Investigating the Effect of Temperature, Molar Ratio of Ethylene Glycol to Oil, and Catalyst Amount on BioLube Production Yield of Neem Seed Oil

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ABSTRACT: *Excessive use of petroleum-based lubricants and their hazardous disposal has increased environmental pollution; hence the need for eco-friendly lubricants has been increased to meet the requirement of the industry and automotive. Due to the oil crisis, the world's attention has been diverted to producing bio-lubricants from non-edible sources. The use of non-edible sources can overcome the problems of toxicity, hazardous nature, and non-biodegradability. This study discusses the effect of various parameters on transesterification reaction to produce bio lubricant from Neem oil. The dried neem seeds were crushed, and the oil was extracted using the Soxhlet extraction method using n-hexane. The bio lube was produced by a double transesterification process using CaO as a catalyst. The effect of temperature, the molar ratio of ethylene glycol to oil, and catalyst wt% on yield bio lubricant was observed. The temperature varied from 110 to 140 °C, molar ratios of ethylene glycol to oil varied from 2:1 to 8:1, and the catalyst wt% was 0.8 to 1.6%, keeping the reaction time and other conditions constant. During the experimentation, it was observed that the yield was low at 110°C, but as the temperature increased, yield also increased, but no significant change in yield was observed beyond 130°C. The maximum yield observed at 130°C, and 140°C was 93.7% and 94.37%, respectively. Similarly, as the molar ratio increases, the yield of bio lube also increases, and the maximum conversion was 94.3% achieved at an ethylene glycol to oil molar ratio of 8:1, but a molar ratio 6:1 may be considered optimum because there is no substantial increase in conversion after 8:1. Moreover, the profile was observed by varying the amount of catalyst and it is evident from the results, that the amount of catalyst increases as the conversion increases from 66% to 95%; however, at a catalyst ratio of 1.6 wt %, the yield decreased slightly to 94.2. It has been observed that the temperature significantly impacts the production yield of Biolube.*

KEYWORDS: *Biolube; Transesterification; Soxhlet extraction method; Neem; Biomass.*

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INTRODUCTION

Energy plays a vital role in human life and the nation's economy. Expeditious increase in the world's population and industrialization has increased the consumption of fossil fuels. Coal, oil, and natural gas have been playing their roles in fulfilling the energy demand for centuries, causing depletion in fuel reserves. The global campaign has been started to replace petroleum-based lubricants due to depletion of fuel reserves and emission of greenhouse gases from fossil fuels resulting in climate change, the emergence of drough, and the spread of diseases [1-5].

Lubricants are essential for the proper functioning and smooth operation of machines as they reduce the friction between the moving parts of the machines [6]. Based on their base oil, they may be categorized as mineral lube (petroleum-based) and synthetic/ bio lube ((e.g., polyalphaolefins, polyalkylene glycols, synthetic esters, silicones, etc.) [7]. The fuel and lubricants produced by edible and non-edible sources are considered as the most promising alternative due to their non-toxic, biodegradable environmental friendly nature [8, 9]. However, the direct use of edible and nonedible oil is not feasible due to their poor stability and low thermal-oxidative properties. Therefore, bio lube is mainly acknowledged as an economical and sustainable alternative. Moreover, it has more advantages over mineral oil-based lubericants because edible and non edible oils are abundant compared to mineral oil-based lubricants produced from petroleum resources only.

