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Optimized production of diesel from karanja oil

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ABSTRACT
The everlasting need of people to find an easily available source of energy has lead man to discover fossil fuel as first choice in energy production. With the advancement of science and technology utilization of fossil fuel like oil, gas and coal as energy source has increased drastically. This massive utilization of fossil fuel has increased air pollution with a threat of diminishing oil, gas and coal reserve around the world. However, properties of crude vegetable oil limit its direct use as fuel in engines. Further, fatty acid methyl esters known as biodiesel are suggested as suitable alternative to diesel. Biofuel is gaining popularity as a renewable source of energy with less environmental harm.

Keywords: "biodiesel", "Karanja", "esterification", "calorific value", "Kinematic viscosity"

1. Introduction

Biofuels can be produced from all living organisms or part of these organisms. This enables us to choose from a variety of raw materials to produce biofuel. But the efficiency of biofuels to use as engine oil varies with raw material used. We also have to control production environment to achieve our desired quality. Bellow some of the renowned works related to biofuels is given to justify this effort of KOME production from Karanja oil.Y.C. Sharma et al.[11] found a maximum yield of 89.5% was achieved at 8:1 molar ratio for acid esterification and 9:1 molar ratio for alkaline esterification, 0.5 wt.% catalyst (NaOH/KOH) using mechanical stirrer. Puneet Verma et al. [1] the maximum HRR (heat release rate) for Karanja biodiesel and its blends was lowered compared to the diesel fuel, specifically, 63.21, 62.8, 62.1, 61.56 and 61.12 J/° CA for LA10 for KOME20, KOEE20, KOPE20, KOBE20, KOPnE20 in comparison with 66.23 J/° CA for diesel. The ignition delay was shorter for biodiesel and its blends than diesel. M. Erdem Günay et al. [14] showed that for the biodiesel production from crude mahua oil, the four variables; namely temperature, ethanol/oil molar ratio, time, and initial CO_2 pressure with relative importance of 39.24, 19.61, 28.57 and 12.58, respectively have strong effects on the fatty acid ethyl ester (FAEE) content. The degree of effectiveness of variables was found to be in the order of temperature > reaction time > ethanol/oil molar ratio > initial CO_2 pressure. S. Jain [15] observed biodiesel mixed with diesel gave lower emissions than diesel fuel, except in the case of NOx. It is observed that in the case of NOx, there is an increase in 2% NOx with B20 use and 10%with B100 use. 10 Ayhan Demirbas [16] in 2007, discussed about different aspects on use of biofuel. A prediction was made, till 2050 biodiesel will serve half of the total energy demand of the world. He found less CO and CO_2 emission from these fuels. With advanced processes the price of biofuels is assumed to be $20 per barrel. Arjun B. Chhetri et al. [17] researched on production of biodiesel from non-edible plant oil and found many non edible plant oil having good amount of fatty acid to produce biodiesel. They effectively extracted 27% biodiesel from 278 gm jatropha seed oil.

Md. Nurun Nabi et al.[18] found that, 20% methanol and .5% NaOH at 55℃ yeilds 77% of biodiesel from cottonseed oil. They also found at a temperature of 20-40℃ 50% biodiesel mixture with fossil diesel works effective. But for low temperature percentage of biodiesel is to be reduced for effective engine performance. May ying Koh et al. [19] in 2010 analyzed, Viscosity of biodiesel or vegetable oil can be reduced by micro-emulsification which consist of three phase oil. They also analyzed 93% extraction of biodiesel from jatropha seed oil using methanol in 11:1 ratio and taking 1.1% KOH at 66℃. M. Mumtaz et al. [20] found that NaOCH3 is better catalyst than NaOH and KOH. This is because NaOCH3 does not produce water like NaOH and KOH in transesterification process of Sativa oil to produce biodiesel. Water reduces the rate of biodiesel formation in transesterification process. S. Keera, S EI Shabagh et al. [21] found that a 9:1 molar ratio of methanol and oil yields to 95% of methyl ester. Tran et al. [22] found that supercritical methanol process can yield at least 80% of final product biodiesel. This process remains unaffected by water released in transesterification. Thus the production rate of biodiesel increases.

