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Comparative Study of Methods of Chemical Modification of Dehydrated Castor oil for Synthesis of Biolubricant

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Vegetable oil blends are the emerging alternative feedstocks, which are being used to synthesize environment friendly lubricants. This work focuses on the Comparison of the methods of chemical modification, which are used for the biolubricant synthesis and also compares the resultant behaviour characterized on their thermal stability and viscosity. The biolubricant products are synthesized using two chemical modification methods. Method 1: Epoxidation of vegetable oil followed by transesterification of epoxidized oil. Method 2: Preparation of methyl esters (Transesterification) followed by epoxidation of methyl esters. Biolubricant derived from method 1 shows better thermal stability (T_{onset} = 338 ºC) compared to method 2 (T_{onset} = 205 ºC). The viscosity indices and pour point values of the products were found to be 222; 4.6 ºC and 216; 6.0 ºC, respectively. Thus, the biolubricant derived from first method showed better physio-chemical properties and were comparable and a blend of non edible oil and waste cooking oil acts as potential basestock for biolubricant synthesis.

Keywords: Dehydrated castor oil, Epoxidation, Transesterification, Biolubricant, Thermal stability, Rheology.

INTRODUCTION

From ancient times, lubricants have been playing very crucial role in serving all the emerging industries like automobile industry and other machinery involving industries. Lubricants serve as the major part in reducing the heat due to friction, minimizing the losses of energy and bringing smoothness among the moving surfaces. In the present scenario, metal working fluids, hydraulic fluids, cutting oils, industrial gear oils and many more fluids are being used as lubricant base oils, in the market [1-3]. The finite resources of crude oil, risen prices and their adverse effect on the environment have clearly indicated that possess harmful characteristics towards the environment. Chemically modified vegetable oils have been emerged out for being the alternative resource for synthesizing environment friendly lubricants as they have relatable properties *i.e.* high biodegradability, renewability, better lubricity, higher flash points, less toxicity and their good anti-wear characteristics. Vegetable oils which have been chemically modified for the synthesis of high temperature biolubricants are cottonseed [4], safflower [5,6], palm [7], *Jatropha curcas* [8,9], karanja [8], mustard [10], castor [11], tilapia [12], canola [6], corn [6], olive [6], peanut [5,6], tobacco seed [13] and soybean [6,14]. Kulkarni *et al.* [10] studied the performance of mustard oil through epoxidation and transesterification and showed excellent properties at diverse operating conditions. Recent researches are mainly focusing on preparation of eco-friendly lubricants derived from waste cooking oils [15] and blend of some of the above vegetable oils of edible and non-edible origin, which are favourable for preparation of biolubricants. Reeves *et al.* [6] studied that a stable polymeric film is formed on the surface of the metal when the synthesized product is used for lubricating application, as they have a better coefficient of friction. Kamlakar *et al.* [16] studied the use of epoxidized thumba oil as a basestock for aviation grade lubricants and its chemical modification (epoxidation) leads to its increasing demand in metal working applications.

This work focuses on the synthesis of biolubricant from a blend of vegetable oils *i.e.* dehydrated castor oil (DCO) and waste cooking ricebran oil (WRBO) *via* chemical modification of the blend. This work also shows the comparison of the methods of chemical modification of vegetable oil through which the biolubricant is synthesized. The economic considerations and presence of unsaturation was the basis for selecting a blend ratio of 60:40 of DCO and WRBO, respectively.

Chemical modification involved processes *i.e.* epoxidation and transesterification. The synthesis of biolubricant involved two methods, which further involved the above two processes. In the first method, epoxidation of vegetable oil was carried out, followed by the transesterification of the epoxidized vegetable oil; and the second method involved the preparation of fatty acid methyl esters of blend oil *i.e.* transesterification, followed by the epoxidation of methyl esters. Epoxidation process involved ring opening, which improves the lubrication properties. The effect of temperature on the physico-chemical properties like thermal stability [9,10,15] and viscosity [17] of the synthesized biolubricant through both methods were deter-mined using thermal gravimetric analyzer (TGA) and rheometer, respectively. The synthesized biolubricant was also analyzed and confirmed by FT-IR, ^{1}H NMR and ^{13}C NMR.

