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Synthesis, theoretical studies and viscosity correlations of ricinoleic acid based methyl ester soap solution

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The use of vegetable oils for industrial applications needs chemical modification to improve its properties. For coolants and lubricant applications, the viscosity of vegetable oils plays a vital role which decides the heat dissipation and flow characteristics of vegetable oils. In this study, the viscosity prediction modeling of binary mixtures has been made using refutes empirical relations. The blend viscosity from the model depends on the blend ratios of the unsaturated fatty acids in castor oil and methanol. This blending of castor oil and methanol follows KOH assisted transesterification process to form Fatty Acid Methyl Ester (FAME) soap solution. The transesterified blend mixes have been experimentally analyzed for viscosity using Brookfield viscometer. The measured values of viscosities of the blend mix have been compared with viscosities obtained from refutes binary blend mix model. The percentage deviation between the experimental viscosities and Refutes viscosity prediction model is about 10.76%. The prepared FAME soap solution can be used as coolants and lubricants in manufacturing industries.

Keywords: Castor oil, Kinetic modeling, Methanol, Refutes Viscosity modeling, Transesterification

The usage of seed oil and animal fat oil has been known from antiquity[1] to make soaps, skin products, candles, perfumes and other personal care and cosmetic products etc.[2]. Now-a-days the requirement being the usage of vegetable oils in wide spectrum to replace petroleum and synthetic based oils for engineering applications is necessary[3]. Off all available vegetable oils, castor oil is unique compared with other oils for its chain length, presence of hydroxyl group with hydrogen bonding[4].

Over thousands of years, castor oil has been used in multiple disciplines such as agriculture, food processing, healthcare, energy fuel, and engineering[5,6]. It is extracted from castor beans yields pale yellow highly viscous liquid. These oils are naturally occurring renewable resources, which are not too expensive and eco-friendly[7,8]. It has by nature possesses anti-bacterial and anti-fungal properties[9,10]. Moreover, the extracts from castor bean also proved to be a potential natural antioxidant agent and anti-inflammatory is a good replacement for synthetic agents and even resist pathogenic strains[11,12]. The physical properties of castor oils are highly reliable and follows product uniformity find its absolute standard long-term usage for its viscosity. The presence of higher hydroxyl groups in castor oil makes it compatible with any natural resins, polymers wax and possesses excellent emollient and lubricating properties [13,14].

Naturally, the oil extracted from plant seeds are highly viscous in nature, and are having linear relationships of shear stress to shear rate obeys Newton's law of viscosity, with poor flow characteristics[15]. Fasina *et al.* experimentally found that, castor oil and palm oil have more specific heat which increases linearly with raise in temperature up to 180°C[16]. This makes castor oil to withstand high temperature in the applications of lubricants and coolants. The viscosity of the vegetable oils plays a major role in wide spectrum of engineering applications which is the measure of resistance to flow against the shear stress developed between the layers of the oils[17]. The flow behaviour of castor oil from 1.7°C to 62°C follows Newton's law of viscosity[18] and is given by the equation 1.

$$\tau = \mu\frac{du}{dy} \qquad \ldots(1)$$

where τ = shear stress, μ = dynamic viscosity, and du/dy = velocity gradient. Diamante *et. al* [19] and Igwe *et. al.*[20] studied that, any raise in temperature of vegetable oils, there would be significant fall in absolute viscosities. In order to improve its flow and high temperature characteristics, the viscosity of the castor oil could be reduced to have many useful derivatives such as paints, detergents, coolants, lubricants[21,22] through nudging the reaction sites such as ester linkages(-O-C=O-), double bonds(CH=CH), and hydroxyl groups(-CH-OH) in their composition. The chemical modification of castor oil almost revolves around these three elements to get desired properties for any applications[23]. Karmakar *et al.* found that, the dynamic viscosity of vegetable oil is reduced to desired level with better stability by blending with suitable additives[24]. Commonly used additives being low viscous alcohols, on binary blending with vegetable oils significantly reduce its high-viscous effect eventually this action plummets the viscosity of blend mix to fresh equilibrium state which could be far lower than the viscosity of base oils.

