





OPTIMIZATION OF BIODIESEL DEVELOPMENT FROM NON-EDIBLE INDIGENOUS FEEDSTOCKS IN NIGERIA

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ABSTRACT

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This study investigated the transesterification of Rubber seeds (*Hevea brasiliensis*), Avocado Pear seeds (*Gratissima persea*), and Nipa Palm Kernel seeds (*Nypa frutican*) locally sourced from Nigeria as an adsorbate for the esterification reaction of oleic acid with methanol. Reaction was catalysed by potassium hydroxide (KOH) to form methoxide oleate (biodiesel) adsorption from aqueous solution in batch model for the production of biodiesel. The results obtained showed that rubber seeds oil (RSO), Avocado pear seeds oil (APO) and Nipa palm kernel seed oil (NPKO) oil gave maximum biodiesel yields at 0.9 % w/v, 0.1 % w/v and 0.1 % w/v catalyst concentration respectively. Their biodiesel yields were 88.0 %, 92.0 % and 96.7% respectively. They recovered biodiesels were characterized as an alternative fuel via specific gravity, viscosity, acid value, flash point and cloud point. The results obtained showed that the specific gravity for RSO (0.886 kg/m³), APO (0.876 kg/m³) and NPKO (0.850 kg/m³) were fairly significant at temperature of 25 °C to other conventional petroleum diesel ASTM D 975, standard biodiesel ASTM D975, and standard biodiesel ASTM 6751 and EN14214.

Contribution/Originality: This study is one of very few studies which have investigated on Nigeria local plants capable of producing biodiesel via catalysed trans-esterification reaction.

1. INTRODUCTION

The need for alternative sources of energy is ever increasing due to the rapid growth in the number of energy dependent industries, energy consuming automobiles and population explosion. Currently at global level, fossil fuels: gasoline and diesels stands as the most conventional sources and readily available energy supply. However, their sources are non-renewable. Its continuous depletion to meet global demands has created the need for renewable sources of energy that are clean and free from environmental pollution such as greenhouse gases and toxic fumes (Abdel-Shafy and Mansour, 2016; Nkwoada *et al.*, 2016).

Moreover, the incomplete combustion from the fossil fuels contribute to higher emissions of particulate matter and polycyclic aromatic hydrocarbons. They possess the ability to form toxic metabolites and bind to DNA, RNA and cause cell mutation (Igwe and Ikaogo, 2015; Abdel-Shafy and Mansour, 2016). Therefore, need for concerted research study towards renewable sources of energy with minimal environmental contamination to meet global energy demand is therefore a research problem.

In Nigeria, the Energy Commission of Nigeria (ECN) has published severally that Nigeria fossil dependent economy is unsteady and unstable. They reported that depletion of oil production and undulating oil price is increasingly threatening Nigerian economy. Worst still, existing hydro – power plants are susceptible to poor management and under-capacity utilization (Osueke and Ezech, 2011; Gadiga *et al.*, 2018). Unfortunately, in Niger Delta regions of Nigeria, oil exploration and production has caused severe damaging environmental pollutions. These however, resulted to ethnic agitations, militancy and unrest among the locals (Uwasomba and Alumona, 2013). Thus making oil prospecting and oil lifting a more expensive and difficult task for companies in Nigeria. Hence, there is exigency to find alternative renewable forms of energy to meet the energy demand for Nigeria and its teeming population. Moreover, the country envisages an energy transition from crude oil to renewable energy by 2025 (Galadima *et al.*, 2011; Iwo *et al.*, 2016). Researches have been made to produce renewable biodiesel by using greener oil resources like the edible and non-edible oils (Shaaban and Petinrin, 2014; Hood, 2016). The renewable alternatives like biodiesel is simple to use, biodegradable, non-toxic, free of Sulphur and aromatics, and does not require a separate storage infrastructure. Thus this research work, will therefore investigate the proficiency of biodiesel production from rubber seeds (*Hevea brasiliensis*), Avocado Pear seeds (*Gratissima persea*), and Nipa Palm Kernel seeds (*Nypa frutican*) found in Nigeria.