EXPERIMENTAL

Dehydrated castor oil (DCO) (iodine value-129 I_2/g, hydroxyl value-7, average molecular weight- 881.15) was obtained from Jayant Agro Organics Limited (Mumbai, India) and waste cooking ricebran oil (WRBO) (peroxide value-13.7, iodine value-101) was obtained from a local restaurant in Kanpur city, India. The reagents used in the synthesis and characterization methods were of analytical grade. Other chemicals and solvents *e.g.* methyl alcohol, sulphuric acid (98%), formic acid (85%), aq. hydrogen peroxide (30%), ethyl acetate, toluene, sodium bicarbonate, sodium methoxide, isoamyl alcohol, *p*-toluene sulfonic acid, Wijs' solution and sodium sulphate anhydrous were purchased from Nath Chemicals Corporation Limited, Kanpur, India.

Epoxidation followed by transesterification (Method 1)

Synthesis of epoxidized blend oil (EBO): Epoxidation of the blend oil was carried out according to the reported method [17]. DCO: WRBO (60:40) (250 g, 0.285 mol), formic acid (22.15 g, 0.48 mol), H_2SO_4 (3 mL, 2% w/w of HCOOH and H_2O_2) were stirred in a three-necked flask, using electric stirrer in a water bath. Hydrogen peroxide solution (220 mL, 2.78 mol) was added gradually to the above mixture through a dropping funnel for duration of 4 h and the temperature of the system was maintained at 60-65 ºC. This was done to avoid the overheating of the system. The above system was then stirred again at the same temperature for the complete conversion. The reaction mixture was then cooled down and washed with water until it becomes acid free. To recover the product from water phase, ethyl acetate is used, followed by the removal of moisture by passing the mixture through anhydrous sodium sulphate. The weight of the synthesized epoxidized oil was 258.5 g (95 wt.% of theoretical yield) with an oxirane value (OV) of 5 using AOCS Cd 9-57 standard method and iodine value of 4 I_2/g using AOCS Cd 1-25 standard method [18].

Transesterification of epoxidized blend oil (TEBO): The synthesis of methyl esters of vegetable oil blend was carried out as proposed by Valle *et al.* [12]. Epoxidized blend oil (EBO) (100 g), methanol (90 mL, 10% excess) and sodium methoxide (1.5 g, 1.5 wt.%) were stirred at 60-65 ºC for 3 h. The unreacted methanol was then recovered under reduced pressure using rotary vacuum evaporator. The product was then passed through anhydrous sodium sulfate. The synthesized transesterified lubricant (TEBO) was then analyzed and confirmed by FT-IR, ^{1}H & ^{13}C NMR.

Transesterification followed by epoxidation (TFE) (Method II)

Synthesis of methyl esters of blend oil (MEBO): The preparation of methyl esters of blends of DCO and WRBO is previously described earlier [19]. Oil blend (200 g) was heated at 60 ºC and sodium methoxide dissolved in methanol (oil: methanol = 1:12) was further added to the reaction system under stirring. The reaction mixture was then maintained at 60-65 ºC under vigorous stirring for 3 h. The obtained product was kept in a separating funnel for another 3 h for letting the glycerol layer separate out from the mixture of methyl esters and glycerol. After separation, the methyl esters were washed with warm distilled water to remove the soluble catalyst impurities. After washing, the obtained methyl esters were passed through anhydrous sodium sulphate. They were then characterized for NMR, GC-MS and other physico-chemical properties. The yield of methyl esters was 98% (wt.).

Synthesis of methyl ester epoxides (EMEBO) and ring opening: The synthesis of methyl esters epoxides was carried out as proposed by Campanella *et al.* [20]. The experimental setup consisted of a three necked flask (1000 mL capacity), equipped on a magnetic stirrer with speed controller, temperature measuring thermometer pocket and a dropping funnel. Methyl esters (20 g), toluene (50 mL for each 20 g of methyl ester, ME) and formic acid (14.2 mL; formic acid: ME, 1:1) were stirred in the flask. After homogenization of the mixture for 5 min, aqueous H_2O_2 (100 mL, molar ratio ME: H_2O_2, 1:4) was poured through a dropping funnel and added for a period of 1 h. The mixture was then vigorously stirred at room temperature for 20 h. The two layers were separated from the mixture after washing with $NaHCO_3$ solution, till it neutralizes completely. The mixture was then passed through sodium sulfate anhydrous, followed by the solvent removal using under reduced pressure. The obtained epoxidized methyl esters were characterized for their physio-chemical properties. The FT-IR, ^{1}H & ^{13}C NMR analysis was also performed for structure determination. The yield of epoxidized methyl esters was 93% by wt.

