Transesterification Kinetics of *Jatropha* Methyl Ester and Trimethylolpropane for Biolubricant Synthesis Using *Paphiaundulata* Shell Waste

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A R T I C L E   I N F O

A B S T R A C T

Non-edibleoil has become an interesting issue for biodegradable lubricant feedstocks. Modification of biolubricant synthesis from *Jatropha curcas* plants-based oil using *Paphiaundulata*shellas solid waste catalyst could minimize the environmental impacts and separation process difficulty compared to fossiel oil and homogenous catalysts. A transesterification of *Jatropha* methyl ester (JME) with Trimethylolpropane (TMP) using *Paphiaundulata*shell waste as heterogenous catalyst to biolubricant was run in a batch stirrer reactor. The effect of temperature reaction on the biolubricant was determined at range 90 to 130^{0}C. The optimum temperature reaction was found at 110^{0}C with 78.67% composition of triester (TE). The exess amount of JME was set up at 4:1 ratio to TMP for forward reaction control. The kinetics of the transesterification reaction were justified. The second order kinetics model best fits the obtained data with an overall reaction rate constants of 0.0427 (%wt/wt min °C)$^{-1}$. The predicted activation energy is nearly 2.2 kJ/mol. The estimated pour point value is -5^{0}C,and the viscosity index (VI) is 81. The physicochemical characteristics of resulted *Jatropha*biolubricant, kinematic viscosity (40^{0}Cand 100 ^{0}C)are close to other oilseed crops based lubricant, mainlyrape seeds oil.

INTRODUCTION

Lubricants oil is widely used for reducing friction in machinery. Mineral based lubricants have long served in application, like automobiles and hydraulic engines. However, million tons of lubricants are dumped into environment through leakage from machine or vehicle and careless disposal methods, where some of these wastes are resistance to biodegradable. In recent years, world is facing with fluctuation price of fossil fuel that becoming worst. This scenario has gain attention among researcher in developing renewable resources for lubricant production to fulfill market demands. Otherwise, there are seen an increases public awareness about contamination and health aspects that caused from petroleum based oils lubricant [1]. Besides that, the major part of biolubricant manufacturing from the fossil fuel using homogeneous catalyst which also polluted the environment, difficulty separation and high cost, thus this work aims is to overcome these issues by using *Paphia*undulata shell waste and Trimetylolpropane (TMP) as chemical modification for *Jatropha curcas* oil conversion to biolubricant.

The biolubricant synthesis can be conducted via transesterification, epoxidation and hydrogenation process. In this case, transesterification of vegetables oil with polyhydric alcohol or polyols helps to minimize the limitation of biolubricant characteristics with the elimination of hydrogen atom from β-carbon of vegetables oil structure [2]. The transesterification takes place three phases with present of catalyst. Trimetylolpropane monoester (TMPME) and trimethylolpropanediester (TMPDE) become intermediate products, where the trimetylolpropanetriester (TMPTE) is the main product of processing.

Generally, catalyst plays important role in the transesterification process where it functions as reducing time of reaction. Homogenous catalysts such as inorganic acids and alkali have been being applied widely in the

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transesterification process either in biodiesel and biolubricant production. Previous researchers reports that the higher conversion of TMP triester can be found in 8 hours reaction time using calcium methoxide as catalyst [3]. Unfortunately, in the reality, it has been decreasing in these catalysts application due to a few problems, such as the catalysts cannot be regenerated or recovered after the reaction, and it also tend to produce toxic wastewater [4, 5]. Next, the heterogeneous catalyst such as metallic or metal oxide has wide potential to use it, where these catalysts can be easily recycled. The metal oxide catalysts are much cheaper than enzyme or other biochemical catalysts. Currently, study on the transesterification process using solid base heterogeneous calayst such as calcium oxide that derived from mollusk shell has been interesting [5, 6]. Thus, the metal oxide from *Paphia*undulata shell waste has been being developing as advantageous catalyst [7].The surf clam (*Paphiaundulata*) is mostly found in muddy area such as in Malaysia and Thailand. In Malaysia, this clam was sold life for human food and also has been being commercializing for food industry. Thus, the shell that discards as waste able to utilize as solid catalyst in transesterificationprocess [8].The *Paphia*undulata waste shell contains calcium oxide (CaO)after activated by calcinations process. The calcium oxide from shell waste not only solves waste of seafood industry but also enhancing cost effectiveness when used as catalyst [9].

