

# Effect Of Process Variables On Biodiesel Production Via Transesterification Of Quassia Undulata Seed Oil, Using Homogeneous Catalyst

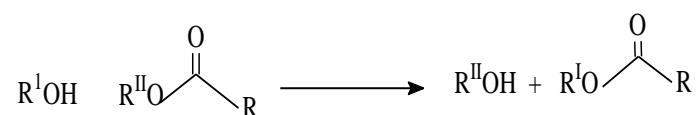
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**Abstract:** This paper studied the extraction of oil from *Quassia undulata* plant using *n*-hexane as solvent. The biodiesel produced was characterized using spectroscopic methods. Important parameters of biodiesel such as methyl ester content, viscosity, specific gravity, acid value, flash point and cetane number were determined. The influence of reactions variables such as methanol to oil molar ratio, reaction temperatures and reaction times on conversion of *Quassia* oil was also measured. The results showed that the maximum conversion of 98.6% was recorded at 60 °C when the methanol to oil molar ratio was 9:1 at time 2 hours, 30 minutes, using fixed catalyst amount of 2.5 wt% ( based on the wt. of the oil).

**Key Words:** *Quassia undulata* oil, biodiesel, reaction variables, parameters, conversion.

## INTRODUCTION

Biodiesel is a fuel produced from vegetable oil, animal fat and recycled oil from food industry.[31] and is known chemically as a 'fatty acid methyl ester. The short chain alcohol is either methanol or ethanol. The most preferable is methanol. Biodiesel is a renewable fuel which consist of fatty acid methyl esters derived through transesterification of vegetable oils, animal fat with methanol. It is recognized as green fuel. It is safe, non-toxic and biodegradable compared to petroleum diesel. It is oxygenated and free from sulphur and aromatics [31], [ 3]. United States and Europe are the world leaders in biodiesel production. The United States produces Biodiesel from soybean, while Europe depends on rapeseed [4], [14]. Transesterification is defined as the process of exchanging the organic group R<sup>11</sup> with the organic group R<sup>1</sup> of an alcohol . This `reaction is often catalyzed by the addition of an acid or base.



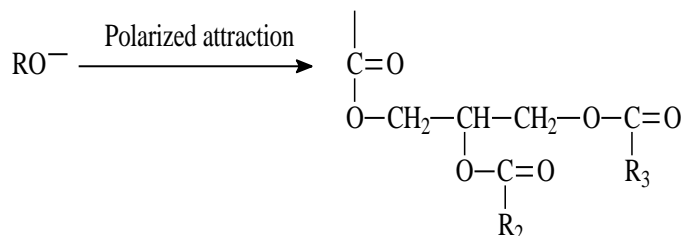
Where R<sup>1</sup> and R<sup>11</sup> are alkyl groups.

The chemical reaction that occurs through this process breaks down the oil molecules replacing the glycerin portion of the molecule with an alcohol molecule. The glycerin falls to the bottom and is drained off leaving behind the crude biodiesel.

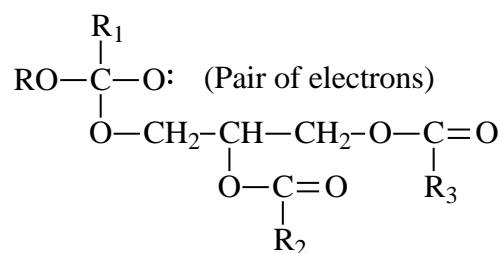
Transesterification is a reversible reaction but in biodiesel production, the back reaction does not occur because glycerol formed is not miscible with the product, giving a two phase system. The stoichiometry of reaction is a 3:1 mol ratio of alcohol to oil to produce 3 mol of biodiesel and 1 mol of glycerol [10], [12]. The presence of a catalyst accelerates the conversion triglycerides to biodiesel [21]. Biodiesel is a renewable, biodegradable, environmentally benign, energy efficient and non detrimental to engine's operational performance [29]. sAs an alternative fuel, biodiesel is to act as complementary supply of energy to reduce total dependence on petroleum gasoline. But when compared with the cost of diesel obtained from petroleum, the high cost is the main drawback in biodiesel production. However, this high cost could be lowered by improving the production process [7]. In this regard oil from the seeds of *quassia* plant, an inedible tropical plant oil has significant potential for biodiesel production was used. *Hannua* plant (*quassia*) belongs to Simaroneaceae family, known as Umopula in Igala language and is widely distributed with many of the plants found around Anyigba area of Kogi State, Nigeria. The tree is native to the Carribean, particularly Jamaica, North Venezuela and Brazil. Knowledge of the properties of the tree were learned from the people in Surinam by a Swede named Daniel Rolander, who brought the knowledge in turn with him back to Europe in 1755. The wood has no smell but has a bitter taste. The main compound which gives the bitter taste is called "quassin." The tree grows up to 100 feet(300 metres) tall, with a trunk up to 3 feet (1 metre) wide. It blossoms with green flowers in October and November [5]. The fruits usually persist till the first rains. In the conversion process triglyceride ester are turned into alkyl esters (biodiesel) using a catalyst (lye) and an alcohol reagent e.g. methanol which yields methyl ester. The methanol replaces the glycerin. The glycerin, the heavier phase, sunk to the bottom. Methyl ester (biodiesel)- the lighter phase- floats on top and can be separated by decantation or centrifuging. This conversion process is called *trans*-esterification. The conventional transesterification reaction in batch processing tends to be slow and phase separation of the glycerin is time consuming, often taking 5 hours or more [24]. The *trans*-esterification reaction which is base catalyzed is capable of deprotonating the alcohol when a strong base is used. Examples, using strong alkali like sodium hydroxide

