



## Thermal Behavior and Fluidity of Biolubricant Synthesized from Mango (*Mangifera indica* L.) Kernel Oil Blended With Cold Flow Property Improvers

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**ABSTRACT:** Fossil fuel and conventional lubricant are not environmentally friendly, non-biodegradable, toxic, and non-renewable. Therefore, the objective of this paper is to evaluate the thermal and fluidity of the biolubricant synthesized from Mango (*Mangifera indica* L.) kernel oil (MKO) blended with cold flow property improvers using standard techniques. A two-step process of esterification and transesterification was employed to produce fatty acid methyl ester (FAME) from MKO mentioned as Mango kernel methyl ester (MKME). Double transesterification of the MKME with trimethylolpropane (TMP) in the presence of sodium methoxide yielded 95 % Mango oil based trimethylolpropane ester (MOTE). Another portion of the MKME was used to synthesize biolubricant using the conventional epoxidation which yielded 70 % Mango oil based epoxidised biolubricant (MEB-L). A simultaneous SDT 2960 TG/DTA from TA instrument analysis reveals MOTÉ to present higher thermal stability (302 °C) with 5 % weight loss compared to MEB-L (250.5 °C) with similar 5 % weight loss. Degradation was endothermic for both MOTÉ and MEB-L and predominantly followed a single step of weight lost. Degradation becomes rapid at temperature above 300 °C for MOTÉ while for MEB-L degradation started at temperature above 250 °C. At about the temperature range of 850 - 900 °C, 95 % of MEB-L and 77 % of MOTÉ were lost respectively. The relative thermal stability of MOTÉ would be attributed to the alcohol substitution of the branch chain Trimethylolpropane (TMP) resulting into a more thermally stable Triester (MOTÉ). The results of the analysis of the cold flow properties reveal that various blend of MEB-L with n-BE portrays better pour and cloud point as they are lower than their counterpart (blends of MOTÉ with n-BE) and (blends of even MOTÉ & MEB-L with EAA for both cloud/pour points).

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The fossil fuels reserves are increasingly being depleted with no hope of replacement, coupled with significant environmental hazards associated with its use all over the world (Sahoo *et al.*, 2007). It is clear that fossil fuel is still the major contributor to the energy demand of the world, it accounts for over 80.3 % (Escobar *et al.*, 2009). Although, Fossil fuel is relatively cheap and give the best benefits; it also causes hazard to the environment. It is an essential fuel

for running power plants, vehicles, motor engines in the transportation, agricultural and industrial sectors (Arshad *et al.*, 2017). The search for green energy has been prompted by fluctuation in fossil fuel prices, environmental problems (air pollution, green-house-gas emission and global warming) and energy security concerns (Balat and Balat, 2009; Ozturk, 2014). According to the Paris Agreement which was adopted by consensus 12 November, 2015, it is a long-term

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goal to keep the increase in global average temperature to less than 2 °C above pre industrial levels; limiting the temperature rise to 1.5 °C recognizing that this would substantially reduce the risks and impacts of climatic change caused by release of green-house gas (GHG) emissions (Jethro *et al.*, 2023; Schleussner *et al.*, 2016). These environmental challenges have prompted the research for alternatives to fossil fuel and conventional lubricants. Biolubricants are produced from vegetable oil-based source such as rapeseed, palm, soybean and mango seed. Due to the ever increasing world population and demand for food, focus has shifted to non-edible oils as potential feedstocks (Ayhan *et al.*, 2016; Sharma *et al.*, 2012; Vasudevan and Briggs, 2008). Oil extracted from non-edible seeds as straight vegetable oils (SVO) are usually not used directly as fuel in compression ignition engine due to higher viscosities of oils but have been used at several stages of human history as lubricant. However, there could arise problems like injector coking, more engine deposits of carbon, ring sticking and problems with the fluidity of engine lubricant are observed using straight vegetable oil as lubricants. The use of *Mangifera indica* L. (*M. indica*) as a feedstock for biolubricant synthesis still remain scanty. There has not been much reports regarding the use of *M. indica* as a potential substitute to conventional lubricant which are known for their non-biodegradability and toxicity, as 40 % of these toxic oleo-chemicals are discharged into the environment (Obasa *et al.*, 2020). The need for environmentally friendly, biodegradable, non-toxic and renewable biolubricant cannot be overemphasized. Therefore, the objective of this paper is to evaluate the thermal and fluidity of the biolubricant synthesized from Mango (*Mangifera indica* L.) kernel oil (MKO) blended with cold flow property improvers.

