[1.]Babatope Abimbola OLUFEMI, [2.]Rachael Aniedi ESSIEN

PRODUCTION OF BIOLUBRICANTS FROM NEEM SEED OIL CATALYZED BY CALCIUM OXIDE FROM SNAIL SHELL

[1.]Department of Chemical and Petroleum Engineering, University of Lagos, Akoka, Lagos, NIGERIA

Abstract: Biolubricants was produced from neem seed oil with methanol by transesterification using calcium oxide as catalyst. Snail shells were utilized as a source of calcium oxide (CaO) in catalyzing the transesterification reaction to produce the methyl ester biolubricant. The heterogeneous catalyst was prepared by calcination method. Experimental results showed that the maximum yield of the methyl ester was 96.7 % obtained at a methanol-oil ratio of 6:1, catalyst of 6 wt.% and a reaction time of 5 hours. Major lubricating properties of the synthesized neem biolubricant such as viscosities at 40°C and at 100°C, viscosity index and pour point were analyzed and found to have values of 54.87 cSt, 6.64 cSt, 136.43 and – 9°C respectively. The biolubricant produced is comparable to other plant based biolubricant and the International Standards Organisation Viscosity Grade 46 (ISO VG 46) commercial standards for light and industrial gear applications.
Keywords: Neem seed oil, heterogeneous catalyst, transesterification, biolubricant, snail shell

INTRODUCTION

Environmental concerns associated with the use of petroleum-based products in various industries, such as forestry, farming and mining to mention a few have led to increased interest in the use of environmentally friendly substitutes. Growing environmental concerns are providing the incentive for increasing the demand and usage of plant oils in lubricants for many applications. The nature of vegetable oil, that is, easily renewable, biodegradable and non-toxic makes it the best alternative to mineral oil in the production of lubricants (Syaima et al., 2014).

Today, the constant use of petroleum-based products and other non-renewable assets are depleting the world's natural resources (Schneider, 2006). Biolubricant production is necessary to serve as a substitute lubricant to supplement or replace conventional lubricants due its numerous advantages such as renewability, biodegradability and lower gaseous emission profile (Bilal, et al., 2013).

It has been recounted that finding the other alternatives sources of feedstock and catalyst is of great interest in biolubricants production (Ghafar et al., 2019). Banu et al., (2018), Bahadur et al., (2015), Thangaraj et al., (2014), Muthu et al., (2010), Woma et al., (2019) among others also stressed the importance of neem seed oil as a viable source of material. According to Idris et al., (2018), neem oil can successfully serve as a lubricant, based on the lubricant properties it displayed for both light automotive systems and heavy automotive system. It has been observed that biolubricants are biodegradable and release less carbon and greenhouse gases compared to petroleum-based lubricants according to Hossain et al., (2018). According to Damjanovic et al., (2016), from an environmental point of view, the advantages of using vegetable oil as compared to conventional mineral oils are expressed as non-toxicity, bio-degradability, renewability, good lubricity, high flash point and viscosity index, low volatility, savings and conservation of non-renewable resources. Others are less dependence on non-renewable resources, reduced emissions of greenhouse gases and increased agricultural production.

Heikal et al., (2017) reported that even though biolubricants are priced twice as high as conventional petroleum lubricants, various industries are investing in research and development programs toward increasing the oil recovery from seeds, reducing the costs of processes and exploring niche application areas. In the continuous activity for sourcing for better biolubricants, it had been reported that chemical modification of vegetable oils causes improved thermooxidative stability, thereby allowing their use in a wider range of operating conditions (Cecila et al., 2020).

Towards the production of suitable and accepted biolubricants, Hassan et al., (2019) reported that the increasing prices of crude oil, depletion of crude oil reserves in the world, and global concern in protecting the environment from pollution have renewed interest in developing and using environment-friendly lubricants derived from alternative sources.

According to Suresha et al., (2020), neem oil has promising benefits in all fields. As a result, neem oil was explored with tribological properties as biolubricant, by exploring the role of graphene nanoplatelets (GNPs) in neem oil as it affects viscosity, friction and wear as well as on seizure load which are important factors that accounts for the right selection of the appropriate lubricant in different sliding components of food processing machines.

Various researchers have contributed towards perfecting the suitability of biolubricants for various domestic and industrial purposes over the years. Bilal et al., (2013) studied the feasibility of producing biolubricant from Jatropha oil by conducting chemical modifications on the Jatropha crude oil. The modification involved improving some of the lubricating properties of the Jatropha crude oil.