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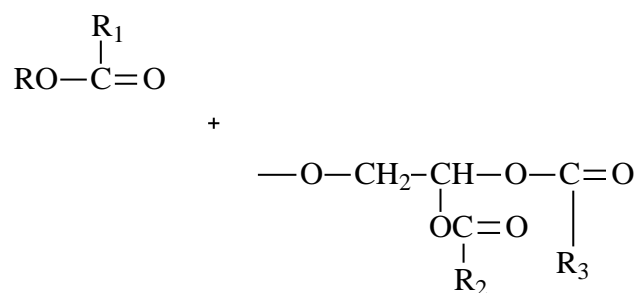
or potassium hydroxide or alkoxides. The base KOH or NaOH is dissolved in the alcohol to make a convenient method of disposing the solid catalyst in the oil. The alcohol needs to be very dry because any water present promotes the saponification reaction, thereby producing salts of fatty acids (soaps) and consenting the base and inhibiting the transesterification reaction. The carbon on the ester of the triglyceride has a slight positive charge and the carbonyl oxygen have a slight negative charge. This polarization of the C=O bond is what attract the RO<sup>-</sup> to the reaction site.



This yields a tetrahedral intermediate that has a negative charge on the former carbonyl oxygen.



These electrons then fall back to the carbon and push off the diacylglycerol forming the ester.



Two more RO groups react via this mechanism at the other C=O groups. Considering limiting factors, RO<sup>-</sup> has to fit in the space where there is a positive charge on the C=O. Methanol works well because it is small in size. Increase in chain length of RO<sup>-</sup> group, increases reaction rate. This causes steric hindrance [30]. This is the primary reason why methanol and ethanol are preferable in the reaction. There are a number of factors or variables which could affect the transesterification process. These include moisture content, reaction temperature, free fatty acid content, reaction time, oil to alcohol molar ratio, mixing intensity, type and amount of catalyst and co-solvent [6], [13], [21], [27]. These variables usually have different effect on the transesterification process. One of the most important biodiesel fuel physical properties is viscosity. The

acceptable kinetic viscosity range at 40°C is 1.9-6.0 mm<sup>2</sup>/s as required by ASTM D6751 specification. The kinetic viscosity of fatty compounds (such as those found in biodiesel fuel) is influence by structure of the compound, chain length, the position, number and nature of double bonds and the nature of oxygenated moieties [11], [15], [24]. High concentration of monoglycerides and diglycerides in biodiesel not only affect the fuel quality, but also indicate incomplete reaction during production. The levels of these compounds were determined using advance methods such as infrared spectrometry (FT-1R), Rama spectra, NMR, gel permeation chromatography (GPC), HPLC, and gas chromatography-mass spectrometry (GC-MS) [9], [19]. In this work, biodiesel was successfully produced by transesterification of *quassia* oil using homogeneous basic catalyst-potassium hydroxide dissolved in methanol. The advantages of homogeneous transesterification was also studied. This paper tried to avoid the problems associated with heterogeneous transesterification which is the formation of three phases with alcohol and oil, thus lowering the rate of reaction [18]. We also investigate the influence of operating parameters (reaction period, reaction time, ratio of oil to methanol and amount of catalyst) on conversion of *quassia* oil to biodiesel and the feasibility of a non-edible oil (*quassia* oil) as alternative feedstock in biodiesel production.

## MATERIALS AND METHODS

All chemicals were obtained from different commercial sources (Pitex, Altran, Aldrich) and were used without further purification. The Infrared (IR) spectra were recorded in cm<sup>-1</sup> on a Bulk scientific 500 spectrophotometer. UV spectra were (max in nm) recorded in spectroscopic grade meoH on a Beckman DU 640 apparatus.

**Sample Collection:** A literature procedure was adopted [10]. The fruits of *Quassia undulata (umopula)* were collected from the farm lands in Anyigba, Nigeria in clear polyethylene bags in the months of December/January and taken to the laboratory and clearly labeled. The sample was air dried on the laboratory bench. Voucher specimen was authenticated by Abdulkadir Fatima of Microbiology laboratory, Kogi State University. Herbarium specimens were deposited with the same laboratory. The dried fruits were reduced to coarse powder and the oil extracted by solvent extraction

**Extraction and Purification of oil:** 50g of the powdered sample was placed in the thimble of the extractor and 250 cm<sup>3</sup> of n-hexane was added. The extraction lasted 3hrs. The n-hexane was distilled off using rotary evaporator, while the oil was in the flask.

**Purification of the oil:** The oil was centrifuged using a 12 bucket centrifuge machine and was further degummed to remove any phospholipids compound.

## Analysis of Parameters in Oil Sample:

### Acid number determination

1g of oil was weighed into 100ml of neutral solvent in a 250ml conical flask. The solution was titrated using 0.01M

KOH and 4 drops of phenolphthalein was added until a persistent colour appeared. [25].