2. MATERIALS AND METHODS

2.1. Materials

2.1.1. Sample Collection and Preparation

Avocado pear fruit (*Gratissima persea*) were purchased from oil mill market in Port Harcourt, Nigeria. The non-edible seed of avocado pear seed were crushed and dried at 110 °C to a constant weight to remove water content and then milled. Rubber seeds (*Hevea brasiliensis*) were collected from rubber research institute in Akwete, Abia State, Nigeria. They were dried at 40 °C, cracked and milled. Nipa palm kernel fruits (*Nypa frutican*) were collected from water side Ogoni along eastern-bypass Port Harcourt, Nigeria. The kernels were cracked and dried at 70 °C to constant weight and then milled. All plants were identified by Department of Crop Science Technology, Federal University of Technology Owerri, Nigeria. Their oils were extracted with 500 ml soxhlet extractor using 200 ml of n-hexane as a solvent at 60 °C for 2 hours (Alamu *et al.*, 2007).

2.2. Methods

2.2.1. Transesterification Procedures

Rubber seed oil (RSO) of 300 cm³ was measured into 1000 cm³ round bottom flask (reactor) and heated to 45 °C (10 mins) over water bath with automatic temperature regulator. Potassium hydroxide (KOH) was used as the catalyst and prepared in serial increment of 0.5 %. A weighed amount (0.5 g) of the catalyst (KOH) was dissolved in 180 mL of methanol and stirred for 10 minutes over hot plate to form potassium methoxide. The mixture was introduced gently into the heated oil in the reactor and continuously stirred to bring it to a temperature of 65 °C over 30 mins. This prevented vaporization of the methanol from the reaction mixture during the biodiesel production process. After 30 minutes, the mixture was removed from heat and carefully poured into a separating funnel. The separation was allowed overnight to build glycerol and biodiesel phases. After 24 hours two distinct layers were formed and separated. The lower layer containing the glycerol, large fractions of catalysts and impurities was recovered after phase separation. The upper layer (ester) containing the unpurified biodiesel was transferred into a beaker and heated at 90 °C for 10 more minutes to evaporate the excess of methanol. The aliquot was divided into two portions and each of them subjected to wet and dry purification processes (Fukuda *et al.*, 2001; Uzoekwe *et al.*, 2015). This procedure was repeated for Nipa palm kernel oil (NPKO) and Avocado peer oil (APO) respectively at 0.6, 0.7, 0.8, 0.9, 1.0, 1.1 and 1.2 increments of catalyst concentrations respectively. The catalyst concentration was obtained by dividing weight of KOH by volume of oil and multiplied by 100.

2.2.2. Wet Purification Process

Acid water was prepared by adding 2 % (v/v) phosphoric acid (H_3PO_4) to distilled water. The unpurified biodiesel was washed with 10 % acid water with a volume that was $\frac{1}{2}$ of the total volume of biodiesel produced in the reaction at 65 °C. This washing procedure was performed in the same transesterification reactor with constant stirring over 5 mins. Obtained mixture was transferred to a separation funnel and the waste water (bottom layer) and biodiesel phases were separated. The volume of the biodiesel formed was determined in a measuring cylinder and recorded. Biodiesel oil sample was stored for further analysis and sample characterization (Aliyu *et al.*, 2013; Uzoekwe *et al.*, 2015).

2.2.3. Dry Purification Process

The dry purification conditions were improved using previous method by Berrios and Skelton (2008). The unpurified biodiesel was heated in programed hot plate (5 mins) and stirred slowly until it reached 65 °C to remove water and volatile components from the oil. The final volumes were determined and obtained biodiesel sample stored for analysis and characterization.

2.3. Characterization of Samples

The physical and chemical properties of the RSO, APO and NPKO and their respective methyl esters were determined and compared with the standard fossil diesel and standard biodiesel.

2.3.1. Determination of Free Fatty Acid (FFA)

About 5 g of the oil was weighed into a flask of hot neutralized ethanol and 2 cm³ phenolphthalein indicators was added and titrated with 0.1 M sodium hydroxide (Canesin *et al.*, 2014).

2.3.2. Determination of Iodine Value

0.1 g of oil was weighed into 250 ml conical flask. 15 mL of carbon tetrachloride was added and stirred continuously with the addition of 25 mL of Wijs's reagent. The solution was stoppered and shaken. The mixture was allowed to cool for 30 minutes in the dark at room temperature of 28 °C. About 150 mL of distilled water and 20 mL of 10 % (w/v) of potassium iodine was added to the mixture and titrated with 0.1 M standard thiosulphate using 10 % (w/v) starch indicator (Canesin *et al.*, 2014).