Bio lube is non-toxic, produces less carbon and greenhouse gases, provides good lubrication, has a high flash point, high viscosity index, low pour point, high oxidation stability, and low volatility [10]. Despite having many advantages, biofuels have some disadvantages, such as poor cold flow properties and stability, which can be overcome by chemical modification [11]. It has been observed that direct blending of edible and non-edible oil with commercial lubricants does not provide satisfactory performance due to their low thermo-oxidative stability, high viscosity, and low volatility. Therefore, edible and nonedible oil modification is required before blending the nonedible oil with conventional lubricant. The most commonly used chemical modification methods reported in the literature are epoxidation, hydrogenation and transesterification [6, 12]. Hydrogenation is carried out by using Ni and Pd catalyst under high temperature and pressure conditions. This process commonly causes isomerization and decreases the fluidity of lubricant, which solidify the lubricant and form waxes. Therefore, the hydrogenation method is not considered adequate for improving physicochemical properties. Furthermore, using alkyl hydroperoxides, peroxy acids, dioxiranes, or peracids as sources is epoxidation of unsaturated bonds. Epoxidized products exhibit poor temperature properties, due to which esterification is necessary to improve their properties [3, 13]. Moreover, transesterification is a reaction with alcohol to form esters and glycerol, which is carried out at low temperatures and pressure in the presence of a catalyst, such as HCl, H_2SO_4, or H_3PO_4. Transesterification reaction plays a vital role in biofuel production, and it is generally preferred over other methods due to low temperature, pressure, and capital requirements [14]. Furthermore, it has been investigated that it enhances lubricity and increases the metal surface's wear resistance [10, 15, 16]. Methods commonly utilized for producing bio lube involve the extraction, purification, and transesterification. The yield of lube produced by the transesterification method is affected by various operating parameters such as temperature, molar ratio, reaction time, and catalyst weight [7].

Several studies have been carried out using various non-edible sources, in which characteristics of bio lubes have been studied [1, 6, 17, 18]. The key works pertaining to biofuels indicate that they exhibit good properties comparable to mineral-based fuels and provide good lubrication properties. The properties of bio lube are also improved by the addition of antidepressants, corrosion inhibitors, pour point depressants depending upon the application [13]. *Soufi et al.* discussed the need for renewable and environmentally friendly products in bio-lubricants for sustainable development. After chemical modification, the authors concluded that edible oil could be used as sustainable products for refineries and lubricant industries [19]. Several studies have proved that Jatropha, Jojoba, and castor oil also provide promising results comparable to mineral-based oil [18, 20-24].

The study of *Bilal et al.* focused on producing bio lubricant from Jatropha oil by chemical modification. As a result of the modification, some physiochemical properties of lube were improved, which were comparable with standard properties of lubricants [18]. *Bokade* and *Yadav* studied a transesterification process for ethyl ester production; as a result of this reaction, bio lube (methyl and octyl fatty acid ester)was produced in the presence of heteropolyacids as catalysts [25]. Finally, *Samuel et al.* investigated the chemical modification of bio lube produced from castor, palm, kernel, and coconut oils using transesterification. The physiochemical properties of the bio lube showed good characteristics comparable to SAE 40 applicable for motor engines [10].

Neem tree is native to Asian countries, containing 25 - 45% non-edible oil with high free fatty acid contents. There are two ways of utilizing this non–edible oil, either by directly blending oil with commercial lubricant or by chemical modification. Different studies have shown that Neem oil is a promising source for producing bio lube with good physicochemical properties [12, 26, 27]. *Idris et al.* has proved that neem oil can serve as bio lubricant as it displayed good characteristics for automotive system [12]. According to the study of *Suresh et al.*, neem oil was beneficial in all fields. It was investigated for tribological properties by exploring the effect of nanographene platelets, which affect its viscosity and friction, and found to be suitable for the gear oil applications [27].

Betiku et al. presented the work in which neem seed oil was converted into fatty esters by catalytic reaction. The catalyst was obtained from cocoa pod husk. The research showed that ester was obtained at 65 °C using 0.73 (v/v) methanol to oil ratio and 0.65 catalysts wt%, keeping the reaction time 57 min at a yield of 99.3% [28]. *Tanwar et al.*, proved that neem oil has the highest potential for producing methyl esters among the available wild oils. Moreover, Neem contains 25%-45% nonedible oil on a dry basis, which can be utilized to produce bio lube [29]. *Chauhan* experimented with neem oil by transesterification and partial hydrogenation to study the lubrication properties [30]. *Chan et al.* showed the tribological aspect and proved that a good bio lubricant base stock could be obtained by optimizing the stability and adhesiveness of obtained tribofilm. These conditions could be achieved easily if the lubrication regime's desired properties and the lubricant are according to the application requirements [31].

