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STUDIES OF SOME ENGINE OILS MANUFACTURED IN NIGERIA

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ABSTRACT

Nigeria blending plants were few and mostly dominated by the multinationals. Lubricants produced in the past were of high quality but expensive. With time the lubricant market boomed and more investors entered the operations leading to quality challenges and adulterated products began to litter the market with some manufacturers' addresses becoming elusive. In the present work some physical and chemical properties of selected multi- and mono-grade lubricants manufactured in Nigeria have been investigated. The values of viscosity in this investigation were found to decrease gradually with temperature for both multigrade and monograde lubricants. For most lubricants a high flash point is desirable to prevent ignition in a motor. Mobil super (20W50), Conoil (20W50) and Total (SAE40) were found to have high flash points, while Oando SMOR (SAE40), Texaco Havoline (20W50) and AP Super V (20W50) have low flash points. The general specification for density of petroleum lubricants is 0.85-0.95. This investigation has confirmed that Nigerian manufactured lubricants are of good quality with densities above 0.85. The deposition of the sludge increases the exhaust back pressure and reduces over time the fuel economy. Verification of sludge formation is a guide to the formation of carbon deposits of lubricating oil in the cylinders of internal combustion engines. In this test low values of carbon residues were obtained indicating very small sludge formation from the lubricants. However Amasco (20W50) and Total (20W50) produced high carbon residues of 2.8800 and 1.5018 respectively. The Total Base Number (TBN) which is a measure of the reserve alkalinity of oil is another manipulated property and it is the lubricants acidity. The values from this work indicate satisfactory ability of the lubricants to neutralize any acid formed during operation and minimize oxidation. The trace element is a measure of the additives in the lubricant and their functions include keeping the engine clean, extension of the range of applicability and useful life of the lubricant. Na, K and Ca which serve these functions are found in appreciable quantities in this investigation. The hydrogen content of oil indicates the level of contamination by other lipids and the parity with lubricants processed elsewhere. The values ranged between 11.0 to 12.30 by weight percent and compares with the results of other investigators.

1.0 INTRODUCTION

The purpose of proper lubrication is to provide a physical barrier (oil film) that separates moving parts, reducing wear and friction. Many liquids, including water, have been used as lubricants to minimize the friction, heat and wear between mechanical parts in contact. Nowadays lubricating oil, or lube oil, is mostly used because of its wide range of possible applications, the basic categories being mineral (refined from naturally occurring petroleum, or crude oil) and synthetic which is manufactured poly-alpha-olefins or hydrocarbon- based poly-glycols (Nadkarni, 1991). Crude oil has the advantage of availability and hence inexpensive and could be produced in a wide range of viscosities. Synthetic lubricants are usually formulated for specific applications especially where the lube oil must be fire resistant. Early lubrication began with animal fats and oils and slowly evolved to petroleum based oils. Early processes such as acid treating and solvent extraction improved the quality of base oils by removing some of the contaminants from the oil. Later processes like hydro-treating, catalytic hydro-cracking, catalytic de-waxing and modern wax hydro-isomerization transformed feed molecules into molecules with improved lubricating properties (Kramer et al, 2001).

Lube oils are just one of many fractions or components that can be derived from raw petroleum, which emerges from an oil well as yellow-to-black, flammable, liquid mixture of thousands of hydrocarbons. The nature and percentage of hydrocarbons vary widely due the different rates at which organic material decomposed in various places. Also the physical and chemical characteristics of the crude oils extracted from different places vary. For example, the overall specific gravity of crudes ranges between 0.80 to 0.97

grams/millimeter (Nadkarni, 1991) and depends on the refining method and the additives present.

Common additives include metals such as lead or metal sulphide, which enhance the lube's ability to prevent galling and scoring when metal surfaces come in contact under extremely high pressures. High-molecular weight polymeric is another common additive which improves viscosity, counteracting the tendency of oils to thin at high temperatures. Nitrosamines are employed as antioxidants and corrosion inhibitors to neutralize acids and form protective films on metal surfaces. Other functions of additives include protection of metal surfaces, extend the range of lubricant applicability and extend the lubricant's life.