2.3.3. Acid Value Determination

About 2g weight of oil was weighed into 250 ml conical flask and 25 mL of a mixture of diethylether and ethanol (1: 1 ratio) was added. Then 4 drops of phenolphthalein were added to the mixture and titrated with 0.1 N NaOH until a persistent color appeared (Canesin *et al.*, 2014).

2.3.4. Determination of Saponification Value

About 1 g of oil was weighed into 250 mL conical flask. Then 3 mL of fat solvent was added followed by 25 mL of ethanolic Potassium Hydroxide into the flask using a pipette. The content of the flask was stirred and refluxed for 30 mins at 65 °C to ensure complete saponification. Then the content was cooled and titrated with 0.5 M HCl using phenolphthalein indicator (AL-Harbawy and AL-Mallah, 2014).

2.3.5. Specific Gravity

An empty 5 mL specific gravity bottle was weighed out and the weight was recorded (y). It was filled with water and reweighed (x). Finally bottled was dried and filled with the oil sample and weighed (z). The specific gravity of the oil samples was calculated using Equation 1 shown below (Tat and Gerpen, 2000).

$$\text{Specific gravity} = \frac{\text{weight of oil}}{\text{weight of water}} = \frac{z-y}{x-y} \quad (1)$$

2.3.6. Determination of Relative Viscosity

A standard volume of the oil was transferred into the viscometer. The oil was then drawn into the upper reservoir bulb of the viscometer by suction. Efflux time (t_s): time required for the oil to fall from the two bulbs to the bend mark and emptying the bulbs was noted with a timer. The process was repeated using water and the time taken (t_o) for the water to flow down the bulb was also noted. The relative viscosity was then determined using the Equation 2 stated below:

$$\text{Relative viscosity} = \frac{t_s - t_o}{t_o} \quad (2)$$

2.3.7. Determination of Flash Point

The sample was poured into a metal container and heated at 5 °C interval with a flame being passed over the surface of the sample until a flash appeared on the surface of the sample (Tat and Gerpen, 2000).

2.3.8. Determination of Ester Content

Ester value of oil is the number of milligram required to saponify the ester contained in 1.0 g oil. The value is obtained as a difference between the saponification value and the acid value (Tat and Gerpen, 2000).

2.3.9. Cloud Point

50 mL sample kept at 28 °C was filtered using Lintless filter paper to remove moisture in sample. Filtered sample was poured into a test tube and stoppered firmly using a cork fitted with thermometer touching the bottom of the test-tube. The set-up was immersed in beaker containing 450 mL of methanol and stirred continuously. Dry ice was slowly added at every 2 °C/mins until that temperature when the sample showed distinct cloudiness at the bottom of test tube (Tat and Gerpen, 2000).

3. RESULTS AND DISCUSSIONS

3.1. Properties of Oil

Specific gravity at 25 °C: The specific gravity was 0.920 for RSO, 0.916 for APO, and 0.910 for NKPO. This indicated that the feedstock was less dense than water. Hence it is suggested that RSO had more OH molecules present and there was an indication of absence of heavy metal impurities.

Viscosity at 40 °C: NPKO had the highest viscosity of 73.5. While APO at 43.5 was slightly denser than RSO at 35.2. These values were of high viscosity when compared to groundnut oil, palm oil and coconut oil as shown by Satyanarayana and Muraleedharan (2011) may be due to unsaturation; hence NPKO is the most unsaturated oil. The lower viscosity of RSO and APO may be due to the impurities and contaminant present in the oil such as pigment, non-hydratable lecithin, oxidized fats and non-lipid materials (Moser, 2009; Kai *et al.*, 2010).

Acid value Mg/KOH/g: Low acid value (3.20 mg/KOH/g: NPKO and 5.14 mg/KOH/g: APO) is an indication of low free fatty acid while high acid value (40.3 mg/KOH/g: RSO) is an indication of high free fatty acid and also rancidity. This result agreed with the specific gravity trend.

Saponification value mg/KOH/g: APO gave the highest saponification value of 226 mg/KOH/g which may arise due to higher percentage of low molecular weight fatty acid. RSO had a value of 193 mg/KOH/g while NPKO at 56.7 mg/KOH/g was the lowest.