As per author's knowledge, the effect of various parameter on the production of Biolube oil from neem seed using CaO as a catalyst has not been explored before in the literature. Hence, this work aims to study the potential use of bio lube produced from neem oil by transesterification process comprised of two steps. In the first step, transesterification was done by using methanol and Potassium hydroxide. The next step was transesterification with Ethylene glycol using CaO. The factors affecting second transesterification reaction such as temperature, catalyst wt%, and ethylene glycol to oil molar ratio on conversion, and among these, the optimal conditions were analyzed. Moreover, the properties of neem bio lube were also compared with other bio lubes and ISO VG 46 to study its applicability for industrial and domestic purposes.

EXPERIMENTAL SECTION

The neem seeds were purchased from local market and chemicals which are used for transesterification such as Pottasium Hydroxide (90% purity) ,Methanol (99% purity) and Calcium oxide (90% purity) were purchased form Sigma Aldrich.

The dried, clean and crushed neem seed kernels were used for the extraction of oil. Neem oil was extracted using the facility of traditional Soxhlet extractor, available at Department of Polymer & Petrochemical Engineering, NED University of Engineering & Technology. The solvent used for extraction was n-Hexane, which was utilized in the proportion of 6:1. A reflux condenser is attached with the facility, and the mixture was kept on stirrer at 60°C for 8 hours. After numerous cycles, the ideal compound was collected in the flask. After neem oil extraction, the solvent was removed by a rotary evaporator at 50°C, yielding the extracted compound. The solution was then heated to remove the traces of n-hexane by temperature-controlled water bath, and finally, the oil was collected. The properties of Neem oil are mentioned in Table 1.

The refined neem oil was subjected to esterification followed by transesterification. The chemistry behind the transesterification is shown in Fig. 1, in which triglycerides react with an alcohol to produce ester and glycerine. To speed up the reaction catalyst is added.

The esterification reaction was performed in a glass reactor with a mechanical stirrer. The glass reactor was initially filled with the desired oil and then placed on a hot plate. After reaching the temperature of 45°C, methanol was added in molar ratio of 8:1 (methanol /oil) and 4.5% H_2SO_4 catalyst (w/w oil) , and the mixture was heated for 60 mins. After the esterification, the product was kept in a separating funnel for 24 hours for gravity separation to separate the acid catalyst. Finally, the upper layer formed was separated as reduced FFA oil.

Table 1 Properties of Neem oil

	Properties	Results
1	Density (kg/m^3)	926
2	Pour Point (°C)	23
3	Flash Point (°C)	263
4	Viscosity Index (VI)	131

The transesterification reaction was performed in a stirred glass reactor at 300 rpm for 1 hr. The sample were prepared at the reaction temperature of 55°C, methanol to oil molar ratio from 6:1, and Potassium hydroxide ratio 1 wt.%. After the completion of the reaction, the product was kept for 24 hours in a separating funnel. The bottom layer was dark brown, comprised mainly glycerol. The upper layer was Fatty Acid Methyl Ester, which also contained traces of catalyst, glycerol, and methanol, which were removed by washing with 10% warm water [32, 33].

The biolube is produced by catalyzed biodiesel transesterification with ethylene glycol using Calcium Oxide (CaO) as a catalyst in the next step. The heterogeneous catalysts are considered more effective for transesterification reaction due to ease of separation and regeneration [34, 35]. The effectiveness of CaO in transesterification reaction has been validated through several studies due to its good catalytic efficiency, low cost, non-toxic and non-hazardous nature. The reaction was carried out in a two-neck round Pyrex glass flask; connected with a water condenser and thermometer. The reactants were heated by using a hot paraffin oil bath. When the desired temperature was reached, the reaction mixture was added to carry out the reaction. The water condenser was connected to condense the evaporated methanol during the reaction. The reaction is reversible; therefore, the methanol was removed continuously in the methanol collecting flask and the reaction was continued. Fig. 2 shows the overall flow diagram for the synthesis of biolubricant from neem seed.

