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# Synthesis of Biolubricant *via* Chemical Modification from Vegetable Oil Blend using Heterogeneous Catalyst

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Present study reports the synthesis of biolubricant synthesis from the vegetable oil blend [dehydrated castor oil (DCO) and waste ricebran oil (WRBO)] using heterogeneous catalyst and its physico-chemical properties. The biolubricant was synthesized from blend of vegetable oils using two chemical modification processes *i.e.* synthesizing methyl esters of blend oil using sodium methoxide as catalyst and then epoxidation of synthesized methyl esters using Amberlite IR-120 strongly acidic ion-exchange resin. The biolubricant sample was examined by $^1$H & $^{13}$C NMR and FT-IR analysis. Viscosity index, rheological properties, thermal stability and pour point values of chemical and structurally modified blend oil (*i.e.* biolubricant) ($T_{onset}$ = 215 ºC, iodine value (IV) = 197.5, pour point (PP) = -4.5 ºC) showed notable improvement than that from blend oil methyl esters ($T_{onset}$ = 181 ºC, iodine value (IV) = 167.65, pour point (PP) = -9.5 ºC). Thus, a blend of non-edible oil (dehydrated castor oil) and waste cooking oil (WRBO) is likely to be used potentially as biolubricant basestock for hydraulic applications.

**Keywords: Epoxidation, Dehydrated castor oil, Ion-exchange resin, Thermal stability, Transesterification, Triglyceride.**

## INTRODUCTION

In recent years, an increase in rate of synthesis of lubricants from chemically modified vegetable oils has given a clear senario that people have become more sensitive towards pollution free environment. In present time, the mineral oil based and synthetic lubricants are most common products used by the lubrication industry, because of their toxic nature towards environment, modified vegetable oils have grown their importance. Due to deleterious impact of lubricants derived from mineral oil, lubricant industries have formulated few renewable, non-toxic, environment friendly biolubricants, better physico-chemical characteristic properties than mineral oil based or synthetic lubricants [1-3].

Chemical and structural modifications of vegetable oils have emerged out for its alternative use in synthesis of eco-friendly lubricants because of their relatable properties like high renewability, biodegradability, superior lubrication, high flash points, lower toxic values and better anti wear characteristics. Vegetable oils that were carried out for structural and chemical modification in synthesizing biolubricants are cottonseed [4], safflower [5,6], peanut [5], palm [7], *Jatropha curcas* [8,9], karanja [8], mustard [10], castor [11], tilapia [12], canola [6], corn [6], olive [6], peanut [6], tobacco seed [13] and soybean [6,14,15]. The performance of mustard oil for the synthesis of biolubricants using epoxidation and transesterification processes was studied and implied remarkable properties at varying operating conditions [10]. Recent literature has focussed on synthesis of biolubricants using waste cooking oil as basestock [16] or *via* blend of above vegetable oils and waste cooking oil, are considerate for biolubricant synthesis. Salimon *et al*. [17] studied the synthesis of biolubricants that are used in low temperature hydraulic oil applications. Suarez *et al.* [14] studied the epoxidation of methyl oleate using commercial alumina as heterogeneous catalyst and compared its catalytic activity with laboratory synthesized alumina fused with Lewis acid metals, in the presence of aq. hydrogen peroxide resulting in the increase in yield of the product, when the concentration of aqueous hydrogen peroxide, the quantity of catalyst or its surface area were increased. Borugadda & Goud [18] investigated the epoxidation of castor oil methyl esters using heterogeneous catalyst (Amberlite IR-120) as a chemical modification process for the synthesis of biolubricant.

This study reports the biolubricant synthesis from a blend of non-edible vegetable oil and waste oil which is not suitable for cooking purpose *i.e.* dehydrated castor oil (DCO) and waste ricebran oil (WRBO) through chemical and structural modification using strongly acidic cationic resin; Amberlite IR-120 as heterogeneous catalyst. The economic considerations and presence of desired percentage of unsaturation for chemical modification process was preferred for the selection of ratio of blend of DCO and WRBO to be 60:40. The processes involved in chemical and structural modification are transesterification and epoxidation. The first process *i.e.* transesterification intricated the synthesis of methyl esters of vegetable oil blend, after which the second step *i.e.* epoxidation of methylester was executed. The temperature effect on the physico-chemical properties of synthesized biolubricant *i.e.* viscosity and thermal stability was investigated. The final product *i.e.* biolubricant was characterized by $^1$H & $^{13}$C NMR and FT-IR spectral analysis.