The physicochemical properties of Jatropha biolubricant were also compared with a certain standard properties of lubricants. It was found that the biolubricant produced is comparable to the ISO VG 46 commercial standards for light and industrial gears applications and other plant based biolubricants. Bokade and Yadav (2007) presented a reaction for the preparation of alkyl esters by transesterification process. They produced methyl/ethyl/propyl fatty acid esters called biodiesels, octyl fatty acid ester called biolubricants, which comprises contacting fatty acid glycerides / triglycerides with or without free fatty acids of vegetable oil with alcohols in the presence of a solid heteropolyacids catalyst which is substantially insoluble in the reaction mixture under reaction conditions.

From the study, optimized heteropolyacids (10%TPA) supported on clay was observed to be the best, stable, free fatty acid tolerable catalyst for the transesterification of edible and non-edible oil with lower and higher alcohol to get biodiesel and biolubricants.

The importance of sustainable development based on renewable and environmentally friendly products in the production of biolubricants was also presented by Soufi et al., (2018). They observed that biolubricants that are mainly produced by chemical modification of vegetable oils are sustainable products for biorefineries and the future of lubricant industries.

Biofuels and biolubricants production for industrial application from some common oil as feed stock was considered by Abdalla (2018). Studies on lanthanum and zinc (10-30 mol%) incorporated Mg-Al hydrotalcites for transesterification of soybean oil with methanol and n-octanol to produce biodiesel and biolubricant was presented by Rahul et al., (2011). They observed that the catalyst with 20 mol% of lanthanum showed a soybean oil conversion of 100% and biodiesel yield of 95% at 423 K in 4 hours. Basicity also affected catalyst activity.

Economical and safe ways to improve the properties of biolubricants, such as increasing their poor oxidative stability and decreasing high pour points, among others was presented by Salimon et al., (2010). They concluded that plant bio-based oils are an important part of new strategies, policies, and subsidies, which aid in the reduction of the dependence on mineral oil and other nonrenewable sources.

Betiku et al, (2017) worked on two-step conversion of neem (azadirachta indica) seed oil into fatty methyl esters using a heterogeneous biomass-based catalyst from cocoa pod husk. Their results confirmed that neem seed oil methyl ester (NSOME), which conformed to ASTM D6751 and EN 14214 standards, was produced at an optimum yield of 99.3 wt % using a methanol/oil ratio of 0.73 (v/v), catalyst amount of 0.65 wt%, and reaction time of 57 min while maintaining a constant reaction temperature of 65 °C. The transesterification of neem seed oil using a two step acid-base transesterification method using H_2SO_4 and NaOH had been reported with results that conformed to American Standard Testing Method (ASTM D6751), European Standard and Ghana Standard Authority (Abdul-Wahab and Takase, 2019).

According to Tanwar et al., (2013), neem oil has the highest potential and production among the available wild oils for neem oil methyl ester (NOME) production in India. About 75-80% oil potential is available in surplus which could be harnessed. This is a promising future for biolubricants production from neem oil. In addition, neem seed which contain 25%-45% oil on dry matter basis which is a non edible oil was used in transesterification reactions (Sathya and Manivannan, 2013).

Chauhan (2015) reported the experimental analysis of neem oil by transesterification and partial hydrogenation with better lubricity properties. Chan et al., (2018) added that in terms of the tribological aspect, a good biolubricant base stock can be made by optimizing two characteristics, which are the stability and adhesiveness of its generated tribofilm within the lubricating conditions. These could be met provided that the viscosity range of the lubricant and the lubrication regime are suitable and tally with the application requirements.

Tribological additives may then be added to further improve the base stock. In a recent study, it was proved that the production of different biolubricants having suitable chemical and rheological properties fitted to be used as commercialized industrial lubricants (Attia et al., 2020).

