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Production of Biolubricants from *Balanites aegyptiaca* Seed Oil via Epoxidation and Double Transesterification Techniques

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ABSTRACT: In this study, *Balanites aegyptiaca* seed oil was extracted from the ground seed using the Soxhlet extraction method with n-hexane as the extraction solvent to produce two biolubricants; one via epoxidation to produce *Balanites aegyptiaca* Lubricant (BAB-E) and the other via transesterification with trimethylolpropane to produce *Balanites aegyptiaca* Lubricant (BAB-T). Both biolubricants were characterized using Fourier Transform Infrared Spectrometry. The physicochemical properties, lubricity and thermal properties of the produced biolubricants were determined. Results showed that the biolubricants had wavelengths of 3008.0 cm^{-1} associated with C-H stretching, 2922.8 cm^{-1} to -OH (acid), 1740.7 cm^{-1} to C=O, 1488.8 cm^{-1} to C=C stretching, 1364.2 cm^{-1} to NO_2, 1237.6 cm^{-1} to C-O, 1159.2 cm^{-1} to C-C and 723.1 cm^{-1} to C-H bending. BAB-T had viscosities of 58.29 and 10.36 cSt at 40 and 100 ^{O}C respectively while those of BAB-E were 54.37 and 9.56 cSt. They had viscosity index of 168 and 161, and pour points of -9 and -8 respectively. These values were observed to be within acceptable range. BAB-T had coefficient of friction of 0.094 ± 0.014 while that of BAB-E was 0.080 ± 0.010 respectively. The biolubricants were observed to have thermal stability and showed properties similar to those of lubricants and show remarkable potentials to be used as alternatives to fossil-based lubricants.

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There is an increase in the demand for energy sources worldwide. The Energy Information Administration of the United States forecasts an estimate consumption of 100.6 million b/d of petroleum and liquid fuels in year 2022 and a global consumption of 102.2 million b/d in year 2023 (EIA, 2022). Annual production of lubricants is about 30-40 million tons. Primary usage is often in industrial applications where it is mainly to decrease friction and heat, protect against corrosion and wear, transmit energy, and eliminate contaminants or sealing processes, etc. (Mang and Dresel, 2006; Saidur *et al.* 2011). Conventional lubricants are primarily fossil-based and have been causing controversy in the energy and environmental community. Fossil fuels have been reported to negative effects on the environment, water bodies, atmosphere, plants, animals and humans. For instance, fossil fuels release large amounts of greenhouse gases in to the atmosphere. Lubricant causes contamination to soils, groundwater and can accumulate in plant tissues as well as tissues of terrestrial and aquatic animals. They have also been reported to be carcinogenic and harmful when they come in contact with human tissue (Abbasi and Abbasi, 2010; Bessou *et al.*, 2011; Pareek *et al.*, 2020). Undoubtedly, fossils are the only sustainable energy sources in the short and medium term even though they are available in limited quantities in the earth crust. Nonetheless, there is a

need to employ a more long-term approach which involves cleaner and renewable energy sources. Great advances have been made in solar, wind, hydro, tidal and geothermal energy sources but none of these can comfortably and cheaply substitute for fossil based lubricant (Zhukovskiy *et al.,* 2021). Thus, the search for renewable, cleaner and cheaper sources of lubricants which has led scientists to the discovery of biolubricants. Biolubricants are non-toxic biodegradable lubricants produced from plant or animal oil (Heikal *et al.,* 2017). Biolubricants are primarily produced from non-edible seeds. Previous studies have reported production of biolubricants from seed oil of plants such as jatropha, rapeseed, sunflower, soybean, castor, linseed etc. have been used to produce biolubricants (Salimon *et al.,* 2010). The biolubricants produced were reported to have many of the desirable qualities of fossil-derived lubricants while having little or no toxic effect on the atmosphere. For instance, studies by Bilal *et al.* (2013) revealed that *J. curcas* biolubricant has viscosity of 66.74 and 14.28 cSt at 40 and 100 ^{0}C respectively while having minimal toxicity. Similar studies on castor bean oil by Silva *et al.* (2015) reported that biolubricants produced had qualities akin to fossil-based lubricants. Although tremendous progress has been made in the field of alternative fuels, there is a long time to go before they can reliably substitute for fossils fuels, thus the need for more research.