For manufacture, lube oil is extracted from crude oil and allowed to undergo a preliminary process or sedimentation before it is pumped into fractionating towers constructed from high grade steels to resist the corrosive compounds present in crude oils, and fitted with ascending series of condensate collecting trays. The thousands of hydrocarbons in crude oil are separated from each other by fractional distillation. As the vapors rise up through the tower, the various fractions cool, condense and return to liquid form at different rates depending on their boiling points (the higher the boiling point of the fraction the higher it rises before condensing). Natural gas reaches its boiling point first, followed by gasoline, kerosene, fuel oil, lubricants and tars. The lube oil that has been collected from the fractionating towers is filtered to remove impurities such as aromatics that affect the lube's viscosity. Finally additives are mixed with the oil to give it the desired physical properties; and subjected to various quality control tests that assess its viscosity, specific gravity, color, flash and fire points, before being packaged for shipping,

Most applications of lube oils require that they be non-resinous, pale colored, odorless and oxidation resistant. The lube's color indicates the uniformity of a particular grade or brand. The oil's flash and fire points vary with the oil's origin. The flash point is the temperature to which oil has to be heated until sufficient flammable vapor is driven off so that it will flash when brought into contact with a flame. The fire point is the higher temperature at which the oil vapor will continue to burn when ignited.

Engine oils are classified by viscosity and performance according to specifications established by the Society of Automotive Engineers (S.A.E). Viscosity or lube oil's resistance to flow at specific temperatures and pressures is the single most important property of a lubricant. The application and operating temperature range are key factors in determining the viscosity of oil. For example if the oil is viscous, it offers too much resistance to the metal parts moving against each other. On the other hand, if not viscous enough, it will be squeezed out from between the mating surfaces and will not be able to lubricate them sufficiently. The performance factors include wear prevention, oil sludge deposit formation and oil thickening.

In the present work engine oils from different manufacturers are subjected to some of the tests to verify their performance standards. In 2000 Dauda and Obi determined some properties of engine oils manufactured in Nigeria. Obi et al, (2010) also verified the performance standards further in a paper titled, 'Conformity to specifications of properties of engine oils manufactured in Nigeria', and found that progress had been made in maintaining standards by some manufacturers. The present work is a longitudinal follow up using improved measuring equipment.

2.0 MATERIALS AND METHOD

2.1 Materials and Equipment

The samples of automobile lubricants produced in Nigeria which were analyzed in this work are stated below;

Total SAE 40 (mono grade)
Total 20W50 (multi grade)
Total 15W50 (synthetic)
Mobil HD SAE 40(mono grade)
Mobil super 20W50 (multi grade)
Oando SMOR SAE 40(mono grade)
OandoOleum20W50 (multi grade)

Conoil20W50 (multi grade)
Amasco20W50 (multi grade)
Texaco Havoline20W50 (multi grade)
A-Z 20W50 (multi grade)
AP Super V 20W50 (multi grade)
These lubricants (multi grade and mono grade) were randomly bought sealed from the market and tested for the different properties mentioned above. The properties measured are viscosity, density, flash and fire points, acidity, carbon residue, hydrogen content and trace elements.

2.2 Measurement of Viscosity

The viscosity was determined using Brookfield syncro-electric viscometer, which employs a rotating disk or spindle driven by a constant speed synchronous motor. The drive shaft is connected to the spindle shaft through a torsion spring which provides the restoring force to balance the viscous resistance to rotation of the spindle. The application of the restoring force is accompanied by an angular deflection of the spindle shaft relative to the drive shaft. When the spindle head is dipped into the lube oil sample, there is viscous drag which opposes the rotation of the motor. This drag causes the deflection of the pointer on the dial. In other words, as the spindle rotates, it experiences a torque due to friction which is a measure of the viscosity. This test was carried out at room temperature. The values were computed from the expression

$$Dynamic\ viscosity = \frac{\text{force/sheared area}}{\text{velocity/film thickness}} = \frac{\text{shear stress}}{\text{shear rate}}$$

Oil that exhibit a viscosity which varies with changing shear rates in the above equation is called a non-Newtonian oils, multi-viscosity graded oils formulated with polymeric additives are good examples.

