

Formulation of Bio-Based Lubricants using Antimony Dialkyldithio Carbonate

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Abstract: This study investigated the formulation of neem seed and jatropha seed oil using antimony dialkyldithiocarbamate (ADTC) additives. Two different oil formulations were tested in the Four-ball tribometer to compare the lubricity performance between the two oils. Wear tests with sliding speed of 0.45 m/s (1200 rpm) with time of 1 hour 10 seconds and normal loads of 392 N at oil bath temperature of 75°C were chosen to investigate the lubricant behavior under contact pressures. The objective of the wear tests was to investigate the ability of the lubricants to protect the surfaces against wear under metal forming processes. The results show that the jatropha seed oil presented lower friction coefficients in virtually all tested conditions and hence was concluded to be the oil with better performance.

Key words: ADTC additives, neem seed, Jatropha, formulation, four-ball test.

I. Introduction

Suitable protection of contacting surfaces in a tribosystem against wear and scuffing are key requirements for the selection of a lubricating oil, as well as base oil for formulating lubrication oil that is appropriate for any tribological design (Bart et al., 2012). To meet these requirements, most mineral based lubricating oils formulations involve reasonable dose of heavy metal, sulphur and phosphorus additives compounds such as antimony dialkyldithio-carbamate (ADTC), zinc dialkyldithiophosphate (Lim et al., 2014). Which intensify the environmental hazardous nature of the formulated oils (Yong, 2014).

Additives are substances formulated for improvement of the anti-friction, chemical and physical properties of base oils (mineral, synthetic, vegetable or animal), (Vander and Victoria, 2014).

The main limitations of vegetable oil are oxidation and gumming effect (Mofijuret., al 2012; Ponnekanti and Kaul 2012; Chen et., al 2011). These stabilities and pour point behaviour can be ameliorated by formulation to enhance the friction and wear behaviors of the lubricant performance and extending the equipment life.

Apparently, in order to investigate the tribological behavior of formulations conditions of neem seed and Jatropha seed oil, a four ball tester was used, since it has reduced cost and requires small amounts of oil for the test. The four-ball tribometer was Plint TE92 equipment that enables to apply normal loads up to 392 N and rotational speeds of 1200 rpm, at oil bath temperature of 75°C. In a four-ball tester a rotating ball is pressed against three fixed balls in oil bath, as illustrated in Figure 1 below. The formulations with good lubricating performance in four-ball test as well as good coefficient of friction and wear protection in vegetable based oils were critically looked into. Viscosity is the most important parameter to increase the oil film thickness in order to protect the metal rubbing surfaces against wear (Stachowiak and Batchelor, (2006).

Vegetable oils for some decades had been identified to be environmentally friendly lubricant base stocks (Quinchia et al., 2014), having some attractive lubricating properties in addition to their non-toxic composition, wholesome biodegradable qualities, and renewability (Baumgart et al., 2010; Salih et al., 2013). One of such advantageous properties is high lubricity, due to the atoms of oxygen present in the ester molecules, causing the molecules to form a monolayer over the metal surfaces (Silva et al., 2013).

Virtually, almost all lubricants require further additives to impart other characteristics of a non tribological nature, such as oxidation resistance, corrosion protection, and detergency. Most cutting fluids, vegetable oils and petroleum-based, are compounded or modified to achieve these requirements. Several methods are available to modify these oil lubricants. The important and most commonly used methods are sulfurization and phosphate modification. Schwab and Gast (2015).