Balanites aegyptiaca; known as 'Desert date or Thorn tree' is a member of *Zygophyllacceae*, one of the most common plant species of the dry land areas of Africa and South Asia. In Nigeria, it is found mostly in the Northern region. It is known as 'Aduwa' in Hausa, 'Utazi' in Igbo, and '*Teji'* in Yoruba. It is an evergreen, woody, spiny flowering tree about 10 m height and is highly resistant to stresses such as sandstorms and heat waves, and grows with minimal available moisture (Saed and Isam, 2018). Oil has been extracted from *B. aegyptiaca* seed giving different yields when different extraction techniques are used, for instance, Ogala *et al.* (2018) reported a yield of 36.5 % when Soxhlet extraction technique is used and when n-hexane is the extraction solvent. Zang *et al.* (2018) on the other hand reported a yield of 45.32 % when a mixture of mechanical and Soxhlet extraction methods were used. However, Chapagain and Wiesman (2011) reported that the general yield of oil from *B. aegyptiaca* ranged from 30 % to 50 % regardless of the extraction method used. Physicochemical analysis of the seed oil by Ogala *et al* (2018) revealed that the oil had a moisture content, iodine value, saponification value and peroxide value of 7.16 %, 43.1 %g/g, 13.6 KOH %g/g and 37 meq/kg respectively. Therefore, the objective of this study was to produce two biolubricants from *Balanites aegyptiaca* seed oil extracted from the ground seed via epoxidation and via double transesterification methods.

MATERIALS AND METHODS

Sample collection and preparation: Balanites aegyptiaca were bought at Samaru market, Zaria, Kaduna state, Nigeria and were identified at the Herbarium, Department of Botany, Faculty of Life Sciences, Ahmadu Bello University Zaria. Healthy *Balanites aegyptiaca* seeds were selected, cleaned and sun dried. The seeds were then cracked and the shells carefully removed to obtain the kernels. The kernels were pulverized using mechanical method (mortar and pestle) and then used for extraction (Shivani *et al*., 2011).

Oil Extraction: Oil from the pulverized *Balanites aegyptiaca* seeds was extracted using the Soxhlet extraction method as described by Jock *et al.* (2017). The percentage oil yield was subsequently calculated using the equation below.

$$\% \text{ yield of biodiesel} = \frac{Weight\ of\ the\ oil\ produced}{Weight\ of\ the\ sample} \times 100$$

Synthesis of B. aegyptiaca Biolubricant by Epoxidation (BAB-E): The preparation of the biolubricant was carried out as described by Bilal *et al.* (2013). The crude *B. aegyptiaca* extracts were initially esterified to reduce its free fatty acid (FFA) content. In carrying out esterification, sulphuric acid was used as a catalyst. 100 g of the oil was weight and transferred into a two litre three necks round bottom flask. 20 % w/w methanol and 5 % w/w sulphuric acid were weighted and mixed in a conical flask. Both the methanol acid mixture and the oil sample were placed in a water bath and heated to a temperature of 60 ^{0}C. They were then mixed in the three necks round bottom flask with a mechanical stirrer at 700 rpm at a temperature of 60 ^{0}C for about 60 mins (Bilal *et al*., 2013).

After esterification, the biolubricant was produced via "double trans-esterification" process. The first trans-esterification process involves the production of methyl esters which is subsequently trans-esterified in the second step to produce the biolubricant (Bilal *et al.,* 2013). Esterified oil (100 ml) from above was trans-esterified with methanol (in the oil-methanol weight ratio 3:1) using potassium hydroxide as catalyst. The amount of catalyst used was 0.5 % w/w of the oil and the reaction was carried out at a temperature of 60 ^{0}C for 120 mins (Bilal *et al.,* 2010). This process led to the production of methyl esters.

The methyl esters produced were subsequently transesterified with ethylene glycol in 50 ml batches using 0.5 M sodium methoxide as catalyst. The weight ratio of oil-methanol was 3.5:1, the amount of catalyst used was 0.8 % w/w of the total reactants and the reaction was conducted at a temperature of 120 ^{0}C for two hours to produce the biolubricant (Bilal *et al.*, 2013).