#### Determination of iodine value

0.1g of the oil was dissolved in 15ml of carbon tetrachloride by stirring. The solution was mixed with 25ml of Wij's solution. The flask was allowed to stand for 30 minutes in the dark at room temperature. 100 ml of distilled water and 20 ml of 10 % (w/v) of potassium iodide was added to the mixture. It was titrated with 0.1 ml standard thiosulphate using 10% (w/v) starch indicator.

Blank titration was done in the same way.

#### Saponification value

10 g of oil was weighed into 250 ml conical flask. 25 ml of alcoholic hydroxide solution was equally added into the flask using a pipette. The content of the flask was stirred and connected to a reflux condenser and allowed to boil for an hour to ensure complete saponification. The content was allowed to cool and was titrated with 0.5 M HCL using phenolphthalein indicator. A blank titration was carried out in the same fashion.

#### Refractive index determination

Abbe's refractometer was used for the measurement of refractive index of the crude oil. The oil was smeared on the lower position. After some adjustment, the refractive index was read off directly at room temperature (25 °C).

#### Flash point determination

The oil was poured into a metal container and heated at 5 °C interval with a flame being passed over the surface of the sample. A temperature reading was taken immediately a flash appeared on the surface of the sample.

#### Specific gravity determination

An empty 5 ml specific gravity bottle was weighed out and the weight was recorded. It was filled with water and reweighed. The water was poured off, and the bottle weighed again.

**Viscosity determination:** The viscosity of the oil was determined using a universal torsion viscometer. The oil was inserted into a vessel on the sample table so that the cylinder was centrally immersed from 0-360. The fly wheel was rotated from rest. The viscosity in centipoises, at a given speed cylinder combination was obtained by multiplying the instrument readings with appropriate multiplying factor given on the calibration chart.

#### Peroxide value

1.0 g of the oil was weighed into dry test tube, followed by addition of 1.0 g of powdered potassium iodide and 20 ml of a solvent mixture of glacial acetic acid and chloroform (2:1). [25]. This was placed in a boiling water bath so that the content are boiled for 30 seconds. The contents were quickly poured into a flask containing 20 ml of 5 % potassium iodide. The tube was washed twice with 25 ml of distilled water and poured into the flask. It was titrated with 0.002 M sodium thiosulphate using starch indicator. The blank titration was done also.

#### Determination of free fatty acid (FFA)

Free fatty acid is the acid value expressed in percentage. 5 g of the oil was weighed into a flask of hot neutralized ethanol and 2 cm<sup>3</sup> phenolphthalein indicator was added and titrated with 0.1 M sodium hydroxide, [25].

#### Determination of ester value

Ester value of oil is the number of milligrams 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.

#### Determination of moisture content

The loss in mass of a substance when heated at 105 °C for about 4 hours is known as the moisture content of the substance. 10g of the oil sample was weighed into an empty crucible (W<sub>1</sub>). The crucible and oil sample was weighed (W<sub>2</sub>). The crucible was placed in an oven at 105 °C for 5 hours. The crucible and content was removed after 5 hours, cooled in a desiccator to room temperature and reweighed. The process of heating and cooling was repeated until a constant weight (W<sub>3</sub>) was obtained.

**Determination of ash content** An empty porcelain crucible was weighed empty (W<sub>1</sub>), 2.0 g of the oil was weighed and poured into the crucible and the crucible reweighed (W<sub>2</sub>). The crucible was later burnt over a bursen burner to remove all the solvent present. It was taken to the muffle furnace for ashing at 55 °C for two hours. The crucible was removed and placed in a desiccator and allowed to cool. It was reweighed (W<sub>3</sub>).

#### Production of Biodiesel (Methyl ester):

The *quassia* oil was stored in a cold and dark room at 5 °C under argon to prevent oxidation. [4]. 5.6 g (0.1M) of 90% analar potassium hydroxide (Mw, 56.1 g) was poured into 200 cm<sup>3</sup> of methanol (90% pure) contained in a two necked quick fit reagent bottle. The mixture was swirled for 6 to 10 times to give proper mixture of potassium methoxide to examine the effect of catalyst amount. 400 cm<sup>3</sup> of the *quassia* oil was preheated to 55 °C and poured into the mini processor (1.0 litre reagent bottle). 80 cm<sup>3</sup> of the prepared methoxide was added gradually unto the oil in the mini processor and covered properly with the lid. This was placed on a magnetic stirrer hot plate and heated between 55-65 °C and allowed to mix for 1-4 hours. The content was poured into a 1.0 litre sample bottle and allowed to cool for 24 hours with the lid of the bottle tightened. The mixture was separated into two layers. The bottom was glycerol and was drained off using a separatory funnel. The upper ester was washed with hot water and dried over anhydrous sodium sulphate. The methyl ester was allowed to cool.

#### Analysis of Parameters in the Biodiesel:

##### Viscosity determination:

The viscosity of the biodiesel was determined using VM/A cylinder combination viscometer. The viscometer in poise at a given speed cylinder combination was obtained by appropriate Multiplying factor given on the calibration chart and viscosity in centistokes was obtained.

### Flash point determination

The flash point of the biodiesel was determined by pouring the methyl ester into a metal container and heated at 5 °C. At interval the flame was passed over the surface. Immediately a flash appeared on the surface of the sample, a reading of the thermometer was taken.