## EXPERIMENTAL

Dehydrated castor oil (iodine value: 129 $I_2$/g, hydroxyl value: 7, average molecular weight: 881.15) was procured from Jayant Agro Organics Ltd. (Mumbai, India) and waste rice bran oil (peroxide value: 13.7, iodine value: 101) was procured from the residential hostel of Harcourt Butler Technical University (HBTU), Kanpur, India. The reactants involved in the experimental procedure and characterization techniques were of analytical grade. Methyl alcohol, glacial acetic acid, aq. hydrogen peroxide (30%), sodium methoxide and anhydrous sodium sulphate were acquired from Qualikems, India.

### General procedure

**Synthesis of blend oil methylesters (BOME):** The synthesis of blend oil (DCO and WRBO) methyl esters was performed according to the reported procedure [19]. Oil blend (200 g) was taken in a two-necked flat-bottomed flask, then sodium methoxide and methanol (oil:methanol = 1:12) were added to the flask, heated the mixture to 65 ºC and stirred mechanically for 3 h. The mixture was kept in a separating funnel and left overnight for the separation of methyl esters from glycerol and methyl esters. Further, luke warm distilled water was used for washing the methyl esters. Formation of methyl esters was confirmed using FT-IR and GC-MS analysis. Percentage methyl ester yield obtained was 98% wt. Methyl esters were further used for the synthesis of blend oil fatty acid methyl ester epoxides.

**Synthesis of methyl ester epoxides:** The epoxidation of BOME was accomplished according to Borugadda & Goud [18]. A two-necked flask (250 mL) was equipped on magnetic stirring with speed controlling regulator, one neck with a temperature measuring thermometer pocket and in the second neck a drop-ping funnel was attached. Blend oil methyl ester epoxides were synthesized by carrying out epoxidation reaction using methyl esters of blend oil (BOME), acetic acid and $H_2O_2$ in the molar ratio of 1:0.5:1.5), using heterogeneous catalyst Amberlite IR-120, a strong cationic ion-exchange resin in (15% wt.). BOME (50 g, 0.29 mol) was heated till 60 ºC to maintain the temperature at which the reaction takes place, followed by the addition of 8.7 g of acetic acid (0.5 mol) and 7.5 g of catalyst into the reaction system. After reaching the homogenization state of mixture, aq. $H_2O_2$ was added to the reaction mixture for 30 min. Then, continuous stirring of the mixture was allowed for 12 h for uniform mixing in the reaction system. After the completion of reaction, the product was washed with warm distilled water repeatedly, until all the catalyst and unreacted reagents were washed out to adjust the pH to neutral. The excess amount of solvent and water (in traces) were recovered using rotary evaporator. The yield of epoxidized methyl esters was 94%.

### Analytical methods

**Iodine value:** The iodine number of blend was determined by Wij's AOCS Cd 1-25 standard method, by titrating it against sodium thiosulfate solution. The iodine value was determined in all the three steps of the reaction.

**Acid value:** The acid value of samples of blend and its methyl ester were determined by AOCS Te 1a-64 standard method.

**Oxirane oxygen content (OOC):** The OOC content of the epoxide product was determined by AOCS Cd 9-57 standard methods. Theoretical oxirane oxygen content ($OOC_{th}$) and relative conversion to oxirane (%) was calculated using the following equation:

$$\mathrm{OOC_{th}} = \left( \frac{(\mathrm{IV_o})/(2\mathrm{A_i})}{100 + (\mathrm{IV_o})/(2\,\mathrm{A_i})\,\mathrm{A_o}} \right) \mathrm{A_o} \times 100 \quad (1)$$

where, $IV_o$ is the iodine value of BOME, $A_o$ and $A_i$ are the atomic weights of oxygen and iodine *i.e.* 16 and 126.9, respectively. The percentage relative conversion to oxirane was calculated as follows:

$$\text{Relative conversion to oxirane (\%)} = \left( \frac{\mathrm{OOC_{exp}}}{\mathrm{OOC_{th}}} \right) \times 100 \quad (2)$$

where $OOC_{exp}$ is the experimental value of oxirane oxygen and $OOC_{th}$ is the theoretical maximum oxirane oxygen present in 100 g of BOME epoxide.