Iodine value g I₂/ 100g: Iodine value for RSO was 135 indicating the presence of free fatty acid. APO at 74.6 and 30.4 for NPKO as the lowest showed a lower degree of carbon to carbon unsaturation of oil. The iodine value also validates the results for acid value and specific gravity.

Peroxide value mEq/kg: It was determined as 12.6 for RSO which was the highest followed 3.50 for APO and 0.800 for NPKO which is the lowest. High peroxide value of RSO when compared to the other two is an indication of oxidation and rancidity and further authenticates iodine values.

Free fatty acid %: The percentage for free fatty acid of RSO was 27.6 which was an indication of excess free fatty acid. 0.526 was obtained for APO and 0.478 for NPKO which is the lowest.

Flash value °C: APO has the highest flash point of 245 °C followed by RSO at 216 °C and 170 °C for NPKO which is the lowest. Hence, APO is least flammable than RSO and NPKO oils. This is because low flash points usually mean increased flammability.

Ester Content: The ester content of APO was 220, while RSO was 152 and NPKO had 53.58.

pH: The pH for RSO oil was determined as 6.0 for RSO, 6.2 for APO and 6.1 for NPKO. This is an indication of the presences of free fatty acid in the oil.

Colour: RSO showed yellow coloration while NPKO was light yellow. APO only had dark brown.

Percentage yield of oil (%): The Table 1 below shows that oil yield decreases in each successive extraction. RSO volume was highest at 663 ml > NPKO at 254 ml > APO at 148 ml; hence RSO has a high prolific rate for oil production while NPKO showed the least yield variation. The % yield of oil was obtained by dividing weight of oil by the weight of sample and multiplied by 100.

Table-1. Yield of extracted oil from non-edible Nigerian feedstocks.

Samples	First extraction(ml)	Second extraction(ml)	Third extraction(ml)	Mean S.V of yield	% yield
RSO	663	656	646	655 ±7.0	52.4%
AP0	148	141	143	144 ±2.9	11.52%
NPKO	254	234	248	247 ±6.6	19.73%

Source: This research work.

3.2. Properties of Biodiesel

Percentage yield of biodiesel (%): From Figure 1 below, the production of biodiesel was observed to steadily increase in volume from 0.5 % w/v concentration of the catalyst (KOH) until it peaked at 0.9 % for RSO with maximum biodiesel yield of 88 % and peaked at 1.0 % for APO and NPKO with maximum biodiesel yield of 92% and 96% respectively. Hence an indication that increase in catalyst concentration after 0.9 % for RSO and 1.0 % for APO and NPKO would not yield further increase in the volume of biodiesel produced. From the figure above, it gives a clear comparison of the effect of catalyst concentration in RSO, APO and NPKO.

This is explained from the view point that maximum yield of biodiesel 94 – 99 % occur at concentration range of 0.5 - 1 w/v % as were observed by Refaat *et al.* (2008). They result is also explained from by the reversible nature of trans-esterification process after certain concentration of catalyst is reached (Ugheoke *et al.*, 2007). Thus, catalyst concentration level greater than 1 may have favored the backward reaction and the formation of glycerin. Hence, the yield fraction at concentration 1.0 is lower than the concentration level of 0.5 w/v % for RSO while 1.1 is lower than concentration level of 0.5 w/v % for APO and NPKO. The biodiesel % yield was determined by dividing the volume of biodiesel by the volume of reactant oil and then multiplied by 100.