## MATERIALS AND METHODS

**Plant material:** Mango fruit seeds were collected from farmer of Mango fruit at Ikanekpo, Ankpa local government area of Kogi State, Nigeria. It was determined at the herbarium by Namadi Sunusi, a staff of Botany Department, Ahmadu Bello University, Zaria with a voucher number (v/n) ABU01944 on 22<sup>nd</sup>

May, 2023. The sun drying of mango kernel was done for two weeks until constant weight was achieved, confirming elimination of moisture content at mean daily temperature of 33.50 °C. The mango kernel after drying was cleaned and sorted by removing the immature, broken kernel and unwanted materials. The kernel was then pulverized with mortar and pestle and 25 g was weigh with electronic balance model (METLAR MT-501) and then extracted using soxhlet apparatus and n-hexane as the solvent. The process was repeated five times to determine the mean oil yield.

**Mango Kernel Oil Extraction:** The crushed pulp was loaded into the extraction chamber of the soxhlet extractor and allowed to reflux at 65 °C for about eight hours. To isolate the free flow lipid from the solvent, the extract was filtered (to remove impurities) and evaporated using a rotary evaporator. The extracted oil was then evaporated on a magnetic stirrer at 120 °C to remove any moisture and residual solvent. The percentage of oil content was calculated by weighing the oil produced and the residue using Equation (1), as reported by (Jethro *et al.*, 2023).

$$\text{Oil yield (\%)} = \frac{W_2 - W_1}{W} \dots\dots (1)$$

Where,  $W$  = weight of mango kernel used (g);  $W_1$  = weight of beaker (g);  $W_2$  = weight of beaker with oil (g);  $W_2 - W_1$  = weight of oil. (g).

**Esterification:** Esterification Mango kernel Oil (MKO) was paramount due to high free fatty acid value, the pretreatment was done using few drops concentrated  $H_2SO_4$  as a catalyst at 60 °C and excess methanol to obtain a mixture of fatty acid methyl esters and triglycerides. The order of addition was MKO followed by a mixture of methanol and concentrated  $H_2SO_4$  that was continuously stirred on a magnetic stirrer for one hour. The mixture was poured into the separating funnel for separation, the mixture separated into two layers of methanol on top and fatty acid methyl esters/triglycerides on the bottom. The bottom layer was collected, heated to remove any remaining methanol, and left to stand for 24 hours.

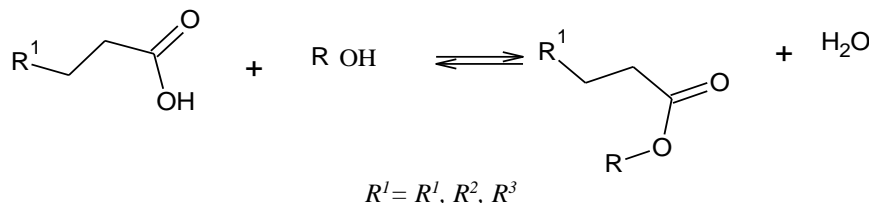


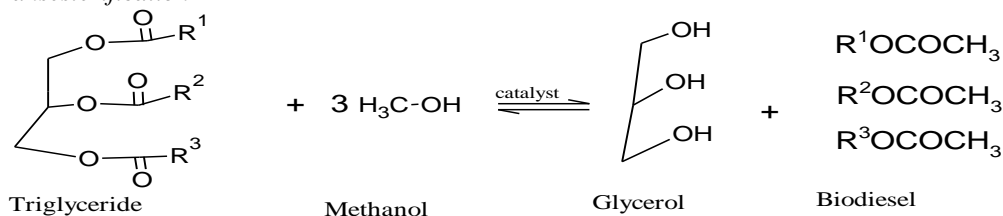
Fig 1. Esterification of Fatty acid. (Jethro *et al.*, 2023)