Tansesterification of *Jatropha* methyl ester, and hence a kinetic was performed by considering the rate of intermediate changes, ME and DE with respect to the concentration of the limiting reactant, TMP. The transesterification process will form trimethyolpropane monoesters (TMPME) and trimethylolpropane diester (TMPDE) as intermediate results, and trimethylolpropane triester (TMPTE) as main product. By product that produce in this reaction, methanol (CH_3OH) is removed during processing to ensure completion of reaction. The approach of reaction kinetics is to provide better understanding about catalyst activity [10]. Thus, in this report, the simple model was applied based on the first and second order kinetics. The best order of reaction was selected with its corresponding reaction rate.This work was focused on developing the simple transesterificationkinetics model of jatropha methyl ester (JME) with trimetylolpropane (TMP) by applying new type of catalyst derived from *Paphiaundalata* shell waste for biolubricantsynthesis.

MATERIALS AND M ETHODS

Materials:

The *Jatropha* seedsoil was procured by Bionas Sdn Bhd, Kuala Lumpur, Malaysia. The *Paphiaundulata* shell waste supplied by the seafood restaurants, Tanjung Lumpur, Kuantan, Pahang, Malaysia. Trimethylolpropane (TMP) and N,O-Bis(trimetyhlsilyl)tri-fluroacitemide (BSTFA), Methanol, Hydrocloric acid, Ethyl acetate, orto-Phosphoric acid, and n-hexane were ordered from Sigma Aldrich and Chemmart Asia,Malaysia.

Methods:

Conversion of Jatropha Seeds Oil to JME:

Firstly, the acid treatment process was applied for free fatty acid removal of *Jatropha* oil. The *Jatropha*seeds oil was converted to JME. The oil was placed in two neck flask equipped with magnetic stirrer and condenser that used as cooling system. Then, the activated *Paphiaundulata*as catalyst solution was poured into a flask. The mixture was heated with constat stirrer up to 60°C for 3 h. After mixing, the mixture was put in a separatory funnel for excess methanol separation. The excess methanol with impurities moves to the surface and removed. The bottom layer, indicated as JME was collected and washed using warm water and orto-Phosphoric acid. Then, the obtained JME was used for the transeterification process in the *Jatropha* biolubricant synthesis.

Experimental Procedure:

The required amount of JME and TMP was weighted and placed in two necks round bottom reaction flask. The reactant and the activated *Paphiaundalata* waste as solid catalyst (3%, wt/wt) were stirred and heated until required temperature on the flask. The neck round bottom flask was completed by a condenser. In order toavoid the backward reaction, excessive usage of JME was provided, where the stoichiometry is 4mol of JME to 1 mol of TMP. The resulted methanol as by product was kept drawn continuously from reaction at vacuum condition. The vacuum pressure was controlled at 50 mbar with the temperature ranged between 90°C and 130°C. Next, the mixture was placed in a vacuum filter for catalyst and other substances removal. The synthesized *jatropha* biolubricant was measured and analyzed using Gas Chromatography (GC) for conversion determination. The samples were collected based on the time interval and stored in a cold room at below 2°C for analysis. Three runs analysis were conducted for each experiment, the average data were recorded. These treatments was done for temperature impacts observation on the synthesized *Jatropha* biolubricant.

Gas Chromatogrphy (GC)Analysis:

The percentage composition of biolubricant was determined by GC equipped with flame ionization detector

(GC-FID) using BD-5HT column. Injector and detector were set at 380°C to 400°C respectively. Initial temperature of the oven was controlled at 100°C with 1 minute of holding time. The increased temperature was observed at 5°C/min to reach a temperature of380°C, and it was held for 25 minutes. Next, 30μl of the sample was mixed with 1ml ethyl acetate (GC grade) and placed in a test tube. After the mixtureswirld for a few minutes, 0.5ml of *N,O*-Bis(trimethylsilyl)tri-fluoroacetamide (BSTFA) was added and transferred to 2ml vial. The sample in vial was heated at 40°C for 5 minutes prior to analysis.

The quantitative results of biolubricant weredetermined using Equation 1, where the percentage composition of biolubricant was estimated based on the external standard from partial glyceride.