The overall aim of this study is to produce biodiesel from non-edible Karanja (*Millettia Pinnata*) oil using 2 stage esterification and transesterification processes and optimize the process parameters (catalyst concentration, methanol to oil molar ratio, reaction time, reaction temperature) using ANOVA software. The specific objectives of this study ARE

1. To produce biodiesel from Karanja (*Millettia Pinnata)* oil.
2. To optimize the process parameters (catalyst concentration, methanol to oil molar ratio, reaction time, reaction temperature) for biodiesel production using ANOVA software
3. To characterize the properties of Karanja (*Millettia Pinnata*) oil methyl ester (KOME).

2. Methodology

Here processes by which biodiesel can be manufactured from Karanja oil in industrial basis is discussed. There are many ways of transforming Karanja oil into

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biodiesel, a simple production process is followed to initiate biodiesel production in Bangladesh. By passing years some modification in these processes will eventually reduce production cost and increase biodiesel production rate.

2.1 Feed stock and catalyst : Biodiesel production from any feedstock requires some catalyst. We are using 2nd generation biofuel production process where biodiesel is produced via transesterification. The items required to perform this process are- (1) Karanja oil (as primary feedstock), (2) Methanol, (3) Sodium hydroxide, NaOH (catalyst), (4) Sulfuric acid, H_2SO_4 (catalyst). Karanja oil is imported from India. Methanol and sodium hydroxide is bought from Taj chemical shop in Chittagong. Sulfuric acid has been provided by our lab.
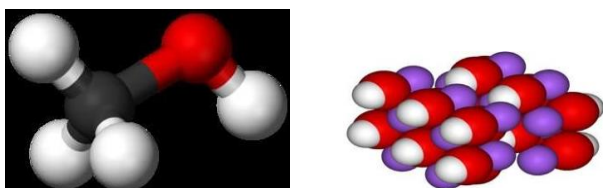

Figure 1 : Molecular structure of Methanol [24] (left) and NaOH (right) [25]

2.2 Transesterification: Transesterification is the process of producing ester with the help of reaction between triglyceride and an alcohol mainly ethanol. In this process glycerol is obtained as byproduct 13 after three consecutive reactions. In this process Carboxylic acids {R-C(=O)-O-H} converted into Methyl esters {R-C(=O)-O-CH3}. There are mainly two types of transesterification. These are –
(1) Catalytic transesterification method.
(2) Non catalytic transesterification method.

2.2.1 Catalytic transesterification method: In this type of transesterification catalyst is used to help chemical reaction to start. Different chemical reagents can act as catalysts. Some of the note worthy catalysts is Methanolic sulfuric acid, sulfonic acid, ferric sulfate, methanolic hydrogen chloride and methanolic boron tri-fluoride. These catalysts produce high alkyl esters but with slow reaction rate. Transesterification reaction can be also done using metal catalyst namely sodium or potassium methoxide.CH3ONa or sodium methoxide is used in almost 60% of industrial plants. This catalyst produces 98% of biodiesel in only 30 minutes which saves time. Main drawback of these catalysts is it only works in absence of water which will gradually increase the production cost for purifying feedstock. In this method the catalyst NaOH, KOH is mixed with methanol by constant stirring which produces hi]op
biodiesel in the reactor which has to be separated from glycerol
2.2.2 Non catalytic transesterification method: There are two basic methods for production of biodiesel by non catalyzed transesterification which includes BIOX co-solvent process This process utilizes Tetrahydrofuran (THF) or Methyl tert-butyl ether as co-solvent. This

process leaves no residue catalyst and acts at low (303K) temperature. Another process uses supercritical methanol to produce biodiesel at a high rate [27]. Having different options in hand catalytic transesterification process is chosen to produce KOME in this project. This process is easy to conduct and the catalysts are easily available. This process requires at least 30 minutes to be completed.