The ring-opening of the methyl ester epoxides was carried out following the methodology studied by Salimon *et al.* [21]. Epoxidized methyl esters (20 g, 0.075 mol) were added to toluene (50 mL) along with 5% *p*-toluene sulfonic acid (w/w) as catalyst and then calculated amount of isoamyl alcohol (molar ratio of ME:alcohol, 1:3) was added dropwise for a course duration of 1.5 h at 75 ºC [22]. The temperature of the mixture was then raised till 90 ºC and refluxed for 3.5 h. At room temperature, the triesters were washed with 10% $NaHCO_3$ solution till it becomes acid free. The mixture was then filtered and passed over anhydrous sodium sulfate. Excess isoamyl alcohol and the solvent (toluene) were recovered through rotary vacuum evaporator. The product was analyzed and confirmed by FT-IR and ^{1}H NMR. For thermal stability, viscosity index (VI), pour point (PP) determination, thermal and rheometer analysis were also performed.

FT-IR: FT-IR spectroscopy analysis was performed using ABB laboratory FT-IR spectrometer MB3000 instrument fitted with DTGS detector and KBr beam splitter.

NMR: ^{1}H & ^{13}C NMR spectra were recorded using the JEOL ECX-500 spectrometer at a frequency of 500 MHz. Deuterated chloroform ($CDCl_3$, Qualikems) was used as a solvent.

Pour point: Pout point was determined using CPP cloud point and pour point apparatus according to ASTM D-97 standard method. The sample was cooled into the apparatus at a certain rate and then checked for every 3 ºC increase in temperature. The pour point was determined by placing a test jar filled with the sample into the cooling medium, which was measured for each 3 ºC increase in temperature, until the sample stops pouring.

Thermal stability: Thermal stability of the synthesized biolubricant was determined using Perkin-Elmer Thermal Gravimetric Analyzer (TGA 4000), from a temperature of 30 to 850 ºC at a rate of 10 ºC/min in the inert (N_2) atmosphere.

Viscosity measurement: The fluid property characteristics of the synthesized lubricant was determined using the Anton Parr Rheometer (MCR-102) and varying the shear rate from 1 to 100 (1/s) against shear stress (Pa) at constant temperature. These parameters were studied at 20, 40, 60, 80 and 100 ºC.

RESULTS AND DISCUSSION

FT-IR studies: FT-IR spectroscopy analysis was performed on epoxidized blend oil (EBO) and transesterified and epoxidized blend oil (TEBO) using ABB laboratory spectrometer MB3000 instrument equipped with DTGS detector and KBr beam splitter. The FT-IR spectrum of lubricant synthesized from method 1 (EFT) and method 2 (TFE) is shown in Fig. 1, respectively. The spectrum reveals: (a) TEBO: 2360 ($-COCH_3$), 1728 (C=O), 1373 (C-H), 1242 (C-O), 1041 (C-O). (b) EMEBO: 2923 (C-H), 2854 (C-H), 2360 ($-COCH_3$), 1728 (C=O), 1442 (C-O-H), 1357 (C-H), 1172 (C-O), 1095 (C-O).

NMR studies: ^{1}H & ^{13}C NMR spectra were recorded using the JEOL ECX-500 spectrometer. The ^{1}H NMR reveals the formation of TEBO (Fig. 2) and EMEBO (Fig. 3), respectively. TEBO: ^{1}H NMR ($CDCl_3$) 4.05-4.22 (m, 4H, $C-O-CH_3$), 2.22-2.25 (t, 1H, C=O), 1.39-1.53 (s, 5H, alkane ($-CH_3-CH_2$)), 1.18-1.24 (d, 1H, C=O), 0.79-0.80 (d, 5H, alkane ($-CH_3-CH_2$)). EMEBO: 5.74 (s, 2H, C=C), 3.58 (s, 4H, $C-O-CH_3$), 2.29-2.32 (t, 1H, C=O), 2.21-2.24 (m, 1H, C=O), 1.43-1.54 (m, 5H, alkane ($-CH_3-CH_2$)), 1.37 (s, 5H, alkane ($-CH_3-CH_2$)), 1.18-1.22 (t, 1H, C=O), 0.79-0.85 (m, 5H, alkane ($-CH_3-CH_2$)).

