
Biodiesel production from crude oil of *Jatropha curcas* and *Pongamia pinnata* by transesterification process

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Abstract: The present study is aimed to produce biodiesel from non-edible oil sources such as crude oil of *Jatropha curcas* and *Pongamia pinnata* by transesterification process which can occur at different temperatures (45°C to 70°C), depending on the oil used. For the transesterification of *J. curcas* oil (methanol 15% of oil and 12 g NaOH/litre), the reaction was studied with different temperatures. It was found that much of the process complexity originates from contaminants in the feedstock, such as water and free fatty acids, or impurities in the final product, such as methanol, free glycerol and soap. Results shows that purified ester yields at 45°C were 76% for 1 hr and 80% for 2 hr. At 50°C, purified ester yields were 79% for 4 hr and 76.75% for 6 hr. In case of *Pongamia pinnata*, purified ester yields at 50°C were 79% for 4 hr and 83% for 6 hr. Further, when the fuel was tested for its authenticity to be used in diesel engine based on the American Society for Testing and Materials (ASTM), it was found that the fuel was coming within the prescribed standards. The study concludes that temperature clearly influenced the reaction rate and yield of esters. [Received: May 6, 2010; Accepted: August 12, 2010]

Keywords: biodiesel; monohydric alcohol; monoalkyl ester; *Jatropha curcas*; *Pongamia pinnata*.

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1 Introduction

Biodiesel is a clean-burning fuel currently being produced from grease, vegetable oils, or animal fats. However, many researches have been conducted to characterise these physical and chemical changes in the vegetable oils (Monyem and Van Gerpen, 2001; Leung et al., 2006). The use of biodiesel is growing quickly because of its excellent fuel properties (Vicente et al., 2004). Diesel engines can be directly fueled with biodiesel without any mechanical change (Zhang et al., 2000; Canakci and Gerpen, 2001; Shah et al., 2004; Etinkaya and Karaosmanoglu, 2005). Because biodiesel is renewable, biodegradable, and environmentally benign (Vicente et al., 2004; Encinar et al., 2005; Baber et al., 2005; Gerhard et al., 1997), engines fuelled with biodiesel reduce the emission of toxic gases, such as SO₂, CO, and hydrocarbons (Vicente et al., 2004; Encinar et al., 2005). The main advantage of using biodiesel fuels as 100% methyl or esters of vegetable oil and animal fat or biodiesel blends (up to 20% blend to the diesel fuel) is reduced production of smoke and particulates, higher cetane numbers and lower production of carbon monoxide and hydrocarbon emissions (Antolin et al., 2002; Encinar et al., 2007).

This fuel is biodegradable and non-toxic and has low emission profiles as compared to petroleum diesel. Usage of biodiesel will allow a balance to be sought between agriculture, economic development and the environment (Allen and Kline, 1945; Boocock, 2001; Canakci and Van Gerpen, 1999; Dasari et al., 2003; Kawahara and Ono, 1979; Mittelbach and Koncar, 1998; Nelson et al., 1996; Trent, 1945). Chemically the oils/fats consist of triglyceride molecules of three long chain fatty acids that are ester bonded to a single glycerol molecule. These fatty acids differ by the length of carbon chains, and the number, orientation and position of double bonds in these chains. Thus biodiesel refers to lower alkyl esters of long chain fatty acids, which are synthesised either by transesterification with lower alcohols or by esterification of fatty acids. Berchmans and Hirata (2008) reported the yield for methyl esters of fatty acids was achieved ca. 90% in 2 h. Typical chemical syntheses of bio-fuels are reported in the literature based on the distribution of the fatty acids in their corresponding vegetable oils (Graboski and McCormick, 1998; Nabi et al., 2009).

Biodiesel consists of mono-alkyl esters of long-chain fatty acid manufactured by transesterification of virgin/vegetable oil (e.g., soybean oil, sunflower oil, rapeseed oil) or animal fat by using alkali catalysts. The majority of biodiesel today is produced by alkali-catalysed (e.g., NaOH, KOH) transesterification with methanol, which results in a relatively short reaction time (Freedman et al., 1984; Ma and Hanna, 1999). Zhou et al. (2006) has also reported that the transesterification reaction is carried out in the methanol phase, and as a result, the reaction shows an induction period.