Solomon *et al.* and Fulton *et al.* noted that, methanol and ethanol are widely used and is increasing day by day, is being alternate source of energy under an act of Energy policy, 1992[25,26]. The blending of methanol with castor oil follows transesterification process in which triglycerides present in castor oil reacts with any alcohol with aid of catalyst to form monoglycerides[27,28]. It is simply an exchange of organic group of an ester with another organic group of an alcohol is given by

$$R'\,OH + RO\,OR'' \rightarrow R''\,OH + ROO\,R''$$

The highly viscous castor oil is chemically modified to attain better oxidation and low temperature stability when compare with straight oils[24]. The mechanism of transesterification reaction between the fatty acids present in the oil with methanol in the presence of KOH catalyst can be studied using the kinetic modelling[29]. It is an integrated approach to help in selection of suitable conditions to increase the formation of required products in available materials and resources. It also explains the rate at which the products were formed, and the pattern followed in the transformation process[30,31].

The formation of fatty acid methyl ester and glycerol blend with reduced viscosity has wide range of applications for coolants and lubricants in engineering industries as metal working fluids. The viscosity of the blend can be obtained theoretically using Refutes empirical model for the different ratios of the triglycerides and methanol. Here, the experimental process was studied with a kinetic model and the blend viscosities were validated using theoretical modelling.

Theoretical background

The theoretical background extensively discusses about the chemical bonding in castor oil and methanol and its chemical composition. The reaction between R-fatty acids in castor oil and -OH group in methanol follows transesterification reaction is discussed.

Chemical Bonding in castor oil

The oils extracted from cold pressing of seeds of *Ricinus communis* also called as castor oil. The obtained oils from cold press technique contain lipid compounds in addition to triglycerides which improves the stability of the compounds[32]. It predominantly contains a triglyceride of fatty acid about 85-95 % of monounsaturated component 12-hydroxy-9-octadecenoic acid also called Ricinoleic acid[7,33]. The castor oil consists of 85-95 % of Ricinoleic acid, 2-6 % of Oleic acid, 0.5-1% of Linoleic, Alpha-linoleic, Steric acid and acid Palmitic acid.

The basic structure of castor oil and the six different components of R-fatty acids is shown in the Fig. 1. It consists of one double bond and alcoholic hydroxyl group on 12th carbon in ricinoleic acid. This made castor oil more reactive on assortment with alcohol at room temperature with base catalyst[34]. In castor oil, the components oleic acid and alpha-linoleic acid, consist of one double bond, which are monounsaturated in nature and the component linolenic acid consist of three double bond which is polyunsaturated. These unsaturated components are actively participated in reaction with alcohol[35]. The other two components steric and palmitic