**Specific gravity:** The specific gravity analysis of the blend oil was performed using a specific gravity bottle according to standard method ASTM D1298. The specific gravity was calculated at 30 ºC.

GC-MS**:** GC-MS analysis was performed using Agilent 5975C and 7890A model equipped with a split injector and auto-sampler and a capillary column (HP-5MS crosslined 5% PHME siloxane, 30 m × 0.25 mm i.d., 0.25 µm film thickness) and MS detector. The oven temperature was initially set at 60 ºC, hold for 5 min and then increased by the rate of 1.5 ºC/min to 230 ºC and then further hold for 3 min, increased to 280 ºC at 30 ºC/min, hold for 10 min. The temperature of injector was 250 ºC. Helium was used as the carrier gas. The split ratio was 1:12 and 1.0 µL of the sample was injected into GC-MS system.

**FT-IR:** Fourier transform- infrared spectral analysis was executed on ABB laboratory FT-IR spectrophotometer MB 3000 equipped with DTGS detector and with KBr beam splitter.

**NMR:** $^1$H NMR and $^{13}$C NMR spectral analysis was performed using the JEOL ECX-500 NMR spectrometer using $CDCl_3$ as solvent.

**Pour point (PP):** The pour point analysis was performed using pour point apparatus as stated in ASTM D97 standard methods. The specimen was cooled in the flask at a fixed rate and is exam-ined for every 3 ºC rise in temperature. The pour point deter-mination was done by keeping a test jar with sample filled in it in the cooling media and that was measured for every 3 ºC rise in temperature, prior to the sample stopped pouring.

**Thermal analysis:** Thermal characteristics of the epoxide product were established with the help of Perkin-Elmer thermal gravimetric analyzer (TGA 4000) instrument, varying the temperature range from 30 to 850 ºC with 10 ºC/min rise in temperature under nitrogen atmosphere.

**Fluid flow characteristics:** The flowing characteristics of the epoxidized product were studied using steady state rheology in Anton Parr Rheometer MCR102 equipment by varying shear rate (0.1-100 $s^{-1}$) with shear stress (Pa), while keeping the temperature factor constant. The above variables were calculated at 20, 40, 60, 80 and 100 ºC.

## RESULTS AND DISCUSSION

**Iodine value, acid value and specific gravity:** Epoxidation of BOME was carried out to chemically modify the structure of triglyceride by reducing percentage unsaturation content [calculated by iodine value (IV)] and converting the unsaturation to epoxy ring (OOC analysis), so that the thermal stability and lubrication properties of epoxidized product shows better result when compared to unmodified BOME [1]. This is because the double bond present in the triglyceride structure restricts its use as direct basestock for synthesizing biolubricant. Therefore, through the process of epoxidation, the structure of blended vegetable oil can be amended for its conversion from linoleic fatty acid group to its respective epoxide and simultaneously improving its thermal stability [30]. Lower the iodine value higher will be the thermal stability and *vice versa*.

The physico-chemical properties of BOME and epoxidized BOME *i.e.* biolubricant are given in Table-1. The iodine value (IV) of BOME was 128 $I_2$/g and after epoxidation it decreased down to 2 $I_2$/g. The lowering in IV of BOME epoxide depicted that almost 98% of double bond present in triglyceride was transformed into oxirane ring, resulting in the improvement of thermal stability.