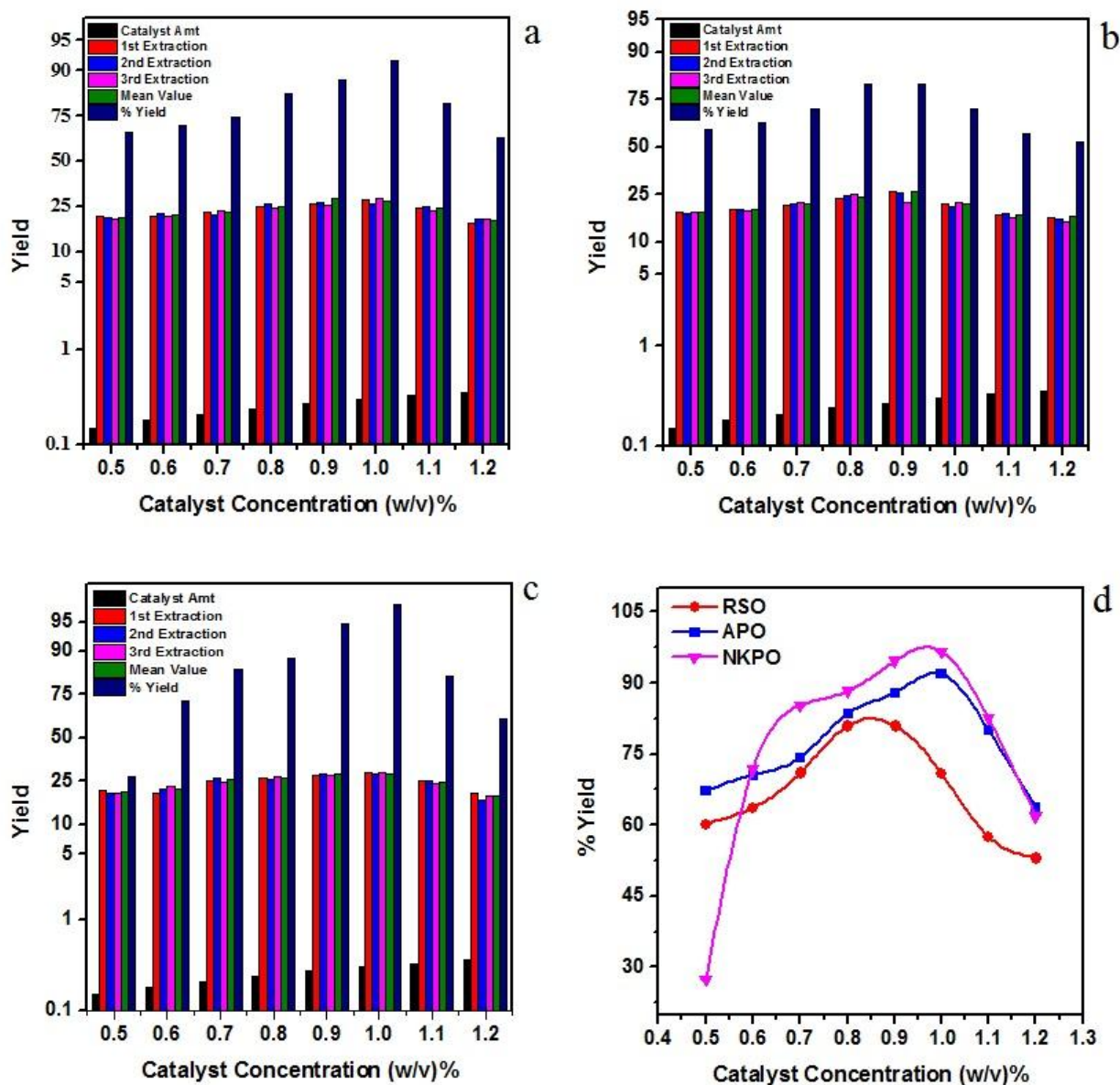


Figure-1. Biodiesel % yield of oils: (a) APO; (b) RSO; (c) NKPO and (d) comparative biodiesel % yield.

Source: This research work.

Biodiesel pH: The pH for RSO, APO and NKPO were determined as 6, 8.4 and 8.6. Hence, only RSO was slightly acidic while NKPO and APO were slightly alkaline. On the other hand, the standards pH for Petroleum diesel ASTM 00975 and biodiesel ASTM D06751 is 7, while Biodiesel EN 1424 has a pH of 6 which was similarly recorded in RSO.

Biodiesel acid value (mg/KOH/g): The acid value for APO determined as 0.500 while NKPO was 0.370 and then 0.340 for RSO which was the lowest. The acid value obtained for APO, NKPO and RSO compares favorably with petroleum diesel at 0.350 while standard biodiesel for both ASTM D664 (< 0.80) and EN14111 (< 0.50). Acid value indicates the degree of fuel ageing during storage as it increases gradually due to degradation of biodiesel causing formation of deposits in automobile engines. More so, free fatty acid such as weak carboxylic acid poses far lower risk than strong mineral acids (Refaat, 2007). High acid value causes corrosion of oil tank and other relevant component; hence, APO is more likely to cause tank corrosion and deposition (Atabani et al., 2012).