**Transesterification:** The transesterification of MKO with methanol produces Mango kernel methyl ester (MKME) and glycerol, catalyzed by both homogeneous and heterogeneous catalysts. Portions of MKO were reacted with 25 cm<sup>3</sup> methanol and 0.5 % catalyst calcium oxide (CaO), sodium hydroxide (NaOH), and potassium hydroxide (KOH), in separate beakers. Temperature was set at 60 °C on magnetic stirrer for 60 mins. Mixture transferred into the separating funnel and allowed to stand for 24 hours for proper separation of the Biodiesel and Glycerol layers. The Glycerol layer was removed, and the Biodiesel was washed with warm water to remove methanol and traces of the catalyst contained in the Biodiesel. Mango Kernel Methyl Ester (MKME) formed was

heated to 120 °C to remove water and methanol still present (Jethro *et al.*, 2023).

**Double Transesterification Method (Biolubricant Synthesis):** Double transesterification of MKMS was done by weighing 50 cm<sup>3</sup> of the MKMS reacted with 5 % Trimethylolpropane in a 100 cm<sup>3</sup> beaker. The mixed substance was then heated to about 120 °C for 2.5 hours while continuously been stirred on magnetic stirrer, 1 % w/w catalyst potassium hydroxide solution in methanol (potassium methoxide) was added slowly to the reaction mixture to avoid spillage. The resultant product Mango oil based trimethylolpropane ester (MOTE) was produced (Jethro *et al.*, 2023).

#### Step 1: Transesterification



#### Step 2: Double Transesterification

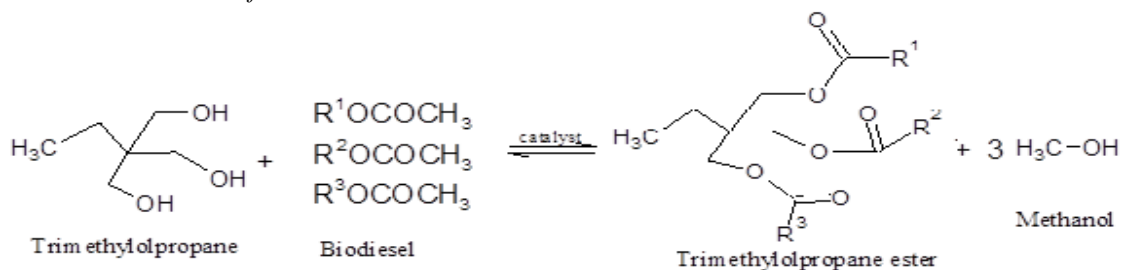


Fig 2. Transesterification and Double transesterification reaction. (Jethro *et al.*, 2023)

**Epoxidation Method for Biolubricant Synthesis:** The classical Prilezhaev method of epoxidation was employed for the synthesis of mango oil based epoxidized biolubricant (MEB-L). Portion of MKMS (40 cm<sup>3</sup>) was measured into 250 cm<sup>3</sup> beaker, 20 cm<sup>3</sup> of acetic acid was added to the oil in the beaker and heated at 60 °C before 20 cm<sup>3</sup> of hydrogen peroxide 30 % mixed with 1 % H<sub>2</sub>SO<sub>4</sub> was added to the mixture dropwise to avoid a spillover of the reaction mixture, the sequence of these addition was reported by (Ashrafi *et al.*, 2017; Cecilia *et al.*, 2020; Jethro *et al.*, 2023). The setup was heated for two hours on a magnetic stirrer and the progress of the reaction monitored at interval by FTIR for completion. The mixture was transferred into separatory funnel where oil layer (epoxide) was seen at the top and hydrogen peroxide/acetic acid clear aqueous layer was seen at the bottom. The aqueous layer was drained off and the epoxide washed repeatedly with warm water. The

epoxide was heated to 120 °C to remove any water present (Jethro *et al.*, 2023).