2.3 Equipments :
1. Magnetic stirrer bits, 2. Measuring beakers, 3. Thermometer, 4. 500 ml 3-neck flat bottom conical flask, 5.Goggles, 6. Gloves, 7. Apron, 8. Separator funnel, 9. Digital weight scale. All of these equipments were procured from metallurgy lab.

2.4 Procedure :
The whole procedure of production of KOME is described in brief in later part of this section. →First step of production of KOME from Karanja oil is esterification. In this phase the oil is mixed with sulfuric acid and methanol. Sulfuric acid and methanol reacts with large carboxylic acid molecules to produce ester. Here sulfuric acid modifies carboxylic acid to react with methanol. → In second step transesterification is done. Here alkyl hydroxide (NaOH) acts as a catalyst which breaks apart molecules of glycerol and fatty acids. It makes a favorable environment for methanol to react with fatty acids to produce esters (Biodiesel). The whole procedure is done when the mixture is stirred with the help of a magnetic stirrer. →In third stage the whole mixture is left to settle down. It is called Settling Period. As glycerin is heavier than biodiesel it settles down to the bottom of the reactor. Glycerin is drained out of the reactor and the left out is impure biodiesel which is further purified by washing.

2.4.1 Experimental Setup: For these experimental purposes we have used a 500 ml 3-neck flat bottom conical flask was used as reactor. For heating and mixing the mixture in the flask an automatic magnetic stirrer with hot plate arrangement was used. The mixture was stirred in range of 500-700 rpm for all test runs. The 15 temperature range of 55-65ºC was maintained during this experiment. The separating funnel was used to separate the methanol- water mixture after acid pretreatment and the glycerol after transesterification.
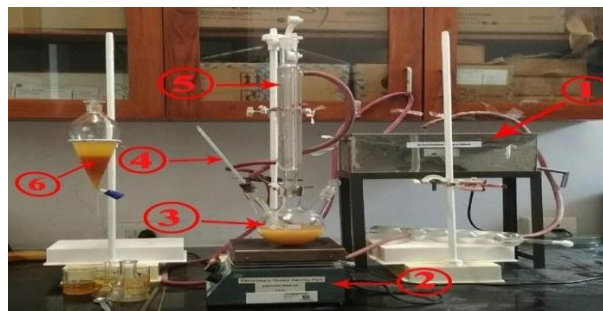

Fig **2** : experimental setup for biodiesel production
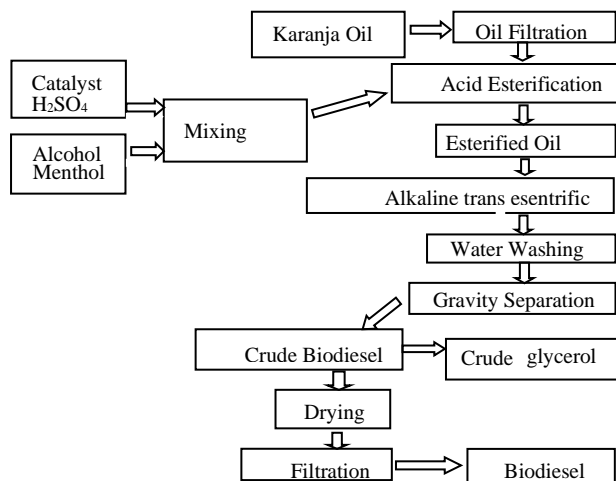
2.4.2 Process flow chart :

Fig 2 : Flow chart of biodiesel production

2.5 Production Procedure Biodiesel : Oil filtration, acid esterification and alkaline transesterification are the main steps for biodiesel production.

2.5.1 Filtering: Karanja oil was filtered by using filter paper to remove ingredients. It had warmed up a bit first to get into run freely, 60ºC was enough.