### Acid value determination

5 g of the biodiesel was weighed into 100 ml of the neutral solvent in a 250 ml conical flask. The solution was titrated with 0.1M KOH with addition of 4 drops of phenolphthalein indicator until a persistent pink colour appeared.

### Cetane number determination

The fatty acid methyl ester composition of the produced biodiesel was used to estimate the cetane number using correlations developed by Bamgboye and Hansen (2008). The obtained cetane number was then compared to cetane numbers obtained from ASTM Standard D613.

### Peroxide value determination

1.0 g of the biodiesel was weighed into a dry test tube, followed by the addition of 1.0 g of powdered potassium iodide and 20 ml of solvent mixture of glacial acetic acid and chloroform (2:1). The tube was heated in a water bath for 30 seconds. The content is boiled for 30 seconds, and the content was quickly poured into a flask containing 20 ml of 5% potassium iodide. The tube was washed twice with 25cm of distilled water and poured into a flask. It was titrated with 0.001M sodium thiosulphate using starch as indicator. The blank titration was done also.

**Determination of free fatty acid (FFA)** Free fatty acid is the acid value expressed in percentage. 5 g of the biodiesel was weighed into a flask of hot neutralized ethanol and 2 cm<sup>3</sup> phenolphthalein indicator was added. This was titrated with 0.1 M sodium hydroxide solution.

### Iodine value determination

0.1 g of the biodiesel was dissolved in 10 cm of chloroform contained in 500 ml stoppered conical flask and mixed with 25 cm<sup>3</sup> of Hannus iodine solution. The flask was kept in the dark for 30 minutes with occasional shaking. After 30 minutes, 10 cm<sup>3</sup> of 15% potassium iodide and 100 cm<sup>3</sup> of distilled water were added into the flask. The content of the flask was titrated with 0.1cm<sup>3</sup> Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution to a faint yellow colouration. 2 cm<sup>3</sup> of starch indicator was added and the titration was continued until the violet colour disappeared. Also, the blank titration was done using another conical flask that does not contain the oil sample. The average titre values of the sample titration and blank titration were obtained.

### Analysis of parameters of petroleum diesel (Automotive Gas Oil -AGO):

The following parameters of petroleum diesel were analyzed in order to compare them with those of biodiesel produced.

**Viscosity:** The viscosity is poise at a given speed and cylinder combination VM/A was obtained by multiplying the instrument reading by a multiplying factor given in the calibration chart.

**Flash point:** The flash of the diesel was determined by the use of ASTM D93 test method of flash point.

**Specific gravity determination, Cetane number, Peroxide value and Iodine value** were determined as in biodiesel.

## RESULTS AND DISCUSSION

The results of the analysis of parameters in the oil of *Quassia undulate*, biodiesel produced and petroleum diesel are presented in fig 1. The yield and percentage yield of the oil sample are 1281 g and 64.05% respectively, while the maximum percent yield of biodiesel is 98.6%. The Infrared (IR) spectra of the oil and biodiesel are shown in tables 1 & 2. It is observed that the IR spectra of *Quassia* oil and biodiesel are very similar. This is probably due to high chemical similarities that exist among triglycerides and methyl ester. Despite the existing chemical similarities, small difference was noticed in the following regions such as C=O ester, CH<sub>3</sub>, and C-O ester. This is attributed to the fact that biodiesel had a different compound bonded, compared to the *quassia* oil. This is evidenced by the strong ester peaks at 1750-1725 cm<sup>-1</sup> for C=O ester and 1150-1050 cm<sup>-1</sup> for C-O ester. Also, is the characteristic peak for CH<sub>3</sub> group at 1389-1370 cm<sup>-1</sup> [28], [31].

**Effect of methanol to oil molar ratio:** In transesterification process, excess amount of catalyst produces excess soap over time and gives lower yield. [4]. For this reason, a fixed amount of catalyst was used, while the methanol to oil molar ratio was varied from 5:1 to 10:1. Reaction temperature was 60 °C and reaction time 2 hour, 30 minutes. The results are given in Fig. 2.

**Effect of reaction temperature:** The effect of reaction temperature on the conversion of quassia oil to biodiesel was also studied. A fixed amount of catalyst (2.5 wt% ) was employed, while the methanol to oil molar ratio was 9:1 and reaction time maintained at 2 hours 30 minutes. The investigation was carried out with three different temperatures. The results are shown in Fig. 3.

**Effect of reaction time:** The effect the reaction time on quassia oil conversion was investigated. The reaction time was varied between 1-4 hours, while maintaining the methanol to oil molar ratio at 9:1 and the reaction temperature at 60 °C. Fixed amount of catalyst (2.5 wt% ) was employed. The percentage yield of *quassia undulata* oil and biodiesel obtain from the result are 69.05% and 98.6% respectively. The oil yield percentage was in agreement with 60-80% reported by Belewa and co-workers [2] indicating extraction with n-hexane gave a higher yield. The effect of methanol to oil molar ratio on conversion of oil to biodiesel is indicated in fig. 2. The results showed that percent conversion increased rapidly with the increase in the methanol to oil ratio. The maximum conversion was obtained when the methanol to oil molar ratio was 9:1. This shows that to increase the conversion, an excess methanol was required only to a certain period, but no effect at the later stage of the reaction. From fig. 3., it can be seen that the percent conversion of quassia oil to biodiesel increased as the reaction temperature increases. The maximum