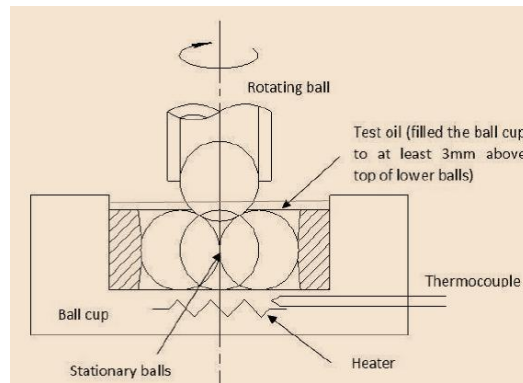


Fig. 1 Schematic of four ball arrangement

The tested oil were developed for application in elevated temperature wire drawing

II. Experimental Methodology

The experimental methodology test was carried out based on ASTM D4172 method. A steel ball, 12.7mm in diameter is thoroughly washed using acetone, wipe dried, it is then mounted on the motor spindle of a four ball tester, and pressed into the cavity of three balls (of the same material, cleaned by the same procedure) clamped in a ball cup filled with lubricant to at least 5mm above the three balls. The ball in the spindle, refer to as the rotating ball normally makes a point contact with each of the three balls in this arrangement (Figure 1 above). The setting was loaded to 392 N - static load (indicated by the controller), then the lubricating oil heated to 75°C using an inbuilt heating device, after which the motor spindle was set rotating at 1200 ± 60 rpm for 60 minutes, 10 seconds. The loading, temperature, speed and time were set on a controller, interfacing the four ball machine and a Winducom 2010 software installed on a PC, for the purpose of extracting the experimental data. After the 60 minutes 10 seconds, the three lower balls were removed, cleaned with methane and the scar diameters made on them owing to friction between their contacting surfaces with the top rotating ball were measured using a metallurgical microscope. All relevant data from the software and microscope were recorded and analyzed.

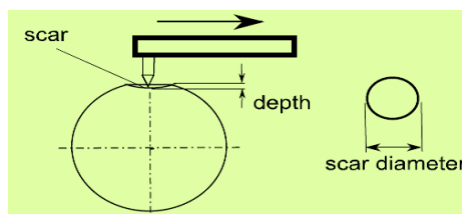


Fig. 2 Scar Depth Measuring in a Fix Ball with a Profilometer



Fig. 3 Steel ball after experiment

III. Wear Test

The wear tests was conducted in the four-ball tribometer. The test conditions were chosen from ASTM 4172-94 (2004) standard, that establishes wear tests with sliding speed of 0.45 m/s (1.200 rpm) during 1 hour with normal loads of 392N, the oil bath temperature of 75°C. The higher normal load, 392N, was chosen to investigate the lubricant behavior under high contact pressures. The objective of the wear tests was to investigate the ability of the lubricants to protect the surfaces against the wear under high contact pressures.

After the tests in the tribometer, the scar diameters were measured in all the fixed balls, in optical microscopic with x-y table, which movement is performed by two micrometers with resolution of 0.01 mm. Each scar diameter was measured in two orthogonal directions.

The CoF in the table 1 shows that jatropha oil has better lubricity performance for almost all the regime of loading under EP test. Even at higher loads, it has manifestly demonstrated lower CoF than the neem seed formulated oil. This proves why wax ester extracts from vegetable oils are proposed for used as friction modifiers in industrial and automotive lubricants (Bart et al., 2012).

From Figure 11, point A to point B represents the compensation line of the tribosystem with neem oil as the lubricating oil, where the loading on the lower three balls neither cause seizure nor welding. Point B is the last non-seizure load. The average wear scar diameters within the stated load regime in conventional oil are quite interestingly low. B to C is incipient seizure region, where the lubricating film experiences a temporal break-down. Point C to D is region with immediate seizure, indicating increasing wear scar, leading to the welding of the four balls together at point E (ASTM, 2009) under the lubrication of the commercial oil

The corresponding regions and locations with jatropha oil as the lubricant in the four ball cup tribosetting are Q to R (compensation line), R (non-seizure load point), R to S (incipient seizure region), S to T (immediate seizure region), and U as the weld point. Points E and U have indeterminate average wear scars diameters, as the four balls were actually welded together in each case. The immediate seizure region in jatropha oil is quite longer than in neem oil, implying a more durable resistance to wear at higher load. The attachment of the polar heads of the hydrocarbon chains of these oil molecules to the contacting surfaces of the balls were so strong for the intermediate mechanical loads to brush them off. Moreover, the hydrocarbon chains must be the long type, which immune the surfaces from severe wear at these loads contrary to the case in engine oil. jatropha oil demonstrated a higher weld load (2158N) compare to neem oil. These may suggest that extreme pressure is not a very critical subject as friction and wear. Hence, the oil may just need to be sufficiently equipped against friction and wear. This may be the reason for the quite lower EP load of the neem oil, and this will give preference to jatropha oil which had demonstrated higher weld load. The picture of the welded steel balls in the two lubricants are shown in Figure 10