2.5.2 Acid esterification: Varity of ratios (molar weight ratio) of methanol to oil was taken. Molar weight of karanja oil is 892.7 gm/mole and molar weight of Methanol is 36 gm/mole. 100 to 50 gm karanja oil was taken for different samples. The amount of sulfuric acid was 1%-2% (w/w oil) .Oil was poured into 3-neck flat bottom conical flask and heated at 60ºC temperature. Then measured amount of methanol was mixed. After some time, when the temperature was fixed at 60ºC, sulfuric acid was poured into the mixture. This mixture was stirred at 600 rpm. This stirring and heating was continuing for one hour. The 3-neck flat bottom conical flask was merged in a water bath for proper heating. All

2.5.3 Titration: After acid esterification, esterified oil was poured into separator funnel. And separation was continuing for two hours. Then upper layer was collected. Titration was performed on the esterified oil for measuring the acid value. The acid value was 1.3 The acid value was 1.3 (<2) mg of NaOH/ gm of oil. Isopropanol was used as a alcoholic solvent for oil. 2 gm of oil was dissolved by heating alcohol. 2-3 drop of phenolphthalein was used as an indicator. 1N NaOH was mixed to the solution to know the acid value.

2.5.4 *Transesterification:*

Acid value of esterified oil had to be measured. So, the esterified oil was ready to go through transesterification process. For transesterification process, methanol was taken at various molar ratios to oil (5:1, 6:1, 7:1). And sodium hydroxide was taken at 1%-2% (w/w oil).

First, esterified oil was poured into the 3-neck flat bottom conical flask and heated at 60ºC. Methanol and sodium hydroxide were mixed and warmed up. Then, methanol-catalyst was added to oil carefully. This transesterification was performed on 55ºC-60ºC because

the boiling temperature of methanol is 65ºC and stirring speed was 500- 600 rpm for 70-90 minutes. All these setup were on the plate of magnetic stirrer. The system was kept close to the atmosphere to prevent loss of methanol during the reaction.

2.5.5 *Separation:* As soon as the transesterification reaction was completed, the mixture was poured into separator funnel for separating glycerol and raw KOME. byproduct was at the bottom with raw KOME was on top. The biodiesel varies in color depending on the oil used. The color of produced KOME was light Yellow. The lower layer was drained off. And the upper layer was kept in separator funnel for washing.

2.5.6 *Washing:* Excess methanol, sodium hydroxide and other impurities was found from raw biodiesel. Hot water was used to wash these excess materials. The water was drained from the bottom of the separator funnel. After water washing, washed biodiesel was heated. The cause of heating was to vaporize the impurities. Hence, more pure biodiesel was found. After washing filtration have been done.

2.5.7 Produced Biodiesel:

Finally KOME was obtained from karanja oil. But the rate of production of KOME from karanja oil was much less according to the karanja oil used.
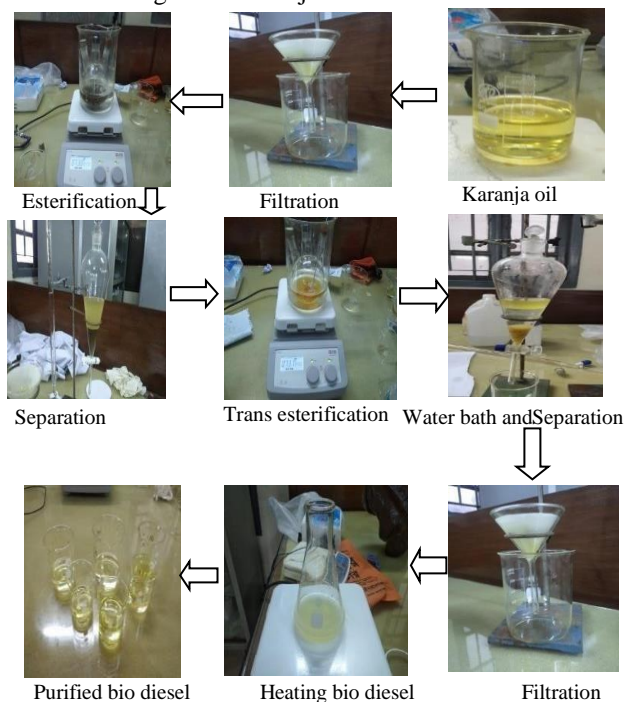

Fig 3 : Biodiesel production

3. Determination properties of biodiesel :

We need to find out different properties of the produced biodiesel to evaluate its potential to be used in diesel engine. We have to find out its calorific value, flash point, fire point and viscosity. Bomb calorimeter will be used in determining the calorific value of the produced biodiesel. Pensky-Martens closed cup tester will be used to find flash and fire point.