Synthesis of B. aegyptiaca Biolubricant using Trimethylolpropane (BAB-T): The synthesis of biolubricant using TMP (Trimethylolpropane) involves two steps transesterification, the first step aimed at producing an intermediate product (methyl ester of the oil), while the second step uses the methyl ester as reactant to produce the desired product (a polyol ester product).

Step 1: Synthesis of B. aegyptiaca methyl ester (BAME): 100 g of the sample of esterified seed oil was transesterified with methanol using potassium hydroxide as catalyst. The weight ratio of oil to methanol was 3:1 and amount of catalyst used was 0.5 % w/w of the oil. The reaction was conducted at 60 ^{0}C for 2 hours.

Step 2: Synthesis of B. aegyptiaca trimethylolpropane ester biolubricant (BAB-T): This was achieved by transesterification of *B. aegyptiaca* methyl esters with trimethylolpropane (TMP) in 100 ml batches using sodium methoxide (in 30 % methanol) as catalyst. The weight ratio of methyl ester to trimethylolpropane was 3.5:1, the amount of catalyst used was 0.8 % w/w of the total reactant and the reaction was conducted at 120 ^{0}C for two hours thirty minutes (2.5 hours) as described by (Bilal et Al., 2013).

Characterization of the biolubricant: Fourier Transform Infrared Spectrometry (FTIR) was used to study the functional groups present in the prepared biolubricant. While Gas Chromatography - Mass Spectrometry (GC-MS) was used to evaluate the biolubricant. A Shimadzu QP2010 plus series gas chromatography coupled with Shimadzu QP2010 plus mass spectroscopy detector (GC-MS) system was used for the evaluation. The mass spectra were compared with the NIST05 mass spectral library (Warra and Abubakar, 2015).

In carry out the analysis, the plates were thoroughly cleaned using acetone. Using a syringe pipette, a drop of the oil sample was placed between the two KCl plates. The chloride plates were then attached to a Fourier Transform Infrared Spectrometer coupled to a computer with print out system. The sample in the plate was irradiated by infrared lamp source at one end of the spectrometer and each sample was analyzed between ranges of 600 cm^{-1} to 4000 cm^{-1}.

Determination of kinematic viscosity: A Redwood viscometer was used to measure the kinematic viscosity of the samples. The determination of kinematic viscosity was carried out by introducing about 50 cm^3 of the sample into a clean dried viscosity tube. This was done by inverting the tube thinner arm into the sample and then using suction force to draw up the oil to the upper timing mark of the viscometer. The instrument measured the time of gravity flow in seconds of fixed volume of the fluid (50 cm^3) through specified orifice made in an agate piece.

The viscometer was placed into a holder and inserted into a constant temperature bath set at 40 ^{0}C temperature and allowed for 30 minutes for the sample to come to the bath temperature. The suction force was then applied to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time was recorded by timing the flow of the sample as it flowed freely from the upper timing mark to the lower timing mark. The kinematic viscosity (ν) was calculated by means of the equation below adapted from Sandford *et al.* (2009).

$$V = C \times t$$

Where V = Kinematic viscosity (mm^2/s); t = Time for of 50 cm^3 sample in seconds; C = viscosity tube constant (0.09757)

Determination of pour point: The ASTM D-97 standard method was adopted for the pour point test. The sample (50 cm^3) was filtered and poured into a test jar to the level mark. The sample was heated in a water bath until it was just sufficiently fluid to pour into the test jar. The heated sample in the test jar was kept at room temperature for 24 hours before testing. The test jar was covered with the cork carrying the high-pour thermometer with its bulb immersed completely into the test sample and the beginning of the capillary was 3 mm below the surface of the sample. The sample was cooled inside a cooling bath to allow the formation of wax crystals and for every subsequent 3 ^{0}C, the test jar was removed and tilted to check for surface movement or flow. When the oil failed to flow when tilted, the jar was held horizontally for 5 seconds. If it failed to flow again, 3 ^{0}C was added to the corresponding temperature of the oil.