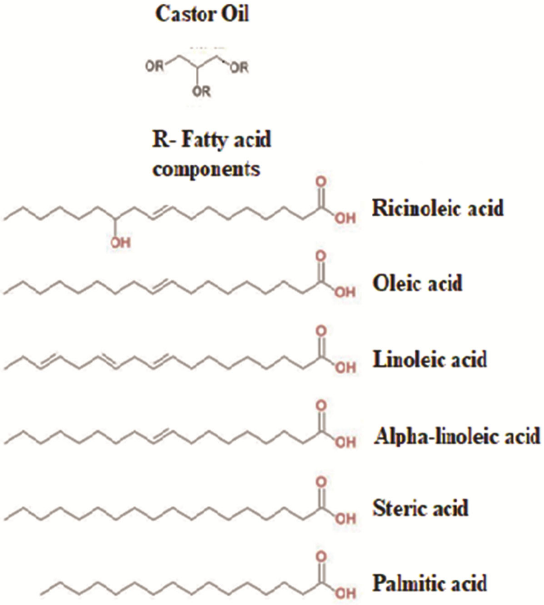

Fig. 1 — Fatty acid components of castor oil

Table 1 — Bonding in castor oil

S. No.	Composition of Castor oil	Saturated/Unsaturated	Number of carbon atom
1	Ricinoleic acid	Unsaturated	18:1 (-OH group on 12th Carbon atom)
2	Oleic acid	Monounsaturated	18:1
3	Linoleic acid	Polyunsaturated	18:3
4	Alpha-linoleic acid	Monounsaturated	18:1
5	Steric acid	Saturated	18:0
6	Palmitic acid	Saturated	18:0

acids are saturated components with no double bond are poor reactive than other components in castor oil is given in the Table 1.

About 85-95 % of chemical present in the castor is ricinoleic acid and the presence of this unsaturated components highly centred on take part in the chemical reaction. In this work, this extremely reactive ricinoleic acid[36] is considered for the analysis on transesterification reaction.

Chemical Bonding in Methanol

Methanol is an organic component in which a methyl group is associated with a hydroxyl group categorized under polar solvent[37]. It is soluble in water and insoluble in oils. It is produced from the addition of hydrogen atom with carbon monoxide or carbon dioxide in the presence of catalyst[38,39] described by the following reactions.

$$CO + 2H_2 \rightleftharpoons CH_3\,OH$$

$$CO_2 + 3H_2 \rightleftharpoons CH_3\,OH + H_2O$$

The chemical structure of methanol consists of one carbon atom linked with three hydrogen atom and a hydroxyl group. It is colourless, volatile, easily available biodegradable chemical fuel completely soluble in Fatty Acid Methyl Ester (FAME) and glycerol to from renewed equilibrium state. A range of alcohols were explored to reduce viscosity of the castor oil, yet methanol and ethanol being the notable alcohols in which methanol suits good for its highly reactive nature with vegetable oils, its physical and chemical advantages over ethanol, as an acyl acceptor, availability, and reasonable cost[40,41]. The dynamic viscosity of castor oil and methanol at 32 °C are 501.96 cP and 0.50 cP considered for the study.

The process of displacement of any alcoholic group from an ester using another alcoholic group element, called as alcoholysis predominantly used to reduce the viscosity of triglycerides in the oils[42]. The schematic diagram of transesterification process is shown in the Fig. 2.

It follows a reversible reaction in the presence of strong acid or base catalyst which accelerates the conversion to forms FAME-soap emulsion[43]. An excess alcohol in little more than oil may be used to move the equilibrium state on the side of ester formation. By the way, it calls pseudo first order forward reaction, and its reverse would be second order[44].

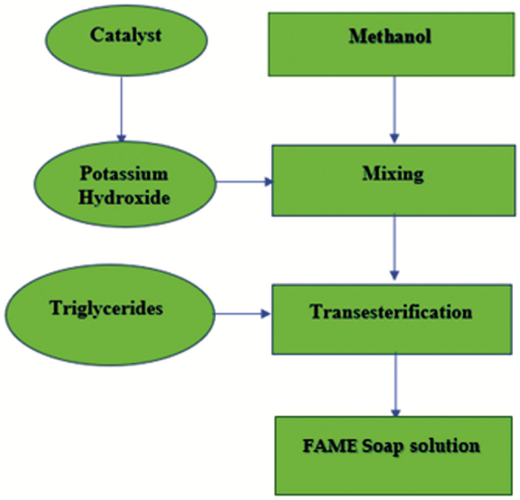

Fig. 2 — Transesterification process chart

Initially, when a pellet (1 g) of KOH catalyst mixed with methanol, KOH separates into K+ ion and OH- ion in methanol. This OH- ion of KOH reacts with the hydrogen ion which forms water and left out -OCH3 ions of methoxide solution and it combines with potassium forms potassium methoxide solution[45]. It is given by the reaction as follows.

$$\underline{CH_3\,OH + K^+ + OH^- \rightarrow K^+ + {}^-OCH_3 + H_2O}$$

Hence, this reactive simple short chain organic compound methanol reacts with fatty acids very quickly and it easily dissolved in –OH functional group catalysts. This intermediate solution reacts with the fatty acid of vegetable oils. The stepwise transesterification reaction is shown in the Fig. 3.