Catalyst is a fundamental requirement for biodiesel production, the choice of catalyst has always resulted into certain level of differences in the course of producing methyl esters (Singh et al., 2006). Difficulties with using homogeneous catalysts centre on their sensitivity to free fatty acid (FFA) and water in the source oil (Ma and Hanna, 1999). Heterogeneous catalysts are promising for the transesterification reaction of vegetable oils to produce methyl esters and have been studied intensively over the years. Unlike the homogeneous catalysts, heterogeneous catalysts can be easily separated from reaction mixture and reused for many times (Omotoso and Akinsanoye, 2015). Non-conventional catalysts are natural or biological materials that can serve as heterogeneous catalysts. These include egg shells, waste shells of mollusk, industrial egg shell wastes, carbonate rocks, oyster shell, crab shells, chicken shells, duck shells, quail egg shells, laterites and lots more (Nakano et al., 2003). Most of these are wastes generated in various nations; they can serve as cheap source of catalysts that are employable in biofuel and biolubricants production. This will lead to the reduction of cost, waste recycling and clean environment, thereby enhancing waste to wealth (Omotoso and Akinsanoye, 2015).

This study was carried out with the objective of extracting oil from neem seed and chemically modifying the crude oil using calcium oxide from snail shell to produce synthetic ester biolubricant. The physico-chemical properties of neem biolubricant produced were compared with standard properties of lubricants. The parameters effecting on the transesterification such as methanol-oil ratio, catalyst ratio and reaction time were optimized.

EXPERIMENTAL

— Materials

The materials and reagents used in carrying out the research are as follows: crude neem oil, anhydrous methanol and orthorphosphoric acid.

The instruments and equipment used in carrying out the study are: beakers (50-500 ml), burettes (50 ml), conical flasks, glass funnels, pH meter, retort stand and clamps, measuring cylinders (50-1000 ml), furnace, oven, electronic weighing balance, water bath, magnetic stirrer, heating mantle and the Gas Chromatography Mass Spectroscopy (GCMS) instrument.

— Preparation of Catalyst

The CaO catalyst was prepared from waste snail shells by calcination method. The snail shells were washed using distilled water and sun dried. Then the solid catalysts was crushed and calcined in the furnace at 800^oC for 1 hour. After cooling, the resultant solid product was ground, sieved and kept in air tight sample bottles.

The sample bottles were kept in the desiccator to prevent air contact. The catalyst was activated by impregnation of a known quality of the powdered sample using orthorphosphoric acid as the impregnation agent. Impregnation ratio of 15 g of activation agent to 5 g of the calcined snail shells was mixed. This mixture was stirred for 30 minutes, until a paste was formed and was allowed to stand for 24 hours.

The activated substrate was filtered using a filter paper. The mixture was washed by pouring distilled water gradually over the filter paper containing the sample while the filter paper was placed over a conical flask. The pH of the filtrate was checked regularly using a pH meter until the pH was within the range 6 – 8. The activated substrate was then dried in an oven at 105^oC for 10 minutes.

— Biolubricant Synthesis

Trans-esterification reactions were performed in a water bath. The oil was heated at 55^oC for 5 minutes in a heating mantle to evaporate water and other volatile impurities.

The trans-esterification process parameters such as amount of catalyst, methanol to oil ratio and reaction time were varied to attain maximum methyl ester conversion. This was achieved by stirring a mixture of oil and catalyst (6%, 8% and 10% of the weight of the oil) with a magnetic stirrer, then, adding a designated amount of methanol (6:1, 8:1 and 10:1 mole ratio of methanol to oil). Each experiment was allowed to continue at 65^oC at variable reaction time (3, 4 and 5 hours).

After the reaction was completed, the mixture was allowed to cool down and filtered to remove the catalyst. After, the mixture was poured into a separating funnel and allowed to stand for 24 hours to separate layers clearly.

— Characterization of Neem Biolubricant

According to ISO VG 46, the properties of the crude oil which are pertinent to lubricity are the viscosities at 40°C, 100°C, viscosity index and the pour point. The synthesized biolubricant was characterized for these physico-chemical properties.

» Viscosity

The viscosity was measured at two different temperatures of 40°C and 100°C respectively. A proper viscometer spindle was chosen. The sample was transferred to a beaker large enough to hold the viscometer spindle. The beaker was placed on a heating mantle which was set to a desired temperature, while the temperature of the sample was raised. The temperature of the sample was checked using a thermometer.

When the desired temperature was reached, the sample was then removed from the heat source and the viscosity was recorded. The spindle was attached to the upper coupling by holding it between the thumb and forefinger while cautiously rotating the spindle counter clockwise. The knob was set to the minimum speed which includes the centipoise range of the material to be tested.