TABLE-1
PHYSICO-CHEMICAL PROPERTIES OF BLEND OIL METHYL ESTER (BOME) AND EPOXIDIZED BOME (BIOLUBRICANT)

| Properties | Blend oil methyl ester (BOME) | Epoxidized BOME |
|---|---|---|
| Iodine value ($I_2$/g) | 128 | 2 |
| Acid value | 1.44 | 0.98 |
| Specific gravity (30 °C) | 0.9158 | 0.9317 |
| Pour point (°C) | -9.5 | -4.5 |
| Thermal stability ($T_{onset}$) (°C) | 181 | 215 |

Acid value (AV) of blended vegetable oil, BOME and its epoxide was found to be 3.25, 1.44 and 0.98 mg KOH/g respectively. Lesser the AV of epoxidized product, it will not cause any problems to the equipment of operation upon its utilization. The specific gravity of epoxidized BOME (0.9317) was found to be higher than its unmodified BOME (0.9158) and blended vegetable oil (0.9286).

**Gas chromatography-mass spectrometry:** The 60:40 ratio of BOME consists of 0.48% of oleic acid, 0.95% of ricinoleic acid, 67.08 % of 9,12-linoleic acid and 21.64 % of 9,11-linoleic acid. The linoleic acid content in the blend is desired for the epoxidation process for maximum conversion of unsaturation to its epoxide.

**Oxirane oxygen content (OOC) analysis :** The experimental value of oxirane oxygen was determined according to AOCS Cd-9, 120 standard methods and was found to be 7.09, while the theoretical OOC content was 7.466. Therefore, the relative percentage conversion of oxirane is found to be 95%.

**FT-IR studies:** The FT-IR spectrum was examined and executed on BOME and BOME epoxide *i.e.* biolubricant. The FT-IR spectrum of blend oil methyl ester (BOME) and BOME epoxide (biolubricant) is shown in Fig. 1. The key IR bands (KBr, $cm^{-1}$) of BOME: 2923 (C-H), 1743 (-C(=O)-$CH_3$), 1442, 1365 (C-H), 1243 (C-O); Biolubricant: 2923 (C-H *str.*), 1735 (-C (=O)-$CH_3$), 1458, 1442, 1365 (C-H), 1242 (C-O).

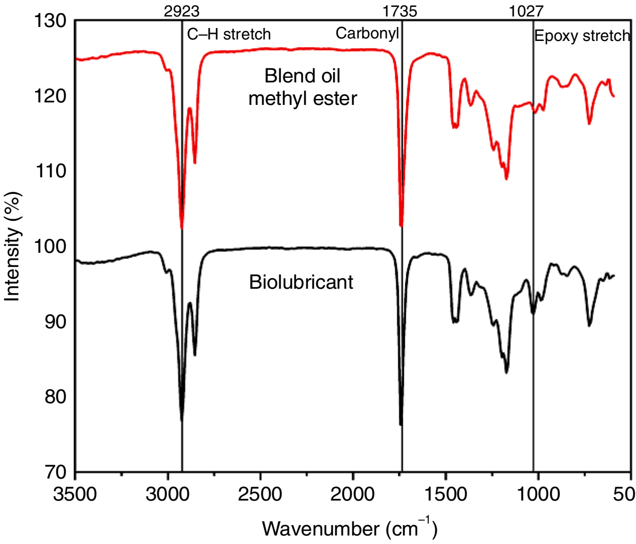

Fig. 1. FT-IR spectral analysis of BOME and epoxidized BOME (biolubricant)

**NMR studies:** $^1$H NMR spectra in Figs. 2 and 3 revealed the formation of BOME and biolubricant, respectively. $^1$H NMR ($CdCl_3$) (BOME): 4.363-4.448 (m, 4H, C-O-$CH_3$), 2.621-2.642 (d, 1H, C=O), 2.367-2.388 (d, 1H, C=O), 1.818 (s, 5H, alkane (-$CH_3$-$CH_2$)), 1.271-1.300 (t, 1H, C=O), 1.204 (s, 1H, C=O),