The study aims to analyze the effect of process parameters on the production yield of bio lube. Therefore, the analysis was conducted using three different parameters; temperature, reactants, catalyst wt%, ethylene glycol to oil molar ratio, while the time and rpm were constant, i.e., 1 hr and 300. The variables were selected according to the ranges mentioned in the literature from 105°C to 140°C. The controlling factors were varied as; the temperature of 110, 120, 130, and 140 °C, the ethylene glycol to oil molar ratio of 2:1, 4:1,6:1 and 8:1, catalyst wt% of 0.8,1,1.2,1.4 and 1.6% (w/w). The bio lube yield was calculated by the equation given below.

$$\text{Yield} = \frac{\text{Amount of produced bio lube}}{\text{Amount of oil used}}$$

Based on the obtained results, it is required to evaluate its properties against the standard and analyze the characteristics possessed by the product obtained as shown in Fig. 3.

RESULTS AND DISCUSSION

The properties of bio lube and the comparison with ISOVG 46 , waste cooking oil bio lube, and Jatropha oil bio lube are shown in Table 2. ISOVG 46 is the standard for gear oil specification, depending on two parameters, i.e., viscosity index and pour point.

Viscosity index indicates the change in lubricant’s properties when it is subjected to temperature variations. The high value of the viscosity index shows that variation in viscosity is lower at high-temperature operations. Therefore, a high value of viscosity index is required for lube. The viscosity index was measured through ASTM D2270-74. The bio lube sample was filled in viscometer tubes, and then the tubes were placed in viscometer baths. The temperature of one bath was set at 100°C however, the other bath was set at 40°C. The time taken for the lube to fall from the start point to endpoint was noted, as the lube flows under gravity. The Viscosity index was calculated with the help of obtained results. The obtained viscosity index from neem lube oil is 212, which is greater than WC and JO lube oils and also satisfies the requirement of ISOVG46. Therefore, obtained neem oil lubricant with high viscosity index is preferable for domestic and industrial gear oil applications [18, 36].

Pour point indicated the lowest temperature at which oil can flow. Pour point is the critical property that indicates the performance of lubricant at low temperatures. In this study, the pour point was determined according to ASTM D97-05. The lubricant is added in a container and pre-warmed, and a cooling stage trails this with the goal that lubricant will be at a temperature of 9°C over its normal pour point. The sample is then examined at each 3°C stretches. The investigation appears as eliminating the vessel from the mechanical cooling assembly and inclining it

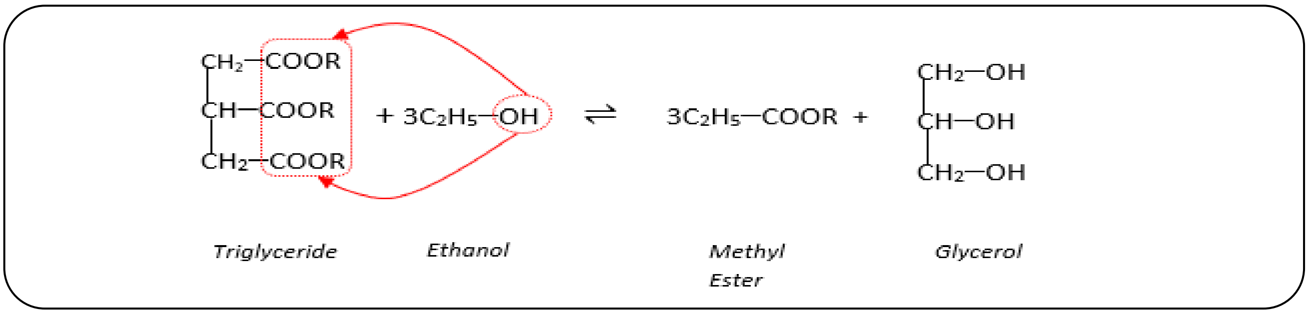

Fig. 1: Transesterification Reaction.