The percentage composition can be calculatedby the following:

$$\text{Percentage composition } \left(\frac{w}{w}\%\right) = \left(\frac{\text{Weight of TE}(w/w\,\%)}{\text{Total weight of ME} + \text{DE} + \text{TE}(w/w\,\%)}\right) \times 100\% \tag{1}$$

, where ME= monoester , DE= diester, TE= triester

Viscosity Measurement:

The kinematic viscosities of *jatropha* biolubricant were analyzed based on the ASTM D-445. The analysis was conducted at 40°C and 100°C. The oil bath was also provided for temperature control. The temperature range for application may require certain magnitude of viscosities. The viscosities index (VI) was measured by ASTM D-2270.

Pour Point Analysis:

The pour point analysis of *jatropha* biolubricant was carried out using ASTM D-97. The pour point was measured by placing a tes jar filled with the processed oil into a cooling media, and it was observed in 3°C increment until it stopped to pour. The pour point was indicated as temperature, where the oil was able to pour.

Kinetics ModelEstablishment:

The kinetics model of JME transesterificationwith TMPfor biolubricant was established based on the effect of temperature and time. The results of kinetics development wereperformed from the decreasing rate of limiting reactant concentration of TMP. Then, the data were plotted to get the best value of reaction either first or second order kinetics.

Activation energy is the energy that initiates the reaction. Arrhenius equation was applied in order to determine the activation energy. The activation energy correlates with the reaction rate constant and temperature. The equation is shown as follows:

$$\log_{10} k = -\frac{E}{2.303RT} + \log_{10} A \tag{2}$$

Where, k is the reaction rate constant, R is the gas constant, T is temperature, A is Arrhenius constant and E or E_A is activation energy.The activation energy, E_A is determined from the slope of the graph $\log_{10}k$ versus 1/T.

Catalyst Reusability:

After reaction termination, the final product was centrifugedfor catalyst collection. Then, the catalyst was dried in oven for the next experiment. The experiment was conducted with the solid catalyst that directly used after drying process. The reaction was made in optimum condition of transesterificationprocess between JME and TMP. The used catalyst for transesterificationwas 3%wt/wt, TMP to JME molar ratio was 1:4, and reaction temperature was 110°C and 3h of reaction time. These procedures were repeated three times for one of four cycles experiment.

RESULTSAND DISCUSSION

Temperature Impact on the Jatropha BiolubricantSynthesis:

The reaction temperature of JME transesterificationand TMP using *Paphiaundulata*shell waste as solid catalyst for the *Jatropha* biolubricantwas carried out at 90°C, 100°C, 110°C, 120°C and 130°C. The final composition of product was shown in Figure 1 that contains triester (TE), diester (DE) and monoester (ME).
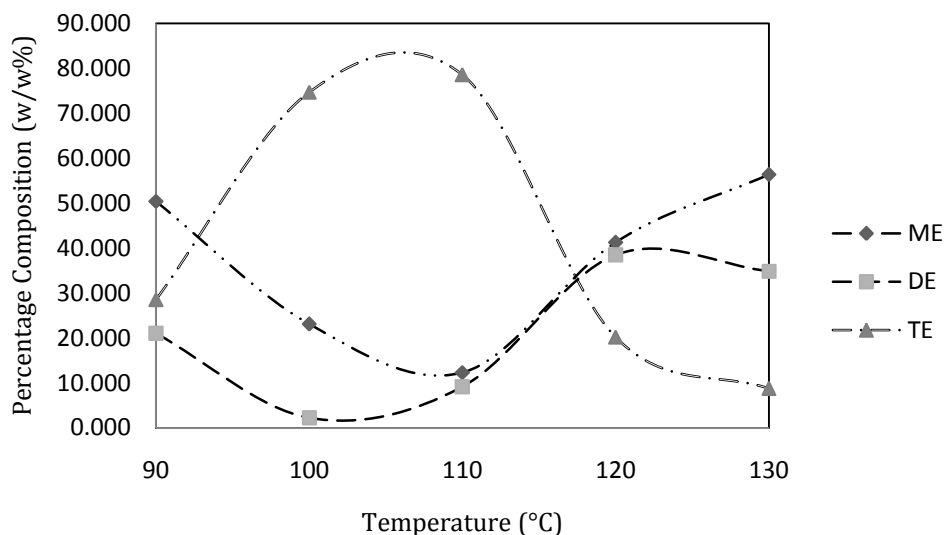
Fig. 1: Temperature impact on the biolubricant synthesis.
         (p=50 mbar, catalyst=3% w/w), TMP:JME=1:4, t=3 h)