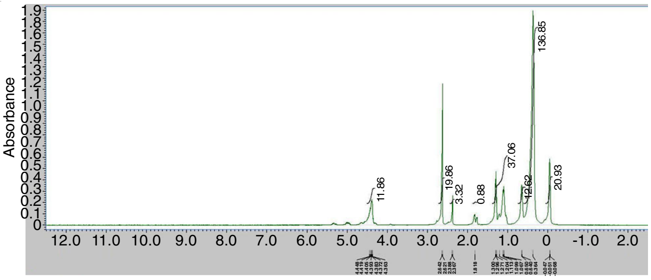

Fig. 2. $^1$H NMR spectra of blend oil methyl ester

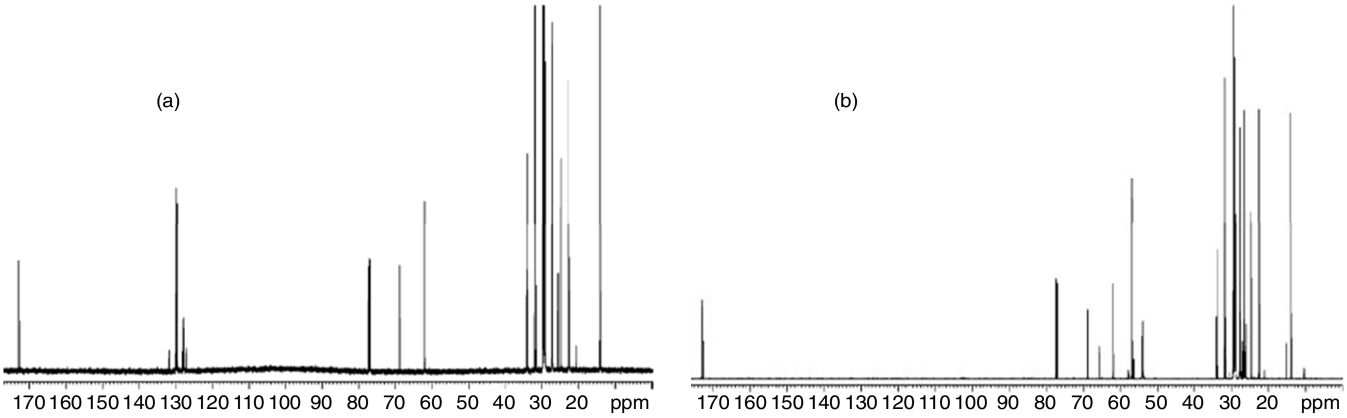

Fig. 4. $^{13}$C NMR spectra of (a) blend oil and (b) biolubricant

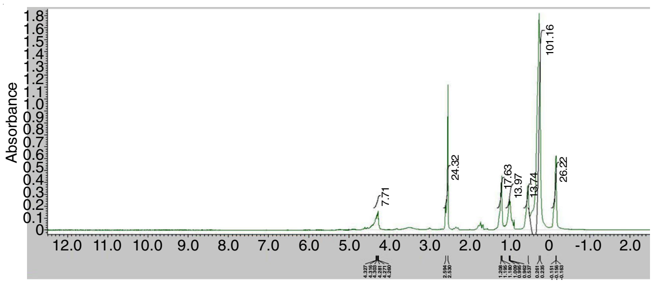

Fig. 3. $^{1}$H NMR spectra of biolubricant

1.087-1.113 (t, 1 H, C=O), 0.640-0.650 (d, 5H, alkane (-$CH_3$-$CH_2$)), 0.364 (s, 3H, alkene (-CH=$CH_2$)). $^{1}$H NMR ($CdCl_3$) (biolubricant): 4.260-4.327(m, 4H, C-O-$CH_3$), 2.530-2.594 (d, 1H, C=O), 1.180-1.208 (t, 1H, C=O), 0.982-1.009 (t,1H, C=O), 0.537 (s, 5H, alkane (-$CH_3$-$CH_2$)), 0.235-0.261 (d, 3H, alkene (-CH=$CH_2$)).