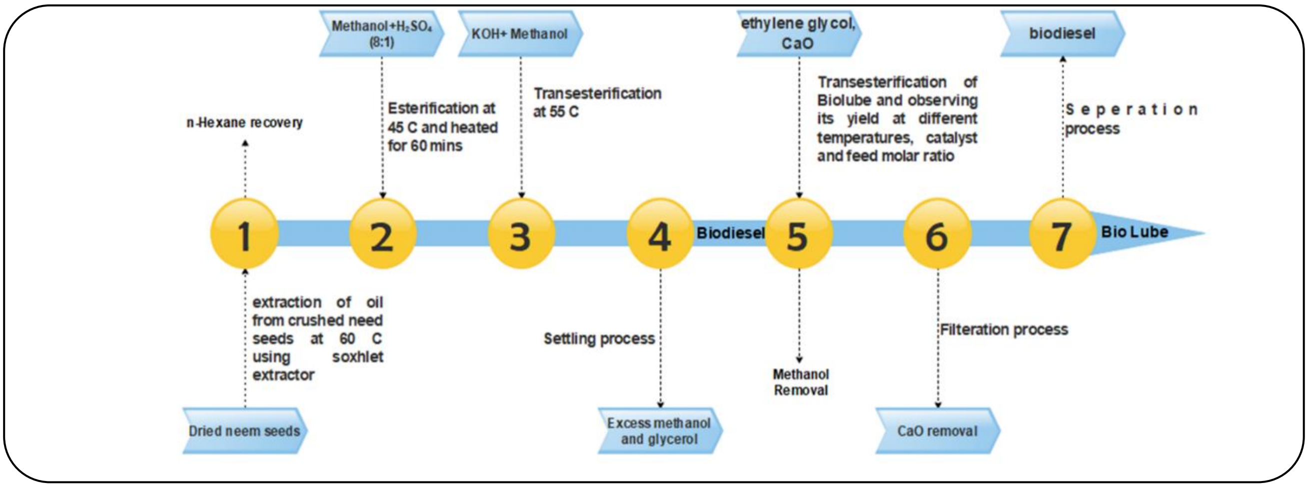

Fig. 2: Flow diagram for the synthesis of biolubricant from neem seed.

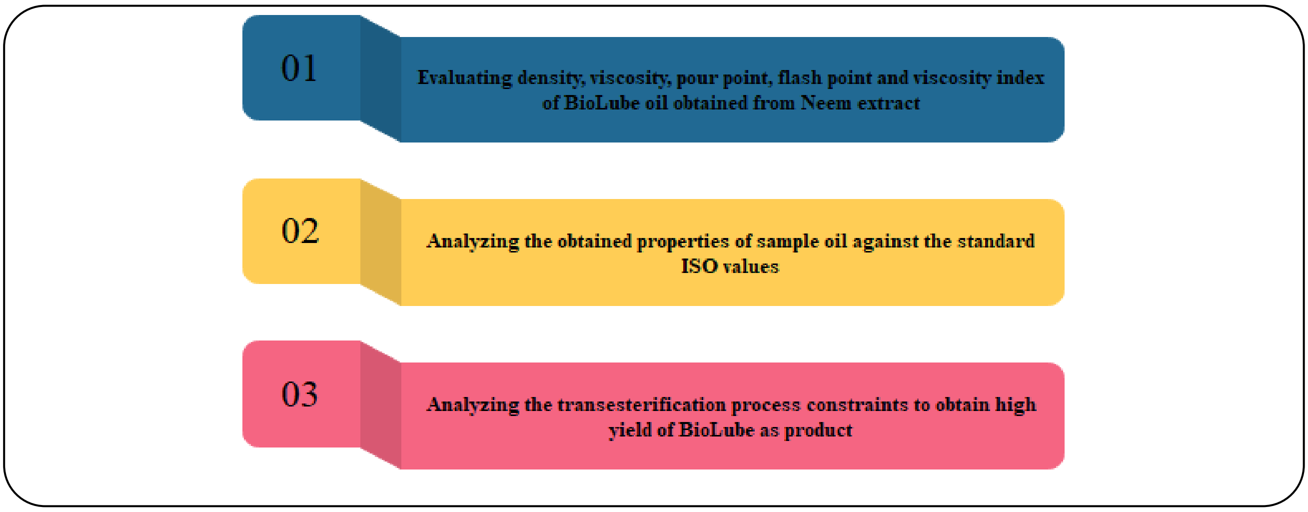

Fig. 3: Bio Lube analysis methodology.