In step I, during transesterification process, the replacement of R-fatty acid in castor oil by OH group of methanol converts triglycerides into diglycerides and produce FAME. It further reacts with alcohol convert into monoglycerides and produce more

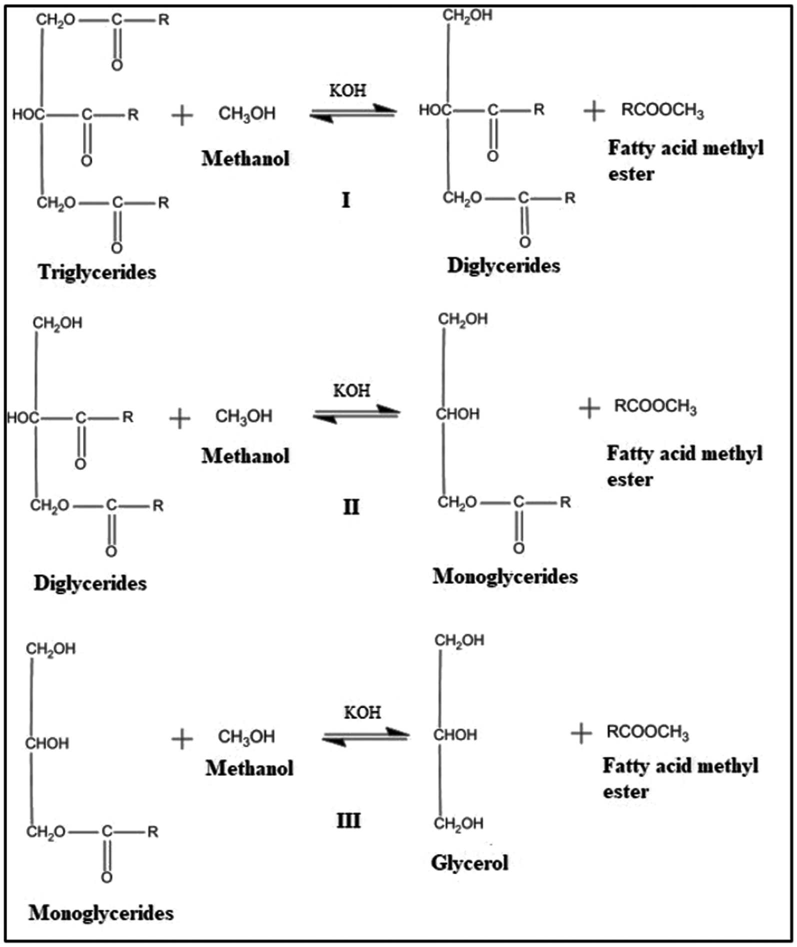

Fig. 3 — General reaction of Transesterification

FAME in step II. In step III, monoglycerides reacts with methanol produce Fatty Acid Methyl Ester glycerol solution.

Output of Triglycerides: FAME-soap solution

The presence of larger composition of monounsaturated ricinolein in castor oil which is the triglyceride of ricinoleic18-carbon hydroxylated fatty acid consist of one double bond and alcoholic hydroxyl group on 12th carbon atom. Because of this nature, ricinoleic acid in castor oil is easily react with methanol which follows transesterification reaction and form methyl ricinoleate and glycerol emulsion[46,47] is shown in the Fig. 4.

Here, the system was kept sealed airtight to react and arrest the loss of methanol and maintained at room temperature which is far less than the boiling point of the methanol and the transesterification reaction took place. It took 20-30 min to react and yield FAME-soap solution.