$^{13}$C NMR ($CdCl_3$) spectrum (Fig. 4) clearly reveals that the biolubricant product (b) shows that the unsaturation [21] which was observed in (a) the 60:40 blend ratio; present in the triglyceride blend of DCO and WRBO at 126.89-129.58 ppm (m, -CH=$CH_2$) has been modified to its epoxide; 12.9-13.1 (d, -$CH_3$-$CH_2$), 24.05-31.78 (m, -$CH_2$-), 34.5 (d,-$CH_2$C=O), 54.71-58.32 (m, -CH-O-CH-), 63.13 (s, -$CH_2$-O-C=O).

**Pour point (PP):** The PP value of the epoxidized BOME (biolubricant) was determined to be -4.5 °C and that of BOME was -9.5 °C, which shows that biolubricant can be used in the equipments involving ambient temperature conditions. The difference in the pour point values of BOME and biolubricant is because of the presence of unsaturation in BOME as its presence shows a positive impact on higher PP values; *i.e.* more the double bond content present in a sample, less will be its pour point temperature [22]. Knothe *et al.* [23] studied about the involvement of long-chain saturated fatty-acids, which were essential for increasing the flow properties was 2 %, but the feedstocks used for biolubricant synthesis the percentage involvement of long-chain saturated fatty-acids to increase the flow properties was 8%. The pour point values of the epoxidized BOME can be enhanced by additivation or implying ring opening reaction using long chain alcohols [12].

**Thermal analysis:** In thermal curve, the temperature extent of dissipation and decomposition of hydrocarbons range between 150-450 °C, focussing on the mortification of hydrocarbon compounds having less molecular weight [24].

In Fig. 5, the TGA of biolubricant in nitrogen atmosphere shows three levels: the first stage, 150-480 °C ($T_{onset}$= 215 °C) shows the decomposition of lower molecular weight hydrocarbon compounds. Subsequently, 2nd and 3rd levels of degradation from 280-365 °C and 367-480 °C depicts the decaying of high molecular weight hydrocarbon compounds. The TGA curve of BOME ($T_{onset}$ = 181 °C) shows single stage from 140-480 °C, which represents the elimination of hydrocarbon compounds. Thus, from the above discussion, it is clear that the biolubricant is thermally stable and its stability could further be improved by additivation, for enhancing thermal stability [16,25].

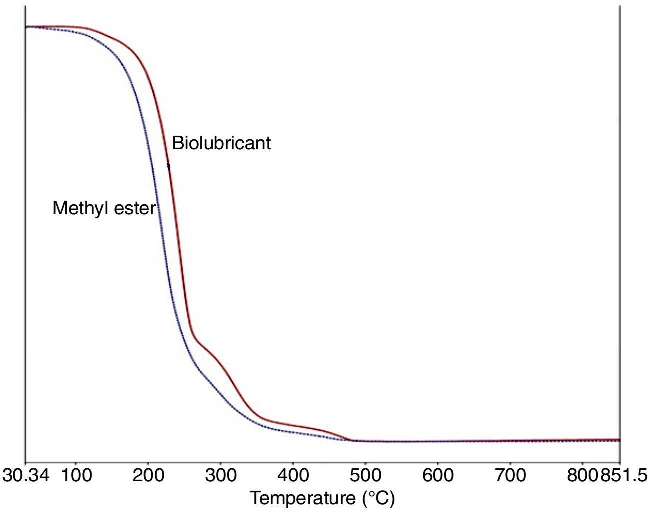

Fig. 5. TGA curve of blend oil methyl ester (BOME) and biolubricant

**Fluid flow characteristics:** The fluid flow property which plays a very crucial role in the physicochemical characteristics of a lubricant is its viscosity as studied by Erhan & Perez [26]. The kinematic viscosity (at 40 °C) of BOME epoxide (31.54 Cst) was more than BOME (10.61 Cst). The reason behind greater viscosity of BOME epoxide may be because of formation of oxirane ring at place of unsaturation, thereby increasing the polarity and molecular weight of the product when compared to unmodified BOME. The rheological variables like viscosity were studied on anton parr rheometer MCR102 with parallel plate of 30 mm diameter, using steady state rheology, in which the shear rate (varied from 0.1-100 s$^{-1}$) was plotted against shear stress (Pa), while keeping the temperature factor constant. These parameters were studied at different temperatures *i.e.* 20-100 ºC (20, 40, 60, 80 and 100 °C) (Fig. 6).