Viscometer will be used to determine dynamic viscosity of the biodiesel.

3.1 Determination of calorific value: In bomb calorimeter the mixture is taken using a crucible.

Weight of the mixture is taken before the experiment. The crucible is placed over a ring and magnesium wire is touched with the mixture. By applying current voltage the mixture is burnt inside the bomb-calorimeter in presence of oxygen at 25 atm pressure. With the combustion of this fuel mixture inside temperature of the calorimeter will increase. We can then calculate calorific value of unit mass of the mixture after measuring the temperature difference of the inside water [30].

3.2 Determination of flash point and fire point:
By pensky-Martens closed-cup tester we can determine the flash point and fire point at a temperature range of 60-190℃. This process is carried out by heating the mixture and blowing a flame over the surface of the liquid mixture. We can easily detect the flash point when the mixture vapor ignites for less than 5 seconds. Further heating of the mixture gives a permanent flame over the liquid surface. These two temperatures are measured with thermometer as flash point and fire point [30].

Fig 4: Pensky-Martens closed cup (left) Bomb calorimeter (right)

3.3 Viscosity measurement:
Viscosity is a measure of liquid's resistance under shear stress to deformation. It is frequently viewed as spreading thickness or resistance. Viscosity depicts inner fluid flow strength and can be considered as a measure of fluid friction. Figure below shows the digital viscometer. Dynamic viscosity was measured by the digital viscometer.

4. Formulation and Data Collection Different data and formulas are used to calculate the yield of KOME. To find acid value of oil before and after esterification, amount of methanol and catalyst at esterification and transesterification, percentage yield of biodiesel, viscosity, density, specific gravity and calorific value different formulas are used.

4.1 Formula: Amount of methanol for A:1 molar weight ratio to oil for X gm of oil at esterification and trans-esterification= ($X\ gm\ of\ sample\ oil \times A \times 32/892.7$)………………………... (1)

Acid value, $A/V = (Volume\ of\ titrant\ in\ ml \times .1 \times 40/mass\ of\ sample\ oil)$.. (2)

Density, $\rho= (Mass\ of\ biodiesel/\ Volume\ of\ biodiesel$) …………………………………….(3)

Specific weight of Biodiesel, $w= (Weight\ of\ biodiesel/\ volume\ of\ biodiesel)$............................ (4)

Specific gravity, $S = \dfrac{Specific\ Weight\ of\ biodiesel}{Specific\ weight\ of\ water}$....................................... (5)

Viscosity, $\mu= \tau/(du/dy)$ CP (Centi poise)………. (6)

Kinematic viscosity, $\upsilon= (Dynamic\ viscosity\ (\mu)/Density(\delta))$………………………………….. (7)

Percentage of yield, y= ($Total\ weight\ of\ KOME\ produced\ /\ Total\ weight\ of\ esterified\ oil\ used$)× 100%... (8)

Higher calorific value=$W \times \Delta T/\ m$ (MJ/kg) …………(9)
Where, w=Energy equivalent of calorimeter, Cal/℃
ΔT= Temperature rise,

4.2 Data Collection : Table 1: Data of Esterification process
Time (minutes) : 70 , Stirring speed : 600 rpm , Temperature : 55°C - 65°C

Trial No	Methanol to oil molar ratio	Weight of oil (gm)	Weight of menthaol (gm)	Weight percentage of H_2SO_4 (w/w oil)	Weight of H_2SO_4 (gm)	Acid value (mg of NaOH/ gm of oil)
1.	6:1	100	21.5	1%	1	1.9
2.	6:1	100	21.5	1.5%	1.5	1.5
3.	6:1	100	21.5	2%	2	1.3