conversion of 98.6% was recorded at 60 °C. At or above the boiling point of methanol (65 °C), the conversion rate decreased. This is probably due to the fact that methanol boils at this temperature which probably led to evaporation and subsequent loss of the solvent, thus lowered the conversion of oil to biodiesel. Fig. 4. Showed the interference of reaction time on conversion. It can be seen that the conversion of quassia oil to biodiesel increased steadily in the first 2<sup>1/2</sup> hours. The maximum conversion of 98.6% was achieved at 2<sup>1/2</sup> hours of reaction. The conversion there after underwent a decrease. The specific gravity of the biodiesel produced (0.816) was in agreement with 0.88 obtained by Belewa [2] and that of fossil oil (0.8667) as indicated in fig. 1. Specific gravity is an important parameter of biodiesel quality. According to [6], fuel injection equipment operates on a volume metering system, therefore a higher density for biodiesel results in the delivery of a slightly greater mass of fuel. Flash point is a measure of the flammability of fuels and an important parameter for assessing hazards during fuel transport and storage [26]. From fig.1., the flash point of the biodiesel 35°C using ASTM D92 method was lower than the specific standard. The flash point of petroleum diesel was also lower using the same method. The open cup was then employed. This gave 122 °C for biodiesel. This is in close conformity with 130°C giving by Belewa and co-workers [2], 170°C by China/US and EU/130°C. The cetane number of the biodiesel (51 units) is in agreement with the international standard of China (49), EU (50) and US (47). The method ASTM 976 was used for both the biodiesel and fossil fuel. Cetane number is a dimensionless indicator of the ignition quality of a biodiesel. High number of cetane leads to incomplete combustion and smoke, and low cetane number gives engine roughness, misfiring, higher air temperatures and also incomplete combustion [26]. The viscosity of the biodiesel is 1.941Cst (fig1). This value is in agreement with the international standard of China (1.9-

6.0); Us 1.9-6.0 and EU 3.5-5.0. The viscosity of biodiesel is higher than that of fossil fuel because biodiesel have lubricating effect which reduces wears and tears of the engine. Viscosity also affects the operations of fuel injection equipment. The refractive index of the biodiesel (1.440) agrees with 1.33-1.5 units as depicted by ASTM D1500. Refractive index and refractive dispersion can be used in conjunction with other properties to characterize fuels. Also from fig. 1, the acid value of the biodiesel is 0.32mg KOH compared with China standard (0.8), US (0.5) and EU (0.5). This value closely agreed with the international standards for biodiesel. Acid value is a good parameter for biodiesel test. Biodiesel with high acid value can cause corrosion of oil tank and other relevant component. It is a measure of mineral acids free fatty acids contained in a fuel sample.

## CONCLUSION

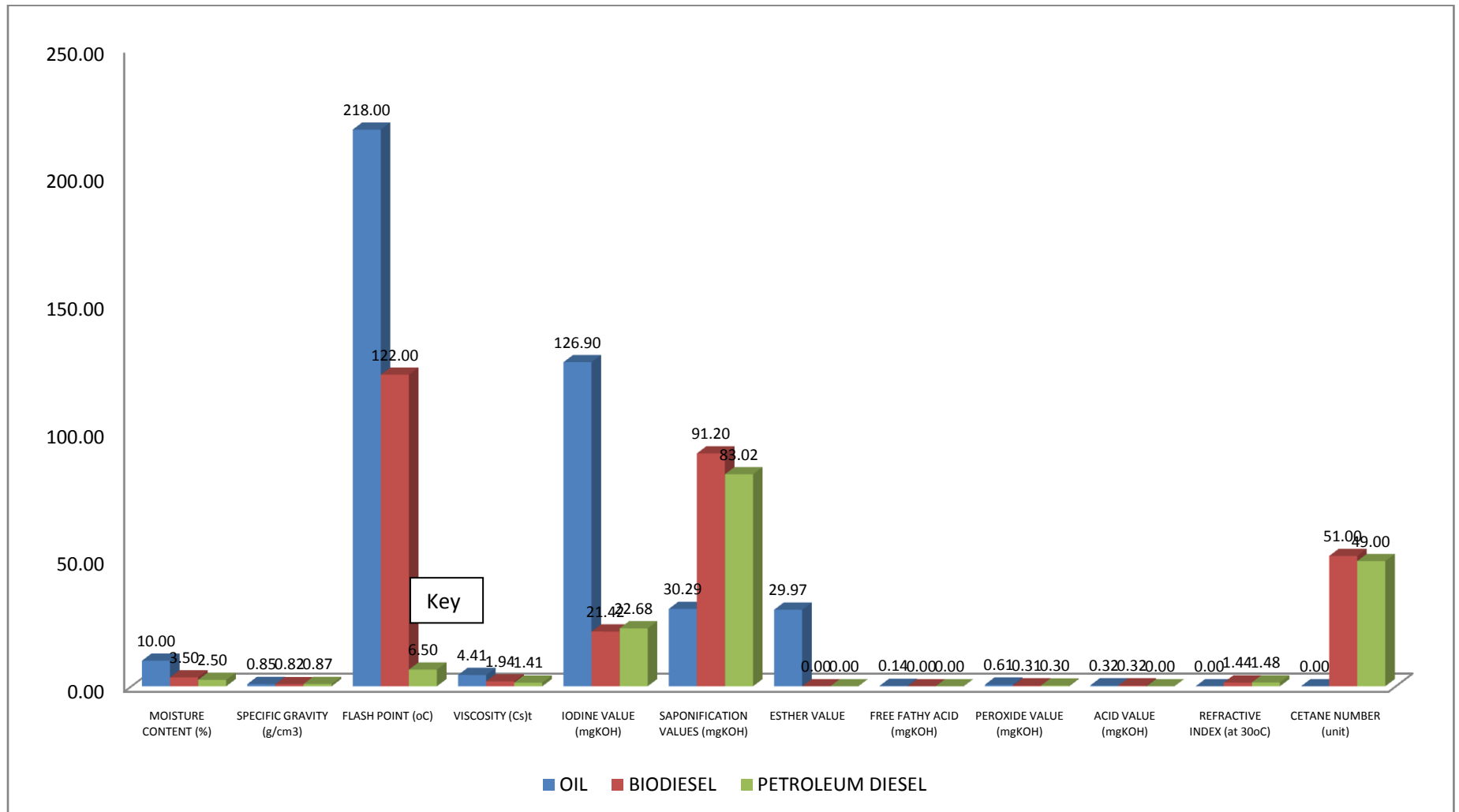
The Quassia plant possessed high percentage (64.05%) of oil as compared to International Standards and can be an alternative feed stock for biodiesel production which will reduce the application of food crops for renewable fuel production. The viscosity of the oil compared favourably with the viscosities of most common vegetable oils. The iodine value of the oil (126.90) agreed with that of other vegetables oils like groundnut oil (88-100): cotton seed oil (103-106); soybean oil (125-140); sunflower (130-150); safflower (130-150). Based on results of the analyzed parameters, the following is concluded on the biodiesel produced: \*The maximum conversion was obtained when the methanol to oil molar ratio was 9:1. \*The percent conversion of oil to biodiesel increased as the reaction temperature was increased. 98.6% conversion was recorded at 60 °C. \*The 98.6% conversion was achieved at the 2<sup>1/2</sup> hour of reaction. \*A 2.5 wt% catalyst was sufficient to complete the transesterification of quassia oil to biodiesel.