The curve indicates the conversion of JME to ME, DE and TE simultaneously. The highest percentage of TE (78.67%) is found at temperature of 110°C. The low temperature tends to reversible reaction; it has been approved by using another vegetable oilsas biolubricant feedstock. On the other hand, it is reflected that at the higher temperature, colour of product is turn to dark due to oil oxidation. Besides that, the sintering process could be taken place, where the tendency of solid catalyst fuses together and forms 'cake' that caused by the catalyst activity reduction. Then, the investigation results using *Paphiaundalata* shell waste as heteregenous catalyst shows higher TE composition compared than homogenous catalyst (NaOH). The formatted *Jatropha*biolubricantusing NaOH as catalyst at 200°C reaches 47 % TE maximal [11]. The risedOtherwise, the increased temperature more than 110°C reflects the decreasing of TE, it may leads to vaporization of the reactant volatile substance. These experiments approves the obtained biolubricant with the higher TE as main product than the ME and DE asintermediate results. The risedTE is observed by the decreasedME and DE at various temperatures.

Kinetics Justification: Transesterification Reactions of JME and TMP for Jatropha BiolubricantUsing Paphiaundulata Shell Waste as Solid Catalyst:
The kinetics constant and reaction order were observed by the different experiment times. The time interval was fixed 0 – 60 min. at various temperatures. Precautionary steps were conducted by the samples storage about 0°Cin a cold room prior to analysis. The transesterificaion kinetic establishment follows the reaction mechanisms as shown below.

TMP + JME ↔ TMPME
                        + CH_3OH (3)
TMPME + JME ↔ TMPDE + CH_3OH (4)
TMPDE + JME ↔ TMPTE + CH_3OH (5)

The overal reaction can be written, as in
    TMP + 3JME ↔
TMPTE + $3CH_3OH$ (6)
Next, the time gives the significant impact on the biolubricant (TE) synthesis at various temperatures. These phenomenons can be shown at the curve below (Fig. 2).
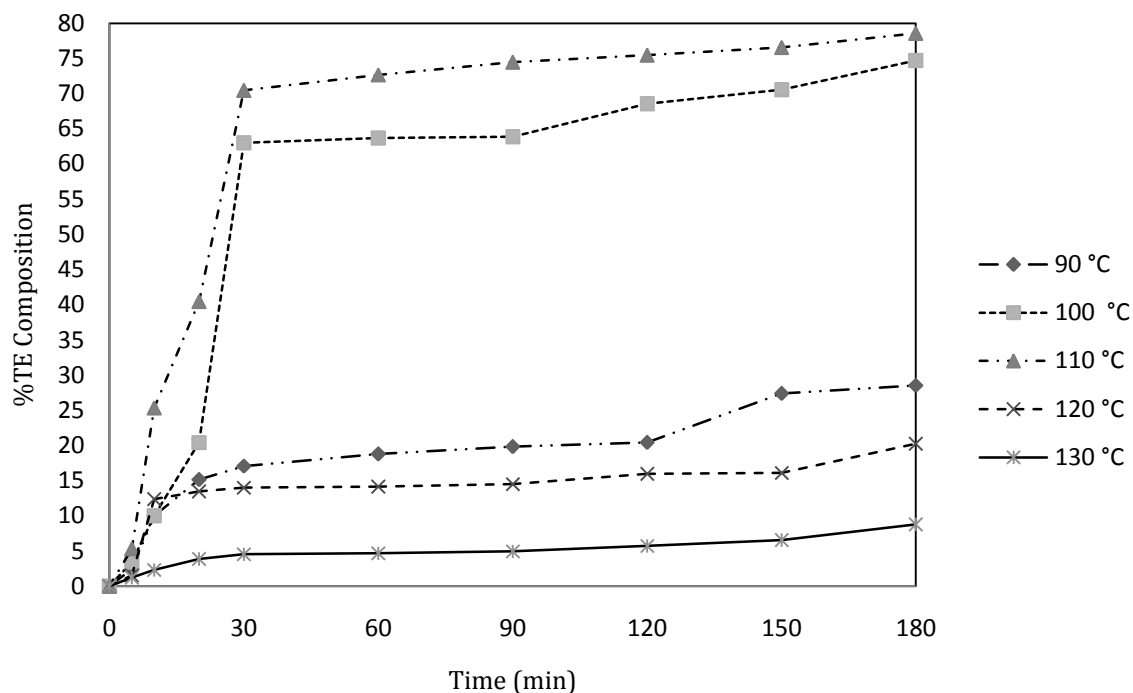
Fig. 2: Time impact on the biolubricant (TE) synthesis at various temperatures.
(p=50 mbar, catalyst=3% w/w), TMP:JME=1:4)