Biodiesel specific gravity Kg/M³: The specific gravity of biodiesel obtained from RSO, APO and NKPO were 0.886, 0.878 and 0.850 respectively. RSO and APO had values which falls within the limit for standard diesel ASTM D6751 (0.870 – 0.900) and above biodiesel EN ISO 3675 (0.860). NKPO value falls within the limit of petroleum

diesel (0.810 – 0.860) and standard biodiesel ASTM D6751 and EN ISO3675 (0.860). The specific gravity obtained for RSO, APO and NPKO were within similar specified international standards such as ONC1191 (Austria) 0.8850 – 0.890, CSN656507 (Czech Republic) 0.87 – 0.89. Journal official (France) 0.870 – 0.900, DIN 51606 (Germany) 0.875 – 0.900, UN110635 (Italy) 0.860 – 0.900, SS1554436 (Sweden) 0.870 – 0.90 respectively for biodiesel fuel (Sokoto *et al.*, 2011).

Biodiesel viscosity at 40 °C mm²/S: It is the measurement of the internal flow resistance of a liquid and constitutes an intrinsic property of vegetable oil. The biodiesel produced from RSO were determined to have the highest viscosity 6.89 while APO was 4.24 and NPKO 3.12. The RSO have higher viscosity than conventional petroleum diesel (1.9 – 4.1), standard biodiesel ASTM D445 (1.9 – 6.0) and EN ISO 3104 (3.5 – 5.0). The biodiesel produced from NPKO falls within the limit of standard biodiesel D445 and petroleum diesel D975. APO falls below the limit for biodiesel EN ISO 3104 and within the limit for biodiesel ASTM D445 and above the limit for petroleum diesel ASTM D975 (Knothe, 2005). This implies that all satisfies the fluidity requirement as an alternative biodiesel and will not compromise the mechanical integrity of injection pump drive system. It is of remarkable influence in the mechanism of atomization of the fuel spray.

Biodiesel flash Point °C: Flash point is an important parameter for assessing hazards during fuel transport and storage. The flash point for biodiesel obtained was determined as 226, 194, and 156 for RSO, APO and NPKO respectively. Biodiesel from RSO, APO and NPKO have high flash point compared to standard biodiesel EN ISO 3679 (S120), ASTM D93 (K130) and petroleum diesel ASTM D6751 (60 – 80). However, biodiesel is considerably higher than the prescribed limits, but can decrease rapidly with increasing amount of alcohol (Mittelbach, 1996).

Biodiesel cloud Point °C: The cloud point for RSO, APO and NPKO were determined as 6, 8 and 10. This was compared to the standard biodiesel ASTM D 6751 (- 3 to 12) and with petroleum diesel ASTM D 975 (- 15 to 5). The result was found to be within the limit of standard biodiesel and above the value for petroleum diesel. This agrees perfectly with the general report that biodiesel has higher cloud point than petroleum diesel. The cloud point is another key parameter for low temperature operation of a fuel. It is the temperature at which solidification of heavier component of RSO, APO and NPKO biodiesel resulting in a cloud of crystals within the body of the biodiesel (Alamu *et al.*, 2007).

4. CONCLUSION

Rubber seed gave the highest yield of oil (52.4 %) while avocado pear had the lowest yield. Rubber seed has high oil content but contain cyanogenic glycoside which releases hydrogen cyanide in the presence of enzymes, thus making it a very good reserve for the production of non-edible product e.g. biodiesel. In addition, the physical and chemical parameter of the oil analyzed, showed that rubber seed oil, avocado pear oil and nipa palm kernel oil can be used as an alternative feedstock for biodiesel production. As they compared favorably with biodiesel oil production. Rubber seed oil gave a maximum biodiesel yield at 0.9 w/v % catalyst concentration while Avocado pear oil and Nipa palm kernel oil gave their maximum biodiesel yield at 1.0% w/v at constant mole ratio, time and temperature. Finally, the physical and chemical parameter of the biodiesel analyzed, revealed that all the biodiesel produced from the feedstock RSO, APO, NPKO compared favorably with the conventional petroleum diesel ASTM D 975, and standard biodiesel ASTM D975, and standard biodiesel ASTM 6751 and EN14214.

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