**Table 1.** Infra red (IR) spectra of *quassia* oil

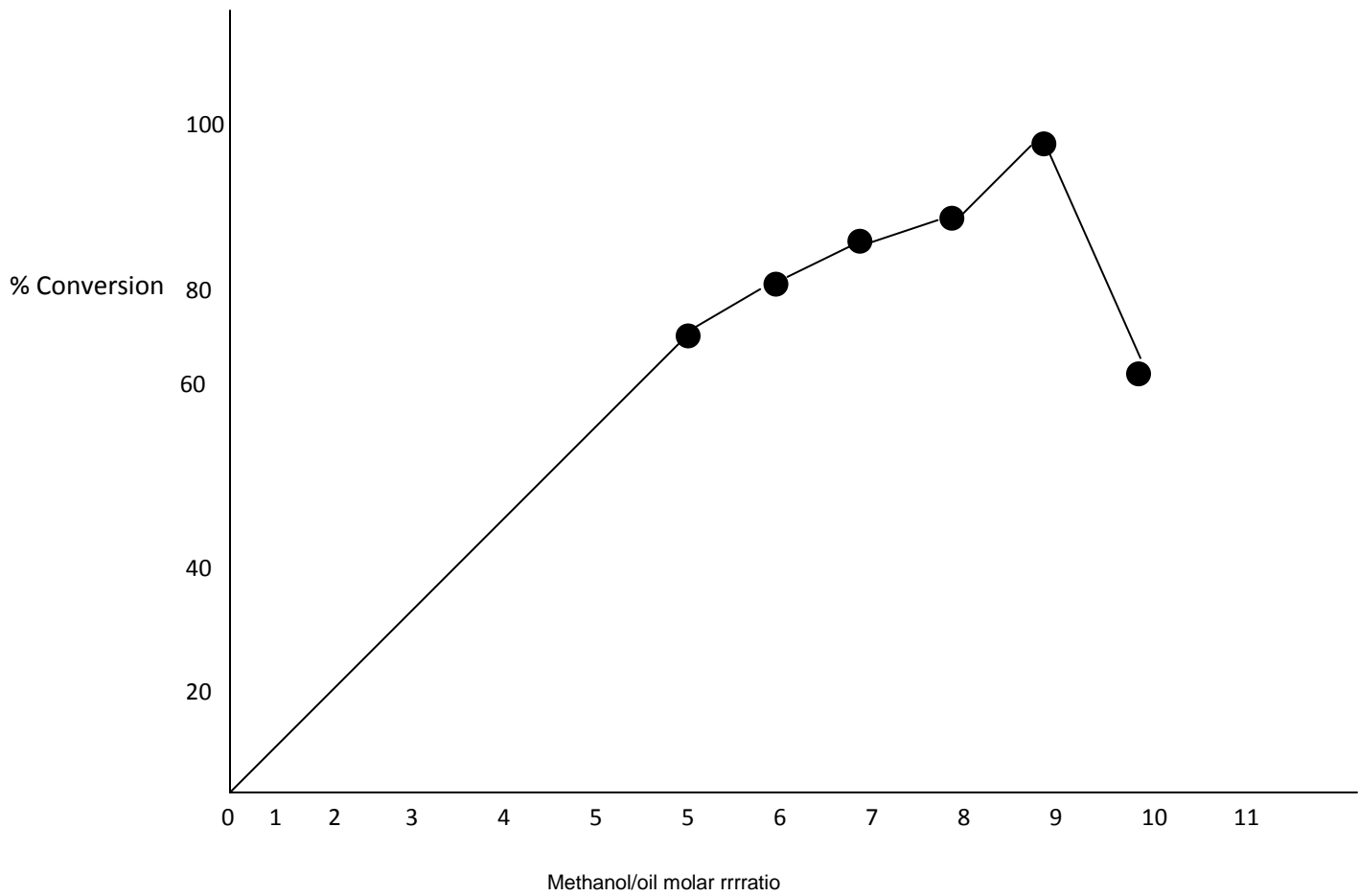
Frequency cm <sup>-1</sup>	band	Compound
3000-2850	C-H stretch of C-H	Alkyl groups CH <sub>3</sub> CH <sub>2</sub> CH
1745-1725	C=O stretch	Ester RC=O
1050-1000	C-O stretch	Alcohols RCH <sub>2</sub> OH R-C
3600S	O-H stretch	Alcohols, ROH <sub>2</sub> phenols ArOH
3400-3200	O-H stretch (H-bonded)	Alcohols ROH <sub>1</sub> phenols ArOH. R=alkyl