Kinetic modelling

The mechanism of blending of reactants and the rate at which the reaction moves forward and backward can be better analysed using kinetic modelling. It is used to predict the behaviour of the reaction system and the comparison with the reactants data gives the entire picture of the system to find the parameters that fit the data. Based on that, a suitable model may be selected for the system.

General Rate equation

The transesterification of triglycerides with methanol with the aid of KOH catalyst reduces the viscosity of the base stock which improves the kinetic behaviour of the blend[44]. In transesterification reaction system, the conversion of triglycerides into diglycerides and to monoglycerides from diglycerides is given by general rate equation.

$$\text{Triglycerides} + \text{Methanol} \rightleftharpoons \text{Diglycerides} + \text{methyl ester}$$

$$\text{Diglycerides} + \text{Methanol} \rightleftharpoons \text{Monoglycerides} + \text{methyl ester}$$

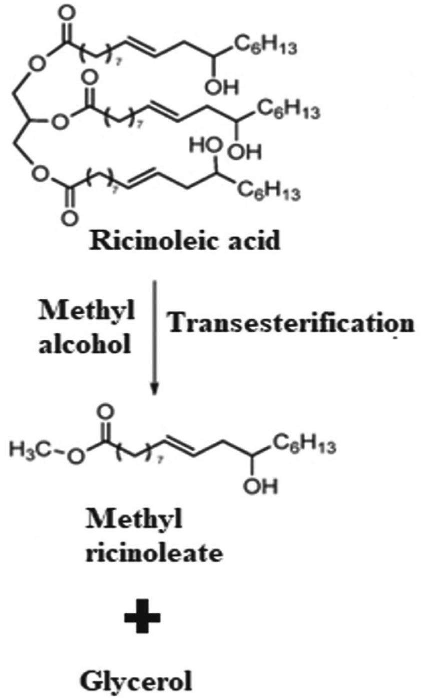

Fig. 4 — Transesterification of Ricinoleic acid

Monoglycerides + Methanol ⇌Glycerol + methyl ester

The complete transesterification of triglycerides present in the oils with alcohol is written as follows.

Triglycerides + 3 methyl alcohol ⇌3 methyl ester + glycerol
Or
TG + 3MA ⇌ 3ME + G
Or
P + 3Q ⇌ 3R +S

Where P, Q, R and S represents TG, MA, ME and G, respectively. For liquids with constant density, the reaction rate with respect to reactant P can be written as

$$-r_P = \frac{-dC_P}{dt} = k_1 y_P y_Q - k_2 y_R y_S \qquad \text{...(2)}$$

Here y_i = molar concentration of species i, where i = P, Q, R and S. The assumption being the reaction is reversible and first order reaction. If X_P = fractional concentration of P and $y_{P0}, y_{Q0}, y_{R0}, y_{S0}$ are the initial molar concentrations of P, Q, R s respectively. At $t = 0$, there is no product has been assumed to be present at $t = 0$.

$$\frac{dX_P}{dt} = k_1 C_{P0}[(1 - X_P)(M - X_P) - 3\frac{1}{K}X_P{}^2] \qquad \text{...(3)}$$

This is the General Rate Equation (GRE) for kinetic modelling of transesterification process. Here, $\frac{k_1}{k_2} = K$ and $M = \frac{C_{Q0}}{C_{P0}}$ is the molar ratio of methanol and Triglycerides in the experiment. It is known that at $t = 0$, then $X_P = 0$. In the experimental part, the flow of reaction is monitored by the measure of concentration of ME(R) with respect to time t. Then

$$\frac{-r_P}{1} = \frac{-r_Q}{3} = \frac{r_R}{3} = \frac{r_S}{1} \qquad \text{...(4)}$$

Or

$$-r_P = \frac{r_R}{3}$$

Hence equation (3) in terms of fractional formation of ME(X_R) which becomes

$$\frac{dX_R}{dt} = k_1 C_{R0}\left[(1 - X_R)(M - X_R) - 3\frac{1}{K}X_R{}^2\right] \qquad \text{...(5)}$$

At $t = 0, X_R = 0$. Here equation (5) is other form of GRE for transesterification reaction.