2.3 Measurement of Density

The picometer or density bottle is a flat bottomed corked bottle of designed capacity (standard density bottle by Pyrex, England). This employs a constant volume variable weight method for determining accurately the density of the fluids. It involves weighing the corked bottle when empty, W_1 and subtracting this from the weight when filled with the sample, W_2. The density was computed from the equation given by: .

$$\rho = \frac{W_3}{V}\ (g/cm^3)$$

where,
V = Volume of Bottle = 25cm^3
W_1= mass of empty bottle
W_2 = mass of bottle + sample
W_3 = $W_2 – W_1$ = Mass of sample

2.4 Measurement of Flash Point

The Cleveland open cup apparatus was used to determine the flash point of the samples. The apparatus consists of a test cup, heating plate, test flame applicator, heater and thermometer. The test cup was filled with the sample until the top of the meniscus of the test specimen is exactly at the filling mark and then placed on the centre of the heater. Heat was applied initially at such a rate that the temperature indicated by the measuring device increases from 14 to 17°C/min. The test flame was then applied when the temperature was approximately 28°C. Then the temperature for each sample was taken every 5°C, to 100°C for multi grade and 80°C for mono grade until a flash was noticed. The temperature at the flash is the flash point.

2.5 Measurement of Acidity

A solvent mixture of 25cm^3 95% ethanol and 25cm^{3}diethylether was neutralized with 0.1M ethanolic acid using phenolphthalein as indicator. Four grams of the lube oil sample was dissolved in the neutralized solvent mixture and titrated with 0.1M KOH. The acidity was then computed from the equation:

$$Acidity = \frac{M \times T \times V}{W}$$

where,
M = Molecular Mass of KOH
T = Molarity of ethanolic acid
V = Volume of KOH used
W = Weight of sample taken

2.6 Measurement of carbon residue/sludge

This test is a guide to the formation of carbon deposits of lubricants in the cylinders of internal combustion engines. The deposit varies with the type of engine, refining process, and the viscosity of oil. The apparatus used for this test is the *Canradson* apparatus and consists of an insulator with a hollow sheet-metal box containing sand. The residue was determined by heating 10g of oil sample in a cruciblefora short while until vapors are emitted. When the vapor emission ceases, the bottom plate is held *at a cherry red heat* for seven minutes. The crucible is then allowed to cool. The carbon residue is weighed and calculated as a percentage of the original weight of sample (10g).

2.7 Measurement of trace elements

This method was used by Ojeka and Ayodele (1995) and involves the dilution of the sample with kerosene followed by aspiration into the atomic absorption. The flame AAS (Atomic Absorption Spectrum) of Model number *ALPHA 4AAS and PFP 7 JENWAY* was used to determine trace elements in the engine oils. Twenty cubic centimeters ($20cm^3$) of each sample was mixed with nitric acid and *HCl* at a ratio of 3 to 9 using *perchloric* acid to enhance the dissolution. After mixing, the sample was heated till the color of the gas changes from yellow to white. The sample was then filtered and mixed with $100cm^3$ of distilled water. This prepared sample was analyzed with the AAS.

2.8 Measurement of hydrogen content

The Principle of the method is based on the fact that the energy loss of fast neutrons through interaction with the atoms of the medium strongly depends on the mass number of the colliding partners (Obi and Jonah, 1999). The 1 Ci Am-Be source of neutrons was placed\in a paraffin wax of 30cm diameter and the emitted neutrons are slowed down and reflected when they strike the sample (See figure 1). In order to reduce the background of slow neutrons from the shield, the sample is covered with a cadmium layer except for the side facing the source and detector. Counting was carried out by two *BF3* counters each 20 mm diameter and 80 mm long. The background radiation was measured when no sample was used. Then the thermal neutron intensity of a number of organic liquids and water were measured for calibration purposes.