Table 2: Data of Transesterification process varying molar ratio of Methanol Time (minutes) : 80 , Weight percentage of NaOH (w/w oil) : 2% , Stirring speed : 600 rpm, Temperature :55°C- 65°C

Trial No	Methanol to oil molar ratio	Weight of oil (gm)	Weight of methanol (gm)	Weight of NaOH (gm)	Weight of Biodiesel (gm)	Percent yield of biodiesel
1.	5:1	96	17.20	1.92	78	81.25%
2.	6:1	96	20.64	1.92	82	85.41%
3.	7:1	92	23.08	1.84	74	80.43%

Table 3: Data of Transesterification process varying %NaOH ,Time (minutes) : 80 , Methanol to oil molar ratio : 6:1 , Stirring speed : 600 rpm , Temperature : 55°C- 65°C

Trial No	Weight of oil (gm)	Weight of methanol (gm)	Weight percentage of NaOH (w/w)	Weight of NaOH (gm)	Weight of Biodiesel (gm)	Percent yield of biodiesel
1.	94	20.21	1%	0.94	78	82.97%
2.	92	19.78	1.5%	1.38	78	84.78%
3.	96	20.64	2%	1.92	80	81.25%

Table 4: Data of Transesterification process varying Stirring speed ,Time (minutes) : 80 , Weight percentage of NaOH (w/w oil) : 1.5% , Methanol to oil molar ratio :6:1 Temperature : 55°C- 65°C

Trial No	Weight of oil (gm)	Weight of methanol (gm)	Weight of NaOH (gm)	Stirring speed	Weight of Biodiesel (gm)	Percent yield of biodiesel
1.	46	9.89	0.69	500	38	82.6%
2.	96	20.64	1.44	600	84	87.5%
3.	92	19.78	1.38	700	78	84.78%

Table 5: Data of Transesterification process varying Reaction time : Methanol to oil molar ratio :6:1 , Weight percentage of NaOH (w/w oil) :1.5% , Stirring speed : 600 rpm , Temperature : 55°C- 65°C

5. Result and Discussion :

Experiment has been done to optimize the production of biodiesel. Different properties of the biodiesel have been calculated. 4 samples out of 12 samples were selected based on highest yield in every group. Average values of these fselected samples are compared with diesel and processed biodiesel.

5.1 Summary of the result :

The collected data were calculated with accurate formulas to find out dynamic viscosity, kinematic viscosity and specific gravity flash point and fire point. The results are shown with the help of graphs for better understanding. For trial no 2,

Volume of biodiesel= 30 ml

Mass of biodiesel= 25.5 gm , Density ρ= (26.7/30) ×1000= 890

Dynamic viscosity of biodiesel= 20.75 cP Kinematic viscosity of biodiesel= $(20.75 \times 10^{-3})/890 = 23.31$ Specific gravity= 890/1000= .89 Where, density of water is 1000 kg/m^3

5.2 Calorific value:

Higher calorific value= $(2426 \times 2.4 \times 4.2)/(.84 \times .001) = 30.32$ MJ/kg ,W=2426 cal/°C

Table 6 : Calculation of density, viscosity, specific gravity

Sam	Density (kg/m^3)	Dynamic viscosity	Kinematic viscosity	Specific gravity	Flash point °C	Fire point °C	%yield	
1	850	17.25	20.29	.85	72	82	85.41%	
2	890	15.8	17.75	.88	65	76	84.78%	
3	830	16.90	20.36	.83	63	72	87.5%	
4. 4	870	16.30	18.73	.87	69	78	86.36%	
Average	860	16.56	19.28	.86	67.25	77	N/A	