**TGA and DTA of Biolubricants:** In order to investigate the thermal behavior of MOTE and MEB-L, TGA and DTA were employed as depicted in Figure 4.10 and Figure 4.12. The shelf life of a lubricant can be probe via its oxidative/thermal properties evaluation at extreme temperature, pressure and other applications. Lubricants with high oxidative stability does not easily form deposits, plugs, byproducts that are corrosive in engine oil, grease and other oil applications. Also, viscosity of such lubricants does not increases arbitrary during use as the more volatile components vaporizes. There is a direct relationship between the type and amount of unsaturation present at the fatty acid chain of vegetable oils to its oxidative stability. Thermograms show the behavioral 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. Differentials in weight are as a results of cleavage or fusion of several physical and chemical bonds at high temperatures that eliminate volatile products or formation of denser reaction products (Willard *et al.*, 1989). The thermal properties of fatty esters are utmost importance in the science of lubrication. Their usability is always in air hence their susceptibility to oxidative and thermal degradation. It is then imperative to probe their susceptibility using thermogravimetric analysis (TGA), which quantitatively measures all the changes in weight of a substance as a function of temperature (Heikal *et al.*, 2017). TGA may be coupled with differential thermal analysis (DTA) to differentiate the physical occurrences of state change from the chemical occurrences responsible for the change in sample weight. DTA which occurs under inactive environment such as Nitrogen or Helium are characterized by endothermic peak which results from thermal degradation of the substance, conversely in the presence of air or Oxygen; oxidation reaction occurs resulting to an exothermic peak which when superimposed on the endothermic peak correspond to loss of various lower molecular weight products generated during oxidation or thermal degradation of the compound being analyzed via evaporation mainly. TGA results made available to the researcher a quantitative measurement of any change associated with a phase change. However, TGA can directly measures loss in weight with time/temperature which is as a result of dehydration and degradation. The weight change is as a result of cleavage and or formation of various chemical/physical bonds at high temperatures that results into release of lower molecular weight products or formation of heavier reaction products (Dufaure *et al.*, 1999).

*Cold Flow of the Biolubricants:* Low temperature applicability is a paramount property desirable of a

lubricant used as engine oil most especially in extreme cold temperature region. Bio-lubricant on the average rarely possessed this properties, until they are blended with additives of varying amount to give best results. Cloud point (CP) and Pour point (PP) are crucial test which predicts the cold flow property (Verma *et al.*, 2016). The experimental set-up were presented in (Plate 1 & 2), while results were captured in (Table 1). CP and PP were measured using digital electronic thermometer (measures upto -50 °C).

## RESULTS AND DISCUSSION

**TGA & DTA OF MOTE AND MEB-L:** Investigation of the thermal properties of MOTE and MEB-L was performed by Thermogravimetric analysis (TGA) and Differential thermal analysis (DTA). A simultaneous SDT 2960 TG/DTA from TA instrument was used, with a heating rate of 10 °C/min from 30 °C to 950 °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 of onset temperature reveals MOTE to present higher thermal stability (302 °C) with 5 % weight loss (Figure 3) compared to MEB-L (250.5 °C) with similar 5 % weight loss (Figure 5). Degradation was endothermic for both MOTE and MEB-L (Figure 4 & Figure 6) and predominately followed a single step of weight loss (Uni-stage). Degradation becomes rapid at temperature above 300 °C for MOTE while for MEB-L it started at temperature above 250 °C. At about 900 °C 77 % of the MOTE was lost while at about 850 °C 95 % of MEB-L was gone (Dweck & Sampaio, 2004; Jayadas *et al.*, 2005). The relative thermal stability of MOTE would be attributed to the alcohol substitution of the branch chain Trimethylolpropane (TMP) resulting into a more thermally stable Triester (MOTE) (Saboya *et al.*, 2017; Cecilia *et al.*, 2020).