Case I:

Here the reaction is pseudo homogeneous of first order and it is irreversible. When methanol is added in large quantity initially, this assumption become valid and equation (5) modifies to

$$\frac{dX_R}{dt} = k(1 - X_P)$$

$$ln\frac{3}{3-X_R} = kt \qquad \text{... (6)}$$

Here, X_P is the initial concentration of methanol. From equation (5), the pseudo first order homogeneous irreversible model is obtained initially with more concentration of methanol. The plot of $ln\frac{3}{3-X_R}$ with respect to time moves linear and the slope of the curve yields rate constant k.

Case II:

For second order irreversible model, obtained at short initial period of times makes equation (5) as

$$\frac{dX_R}{dt} = k_1{}'(3 - X_R)(M - X_R);\ k_1{}' = k_1 C_{P0} \qquad \text{...(7)}$$

On integration,

$$\ln\left[\frac{3(M-X_R)}{M(3-X_R)}\right] = tk_1{}'' \qquad \text{... (8)}$$

Then,

$$k_1{}'' = (M-3)k_1{}' = (M-3)k_1 C_{P0} \qquad \text{... (9)}$$

Here a plot of $\ln\left[\frac{3(M-X_R)}{M(3-X_R)}\right]$ against t finds a straight line and its slope estimates $(M-3)k_1C_{P0}$ and k_1.

With the proposed model, at earlier stages, the second order kinetic model is accurate. If the initial molar concentration of methanol is high, the conversion of second order model to pseudo first order at short span of reaction time.

Case III:

For modified second order reversible model, the equation (5) becomes

$$\frac{dX_R}{dt} = C_{P0}\left[k_1(3 - X_R)(M - X_R) - k_2 {X_R}^2\right] \quad \ldots(10)$$

At first, the reaction rate is fast, but over a period of time, the rate of reaction plummets as the source of reactants reduced and follows increases in backward rate of reaction when the products are significant. For higher molar concentrations of methanol to oil, the forward reaction follows first pseudo-order compared with oil and reverse reaction follows second order with respect to the end products. With this assumptions, equation (10) will be of form for $X_R = 0 \; at \; t = 0$.

$$\frac{dX_R}{dt} = \left[k_1(3 - X_R) - k_2 {X_R}^2 C_{P0}\right] \quad \ldots(11)$$

Ezzai *et al.* and Darnoko *et al.* were studied similar laws for the kinetics of the reaction follows esterification in palm and sunflower oils[48,49]. However, the rate of reaction varies with the different molar ratios of methanol to oil even with higher oil to methanol concentration follows reversible reaction from case III.

Refutes viscosity modelling

The reaction between ricinoleic acid of castor oil and methanol, in the presence of KOH catalyst yields methyl ricinoleate and glycerol solution. The formation of this solution depends on the percentage addition of methanol to take forward transesterification reaction. The viscosity of the FAME-soap is studied by Refutes viscosity modelling.

Viscosity modelling of Methyl ricinoleate and glycerol solution

The formation of FAME-soap solution at the end of transesterification processes reaches new equilibrium with reduction in viscosity of the castor oil. Zhmud *et al.* used double logarithmic empirical relation for kinematic viscosity of blend[50] us given by the equation 12.

$$v_{ab} = exp\left[exp\left(\frac{A_{ab} - 10.975}{14.534}\right)\right] \text{-}0.8 \quad \ldots(12)$$

Here, $_{ab}$ is the kinematic viscosity of the blend and A_{ab} is the average viscosity blend index which can be calculated by the equation 13.

$$A_{ab} = x_a A_a + x_b A_b \ldots(13)$$

The average viscosity blend index depends on weight fraction (x_i) and viscosity blend index A_a and A_b from equation 14.

$$A_i = 14.534 \ln [\ln (v_i + 0.8)] + 10.975 \ldots (14)$$

A_i is the viscosity blend index of individual component *i*.