The curve shows the slowest reaction at130°C compared to another temperatures. This observation has been approved by using NaOH as homogenous catalyst, but it was found at 120°C[11]. It could be estimated due to the diference of catalysts, amount and its properties. The higher yield and conversion of time range were indicated at 110°C. One side, the interpreted curve indicates the optimal temperature and time are 110°C and 3 h., but another side, the above curve indicates a rapid reaction at first 30 minutes of various temperatures. Therefore, it is appropriate to establish the reaction kinetics during this time range. There are a fewassumptions that made for the preferred kineticsmodel. Firstly, the used catalyst is sufficient to shift the reaction to equilibrium state, thus the catalyst reactions can be ignored and the concentration of catalyst is constant, so it could be assumed insignificant. Secondly, the single step transesterificationprocess is assumed that the excess of JME concentration is also constant and negligible. Thus, the depletion of the limiting reactant is analyzed in order to obtain the reaction rate and kinetics order. The reaction rate law is referred by the following equations.

$$-r_{TMP} = -\frac{d(TMP)}{dt} = k_{TMP}[TMP][JME] \tag{7}$$

Catalyst and JME concentration are assumed negligible, [TMP] = [JME]

$$-r_{TMP} = -\frac{d(TMP)}{dt} = k_{TMP}[TMP]^2 \tag{8}$$

Integrated Equation (8),

$$\left[\frac{1}{TMP} - \frac{1}{TMP_0}\right] = k_{TMP\,(t-0)}$$

$$k_{TMP}\,t = \left[\frac{1}{TMP_0}\right] - \left[\frac{1}{TMP}\right] \tag{9}$$

, where $k_{TMP}$is second order rate constant, t is reaction time, $[TMP_0]$ is initial of TMP concentration, and [TMP] is final of TMP concentration in product. The unit of rate constant k isstated as (%wt/wt min)$^{-1}$.

The collected data were plotted and the best reaction order was identified (Fig. 3 and Fig. 4). The simple

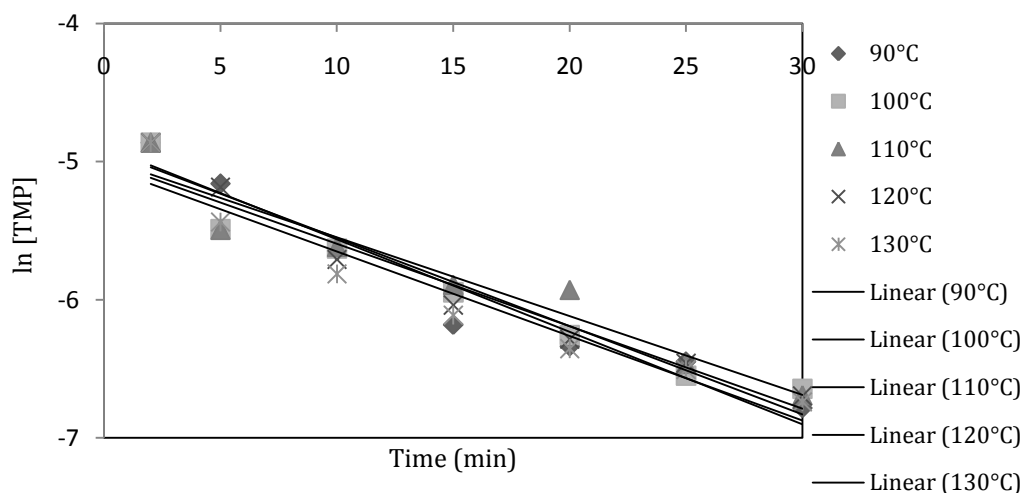kinetics modelsof first and second order was applied by ignoring the intermediates product.