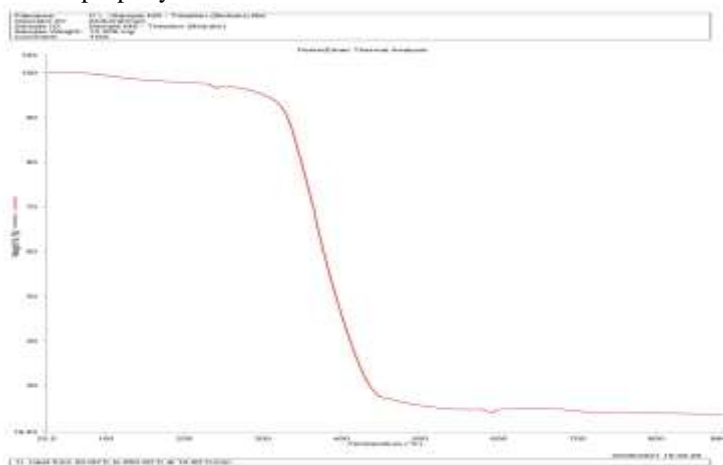


Fig 3: TGA result for MOTE

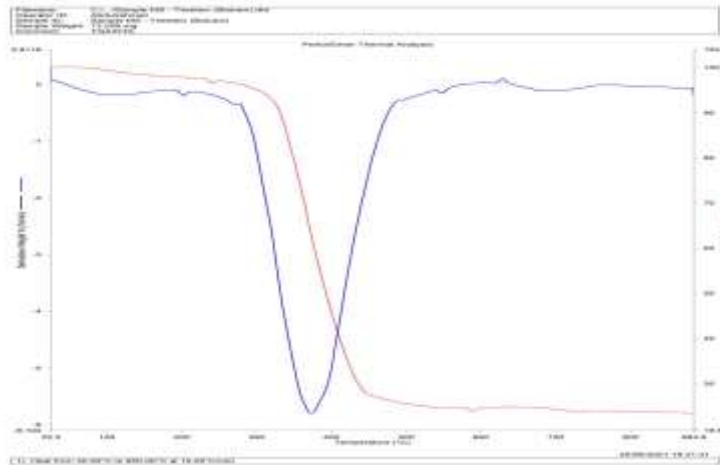


Fig 4: TGA and DTA for MOTE

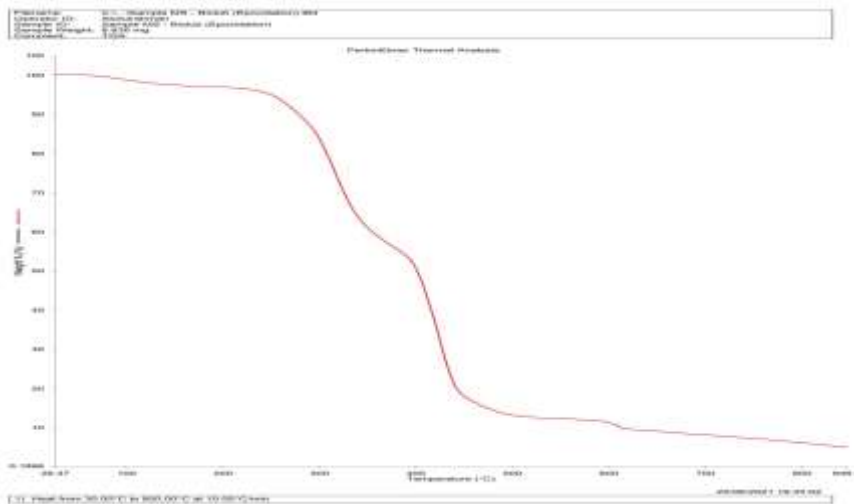


Fig 5: TGA result of MEB-L

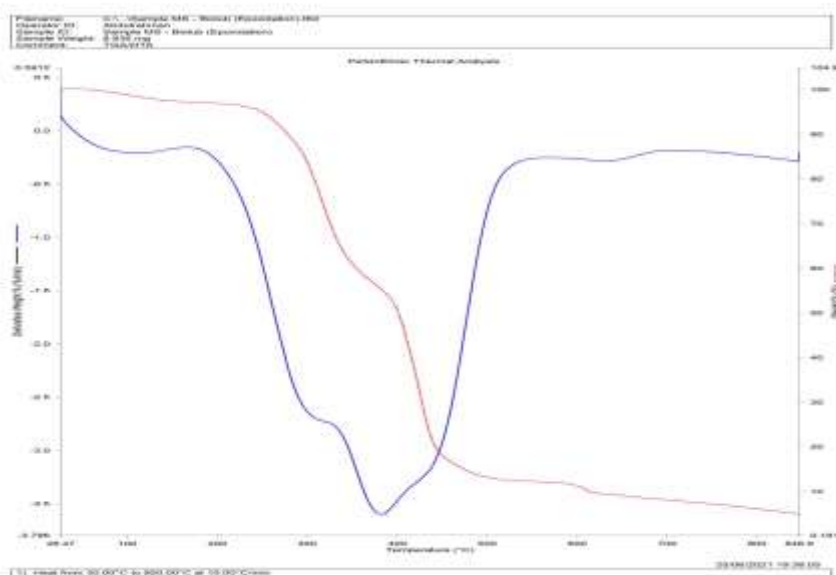


Fig 6: TGA and DTA results for MEB-L

**COLD FLOW PROPERTY OF MOTE & MEB-L:** The cloud point for MOTE and MEB-L when ethylacetate (EAA) was used for blending at various percent shows a continuous decline in cloud point as the amount of EAA increased. The decreased cloud point values were gradual throughout the blend as evidence in (Figure 7). The cloud point values of MOTE and MEB-L decreased significantly when n-butyl ester (n-BE) was used at various blend percent. This is presented in (Figure 8), with blends of MEB-L having cloud point values that decreased most especially at 20 % blend. The various cloud point obtained of MOTE and MEB-L when n-BE was used as cold flow improver additive were generally lower than their corresponding EAA blends (Table 1, Figure

11). The pour point values for MOTE and MEB-L when EAA was used for blending at various percent shows a continuous decline in pour point as EAA amount increased.