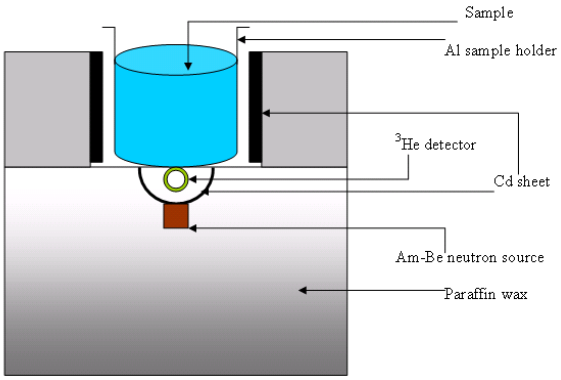

Fig1. The Experimental facility for determination of hydrogen content of the lubricants

The same measurement was made for all the lubricant samples, each of the measurements being performed three times and the average used to compute the increase in thermal neutron intensity. The increase in thermal neutron intensity due to the presence of the samples, (I - I_o) was corrected with the density ρ, of the sample to obtain:

$$\eta = \frac{1}{\rho}\frac{I-Io}{Io}$$

Where, I and I_o are the count rates with and without the samples respectively, and η and ρ are the corrected thermal neutron intensity and density of sample respectively.

Table 1: Calibration Curve data for hydrogen content determination

Sample	ρ g/cm^3	Count n/s	η cm^3/g	H w%
Xylene $C_6H_4(CH_3)_2$	0.864	1221.6	0.98549785	9.43
Water H_2O	1	1362.2	1.06456502	11.11
Methanol CH_3OH	0.7918	1274.2	1.176043534	12.50
Ethanol C_2H_5OH	0.79	1314.2	1.255462914	13.04
N-hexene C_6H_{14}	0.66	1334.2	1.548678663	16.28

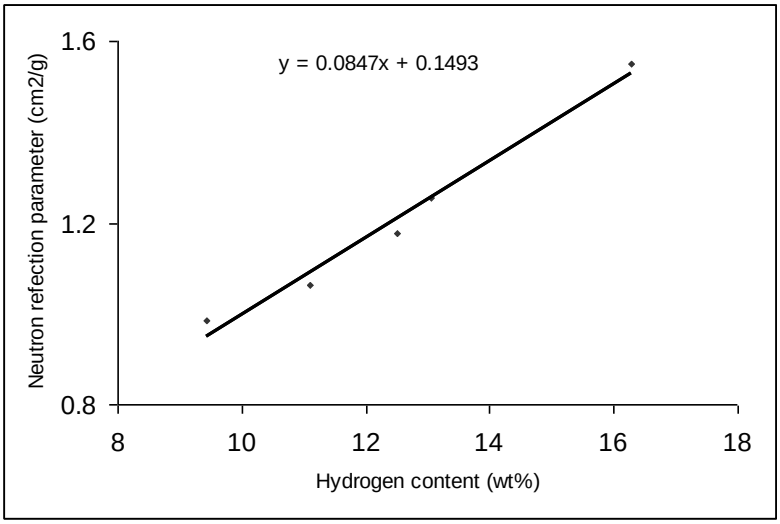

Fig 1: Calibration line for hydrogen determination

30 RESULTS AND DISCUSSIONS

Table 2:Variations of the viscosities with temperature of the multi-grade oil samples

Temp (oC)	Viscosity (Cp.m Pas, 20W50 grades								
	Total (15W50)	A-Z (20W50)	Total (20W50)	Mobil Super (20W50)	AP SuperV (20W50)	Oando (Oleum)	Conoil	Amasco	Texaco (Havoline)
28	392	397.8	314	381.6	356	363	344	310	392
40	122.9	265.69	121	146.0	121.34	126.31	109.43	159.2	167.4
50	90.3	111.96	85.50	93.0	91.4	93.13	87.34	109.3	106.0
60	74.7	88.69	69.06	78.7	75.05	76.31	80.43	89.4	82.16
70	62.8	73.01	57.50	64.0	61.4	63.13	68.56	71.7	65.72
100	39.5	44.99	33.24	38.3	40.39	42.6	40.88	41.8	40.76