IV. Results And Discussions

The results morphology of the surfaces of the three lower balls in each of the oils tested under high magnification (500x) of the microscope are shown in Figures 8 and 9 respectively. Figure 8 shows the worn surfaces in the mineral oil. The surfaces have more uniform wear. This should be due to the effectiveness of the anti-wear additives. The surfaces revealed the strength of the tribochemical reaction between the additives and the surfaces of the balls which made the surfaces very resistant to mechanical shear and abrasive wear. The furrows on the surfaces of the balls in jatropha oil (Figure 10) indicated the absence of anti-wear additives. Figure 11 shows average wear scar diameter against applied load. The bonding between the oil molecules and the ball surfaces are more of physical with weaker chemical layer, being more a boundary lubrication system. Once the physical bonding is sheared by constantly attacking mechanical shearing due to the top rotating ball, the chemical bonding are soon overcome and the direct metal-to-metal contact are ensured. This results in the tearing of particles from each surface. The torn out particles will in-turn aggravate the wear by acting as abrasive substances in the system. That informed the nature of the deep grooves on the scarred surfaces of the balls. Average wear scar diameter against applied load.

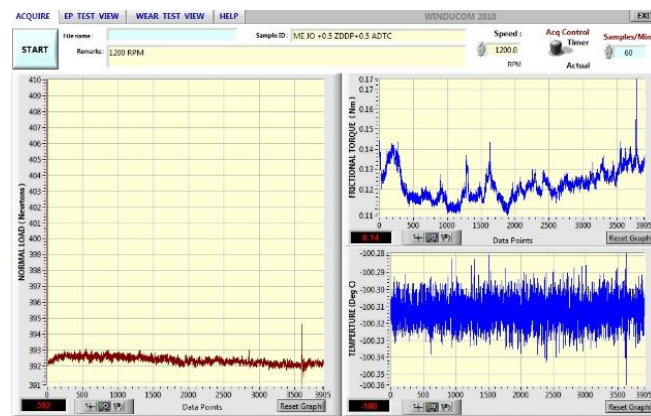