The decreased pour point was also gradual except for a sharp dip at 30 % EAA blend for MOTE and at 20 % dip for MEB-L where their pour point value dropped significantly from 9.5 °C to 5.9 °C and from 8.5 °C to 6.0 °C for MOTE and MEB-L respectively (Table 1, Figure 9). Similarly, the pour point values for MOTE and MEB-L decreased significantly when n-BE was used at various blend percent.



**Plate 1:** Showing the % blend of EAA with MOTE and MEB-L



**Plate 2:** Showing the % blend of BE with MOTE and MEB-L

**Table 1:** Readings of Cloud and Pours points for blends of MOTE and MEB-L

MOTE	10%EAA	20%EAA	30%EAA	40%EAA	50%EAA	CONTROL 100% MOTE
CLOUD POINT °C	21.1	17.0	14.2	12.2	10.5	22.0
MEB-L	10%EAA	20%EAA	30%EAA	40%EAA	50%EAA	CONTROL 100% MEB-L
CLOUD POINT °C	19.8	14.5	13.1	11.1	8.5	16.2
MOTE	10%EAA	20%EAA	30%EAA	40%EAA	50%EAA	CONTROL 100% MOTE
POUR POINT °C	10.5	9.5	5.9	5.3	5.1	14.5
MEB-L	10%EAA	20%EAA	30%EAA	40%EAA	50%EAA	CONTROL 100% MEB-L
POUR POINT °C	8.5	6.0	5.0	4.3	3.8	9.5
MOTE	10%BE	20%BE	30%BE	40%BE	50%BE	CONTROL 100% MOTE
CLOUD POINT °C	14.8	14.1	9.5	6.5	1.5	22.0
MEB-L	10%BE	20%BE	30%BE	40%BE	50%BE	CONTROL 100% BE
CLOUD POINT °C	13.5	9.5	6.2	3.8	1.1	16.2
MOTE	10%BE	20%BE	30%BE	40%BE	50%BE	CONTROL 100% MOTE
POUR POINT °C	11.2	5.3	4.2	0.7	-0.7	14.5
MEB-L	10%BE	20%BE	30%BE	40%BE	50%BE	CONTROL 100% BE
POUR POINT °C	6.0	4.5	2.5	0.5	-1.5	9.5

The cold-flow properties result are presented as bar chart below.