Tribological test: The tribological behaviour of the produced biolubricant was evaluated in accordance with Popoola *et al.* (2021) using an Anton Paar ball-

on-disc tribometer. The standard test method of ASTM G99-05 (2010) was adopted to evaluate the tribological performance of the biolubricant. The Anton Paar ball-on-disc tribometer model TRN used consists of a lever-arm device to hold the ball, a driven spindle and chuck for holding the revolving disc, and attachments that allow the ball to move forcefully against the revolving disc with a controlled load. The tribometer also had a data acquiring system and user interface software which records and controls the experiment. The balls of 6 mm diameter made of stainless steel were fitted into the upper stationary ball holder on the tribometer. The aluminium alloy discs of 70 mm diameter and 6.35 mm thick were machined. Tests were conducted on the bio-lubricant using the loads of 2 N, 5 N and 8 N, speeds of 150 rpm, 200 rpm and 250 rpm. The sliding distance was 50 m and all the tests were done for duration range of 5 – 12 minutes. Balls and disc were cleaned with acetone. Load was applied on the ball by dead weight through pulley-string arrangement. Lubricant was applied between the disc and the ball to satisfy boundary lubrication conditions. The ball specimen was inserted carefully in the holder and it was perpendicularly adjusted to the disc surface when in contact to maintain the necessary contact conditions. Appropriate load was added to the system lever to the selected force pressing the ball against the disc. The speed and revolution counter were adjusted to desired values and the electric motor was turned on. The test was stopped after achieving the desired numbers of revolutions. The specimen was removed and cleaned. The procedure was repeated for all the tests to obtain wear rate and friction coefficient. The values of friction coefficient and wear rate obtained were recorded (Popoola *et al.*, 2021).

Thermogravimetric analyses and Differential Thermal Analysis (TGA/DTA): Thermogravimetric analysis and Differential Thermal Analysis were carried out using Perkin Elmer, TGA 4000 thermogravimetric analyzer. 20 mm of the sample was heated from 30 ^{0}C to 950 ^{0}C under nitrogen flow at 10 ^{0}C/min as described by Tenimu (2019).

RESULTS AND DISCUSSION

The oil yield of Soxhlet extraction of *B. aegyptiaca* seed using n-hexane as the extracting solvent yield 45.62 % and density of 0.9 g/cm^3. This is in line with the observations of Zang *et al*, (2018) as well as Ogala *et al*, (2018) whose yield are 45.32 % fand 36.5 % respectively. The result also agrees with the findings of Chapagain and Wiesman (2011) who reported that the general yield of *B. aegyptiaca* range from 30 % to 50 % regardless of the extraction method used. Haftu (2015) also found the yield of *B. aegyptiaca* seed oil as 87 %.

The Fourier Transform Infrared Spectrum of BAB-T is presented in figure 1. Results revealed wavelengths of 3008.0 cm^{-1} associated with C-H stretching, 2922.8cm^{-1} to -OH (acid), 1740.7 cm^{-1} to C=O, 1488.8 cm^{-1} to C=C stretching, 1237.6 cm^{-1} to C-O, 1159.2 cm^{-1} to C-C and 723.1 cm^{-1} to C-H bending. These wavelengths were observed to be similar to those of biolubricants derived from castor oil and palm kernel seed oil (Musa *et al.*, 2015; Alang *et al.*, 2018). Figure 2 also shows similar findings for BAB-E biolubricant which indicated that both biolubricants have constituents which are similar to those of other biolubricants reported in the literature.