**Table 2:** Infra red (IR) spectra of quassia-biodiesel

Frequency cm <sup>-1</sup>	band	Compound
3600	O-H stretch	Alcohols ROH Phenols ArOH
3000-2850	C-H stretch of C-H	Alkyl groups CH <sub>3</sub> CH <sub>2</sub> , CH
1750-1725	C=O stretch	Esters RC=O
1465-1415	C-H bend	R=alkyl. Alkyl groups
1389-1370	C-H bend	Methyl (alkyl)
1150-1050	C=O stretch	Alcohols, RCH <sub>2</sub> OH, R <sub>2</sub> COH, R <sub>3</sub> COH

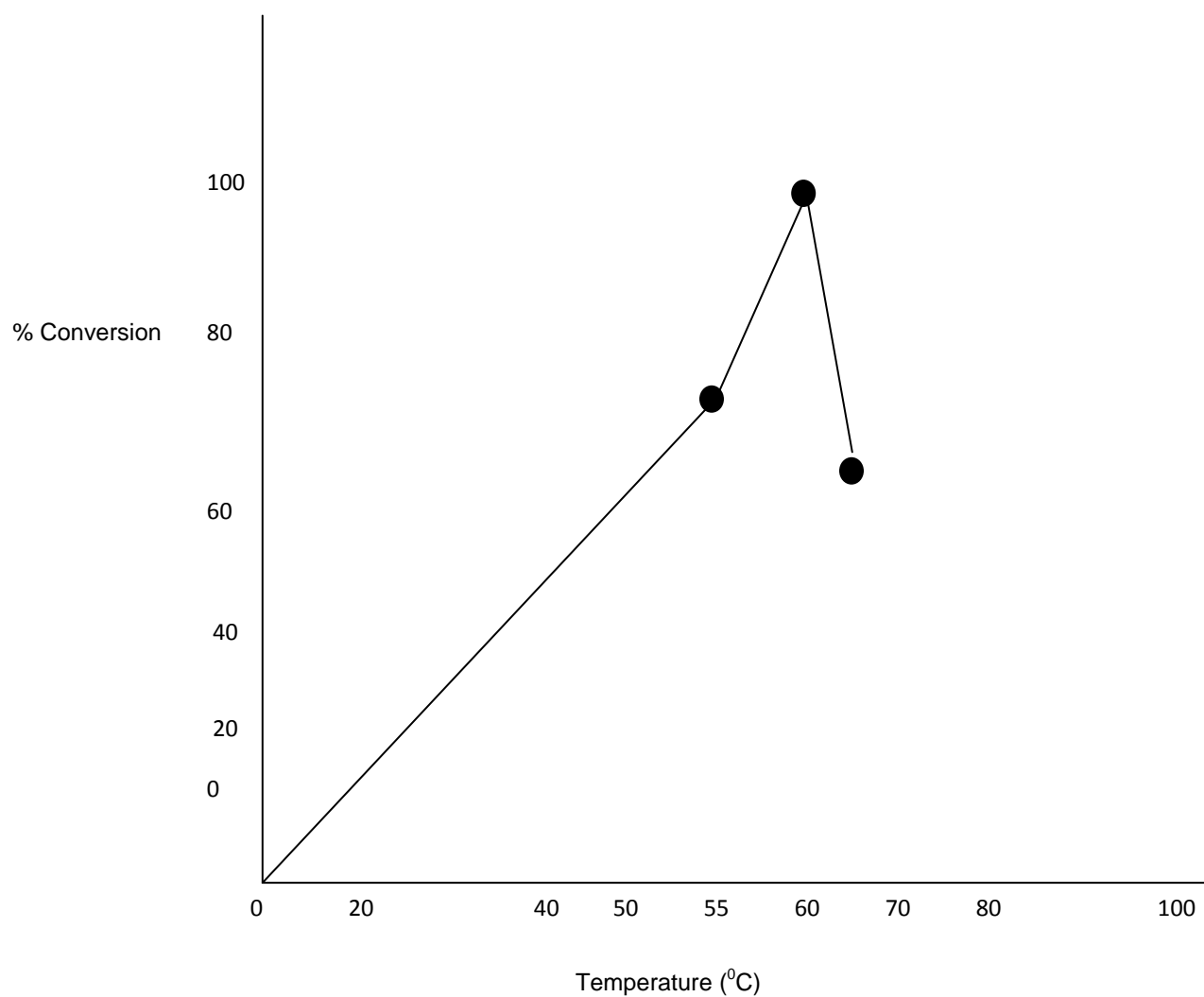


**Fig. 1:** Comparative Bar-Charts of Important parameters in Quassia Oil, Biodiesel and Petroleum Diesel



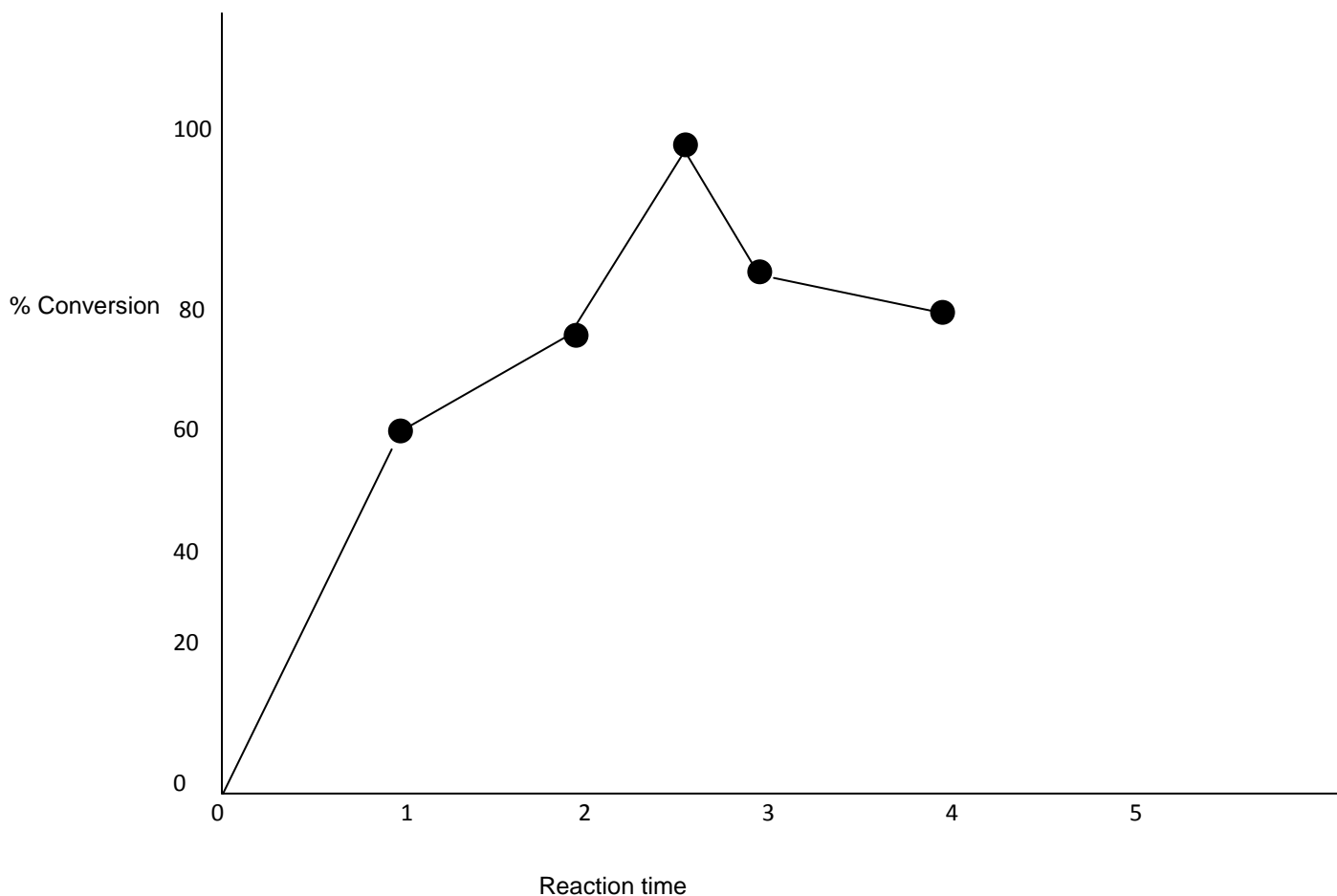
**Fig. 2:** Effect of methanol/oil molar ratio in conversion of oil to biodiesel.

Reaction condition: catalyst 2.5 wt %, reaction time  $2\frac{1}{2}$  hrs, reaction temperature  $60^{\circ}\text{C}$ .



**Fig. 3** Effect of reaction temperature on conversion of *quassia* oil to biodiesel. Methanol oil molar ratio 9:1, catalyst amount 2.5 wt%, reaction time 2<sup>1</sup>/<sub>2</sub> hrs.





**Fig. 4:** Effect of reaction time on conversion of quassia oil. Methanol/oil molar ratio 9:1, reaction temperature 60°C and catalyst amount (2.5wt %).

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