The uppermost number on the knob indicates the revolutions per minute (rpm). The spindle was immersed into the sample up to the middle of the identification in the shaft. The viscometer was turned on and allowed to run until a constant reading was obtained.

» Viscosity Index

The viscosity index is an empirical number. Its value was determined using the values obtained from viscosity. The viscosity index was determined using ISO VG index calculator.

» **Pour Point**

The pour point is the lowest temperature at which a liquid is still able to pour. A quantity of 5 ml of oil was drawn into a capillary tube tied to a thermometer, placed in a 250 ml beaker containing distilled water and immersed together in a water bath for controlled heating. The temperature at which the oil begins to move downwards due to its weight is the pour point.

— Statistical Analysis

The methodology involved optimizing neem biolubricant production using statistical analysis with MATLAB® R2014a The MathWorks Inc. (1994-2020) software.

The optimization of neem biolubricant production via transesterification process was designed using full factorial design. The effects of variables such as the methanol-oil ratio, catalyst ratio and reaction time, on the biolubricant yield was determined in order to find out the optimum conditions required for neem biolubricant production.

RESULTS AND DISCUSSION

— Interaction between variables

Figures 1–3 represent the interaction between the three variables; methanol-oil ratio, catalyst ratio and reaction time. The maximum point on these figures indicates a well-defined optimum condition for the variables.

In Figure 1, high biolubricant yield was favored by high catalyst ratio and high methanol-oil ratio. Similarly in Figure 2, high biolubricant yield was also favored by high reaction time and high methanol-oil ratio. Also in Figure 3, high biolubricant yield was favored by high catalyst, high reaction time and high methanol-oil ratio. However, in Figure 3, the catalyst proportion of 6 wt% favored the oil yield than others.

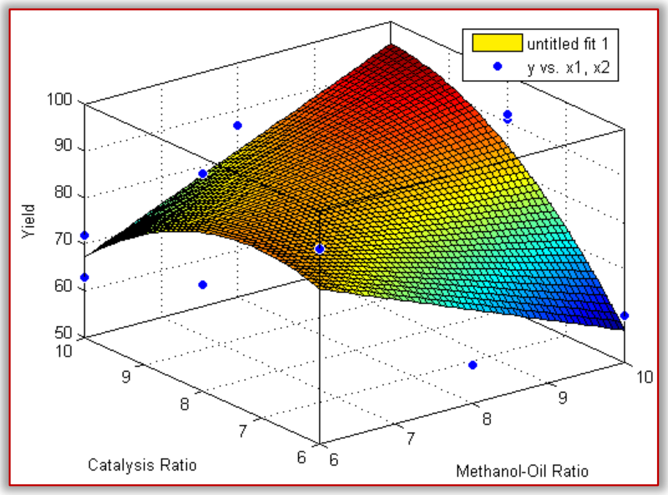

Figure 1: A plot showing the interaction between catalyst ratio and methanol-oil ratio on biolubricant yield

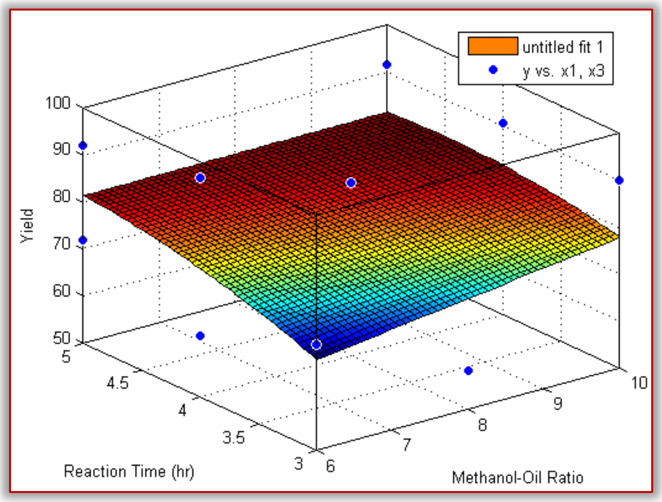

Figure 2: A plot showing the interaction between reaction time and methanol-oil ratio on biolubricant yield