to check whether there is any surface movement from the lubricant. The temperature at which the lubricant stops flowing is the pour point. The pour point of neem oil was -7°C which is equal to the pour point of JO i.e. -7°C, and slightly higher than pour point of WC i.e -8°C.The pour point of ISOVG 46 is -6°C , which is slightly higher than -7°C. This signifies that the produced lube oil is suitable for low-temperature application without filter clogging [18].

The density of neem lube was 0.889 g/cm^3, which was higher than the value of petroleum-based lube

Table 2:Comparison of properties of Neem bio lube with JO ,WC bio lubes and ISO standard [15, 36, 37].

S.N	Properties	Neem Oil Bio-Lubricant	WCO based Lubricant	JO based Lubricant	ISO VG 46
1	Density (g/cm^3)	0.889	---	0.879	0.861
2	Pour Point (°C)	-7	-8	-7	-6
3	Flash Point (°C)	292	240	---	220
4	Viscosity Index (VI)	212	204	195	>90

i.e 0.861 g/cm^3, may be due to the presence of ethylene glycol fatty acid diester, which has a high molecular weight.

Flash point of a bio lubricant is the minimum temperature at which an external source could ignite the vaporized oil. It is considered as an indication of highly volatile and flammable material. It is used to determine bio-lube storage conditions and transportation requirements. This study measured the flash point of lube oil by Pensky Marten Closed Cup apparatus as per ASTM D 93-18. The sample was poured into test cup, and the lid was closed tightly. A thermometer and sensor was immersed in the test sample, and then the machine was started. A test flame was passed across the cup containing the samples at regular intervals. As the flashes appeared, the machine indicated the temperature, which is considered a flash point. The flashpoint of the produced neem bio lube was 292°C , which is quite high compared to flash point of WC lube i.e. 240°C and ISO VG46, i.e., 220°C. This high flash point might be due to the presence of long-chain in their molecules as a result of chemical modification. Thus, the overall flammability hazard is less due to the absence of highly volatile components.

The synthesis of neem bio lube was carried out by transesterification process, the reaction was analyzed by varying the reaction parameters, and the change in conversion was observed. The change in yield by varying the temperature by keeping the other parameters constant is showed in Fig. 4.

It is evident from the results that transesterification is highly dependent on temperature due to the dependence of rate constant on temperature, which influences the reaction rate. The theory of chemical reaction kinetics well explains the dependency of reaction rate on temperature. The speed of the molecular collision is increased by increasing the temperature, resulting in high kinetic rate and increased conversion [17, 38-41].

The effect of temperature on reaction rate and conversion was determined by conducting the experiments

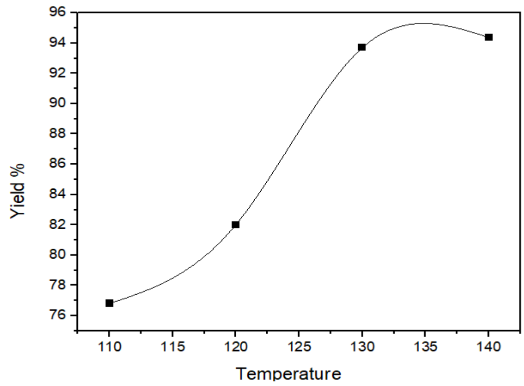

Fig. 4: Effect of Reaction Temperature on production yield of bio lube.

at 110,120, 130, and 140 °C. The experimental results show that the low yield was initially obtained due to low temperature, causing less molecular collision and low kinetic rate. However, from Fig. 5, it can be observed that as the temperature increases, the yield also increases because high temperature accelerates the molecular collision and kinetic rate, resulting in high yield.