Table 2 gives the variation of viscosities with temperature for the selected lubricants. The values of viscosity decreased as the temperature increased. Lubricants are often designed to provide a viscosity that is low enough for good cold weather staring and high enough to provide adequate film thickness and lubricity in hot, high severity service [Kramer et al, 2001]. Therefore when hot and cold performances are required, a small response to changes in temperature is desired as shown in Tables 2 and 3. This behavior is shown also in Figures 2 and 3for both multigrades and monogrades respectively. The bulk of typical motor oil consists of hydrocarbons with between 18 and 34 carbon atoms per molecule [6] and the viscosity of a liquid is an important property which enables the liquid to maintain a lubricating film between moving parts at specific temperatures and pressures. The viscosity must be high enough to maintain a satisfactory lubricating film, but low enough that the oil can flow round the engine parts adequately to keep them well coated under all conditions. The lubricants industry expresses this response as the viscosity index (V.I.) (Dieter, 1984). A higher V. I. indicates a smaller more favorable response to temperature. Obi et al (2010) verified the V.I. s of some of these lubricants and found that Texaco, Total, Mobil and A-Z lubricants had VIs of 131, 114, 112 and 110 respectively for their multigrades and 103,112 and 82 for Texaco, Total and Mobil respectively for monogrades. Control bodies recommend a specification of 95-155 for fresh lubricants. Further investigation is on for Conoil and Oando with VIs less than 95 as obtained in this test (Obi, et al, 2010).

Table 3: Variations of the viscosities with temperature of mono-grade oil samples

Temperature (˚C)	Viscosity (cp/m Pas)		
	Total (SAE40)	Mobil HD (SAE40)	Oando SMOR (SAE40)
28	298.0	385.6	352.0
40	135.07	129.36	113.66
50	95.04	93.60	80.06
60	80.80	76.89	69.65
70	67.80	55.55	58.56
100	35.4	36.39	38.98

Table 4: Properties of some lubricants determined for fresh samples

Samples	Flash Point (˚C)	Density (g/m^3)	Viscosity (cp/m Pas) at 100^{o}C	Shear Rate (%Nm)	Carbon Residue
Total (15W50)	126	0.8493	392	39.2	0.0656
Total (SAE40)	154	0.8678	298	29.8	0.3115
Total (20W50)	208	0.8568	314	31.4	1.5018
Mobil Super (20W50)	162	0.8525	381.6	95.4	0.1023
Mobil HD (SAE40)	134	0.8596	385.6	98.4	0.0721
Oando SMOR (SAE40)	102	0.8647	352	35.2	0.1206
Oando Oleum (20W50)	137.5	0.8600	363	36.3	0.1071
Conoil (20W50)	159	0.8652	348	34.8	0.2653
Amasco (20W50)	123	0.8599	510	51.0	2.8800
Texaco Havoline (20W50)	111	0.8654	396	39.6	0.1028
A-Z (20W50)	134	0.8652	524	52.4	0.2341
AP Super V (20W50)	120	0.8588	356	35.6	0.1025

Flash points and other determined properties are shown in Table 4. Oil is largely composed of hydrocarbons which burn if ignited, hence the importance of flash point which is the lowest temperature at which the oil gives off vapors that can ignite. It is dangerous for the oil in a motor to ignite and burn, so a high flash point is desirable. Mobil super (20W50), Conoil (20W50) and Total (SAE40) have very high flash points, while Oando SMOR (SAE40), Texaco Havoline (20W50) and AP Super V (20W50) have low flash points.

Accurate determination of the density or specific gravity of petroleum and its products is necessary to identify oil from different geographical locations and for conversion of measured volumes to volumes and masses or both at the standard reference temperature of 60^{o}F. For example, while California crude has a specific gravity of 0.92 grams/milliliter, the lighter Pennsylvania crude has a specific gravity of 0.81 grams/milliliter [Nadkarni, 1991]. The specific gravity of a lubricant depends on the refining method and the types of additives present. The general specification for density of petroleum lubricants is 0.85-0.95. Table 4 confirms that the densities of the lubricants are within the specified limits.

The deposition of the oil ash increases the exhaust back pressure and reduces over time the fuel economy. The test is a guide to the formation of carbon deposits of lubricating oil in the cylinders of internal combustion engines. The deposit varies with engine type, refining process and the viscosity of the lubricant. Table 4 shows low values of carbon residues which indicate low ash content of the lubricants. These values are approximately equal as also the viscosities at 100^{o}C shown in Table 4. However Amasco (20W50) and Total (20W50) produced high carbon residue contents of 2.8800 and 1.5018 respectively.