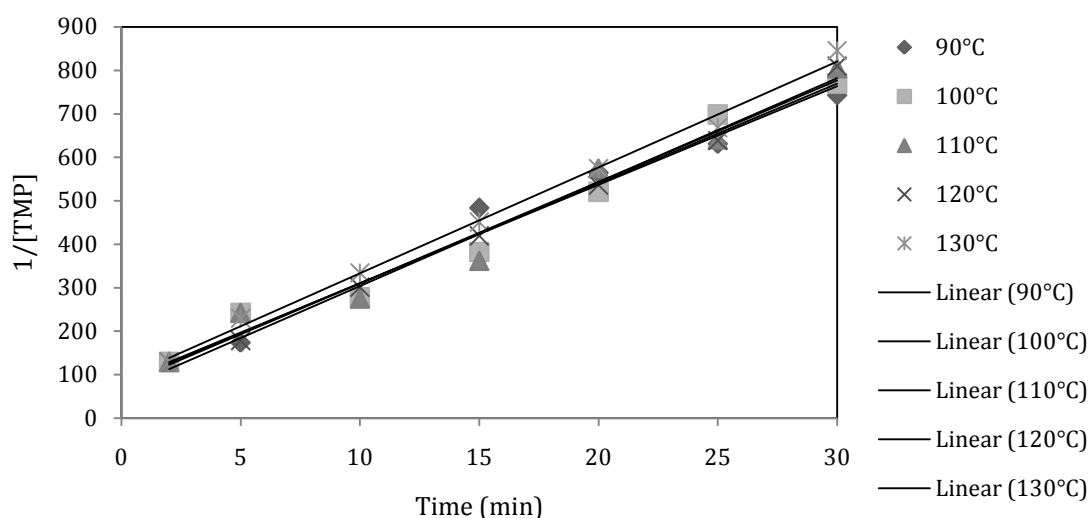**Fig. 3:** First order kinetics model.

Fig. 4: Second order kinetics model.

According to the interpreted data from Fig. 3 and Fig. 4, the reaction rate constants can be distinguished based on the various temperatures as shown in Table 1.

Table 1: Reaction rate constant at different temperature.

Temperature (°C)	k, 1st Order	R^2	k, 2nd Order	R^2
90	-0.0669	0.9478	22.671	0.981
100	-0.0596	0.9446	22.956	0.9789
110	-0.0571	0.9314	23.332	0.972
120	-0.0639	0.9577	23.889	0.9953
130	-0.0611	0.9307	24.337	0.9948

Table 1 approved the higher regression value for the first and second orderkinetics , but the second order model best fit the represented data.The overall rate of the second order kinetics can be shown in Table 2.

Table 2: Overall reaction rate constant of second order kinetics model.

Rate of Reaction	Overall 2nd Order	
	(%wt/wt min °C)$^{-1}$	R^2
k_{TMP}	0.0427	0.9868

Then, the overall rate constant for second order of reaction has been plotted (Fig. 5). Its shows that, the reaction rates is directly proportional with the temperature of reaction. The dependency of temperature and rate
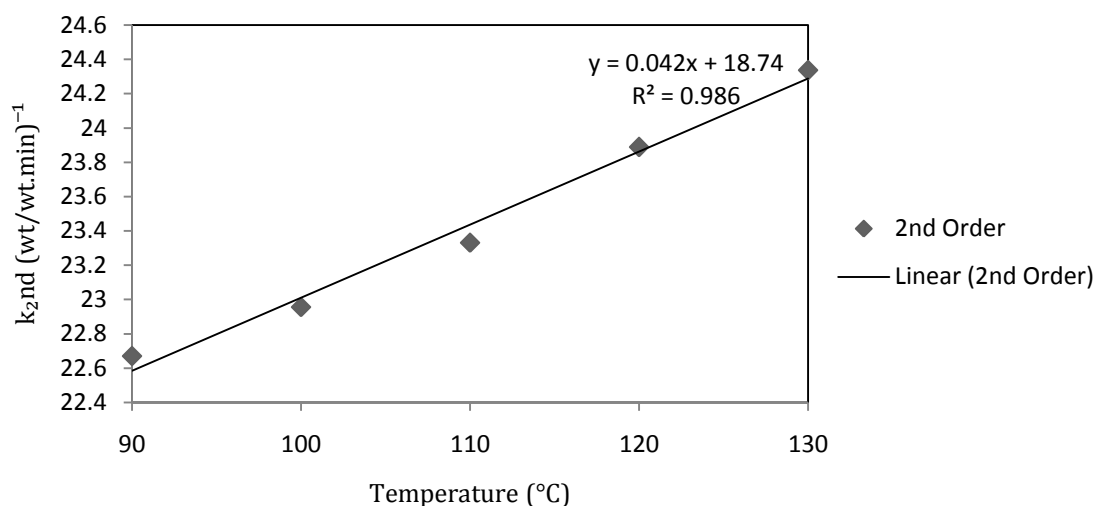
of reaction has also been reported [11].

Fig. 5: Overall rate constant of second order kinetics model.