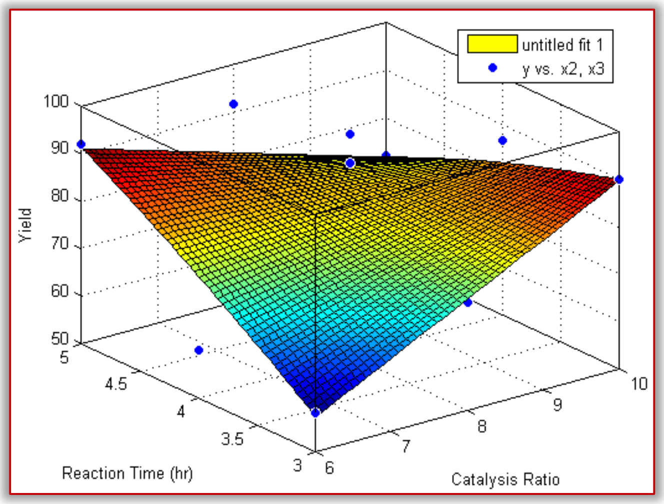

Figure 3: A plot showing the interaction between reaction time and catalyst ratio on biolubricant yield

— Optimization of Parameters

Statistical analysis was used for the optimization of parameters. The model equation based on the coded values (X_1, X_2 and X_3 as methanol to oil ratio, catalyst concentration and reaction time respectively) for the yield of biolubricant from neem oil is expressed in Equation (1) as follows:

$$Y = \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{11}X_1^2 + \beta_{22}X_2^2 + \beta_{33}X_3^2 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2 \quad (1)$$

The β_i and β_{ij} are constants, where i and j are positive numbers 1, 2 or 3.

The result of this statistical analysis generated optimum values at methanol-oil ratio of 6:1, catalyst concentration of 6 wt% and reaction time of 5 hours. The optimized biolubricant yield of 96.7% was produced at a methanol-oil ratio of 6:1, catalyst amount of 6 wt. % and reaction time of 5 hours.

Thus, it showed that the statistical values and the experimental values are in agreement. Therefore, the effect of reaction conditions, which yielded a conversion of 96.7%, were methanol-oil ratio 6:1, catalyst concentration 6 wt. % and reaction time of 5 hours.

— Determination of Physico-Chemical Properties

The ISO VG 46 is one of the grades requirements based on the viscosity range of lubricants, and is one of the two grades that represents over 80% of all lubricants utilized (Lauer, 1995). Requirements for such applications are viscosities at 40 and 100 °C, viscosity index and pour point.

Table 1: Physico-chemical Properties of Synthesized Biolubricant in Comparison with ISO VG 46 and Petroleum Based Lubricants

Properties	Neem Bio lubricant	ISO VG 46	Petroleum based Lubricant
Viscosity at 40 °C (cSt)	54.87	> 41.4	10.801
Viscosity at 100 °C (cSt)	6.64	> 4.1	3.136
Viscosity Index	136.43	> 90	165.4
Pour Point	-9	< - 10	- 9

The viscosities of the biolubricant at 40 and 100 °C are very important lubricity properties. They are useful in determining the fluidity of the lubricant at low and high temperatures. They also show the thermal stability of the lubricant. The viscosity of the synthesized neem biolubricant meets the requirement of the ISO VG 46 since its viscosities are within the standard range presented in Table 1.

Viscosity index shows the characteristics of the lubricant viscosities when temperature changes are applied. The viscosity index obtained for the neem biolubricant was 136.43 and it is comparable to other plant based biolubricant. The high viscosity index of the biolubricant is an indication that changes in viscosities at higher temperatures are going to be minimal. The higher the viscosity index, the more preferable is the lubricant.

Pour point is the lowest temperature at which oil flows. Pour point is crucial for oils that must flow at low temperatures. It is one of the critical properties which determines the performance of lubricants. The pour point of the synthesized neem biolubricant obtained was – 9 °C, which is also comparable to the pour point value of other petroleum based oil.

— GCMS ANALYSIS

Synthesized neem biolubricant was selected for GCMS analysis based on its high lubricity properties. To identify the fatty acid composition of the synthesized neem biolubricant, the gas chromatography coupled with mass spectrometry (GCMS) analysis was used.

Table 2 presents the composition of the synthesized biolubricant produced from the neem oil as extracted from the obtained various peaks of the GCMS used. The table showed that the produced biolubricant consists principally of the fatty acid methyl esters of oleic, palmitic and stearate acids, hence confirming a high quality product.

The values are represented as the relative percentage area from the sum of all identified peaks. Also from Table 2, methyl-9-octadecanoate (methyl oleicate) has the highest percentage. It can also be observed that traces of other methyl esters are present in the composition of the produced biolubricant.

