wt %) were obtained from the standard errors of five count rate for each of the samples. Three out of the five lubricants used in this investigation are sealed engine oil of a popular brand, one is base oil purchased from retail outlet, and the fifth is a spent lubricant.

Conclusion

The main interest of this study was to classify lubricants in Nigerian market. In this regard, a more direct way of doing this classification using total hydrogen content which

Table 2. Measured reflection parameter η and deduced H (wt %) of samples.

Lubricant	Physical density ρ (g/cm^3)	Reflection parameter η (cm^3/g)	Deduced H (wt %)
Fresh Lub-01	0.873	1.23 ± 0.09	10.87 ±0.05
Fresh Lub-02	0.915	1.29 ± 0.09	11.57 ± 0.05
Fresh-Lub-03	0.903	1.31± 0.10	11.71 ± 0.05
Fresh Lub-04	0.936	1.30 ± 0.09	11.84 ± 0.05
Spent Lub-01	0.919	1.39 ± 0.11	12.83 ± 0.05

which is a key known constituent element of lubricants was developed. Further investigations are on-going to determine the useful life of lubricants in vehicles. Results of these investigations can provide information on the optimum mileage/distance for the replacement of a given product to prolong the life spans of vehicle engines.

REFERENCES

Akaho EHK, Jonah S A, Dagadu CPK, Maakuu BT, Anim-Sampong S, Kyere AWK (2001). Thermal neutron reflection method for measurement of total hydrogen contents in Ghanaian petroleum products. Appl. Radiat. Isot., 55: 617-622.

Akaho EHK, Jonah SA, Nyarko BJB, Osae S, Maakuu BT, Serfor-Armah Y, Kyere AWK (2002). Simultaneous use of neutron transmission and reflection techniques for the classification of crude oil samples. Appl. Radiat. Isot., 57: 831-836.

Al-Jobori SM, Szegedi S, Buczko CsM (1978). Determination of hydrogen content in crude oil by the neutron reflection method. Radiochem. Radioanal. Lett., 33: 133-138.

Csikai J, Doczi R (2007). A comparison of the neutron thermalization and reflection methods used for bulk hydrogen analysis. Appl. Radiat. Isot. 65: 764-768.

Berker Technical Document: Lubricating Oils for Berker Dekatorr Pumps; part 1 (1995).

Buczko M, Dezso Z, Csikai J (1975). Determination of the bitumen content in asphalt concrete using a neutron reflection method. J. Radioanal. Nucl. Chem., 25: 179-183.

Jonah SA, Zakari II, Elegba SB (1999). Determination of the hydrogen content of oil samples from Nigeria using an Am-Be neutron source. Appl. Radiat. Isot., 50: 981-983.

Jonah SA, Umar IM (2004). Estimating adulteration of petroleum-based fuels using neutron reflectometry method. Appl. Radiat. Isot., 71: 889-890.

Jonah SA, Okunade IO, Abolude O, Onyike E, Inuwa IM (2011). Bulk H analysis using neutrons for routine quality control of cassava and products. Appl. Radiat. Isot., 69: 988-990.