The correlation between reaction rate constants and activation energy can be obtained usingArrhenius equation.Thevalue of activation energy is determined viacomputed slopbetween logk versus 1/T (Fig. 6).
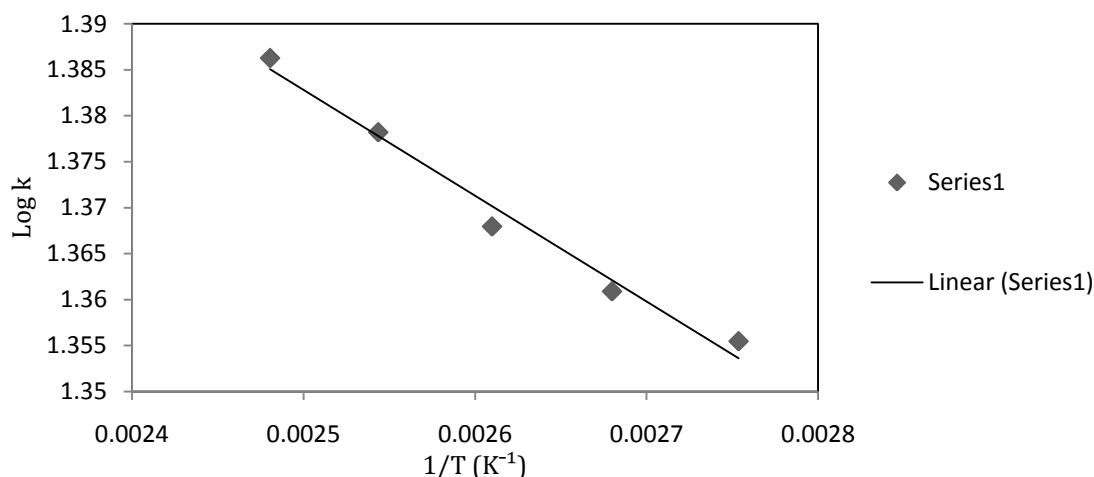

Fig. 6: Correlation of reaction rate constant and temperature.

The estimated value of activation energy for*Jatropha*biolubricant using various catalysts can be found in Table 3.

Table 3: Activation energy of *Jatropha*biolubricant using various catalysts.

Biolubricants	Activation Energy , E_A (kJ/mol)	R^2
*Jatropha*biolubricant using *Paphiaundulata* shell waste catalyst (This work)	2.2	0.982
*Jatropha*biolubricant using sodium methoxide catalyst[11]	1.65	0.861

Table 3 shows the activation energycomparison of*Jatropha*biolubricant between *Paphiaundalata* shell waste and sodium methoxideas used catalysts. The compared result reflects the *Jatropha*biolubricant using*Paphiaundalata* shell waste (2.2kJ/mol)) slightly higher than sodium methoxide(1.65kJ/mol [11]. It can be forecasted due to the difference of used catalysts, so it may be caused by various activation energy. The used solid catalyst is more sensitive to the temperature. Thus, the reaction temperature has also significant impact on the synthesis of *Jatropha*biolubricant.

Catalyst Recyclability:
The recyclability of the used solid catalyst becomes important consideration in the synthesized reaction of

biolubricant. The stability of the catalyst for few hours of industrial operation could be the main concern. Thus, few cycles of the reaction have been made to shows the ability of the catalyst. The simple procedure was applied in the recyclability of the spent catalyst without any treatment involved except drying process. The catalyst recylabilty results for *Jatropha* biolubricant synthesis can be illustrated in Fig 7.
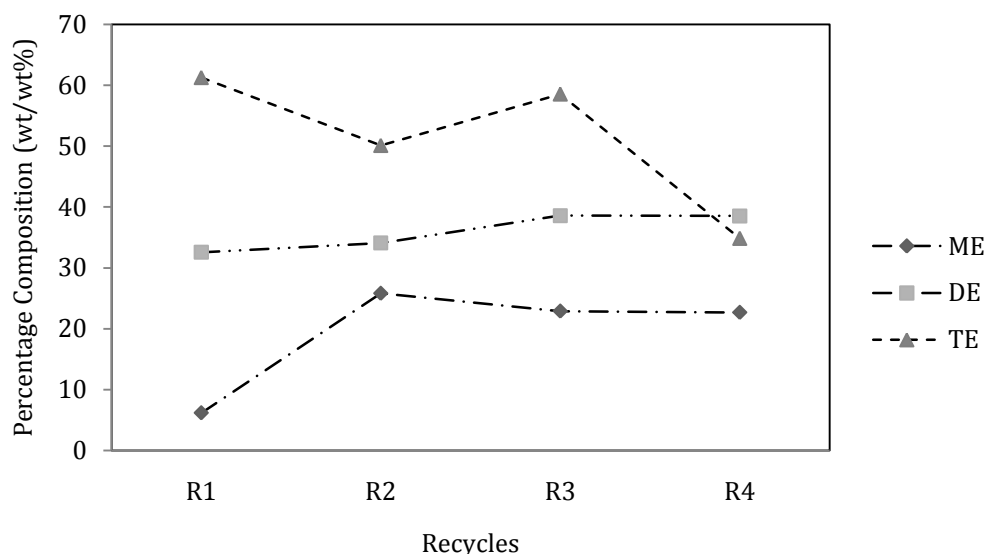