The transesterification reaction in traditional production of biodiesel is catalysed by acid/base catalyst (Zhou et al., 2006). Presently we have used the KOH, NOH as a catalyst. Even though many new catalysts [such as solid base/acids (Cantrell et al., 2004; Satoshi et al., 2004; Kumar and Anju 2005), enzymes (Du et al., 2004; Karl-Erich and Thorsten, 2002; Vanessa and Park, 2004; Scragg et al., 2003), organic bases (Schuchardt et al., 1995), and metal oxides (Suppes et al., 2004)] have been reported to be effective for the transesterification reaction, most of the biodiesel plants at present operate with caustic bases (Boocock et al., 1996).

In the reaction of transesterification, reactants, methanol, and oil are partially miscible (Carmen et al., 2005). Alkali catalyst exists in the methanol phase. Comparing the reaction rates of methanol with butyl alcohol, Boocock et al. (1996) suggested that the transesterification reaction takes place in the methanol phase. So, the reaction rate greatly depends on the solubility of oil in methanol. On the other hand, the formation of glycerol may change the distribution of reaction components in different phases.

1.1 Biodiesel from *Jatropha curcas* and *Pongamia pinnata* as alternative fuel

In present study, we have used *Jatropha curcas* and *Pongamia pinnata* as an alternative plant source for the production of biodiesel. *J. curcas* plants grow on poor degraded soils and are able to ensure a reasonable production of seeds with limited inputs. It is not grazed by animals and is highly pest and disease resistant. Time taken for nut yield is between two and five years based on soil and rainfall conditions. One hectare of jatropha plantation will on an average produce 3.75 tons of seed yielding 1.2 tons of oil. The oil content is 35% by weight of the seed and the extraction efficiency is 91% (i.e., oil that can be converted into biodiesel by transesterification).

The oil of *Pongamia pinnata* (Leguminosae; Pappilo-naceae), commonly known as Karanj, is a non-edible oil of Indian origin (Karmee and Chadha, 2005; Lakshmikanthan, 1978; Agarwal, 2007). It is found mainly in the native Western Ghats in India, northern Australia, Fiji and in some regions of Eastern Asia. This medium sized (max ht. 18 m) tree is found almost throughout India up to an altitude of 1,200 m. It is fast growing and matures after four to seven years yielding flat, elliptic fruits (ca. 7.5 cm long). Each fruit contains one to two kidney shaped brownish red kernels. The oil content of the kernel is 30% to 40% (Karmee and Chadha, 2005; Lakshmikanthan, 1978). A single tree yields 9–90 kg seed per tree, indicating a yield potential of 900–9,000 kg seed/ha (assuming 100 trees/ha), 25% of which might be rendered as oil (Karmee and Chadha, 2005). In general, conventional Indian mills (oil extraction units) extract 24% to 27.5% oil, and the village crushers extract 18% to 22% oil. This dark brown oil has fungicidal properties. Its high viscosity and poor combustion characteristics can cause poor atomisation, fuel injector blockage, excessive engine deposit and engine oil contamination so can be used only after converting it to biodiesel.

2 Materials and methods

2.1 Materials

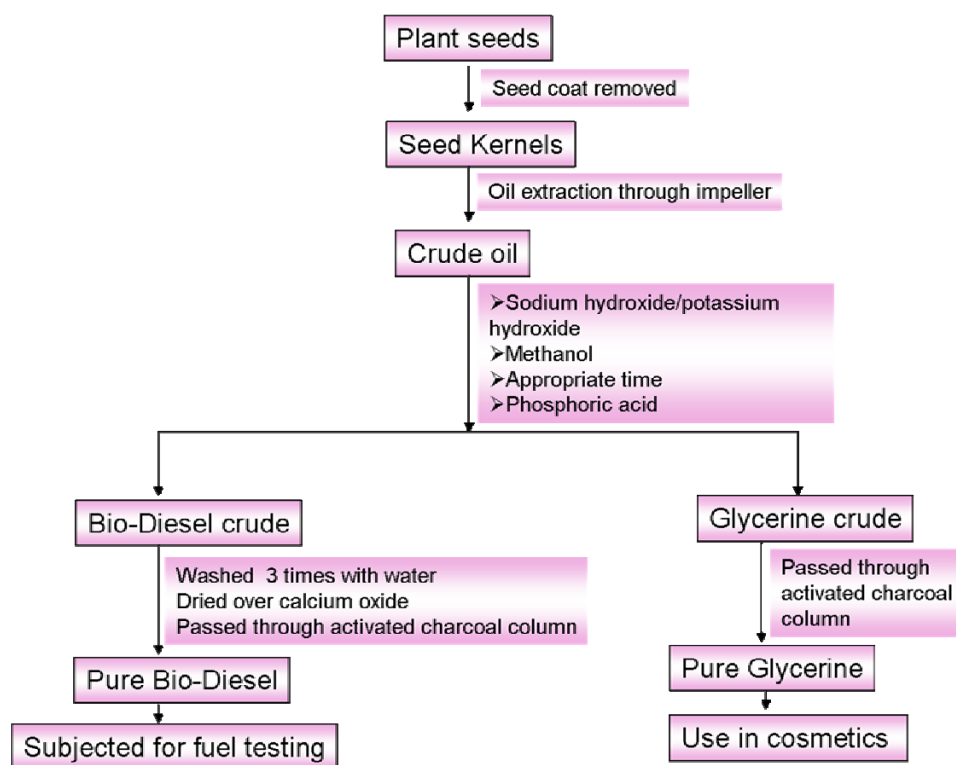
The basic raw material for manufacture of biodiesel is jatropha and pongamia oils. The other raw materials required for manufacture of biodiesel are methanol (methanol was