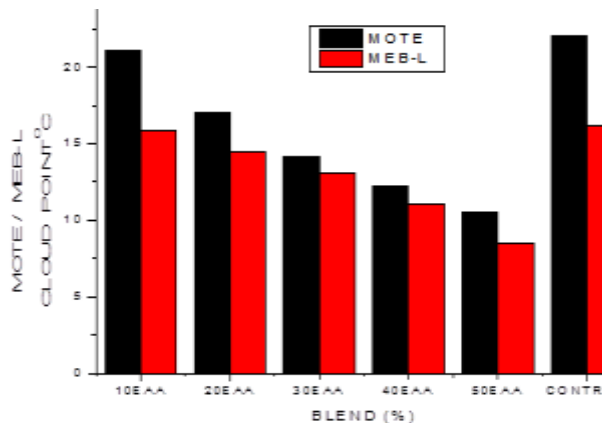


Fig 7: Showing variation of cloud point with increase in blend of EAA.

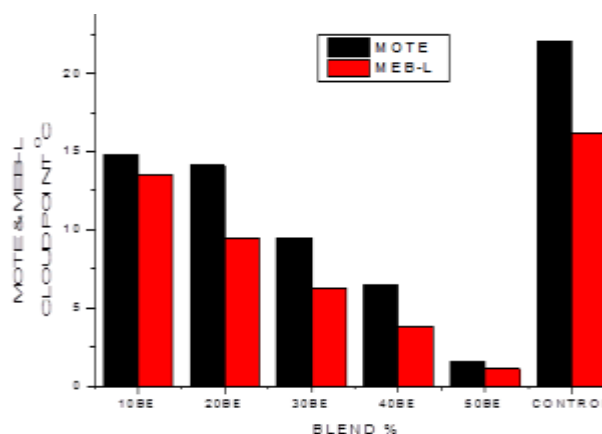


Fig 8: Showing variation of cloud point with increase in blend of n-BE

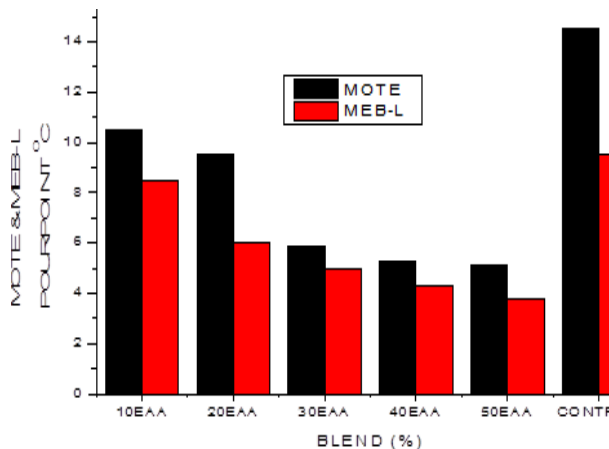


Fig 9: Showing variation of cloud point with increase in blend of EAA.

This is shown in (Figure 10), with blends of MOTE having pour point values that decreased most especially at 20 % blend. Comparative assessment of pour point values for MOTE and MEB-L blends of n-BE were seen to be lower than their EAA counterpart except at 10 % n-BE blend for MOTE which was seen

to be higher (11.2 °C) than its EAA blend of MOTE (10.5 °C) (Figure 10 & Figure 12). However, lower pour/cloud points are preferred (Sharma *et al.*, 2006; Alang *et al.*, 2018).