Results and Discussion

Refutes viscosity blend mix prediction modelling

The viscosity of any oil blend mix depends on the Viscosity Blend Index of the oil, which is required as it does blend linearly with volume and the proportion of the oil used is given by equation (12). The average viscosity blend index of blend mixes and the viscosity of the blend were calculated from equation (13) and (14) are shown in the Table 2.

As an example, the highly viscous castor oil on assortment with low viscous methanol in a ratio of 90:10 (X_C= 0.90 and X_m = 0.10) is demonstrated as

Table 2 — Viscosity blend index and blend viscosity

S. No	% Castor oil	% Methanol	Average viscosity blending index	Blend viscosity (cSt)
1	100	0	37.64	523.42
2	90	10	33.50	110.50
3	80	20	29.37	33.87
4	70	30	25.24	13.62
5	60	40	21.11	6.65
6	50	50	16.97	3.73
7	40	60	12.84	2.32
8	30	70	8.71	1.55
9	20	80	4.58	1.10
10	10	90	0.45	0.82
11	0	100	-3.69	0.64

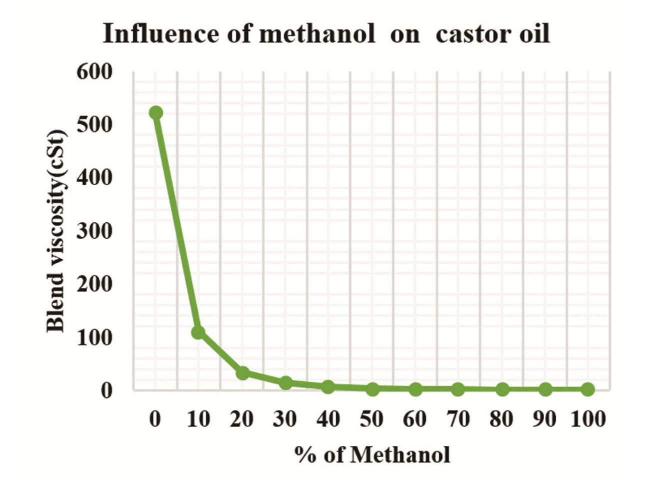

Fig. 5 — Influence of methanol on castor oil

follows: The viscosity blend index of castor (A_c) and methanol (A_m) are calculated as 37.64 and -3.69 respectively. Further, the average viscosity blend index of castor methanol blend is calculated as 33.50. Then, according to Refutus empirical relation, the kinematic viscosity of the castor methanol (v_{cm}) blend mix is obtained as 110.4959 cSt. The influence of methanol on viscosity of the castor oil is shown in the Fig. 5.

It is observed that for a binary mix of Castor and Methanol blend, the kinematic viscosity decreases drastically by 78% than with castor oil on 10 % addition of methanol. However, on further increases in methanol over 30% does not have significant changes in kinematic viscosity when compared with the range between 10- 30% of methanol theoretically.

Experimental validation

The experimental part starts from preparation of blend mix in laboratory, where the transesterification reaction of ricinoleic acid assisted by KOH catalyst was done at 1 atmospheric pressure and at 32°C. A magnetic stirrer was employed which is immersed inside the reactants, uses rotating magnetic field for 30 minutes. The experimentation was done for all the combinations of oil methanol ratios and the blend viscosities were experimentally measured for validation. The schematic view of the reaction is shown in the Fig. 6.

The viscosities of blend mix is quantitatively measured using Brookfield Rheometer DV 3T and its component diagram is shown in the Fig. 7. The principal of operation of DV 3T is to drive a spindle through calibrated spring. The viscous drag of the fluid against the spindle is measured by the spring deflection. It is measured with a rotary transducer.