Fig. 7: The composition of *Jatropha* biolubricant at various recycles.
(p=50 mbar, catalyst=3% w/w), TMP:JME=1:4, T=110^{0}C)

Fig. 7 shows that the final product was higher at first until third cycle, where more than 50% TE composition is discovered. The deactivation of CaO on the catalyst occrures after the third cyle of reaction. In the forth cycle, the composition of product declines. This investigation validates that the solid catalyst is stable up to the forth cycle without treatment prior to the decreased catalytic reaction.

Physicochemical Properties of Jatropha Biolubricant:
The physicochemical characteristics of biolubricant become the main concern for usage in all types of practical application such as in machine and also in automotive industry. In addition, lubricants also should work in any extreme condition such as in winter season. Thus, the viscosity and pour point are the important parameters to indicate the biolubricant performance following ASTM. Table 4 reflects the indication of kinematic viscosity (c.s.t), viscosity (cp) at 40°C and 100°C, viscosity index (VI) and pour point as measured biolubricant standard.

The obtained viscosity value fullfills the biolubricant standard (32.29 cstat 40°C and 5.14 cst at 100°C). These obtained viscosity is higher than *Jatropha* biolubricant treatment using modified *Polipazime*^{TM}with glycerine (3.53 cst). Generally, TMP ester has higher viscosity due to three acid group content in its structure. The TMP is also found an aternative triol that provides a stable structure in which there is no hydrogen β-to the OH groups[12,13]. Moreover, the high viscosity index is desireble characteristic in industrial biolubricant due to its ability that resistance to oxidation and thermal exposure.

The tested pour point (-5°C) gives slightly higher than limitation of biolubricant (-6°C). It may be caused by unsaturated fatty acid content. The unsaturated fatty acid affects the characteristics of the synthesized *Jatropha* biolubricant, like the pour point, etc. The lower pour point tends to unstable condition of biolubricant synrthesis. The cold flow properties of the vegetables oil are poor and thus the limits their use in subzero temperature. The cold flow temperature can be improved by adding chain length or by branching of the fatty acid chain. Also improvement in low temperature flow behaviour of fatty acid esters can be achieved by attaching ester at double bond sites on the fatty acid chains [14].

Tabel 4: Resulted *Jatropha* biolubricant properties.

Properties	Biolubricant standard	Esterified rapeseed oil	This work
Kinemetic Viscosity (cst) 40 (°C) 100 (°C)	>12 1.9-6.0	35.34 7.99	32.29 5.14
Viscosity Index (VI)	>50	209.2	81
Pour Point (°C)	-6	-15.5	-5

Conclusion:

Process modification of *Jatropha* seeds oil biolubricant using solid waste catalyst produces a proportional physicochemical properties at optimum temperature. The high temperature is able to supply sufficient energy towards maximum reactions, but the obtained best results at 110^0C, 3 h, of catalyst and 4:1 of JME:TMP, 3% w/w of catalyst. The catalyst can be reused up to four times of cycles for effectively JME transesterification, mainly for TE formation. The transesterification reaction follows second order kinetics with a reaction rate constant of 0.0427 (%wt/wt min °C)$^{-1}$and activation energy is 2.2 kJ/mol. The properties of resulted *Jatropha*biolubricant(kinematic viscosity, pour point and viscosity index)using *Paphiaundulata* shell as solid catalyst fulfills the biolubricant standard which corresponds to better pour point and viscosity.The utilization of *Paphiaundulata* shell waste and renewable feedstock could be an alternative or ecofriendly processing for the biolubricantsynthesis.

ACKNOWLEDGMENT

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