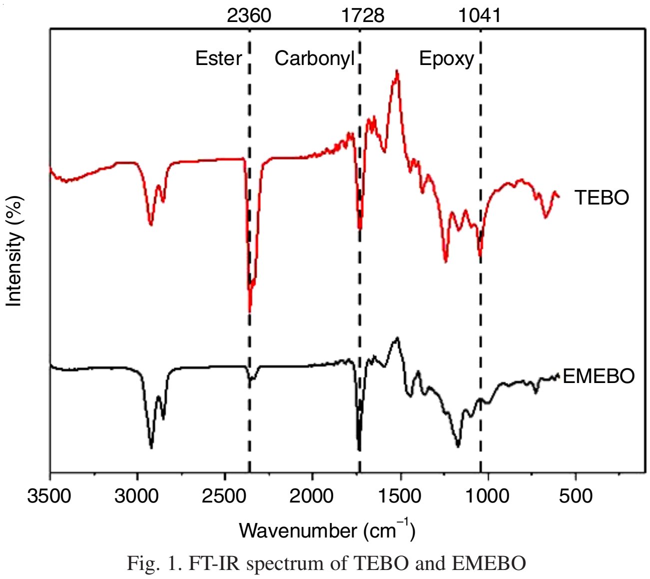

Fig. 1. FT-IR spectrum of TEBO and EMEBO

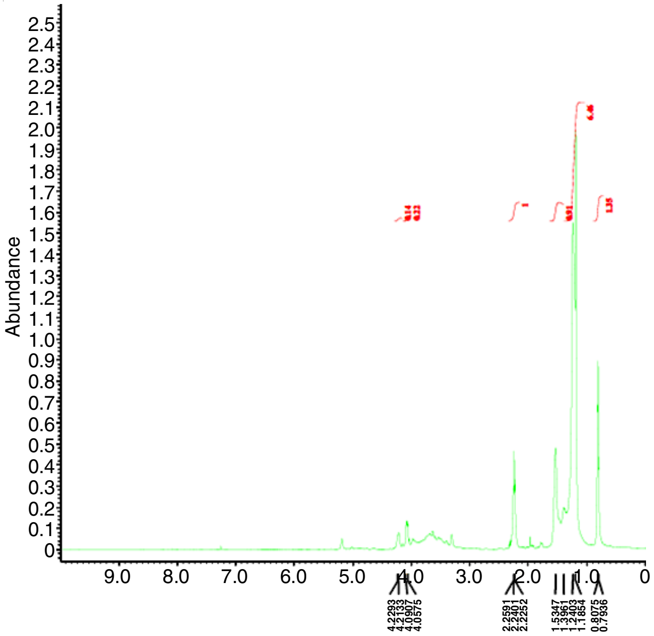

Fig. 2. ^{1}H NMR spectra of TEBO

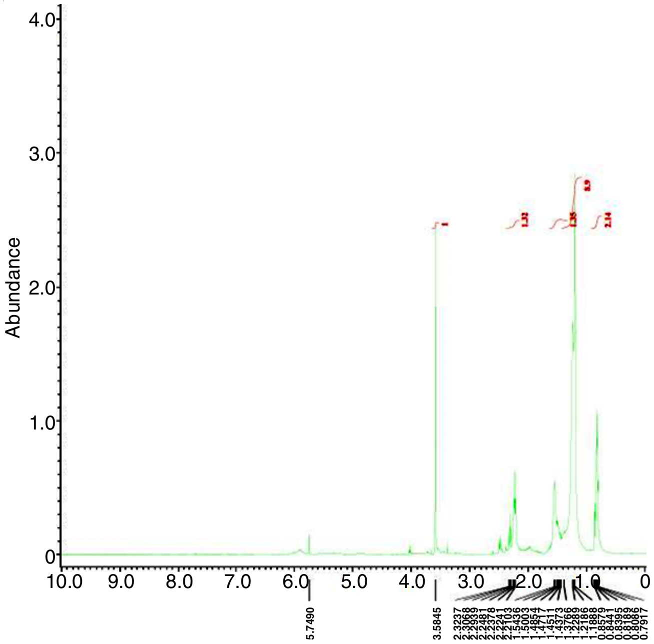

Fig. 3. ^{1}H NMR spectra of EMEBO

C NMR spectra (Fig. 4) clearly revealed an unsaturation at 127-130 ppm in the blend of 60:40 ratio of DCO and WRBO; present in the triglyceride blend. The epoxidized oil shows that the unsaturation [23] which was observed at 127-130 ppm has been transformed and converted to its epoxide; 13.2 (d, $-CH_3-CH_2$), 22.5-34.5 (m, $-CH_2-$), 34.5 (d, $-CH_2C=O$), 54-58.5 (m,