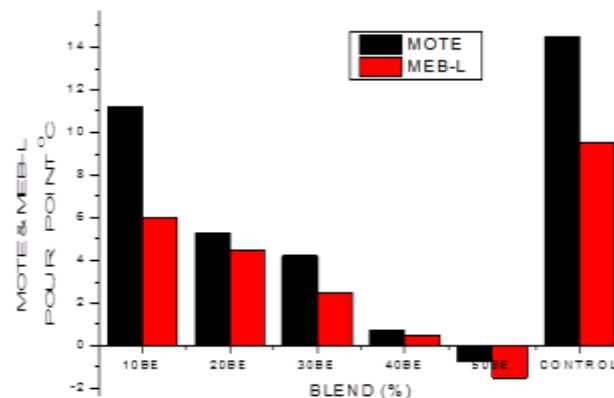


Fig 10: Showing variation of cloud point with increase in blend of n-BE

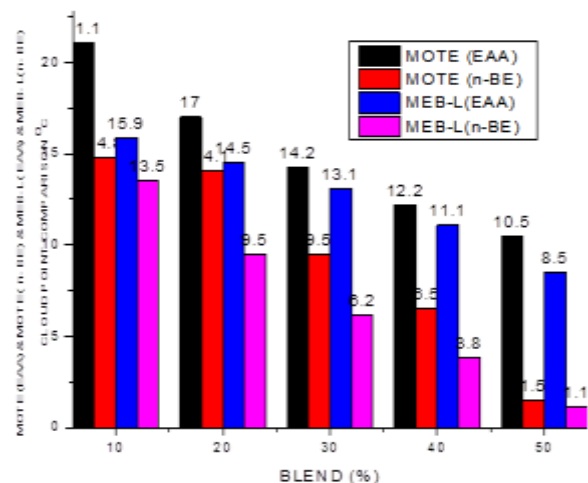


Fig 11: Represent comparison of cloud points of EAA & n-BE for both MOTE and MEB-L

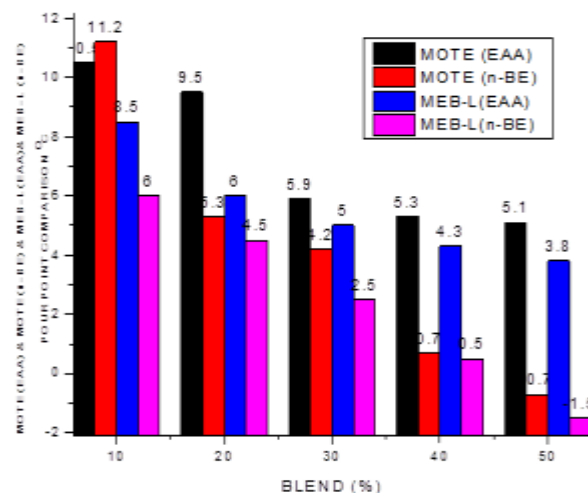


Fig 12: Represent comparison of pour points of EAA & n-BE for both MOTE and MEB-L

The results of the analysis of the cold flow properties reveal that various blend of MEB-L with n-BE portrays better pour and cloud point as they are lower than their counterpart (blends of MOTE with n-BE Figure 9 & Figure 10) and (blends of even MOTE & MEB-L with EAA for both cloud/pour points Figure 7 and Figure 8). The best pour/cloud point for MEB-L for various blend of n-BE occurred at 50 % blend represented by -1.5 °C and 1.1 °C respectively. The best pour/cloud point for MOTE for various blend of n-BE occurs at 50 % blend represented by -0.7 °C and 1.5 °C respectively. The low pour/cloud point values exhibited by blends of n-BE may not be far from the low melting point of n-BE which is -78 °C as compared to -45 °C of EAA.

**Conclusion:** Thermal studies revealed their sump tolerance temperature of above 250 °C for MEB-L and 300 °C for MOTE well above the sump tolerance temperature for conventional lubricant which is within the range of 230 °C to 260 °C, this relative stability of above 300 °C is high enough to support engine operation. The fluidity of the biolubricant improved with adjustment through blending with fluidity improves to suit low temperature applicability, however, among the two blended biolubricant MEB-L possessed a better low temperature fluidity at n-BE blend of 50 %.

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