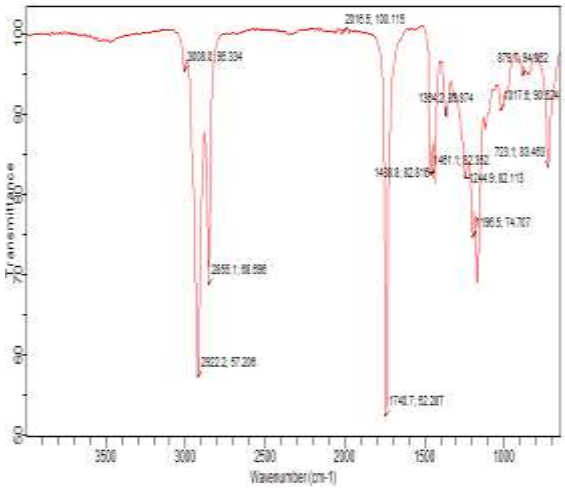

Fig 1: Fourier Transform Infrared Spectrum of BAB-T

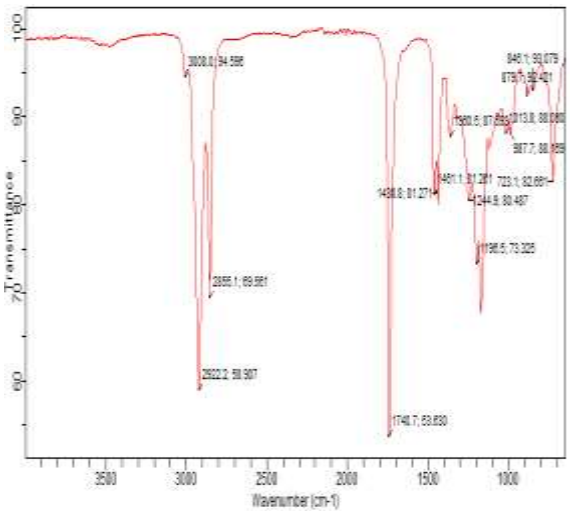

Fig 2: Fourier Transform Infrared Spectrum of BAB-E

The viscosity of BAB-T was observed to be marginally higher than that of BAB-E at 40 ^{0}C and 100 ^{0}C. The 40 ^{0}C viscosity of BAB-T biolubricant was 58.29 cSt while that of BAB-E was 54.37. These values were above the minimum International

Standards Organization Viscosity Grade (ISO VG-46) values for lubricants. Studies by Ahmed *et al.* (2014) reported 40 ^{0}C viscosity of soybean biolubricant to be in the range 50 – 80.40 cSt. Similarly, Bilal *et al.* (2013) reported viscosity of 55.17 cSt for biolubricant from *J. curcas* seed oil at 40 ^{0}C. The viscosities of BAB-T and BAB-E at 100 ^{0}C were also similar to those reported in literature and shows that both biolubricants meets the standard benchmark for viscosity of biolubricants. The viscosity index of biolubricants was 168 and 161 respectively (Table 1). The values were above the ISO VG-46 benchmark value and similar to those reported by Bilal *et al.* (2013) and Ahmed *et al.*, (2014). The pour point of BAB-T is -9 while that of BAB-E is -8 respectively which were similar to the ISO VG-46 standard for lubricants. The physicochemical properties of BAB-T and BAB-E all met standard benchmarks for lubricants and thus highlights their remarkable potentials to be used as alternatives or additives to fossil lubricants.

Table 1: Physicochemical parameters of BAB-T and BAB-E

Parameter	BAB-T	BAB-E	ISO VG-46	ASTM METHOD
Viscosity at 40 ^{0}C (cSt)	58.29	54.37	>41.4	ASTM D445
Viscosity at 100 ^{0}C (cSt)	10.36	9.56	>4.1	ASTM D445
Viscosity index	168	161	>90	ASTM D2270
Pour point (^{0}C)	-9	-8	-10	ASTM D97

The coefficient of friction of the biolubricants produced is as presented in Table 2. The coefficient of friction of BAB-T was 0.094 ± 0.014 while that of BAB-E was 0.094 ± 0.014. This is similar to reports by Baskar *et al.* (2015) who reported coefficient of viscosity of range 0.08 – 0.10 for various nano-based biolubricants. Also, the finding was similar to those of Trajano *et al.* (2014) for various vegetable biolubricants.

Table 2: Friction coefficient of BAB-E and BAB-T

Name	Coefficient of friction	Standard deviation
BAB-T	0.094	0.014
BAB-E	0.080	0.010

Tribology is a science of interacting surfaces in relative motion. It includes application of friction, wear and lubrication. The tribological test was conducted to study the frictional performance of BAB-E and BAB-T. In this test the friction coefficient (μ) was measured, using ball on disc tribometer according to ASTM standard test method (ASTM G-99). Generally, materials with coefficient of friction less than 0.1 are considered lubricious materials and the lower the friction coefficient the better is the performance of the lubricant.

From the results obtained as shown in Table 2. The epoxidized biolubricant BAB-E has lower friction coefficient (0.080) than the transesterified one (BAB-T) with friction coefficient (0.094), the lower value of BAB-E shows its tribological effectiveness. However standard deviation of is BAB-T is (0.014) and that of BEB-L is (0.010), meaning BAB-T is more stable than BAB-E. Moreover, coefficient of friction in this work are better than those reported for palm biodiesel and diesel fuel (Mofijur *et al.*, 2017). Figure 3 and 4 shows the tribological results of BAB-T and BAB-E respectively.