Moreover, if the temperature increases beyond 140°C, no change in conversion may occur because the reaction is reversible and may approach the equilibrium condition. Therefore, the conversion does not change further, and 130°C may be a suitable working temperature.

The effect of the ethylene glycol to oil molar ratio is shown in Fig. 6, keeping all other parameters constant. It is evident from data that the 2:1 ethylene glycol to oil molar ratio gives the lowest yield, and as the molar ratio increases, yield also increases, but 6:1 and 8:1 give very close conversion. Beyond the molar ratio of 6:1, there is no substantial increase in conversion. It is possible that on further increase in molar ratio beyond 8:1, conversion decreases because when the amount is above 8:1, the concentration increases and separation becomes difficult, affecting the production yield of bio lube which is also

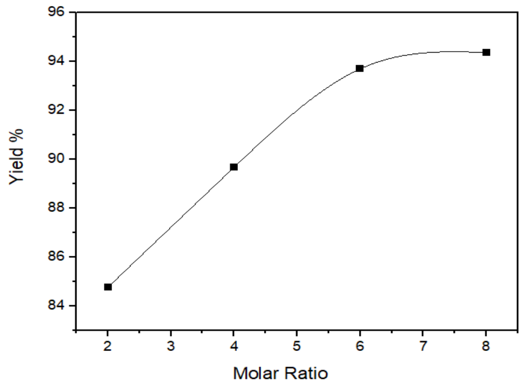

Fig. 5: Effect of molar ratio (ethylene glycol) on production yield of Bio lube.

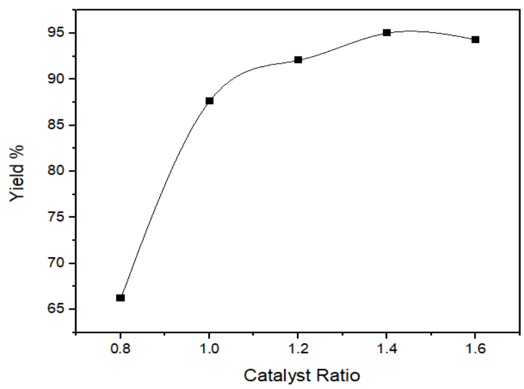

Fig. 2: Effect of Catalyst ratio on production yield of Bio lube

reported in the literature [42]. Thus, the molar ratio of 6:1 may be considered as the optimum working condition.

The effect of catalyst wt% on yield is shown in Fig. 6, keeping all other parameters constant. Data shows that increase in catalyst wt %, yield also increases; however, at a ratio of 1.6 wt%, a slight decrease in conversion is observed. This decrease in conversion may be a result of improper mixing of catalysts in the reaction mixture. Moreover, the solution becomes concentrated and viscous, if the higher amount of catalyst is added in the same amount of reaction mixture, which causes poor mixing and dispersion at the same agitation speed, due to which some amount of catalyst remains unused and mass transfer resistance increases which give low conversion. This behavior is in agreement with literature findings [43-45]. Thus, the optimum catalyst wt% for carrying out this reaction is considered to be 1.4.

CONCLUSIONS

Production of eco-friendly, biodegradable bio lubricant from neem oil has been accomplished. The above study highlights the critical properties of bio lube and the effect of temperature ranging from 110 to 140°C, ethylene glycol to oil molar ratio ranging from 2 to 8, and catalyst wt % 0.8 to 1.6 was studied. It is evident that the optimum reaction parameters for the bio-lube production yield were 130°C, EG ratio 6:1, and catalyst CaO 1.4 wt % with the conversion of 94%. Furthermore, the properties of bio lube were also estimated and found to be closed to ISOVG 46 standard. The viscosity index of obtained bio lube was 212, which fulfilled the requirement of bio lubricant standard. Hence, it may be concluded that the neem bio lube can serve as a lubricant in light and heavy automotive systems. Its production should be significantly encouraged as it is a nonedible crop and would not compete with acreage dedicated for food crops.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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