Table 7 : Comparison among diesel, biodiesel and KOME

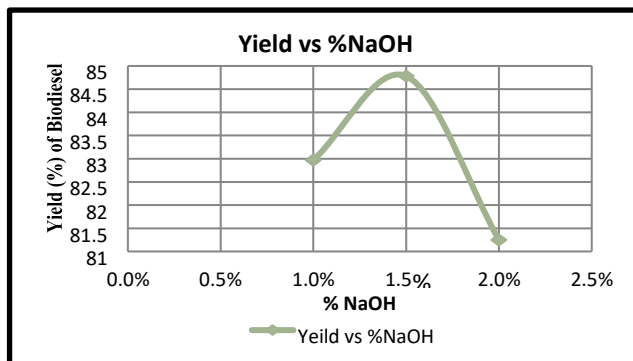| Sample no	Diesel	Biodiesel	KOME (Average)	
Density (kg/m^3)	830	860-900	860	
Kinematic viscosity (mm^2/s)	1.9-3.8	3.6-5	19.28	
Specific gravity	.83	.86-.90	.85	
Flash point	55	150	67.25	
Fire point	65	170	77	
Calorific value (MJ/kg)	45.-46.7	39.3-39.8	30.32	

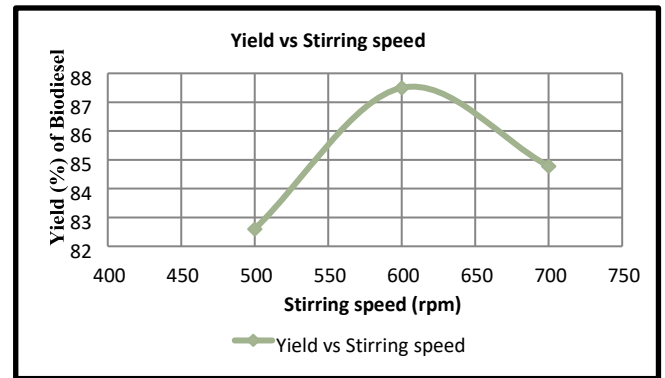Fig 5 : Relation between yield and %NaOH

Fig 6 : Relation between yield and stirring speed
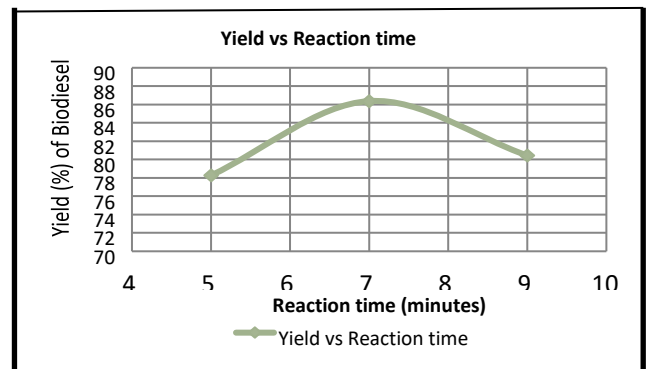

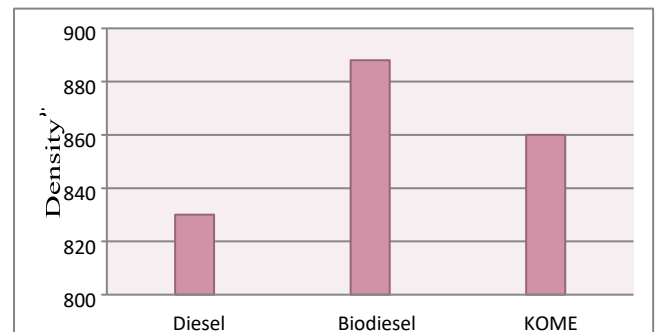Fig 7 : Relation between yield and Reaction time

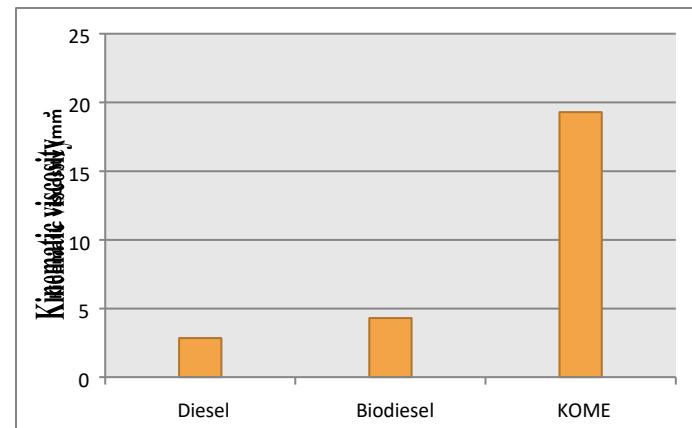Fig 8 : Comparison of density of different fuel