Fig.4 Normal load, f. torque & temp of JO+0.5%ADTC

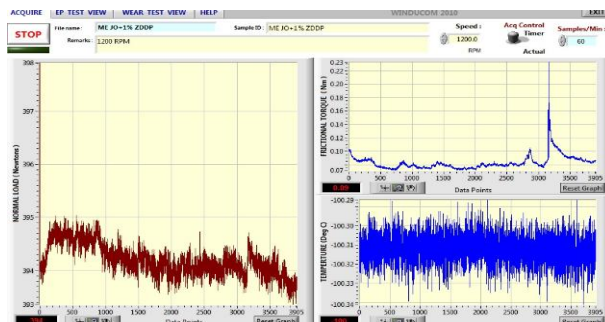


Fig. 5 Normal load, f. torque & temp of JO+1%ADTC

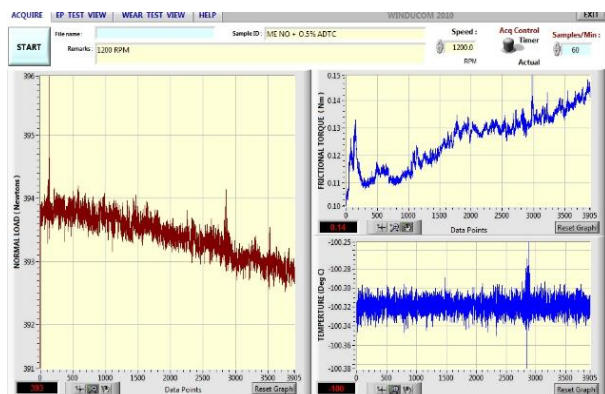


Fig. 6 Normal load, f. torque & temp of NO+0.5%ADTC

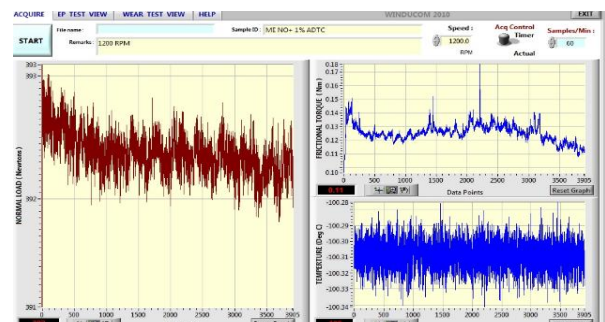


Fig. 7 Normal load, f. torque & temp of NO+1%ADTC

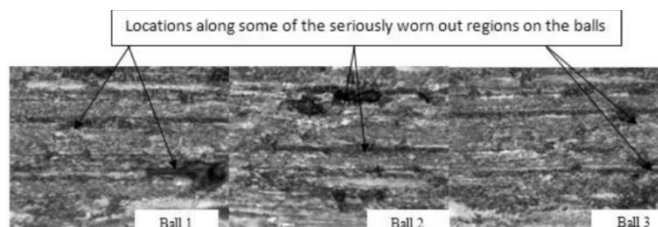


Figure 8 Morphology of worn balls in neem seed oil (scale of 100)

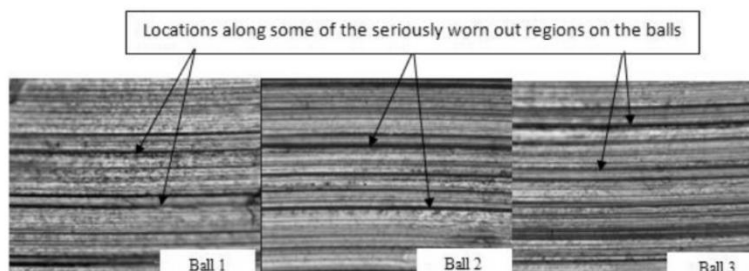


Figure 9 Morphology of worn balls in J. curcas oil (scale of 100)

Table 1 Geometric Wear Results

Lubricant	Friction Coefficient	Scar Diameter (mm)	Wear Depth (µm)	Roughness Ra (µm)	Roughness Rq (µm)
N.oil+0.5%ADTC	0.071	0.52	5.60	0.250	0.400
N.oil+1% ADTC	0.071	0.57	6.48	0.502	0.911
J.oil+0.5%ADTC	0.046	0.11	0.60	0.054	0.083
J.oil+1%ADTC	0.054	0.15	20.05	0.500	0.828



Fig 10 Welded balls in the experimental oil

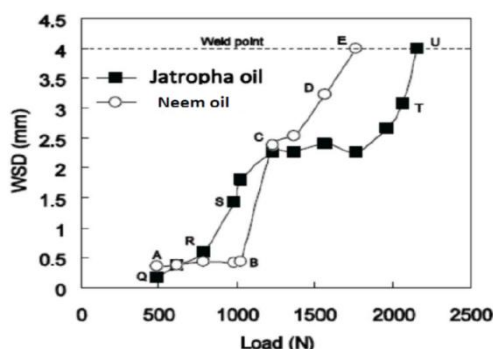


Fig. 11 Average wear scar diameter against applied load

V. Conclusion

In conclusion, the wear tests show the ability of the lubricants to protect the surfaces against wear under metal forming process. The results confirmed that the jatropa seed oil presented lower coefficients of friction in virtually all test conditions and hence was concluded to be the oil with better performance without fracture.

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