Table 2: Fatty Acid Composition of Synthesized Neem Biolubricant

S/ No	Name of Compound	Number of Carbon	Value of Compound (%)	Retention Time (Minutes)
1	Methyl-hexadecanoate (Methyl palmitate)	C17	56.35	11.33.
2	Methyl-9-octadecanoate (Methyl oleicate)	C19	100	13.307
3	Methy-octadecanoate (Methyl stearate)	C19	43.33	13.517
4	Hexa-decanonoic acid (Palmitic acid)	C16	4.97	13.787
5	9-octadecanoic acid (Oleic acid)	C18	83.73	14.136
6	Methyl 18-methyl-nonadecanoate	C21	4.88	15.690
7	Diisoctyl-phthalate	C24	6.74	18.891
8	9-hexylheptadecane	C26	5.22	21.708

Figure 4 is the chromatogram of the biolubricant synthesized from neem oil, many minor and major peaks were observed between retention time of 11 minutes to 22 minutes.

The chromatogram showed three considerable peaks at 11.33, 13.307 and 13.517 minutes and minor peaks with retention time of 13.787, 14.136, 15.690, 18.891 and 21.708 minutes. These peaks are due to the esters of fatty acids and methanol since neem oil is a mixture of various fatty acids.

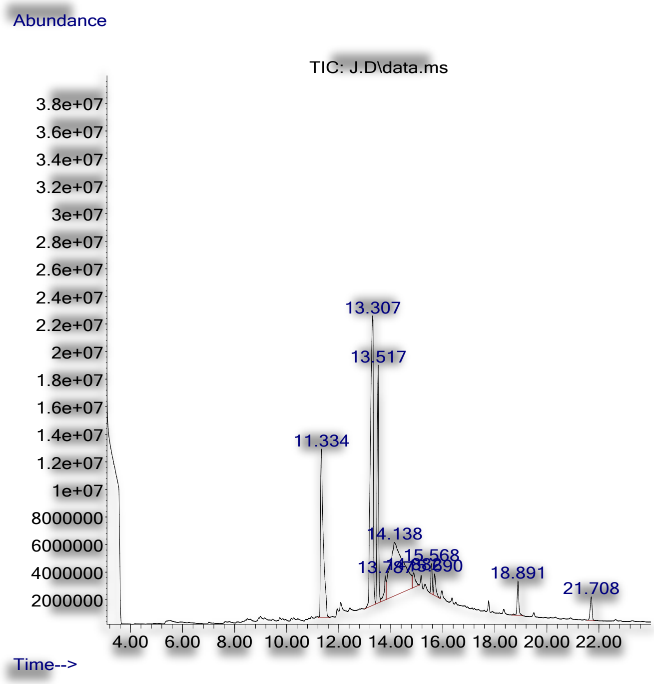

Figure 4: Chromatogram of Methyl Ester of Neem Oil

The peaks of 13.787 and 14.136 minutes indicated unreacted fatty acids. The peak of retention time 18.891 minutes indicated the chemical modification of oil due to either dimerization or condensation reaction of present fatty acids.

The peak at 21.708 minutes was as a result of degradation of fatty acids to alkanols which was promoted by the presence of calcium carbonate from snail shells. The product suggested a beta cleavage of the fatty acid caused by thermal catalytic effects of the carbonates.

CONCLUSION

Neem oil was extracted from neem seeds and chemically modified via trans-esterification using snail shell as a natural source for the production of the catalyst. The catalyst was derived from $CaCO_3$ in the snail shells which was converted to CaO after calcination at a temperature of 800°C for 1 hour. The optimum conditions yielded a conversion of 96.7% for a methanol-oil ratio of 6:1, catalyst concentration of 6 wt. % and a reaction time of 5 hours.

The method of using snail shells as catalyst source reduced the cost of catalyst and was environmentally friendly. Hence, it could be used in large scale industrial process of bio lubricant production, making the process affordable and ecologically benign.

Major lubricating properties of synthesized neem biolubricant analyzed and compared with standards specified by ISO VG 46 showed good agreement. Hence, the synthesized neem biolubricant can preferably serve as a substitute for petroleum based lubricants in light and industrial gear applications. Other natural sources of calcium oxide is recommended as catalyst in the production of biolubricants.

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