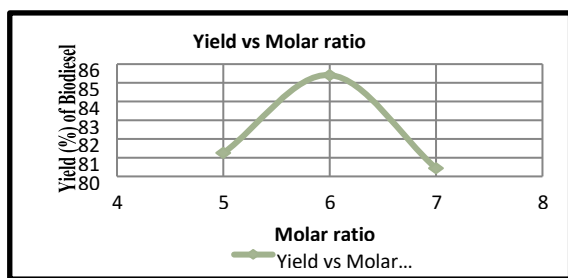Fig 9 : Comparison of viscosity of different fuel

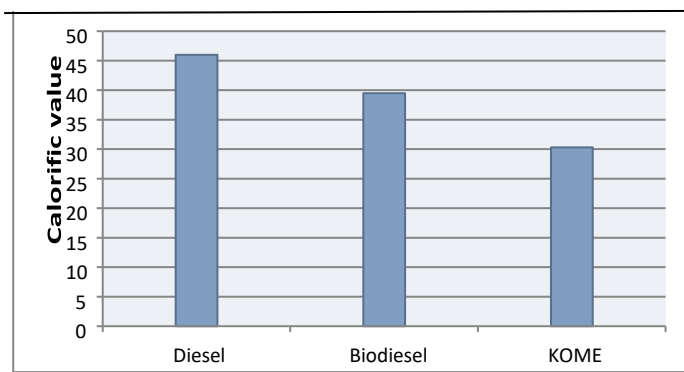Fig 10 : Relation between yield and molar ratio

Fig 11 : Comparison of calorific value of different fuel
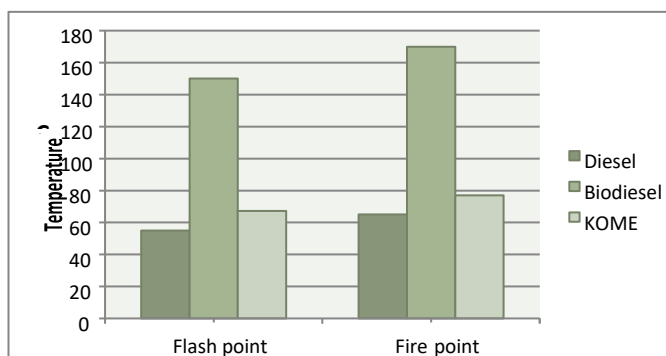

Fig 12: Comparison of flash point and fire point of different fuel

Results of this study comply with previous work done with Karanja oil for biodiesel production. Maximum yield was found for 6:1 molar ratio of methanol to oil, 1.5% NaOH, 600 rpm stirring speed and 70 minutes running time. Though much care has been taken, considerable amount of soap was formed during trans-esterification. The viscosity of produced biodiesel was way more than expected but calorific value was around desired level. Flash point and fire point was more than petroleum diesel which indicates it is a bit safer than diesel for transportation.

6. Conclusion :

This project is designed to produce a cost effective fuel for automobile from Karanja oil. With this project we tried try to optimize the production of biodiesel. We have justified our decision of selecting Karanja oil by producing quality biodiesel. The flash point and fire point of KOME produced was more than diesel but was less than average biodiesel. Viscosity of the KOME was much higher than both biodiesel and diesel. But calorific value was a little bit less. So to cope up with diesel blend at any ratio would give better result.

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Nomenclature

T : Temperature , ℃	S : Speed , rpm
D : Density , kg/m^3	
S : Speed , rpm	K : kinematic viscosity , mm^2/s