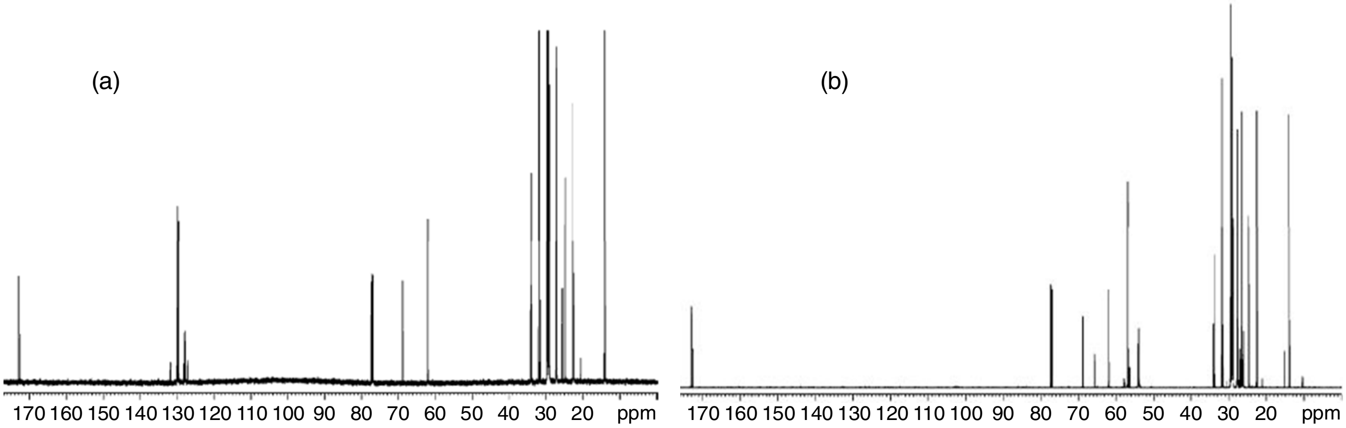

Fig. 4. ^{13}C NMR ($CdCl_3$) spectra of TEBO (a) and EMEBO (b)

-CH-O-CH-), 62-69 (s, $-CH_2-O-C=O$), 78.5 (-CHOC=O), 173.5 (-COO-)

Thermal gravimetric analysis (TGA): The temperature range of both evaporation and degradation of hydrocarbons lies from 150-350 ºC occurs, focusing on the degradation of lower molecular weight hydrocarbons [24]. TGA results of TEBO in N_2 atmosphere represent two stages: the first is from 220-490 ºC (T_{onset} = 338 ºC) shows the elimination of low molecular weight products followed by degradation of hydrocarbon compounds and the second stage of mass loss is from 510-625 ºC shows the decomposition of higher molecular weight hydrocarbons (Fig. 5). TGA graph of EMEBO (T_{onset} = 205 ºC) shows four stages: the first and second are from 190-260 and 260-410 ºC; shows the elimination of low molecular weight products followed by degradation of hydrocarbon compounds and the third and fourth stages of mass loss are from 410-480 ºC and 490-595 ºC; showing the decomposition of higher molecular weight hydrocarbons.