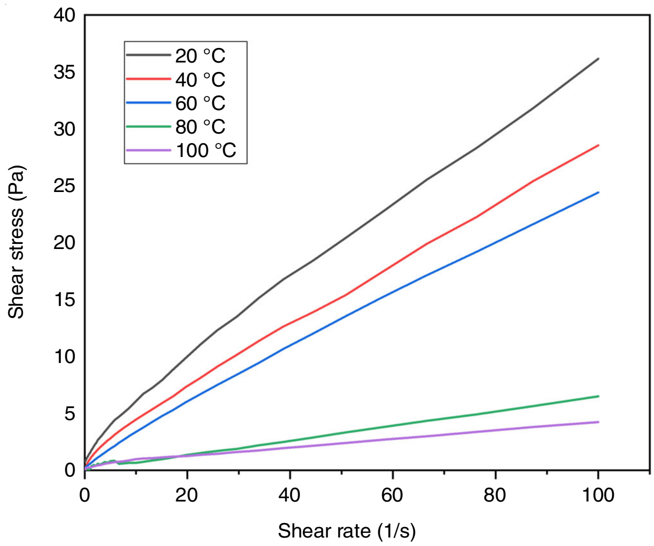

Fig. 6. Rheological study of biolubricant

Power law formula states:

$$\text{Shear stress} = K^* \,(\text{shear rate})^n$$

where, K = Viscosity and n states nature of fluid.

The viscosity index of biolubricant was determined using ASTM D2270 standard method and was found to be 197, which falls in range of IV values of standard lubricants derived from petroleum sources (IV ≥ 117), according to the Brazilian National Petroleum Agency (ANP), that has maintained the standard value of IV for synthetic lubrication to be more than 120 (ANP, 2014) [27].

Table-2 clearly showed that the viscosity (K) at 100 s$^{-1}$ is incessantly falling when the temperature is increased from 20 to 100 ºC. Also, the value of n is less than 1 in all cases, which specifies the nature of biolubricant (BOME epoxide) to be pseudoplastic. The R$^2$ value for the model, calculated from the power law parameter is 0.99~1, indicating the fitted nature of model.

TABLE-2
POWER LAW PARAMETER FOR BIOLUBRICANT

| Temp. (°C) | K (Pa s$^n$) | n (Dimensionless) | R$^2$ |
|---|---|---|---|
| 20 | 2.21759 | 0.76541 | 0.99175 |
| 40 | 0.79541 | 0.79257 | 0.99351 |
| 60 | 0.64891 | 0.80247 | 0.99638 |
| 80 | 0.54621 | 0.69542 | 0.99091 |
| 100 | 0.20811 | 0.71407 | 0.98179 |

## Conclusion

The biolubricant was synthesized from a blend of DCO and WRBO in the ratio of 60:40 in the presence of strongly acidic cationic heterogeneous catalyst Amberlite IR-120 ion-exchange resin. The synthesis involved chemical and structural modification steps *i.e.* transesterification reaction (formation of methyl esters of blend oil) using dehydrated castor oil and waste rice bran oil in 60:40 ratio by weight (yield: 98%) and epoxidation of the synthesized methyl esters using heterogeneous catalyst (yield: 94%, OOC: 7.09). The spectroscopic data *i.e.* FT-IR, $^1$H & $^{13}$C NMR indicated that the unsaturation content (C=C bond) present in the blend oil was transformed to oxirane ring (C-O-C) peaks of epoxy product and carbonyl compound (-COCH$_3$) peaks in transesterified product, leading to the confirmation of final epoxidized product. The pour point and thermal stability were also found to be in the range of the characteristic properties of biolubricants and hence they can be used for hydraulic applications. The biolubricant can further be used in high temperature applications by adding additives like thermal stability and pour point improvers. The viscosity measurement of the biolubricant at 100 s$^{-1}$ shear rate was found to be decreasing while increasing the temperature. The viscosity index of biolubricant was found to be 197.5, which very well lies within the limits of standards of biolubricant.

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## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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