Table 5: Acidity, Trace elements and Hydrogen contents of the lubricants investigated

Samples	Acidity (wt.% of KOH)				
		Na	K	Ca	H_2 (wt%)
Total (15W50)	8.68	10.65	1.49	1.49	11.83
Total (SAE40)	9.07	10.0	1.03	1.06	12.15
Total (20W50)	8.50	9.08	1.04	1.03	11.05
Mobil Super (20W50)	9.34	10.65	1.03	1.49	13.07
Mobil HD (SAE40)	9.28	8.46	1.49	1.01	11.14
Oando SMOR (SAE40)	9.38	8.06	1.37	0.64	11.75
Oando Oleum (20W50)	9.57	8.06	1.37	0.64	11.81
Conoil (20W50)	9.30	10.65	0.68	0.64	11.99
Amasco (20W50)	9.28	8.46	1.2	1.01	12.26
Texaco Havoline (20W50)	9.81	9.35	1.03	1.06	11.34
A-Z (20W50)	9.41	8.50	1.03	1.3	11.98
AP Super V (20W50)	9.65	11.29	1.37	0.64	11.56

The Total Base Number (TBN) which a measure of the reserve alkalinity of oil is another manipulated property and it is the lubricants acidity. Table 5 gives the measured acidity of the investigated lubricants. The values are quite close and indicate satisfactory ability to neutralize any acid formed during operation. Depending on the application, chemicals called additives may be mixed with the refined lubricant to give it desired physical properties. The trace elements in the oil or additives are shown in Table 5 and their functions include protection of metal surfaces (rings, bearings and gears), extension of the range of applicability and that of the lubricants life. The table shows concentration of Na, Ca and K which act as corrosion inhibitor, detergent and alkaline.

The hydrogen content of oil indicates the level of contamination by other lipids and the parity with lubricants processed elsewhere (Obi and Jonah, 1999).Table 4 also shows the values of the hydrogen contents determined the lubricants under investigation. The values ranged between 11.0 to 12.30 by weight percent. Jonah (1995) obtained 13.10-13.91 from Hungarian lubricant.

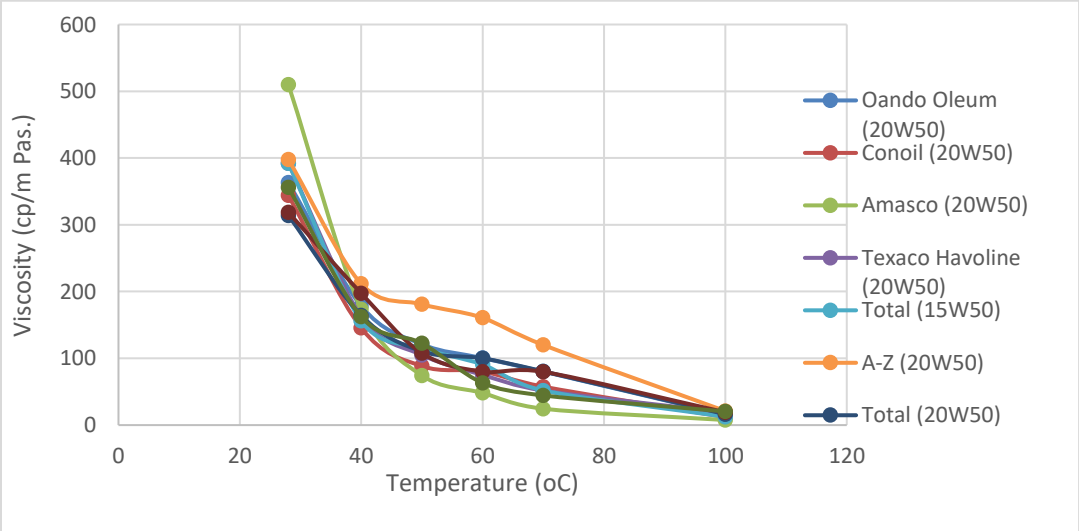

Fig. 2: Variation of viscosity with temperature for multigrade lubricants investigated