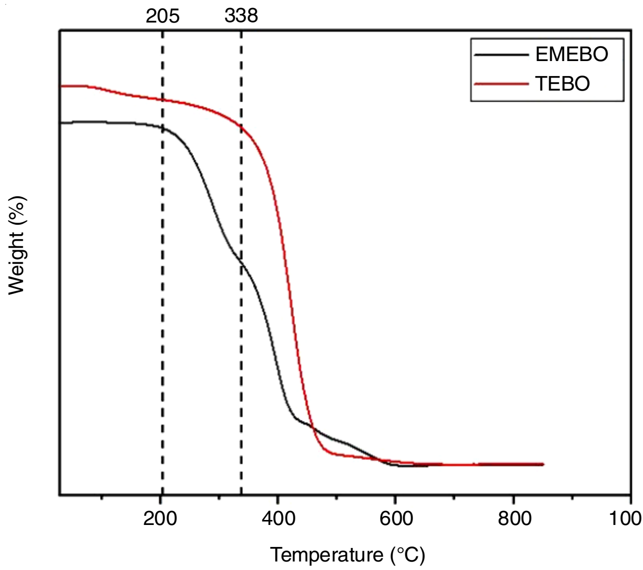

Fig. 5. Thermogravimetric curve of TEBO and EMEBO

Thus, it is clear that the biolubricant derived from the first method (TEBO) is more thermally stable than the biolubricant (EMEBO) synthesized from second method. It can be added that after the addition of additives like thermal stability improvers [15,16], EMEBO lubricant provides better thermal stability.

Pour point: Pour point values indicate the ideal operating conditions of temperature for a lubricant to function for specific applications. The pour point temperature of the TEBO and EMEBO was found to be 4.6 and 6.0 ºC, respectively, which shows that both of the biolubricant samples can be used in the machinery operating at high temperature climates. Generally, the conventional petroleum based lubricants consist of major proportions of *n*-paraffin compounds, which get easily crystallized at lower temperatures when compared to aromatic and cyclic hydrocarbons. The study of Knothe *et al.* [25] shows that the presence of long chain saturated fatty acids required to increase the flow behaviour is only 2% and in the feedstock used for the synthesis of biolubricant, *i.e.* the blend of 60:40 ratios of DCO and WRBO, the percentage of long chain saturated fatty acids is 8%. So, the flow behaviour of the biolubricant samples is a promising part, to be used in warm climates.

Viscosity measurement: Another important parameter, which plays an important role in the physico-chemical characteristics of the lubricant is the viscosity and was studied by Erhan & Perez [26]. Yunus & Luo [27] studied the presence of hydroxyl groups has a very strong impact on the viscosity of the synthesized biolubricant product. This is because of the long chain alcohol used for the ring opening reaction; here in this case, isoamyl alcohol is used, that does not contain branches in its structure, which may lead to much higher viscosity. The rheological parameters like viscosity of TEBO and EMEBO, on the basis of shear rate, shear stress and temperature were performed on Anton Parr Rheometer instrument MCR-102. Temperature was varied from 20-100 ºC (20, 40, 60, 80 and 100 ºC) on a varying shear rate of 0.1-100 s^{-1}, using parallel plate with 30 mm diameter and is depicted in Fig. 6.

The power law was applied by using the formula:

$$\text{Shear stress} = \text{K. (Shear rate)}^{n}$$

where, K = viscosity and n defines the nature of fluid

For a machine to work over a wider range of temperature, it requires the lubricant to pursue a higher viscosity index value. Dabai *et al.* [28] studied the effect of temperature on viscosity of biolubricant; higher the viscosity index, lower will be the