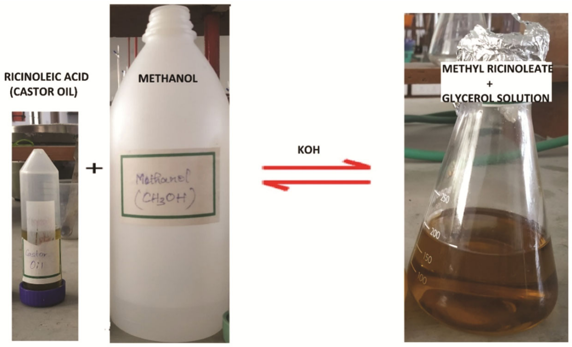

Fig. 6 — Laboratory experimentation model of transesterification reaction

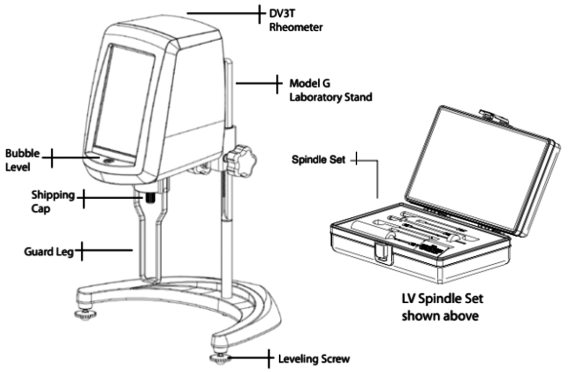

Fig. 7 — Component diagram of Brookfield Rheometer DV 3T

The measurement range of a DV3T is determined by the rotational speed of the spindle, the size and shape of the spindle, the container the spindle is rotating in, and full-scale torque of the calibrated spring.

The measurements on Brookfield Rheometer DV 3T starts with auto zeroising process, followed by mounting cylindrical container on the shipping cap. The cylindrical container is filled with a quantity of 20 mL of oil to test the sample where the cylindrical spindle is rotated at 1 rpm. The setup is maintained at temperature of 32°C. The data points were collected for single step point with 120 sec end condition time. The viscosity data from Brookfield rheometer for all the samples were noted.

Percentage deviation

The experimental values of viscosities were compared with refutes prediction model and the percentage deviation is calculated shown in the Table 3. The viscosity of prepared blend mixes measured from Brookfield rheometer is satisfactorily fitted with modified refutes empirical equation.

Here the percentage deviation between theoretical and experimental blend viscosities were calculated using

Table 3 — Percentage deviation in Viscosity

Castor to Methanol Ratio	Blend viscosity (cp) Theoretical	Experimental	% Deviation
100:0	501.96	452.00	11.05
90:10	104.00	124.65	16.57
80:20	31.28	35.62	12.19
70:30	12.33	13.76	10.38
60:40	5.90	6.54	9.74
50:50	3.25	3.68	11.78
60:40	1.98	2.18	9.39
70:30	1.30	1.44	10.01
80:20	0.90	0.82	9.94
90:10	0.66	0.73	9.89
100:0	0.50	0.54	7.44

equation (15). It is inferred that; the addition of methanol greatly reduces the dynamic viscosity of the blend of even about 10%. However, the closeness of predicted and experimental viscosities is more on addition of methanol over 10%.

$$\text{Percentage deviation} = \frac{Expermiental - Theoretical}{Experimental} \times 100\ \% \qquad \ldots(15)$$

Conclusion

This study explores the chemical modification of castor oil for the application of coolants and lubricants in manufacturing industries. The chemical modification follows transesterification reaction by the addition of methanol assisted with KOH catalyst. This process reduces the viscosity of the castor oil which depends on the percentage addition of methanol. The blend mixes were prepared in laboratory with different proportions of methanol to study its influence on viscosity of the castor oil. The effect of addition of methanol on triglycerides is follows modified second order reversible reaction as per case III of kinematic modelling. The experimental results were validated using refutes prediction model. The percentage deviation in experimental and prediction model on average is about 10.76%.

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