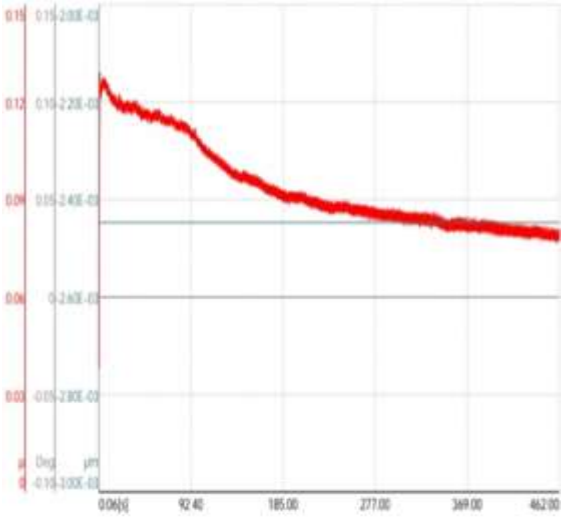

Fig 3: Tribolological result of BAB-T

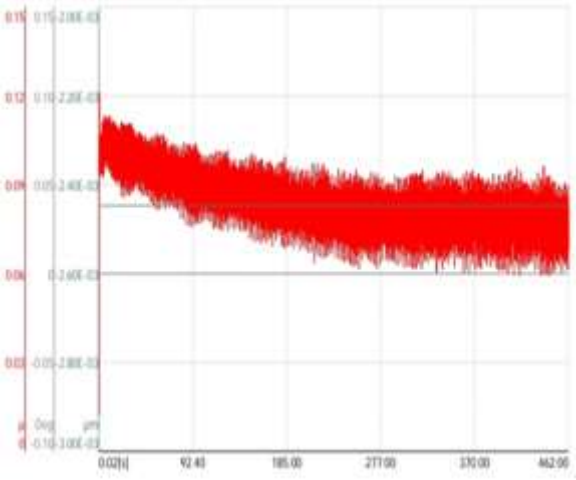

Fig 4: Tribological test result of BAB-E

The thermal behaviour of BAB-E and BAB-T was investigated using Thermogravimetric Analysis

(TGA) and Differential Thermal Analysis (DTA) as shown in Figure 5 to 8. The shelve life of biolubricant can be probed via its oxidative/thermal properties evaluation at extreme temperature, pressure and other applications. Lubricants with high oxidative stability does not form deposits/ plugs by products that are corrosive in engine oils, grease and other oil applications.

Thermograms show the behavioural properties of substance under investigation as a unique order of physicochemical responses over a given temperature range and at a rate that are function of molecular structure. Differential in weight are as a result of cleavage or fusion of several physical and chemical bonds at high temperatures that eliminate volatile products or formation of denser reaction products (Sharma and Sachan, 2019).

Thermogravimetric analysis (TGA) is important for biolubricant because thermal properties of fatty esters are very important in lubricants. They are unstable in air hence, are susceptible to oxidative and thermal degradation, thus making TGA very significant in probing the susceptibility of biolubricant (Heikal *et al*., 2017). TGA may be coupled with differential thermal analysis (DTA) to differentiate the physical occurrences of state change from chemical occurrences responsible for change in weight.

Investigation of the thermal property of BAB-E and BAB-T was achieved by TGA/DTA analysis. A simultaneous SDT 2960 from TA instrument was used, with a heating rate of 10 ^{0}C/min from 30 ^{0}C to 950 ^{0}C. A flow of 100 mL/min of Nitrogen was used as an inactive environment to facilitate the process in estimating the heat of combustion.

From the TG curve extrapolated, decomposition onset temperature reveals BAB-T to present higher thermal stability (305 ^{0}C) with 5 % weight loss (Figure 5) compared to BAB-E (240 ^{0}C) with similar 5 % weight loss (Figure 6). Degradation was endothermic for both BAB-T and BAB-E (Figures 7 and 8) and predominantly followed a single step. Degradation becomes rapid at temperature above 300 ^{0}C for BAB-T while for BAB-E started at temperature above 250 ^{0}C. The relative thermal stability of BAB-T would be attributed to the alcohol substitution of the branch chain Trimethylolpropane (TMP) resulting into a thermally stable Triester (BAB-T) (Egbuna *et al*., 2021; Juan *et al.,* 2020).