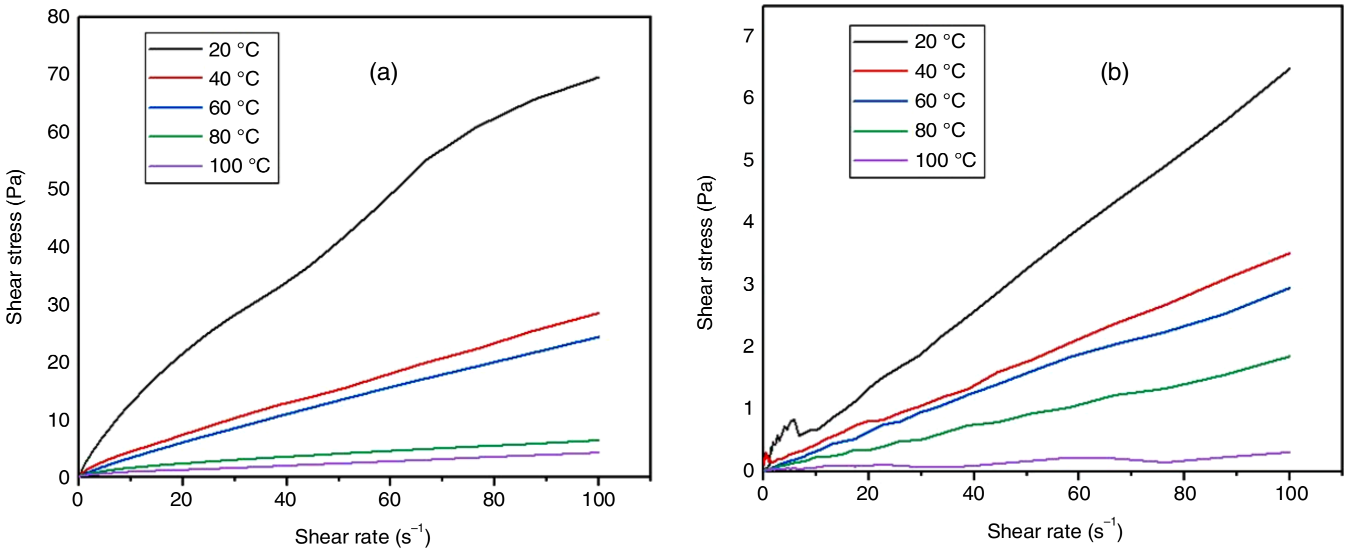

Fig. 6. Rheological study of TEBO (a) and EMEBO (b)

effect of temperature on the viscosity of the lubricant. The viscosity index of TEBO and EMEBO was also determined and found to be 222 and 216, respectively which lies in the range of VI of conventional petroleum based lubricants (VI ≤ 117), according to the Brazilian National Petroleum Agency (ANP), which requires the VI of synthetic lubricants should be higher than 120 [29].

From Table-1, it is clearly shown that the viscosity (K) at 100 s^{-1} is continuously decreasing as the temperature increases from 20 ºC to 100 ºC and the value of $n < 1$ in all the cases, which indicates that the synthesized lubricants either by any methods are pseudoplastic in nature. The adjusted R^2 value are 0.99~1, this indicates that the models are fitted.

Conclusion

The synthesis of biolubricant was performed using two chemical modification methods using a blend of dehydrated castor oil (DCO) and waste cooking ricebran oil (WRBO) in the ratio of 60:40. The two methods were epoxidation followed by transesterification (EFT) and transesterification followed by epoxidation (TFE). In the first method, epoxidation of vegetable oil was carried out with a high yield of 95 wt% and an oxirane value of 5; followed by the second step in which methyl esters of epoxidized vegetable oil were synthesized (transesterification reaction), which also gave 97 wt.% yield of the biolubricant product. In the second method, the vegetable oil blend was transesterified to its methyl esters and gave the yield of 98 wt.%; followed by epoxidation of methyl esters which gave the yield of 93 wt.%. The spectroscopic analysis FT-IR, ^{1}H & ^{13}C NMR clearly indicate the functional group peaks of C=C unsaturation in the blend of DCO and WRBO (60:40), C-O-C peaks of epoxy product and $-COCH_3$ carbonyl group in transesterified product; which confirms the biolubricant product synthesis. The thermal and oxidative stability were also found to be in the range of the characteristic properties of biolubricants. TEBO sample of biolubricant shows the better thermal stability property (T_{onset} = 338 ºC) than the EMEBO (T_{onset} = 205 ºC) biolubricant sample, For this reason, TEBO sample can be used for higher temperature applications. 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 TEBO and EMEBO was found to be 222 and 216, respectively; which very well lies within the limits of standards of biolubricant.

This study was focused on comparing the methods of chemical modification and yield from them; for the synthesis of biolubricants. Thus, it results to the conclusion that the first method, *i.e.* epoxidation followed by transesterification (EFT) method was more effective and efficient in synthesizing the biolubricant, which has the better thermal stability as compared to the latter method (TFE). Therefore, a blend of DCO and WRBO can be used as an alternative and attractive substitute to mineral oil, as feedstock for the synthesis of biolubricants for high temperature applications.

TABLE-1
POWER LAW PARAMETERS FOR TEBO AND EMEBO

Temperature (°C)	K (Pa.s^n)		n (Dimensionless)		R^2	
	TEBO	EMEBO	TEBO	EMEBO	TEBO	EMEBO
20	2.19195	0.09019	0.75734	0.93309	0.99832	0.99924
40	0.67461	0.06305	0.80731	0.91513	0.99814	0.9878
60	0.46457	0.05816	0.85903	0.88333	0.99976	0.97096
80	0.41463	0.02252	0.58819	0.94972	0.99843	0.99587
100	0.19851	0.01976	0.64924	0.54279	0.98746	0.87493

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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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