dried and distilled before use), sodium hydroxide and potassium hydroxide and phosphoric acid (85% pure) The chemicals were purchased from Thomas Baker Chemicals Limited, Marine Drive, Mumbai.

Table 1 Raw materials used in the production of biodiesel

<i>To manufacture 1 ton of biodiesel from crude oil the raw materials required are as follows</i>			
	<i>Material</i>	<i>Quantity</i>	<i>Unit</i>
a	Methanol (up to 99.5% pure)	150	Litres
b	Jatropha/pongamia oil	1,050	Litres
c	Caustic potash	3.8	Kgs.
d	Sodium hydroxide	5.25	Kgs.
e	Phosphoric acid	15	Litres
f	Distilled water	6	Litres
g	Calcium oxide	6	Kgs.
h	Activated charcoal	5	Kgs.

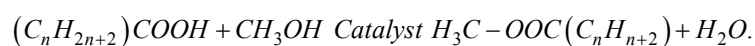
Figure 1 Flow diagram for the procedure of experimental process (see online version for colours)



2.2 Methods

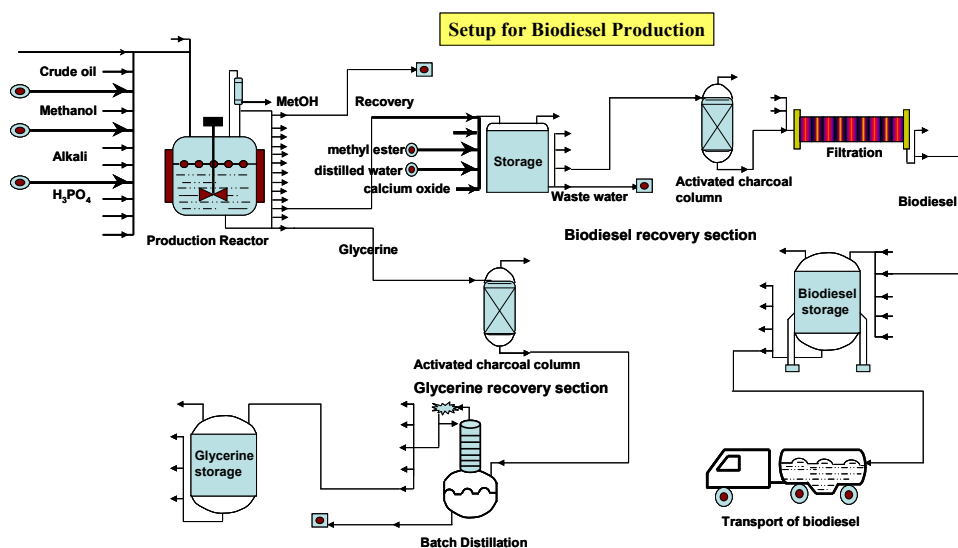
NaOH and KOH being hygroscopic must be kept in air tight container viz. may react with atmospheric CO₂ to form carbonate salts which although do not alter the product but is unable to catalyse the transesterification reaction (Zhang et al., 2003; Watanabe et al., 2000; Meher et al., 2006; Babu et al., 2009). The flow chart explains the experimental procedure adopted (Figure 1). The reaction conditions for best possible yield are

- 1 Reagents used must be 99% pure.
- 2 Potassium hydroxide can also be used as an alternative to sodium hydroxide.
- 3 Crude *Jatropha curcas* oil is converted into biodiesel by transesterification reaction as given below:



- 4 Sodium alkoxide are most efficient catalysts. Alkali catalysts are preferred over acid catalysts as they are less corrosive to industrial equipment.