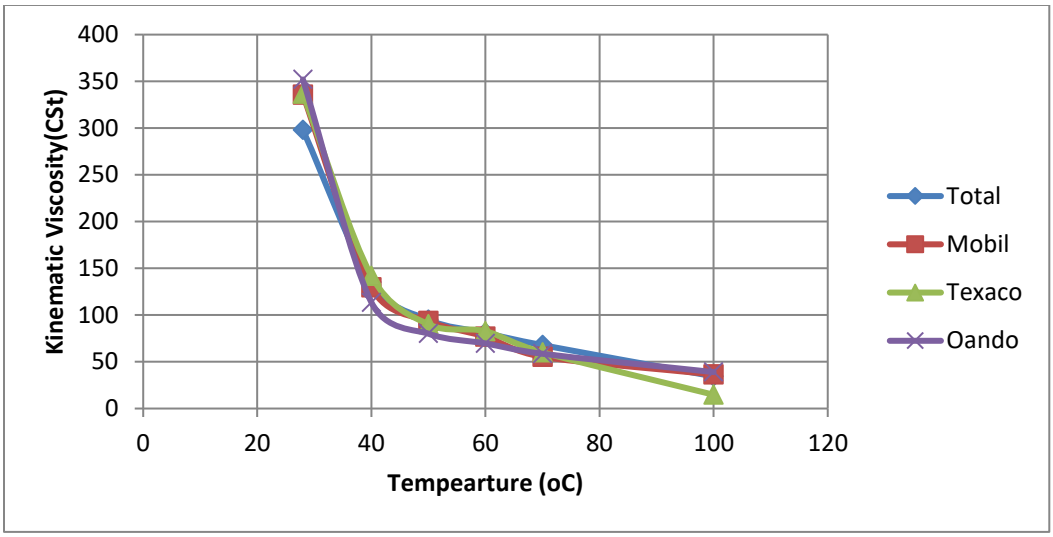

Fig.3. Variation of viscosity with temperature for mono-grade lubricants investigated

4.0 CONCLUSION

Various properties of engine oils manufactured in Nigeria have been determined. Lubricants are often designed to provide a viscosity that is low enough for good cold weather starting and high enough to provide adequate film thickness and lubricity in hot, high severity service [Kramer et al, 2001]. When hot and cold performances are required, a small response to changes in temperature is desired. The values of viscosity in this investigation were found to decrease gradually with temperature for both multigradeand monograde lubricants. The bulk of typical motor oil consists of hydrocarbons with between 18 and 34 carbon atoms per molecule [Nadkarni, R. A.] and the viscosity of a liquid is an important property which enables the liquid to maintain a lubricating film between moving parts at specific temperatures and pressures. The flash point is the lowest temperature at which the oil gives off vapors that can ignite. For most lubricants a high flash point is desirable to prevent ignition in a motor. Mobil super (20W50), Conoil (20W50) and Total (SAE40) were found to have high flash points, while Oando SMOR (SAE40), Texaco Havoline (20W50) and AP Super V (20W50) have low flash points.

Accurate determination of the density or specific gravity of petroleum products is necessary to identify oil from different geographical locations and for conversion of measured volumes to standard reference temperature of 15.6^{o}C. It also depends on the refining method and the types of additives present. The general specification for density of petroleum lubricants is 0.85-0.95.This investigation has confirmed that Nigerian manufactured lubricants are of good quality with densities above 0.85.

The deposition of the oil ash increases the exhaust back pressure and reduces over time the fuel economy. The test is a guide to the formation of carbon deposits of lubricating oil in the cylinders of internal combustion engines. The deposit varies with engine type, refining process and the viscosity of the lubricant. Table 4 shows low values of carbon residues which indicate low ash content of the lubricants. These values are approximately equal and also the viscosities at 100^{o}C shown in Table 4. However Amasco (20W50) and Total (20W50) produced high carbon residue contents of 2.8800 and 1.5018 respectively.

The Total Base Number (TBN) which is a measure of the reserve alkalinity of oil and the values obtained in this work are approximately equal and indicate satisfactory ability to neutralize any acid formed during operation.

The trace elements in the oil is a measure of the additives in the lubricant and their functions include protection of metal surfaces (such as rings, bearings and gears), extension of the range of applicability and that of the lubricants life. Na, K and Ca are found in appreciable quantities in this investigation and they act as detergents keeping the engine clean.

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