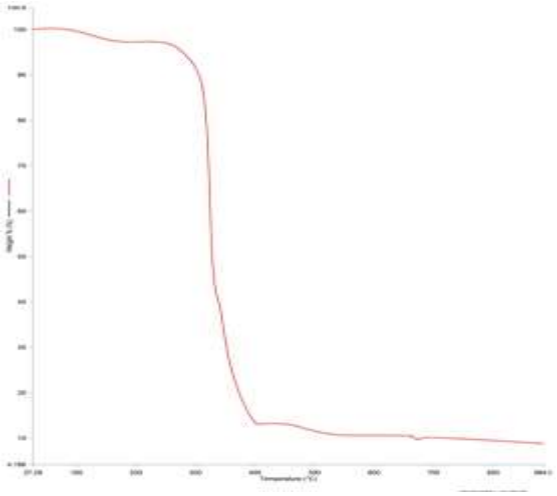

Fig 5: TGA result for BAB-T

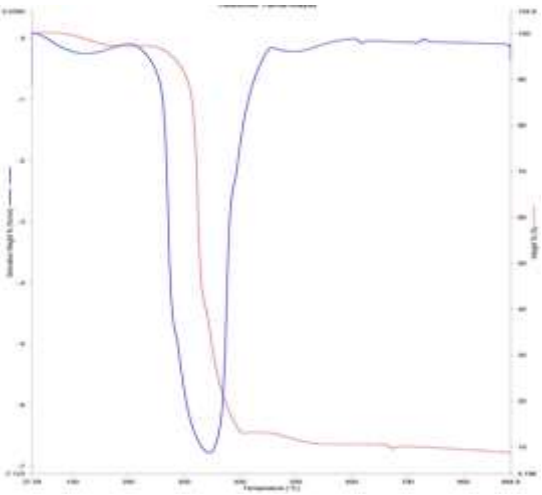

Fig 6: TGA/DTA result for BAB-T

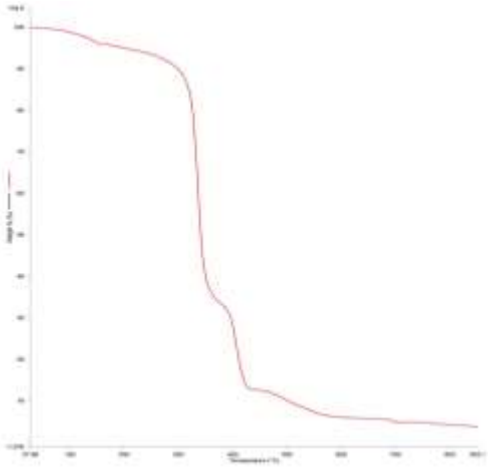

Fig 7: TGA result for BAB-E

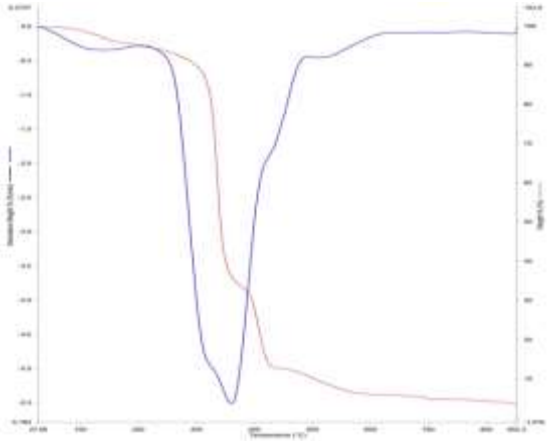

Fig 8: TGA/DTA result for BAB-E

Conclusion: This study produced two biolubricants from *Balanites aegyptiaca* seed oil. One via epoxidation and the other via transesterification with trimethylolpropane (TMP). The physicochemical parameters, lubricity and thermogravimetric properties of the biolubricants were analysed and findings showed that the biolubricants had properties similar to those of fossil-origin lubricants, hence can be used as alternatives to currently used fossil-based lubricants which are non-renewable and toxic to the environment and health.

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