Figure 2 Pilot plant design for biodiesel production (see online version for colours)



2.3 Bioreactor for biodiesel production

A bioreactor has been designed for the transesterification of oil with a working volume of 15 L. It is made up of stainless steel reaction vessel having an internal heating coil, an impeller and a condenser. Heating is monitored through a temperature probe connected to a digital temperature display (DTD) of range 30°C to 80°C and an impeller is used to ensure uniform mixing required for good yield. It can be operated as fed-batch process where substrates are added in required amount on the basis of quantity of oil being processed through input port. Reaction conditions are maintained in the vessel through control panel. Once the transesterification reaction is complete methanol is distilled out

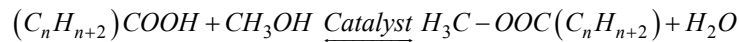
by raising the temperature up to its boiling point and is collected outside after condensation by condenser. Then orthophosphoric acid is added through input port, mixed thoroughly by impeller for 15 minutes and kept for settling. A pilot plant design for biodiesel production is shown in Figure 2.

Once glycerine gets settled down at the bottom which is below the methyl ester, it is collected through product outlet and remaining methyl ester is washed using warm water in the same vessel thrice. Water containing soap and glycerine part is removed through outlet and processed methyl ester is kept in storage tank.

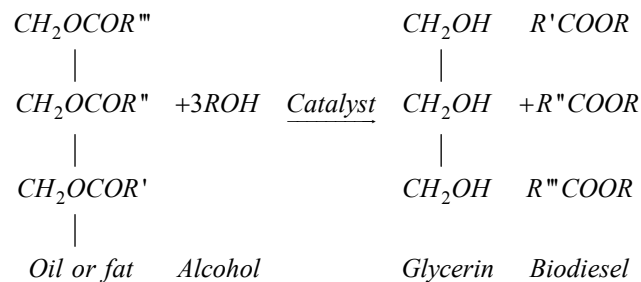
2.4 The process and chemistry of transesterification process

Transesterification or alcoholysis is the displacement of alcohol from an ester by another, a process which is similar to hydrolysis. This process has been widely used to reduce the high viscosity of triglycerides.

The transesterification reaction is represented by the general equation as:



If methanol is used in this process it is called methanolysis. Transesterification is one of the reversible reactions and proceeds essentially by mixing the reactants. However, the presence of a catalyst (a strong acid or base) accelerates the conversion. The general equation of transesterification of triglyceride is



As a catalyst in the process of alkaline methanolysis, mostly sodium or potassium hydroxide has been used.

2.5 Process variables of transesterification

The most important variables that influence the transesterification reaction are:

- reaction temperature
- ratio of alcohol to vegetable oil
- catalyst
- mixing intensity
- purity of reactants.

2.5.1 *Reaction temperature*

The literature has revealed that the rate of reaction is strongly influenced by the reaction temperature. However, the reaction is conducted close to the boiling point of methanol (45°C to 70°C) at atmospheric pressure for a given time.

2.5.2 *Ratio of alcohol to oil*

Another important variable is the molar ratio of alcohol to vegetable oil. As indicated earlier, the transesterification reaction requires 3 mol of alcohol per mole of triglyceride to give 3 mol of fatty esters and 1 mol of glycerol. In order to shift the reaction to the right, it is necessary to either use excess alcohol or remove one of the products from the reaction mixture. The second option is usually preferred for the reaction to proceed to completion. The reaction rate was found to be highest when 100% excess methanol was used. Generally, 15% methanol is taken w.r.t. oil volume.

2.5.3 *Catalysts*

Alkali metal alkoxides are found to be more effective transesterification catalysts compared to acidic catalysts. Sodium alkoxides are the most efficient catalysts. Trans-methylation occurs in the presence of both alkaline and acidic catalysts. As they are less corrosive to industrial equipment, alkaline catalysts are preferred in industrial processes. A concentration in the range of 0.5% to 1% (w/w) has been found to yield 94% to 99% conversion to methyl esters, and further increase in catalyst concentration does not affect the conversion but adds to extra cost, as the catalyst needs to be removed from the reaction mixture after completion of the reaction.

2.5.4 *Mixing intensity*

It has been observed that during the transesterification reaction, the reactants initially form a two-phase liquid system. The mixing effect has been found to play a significant role in the slow rate of the reaction. As phase separation ceases, mixing becomes insignificant. The effect of mixing on the kinetics of the transesterification process forms the basis for process scale-up and design.

2.5.5 *Purity of reactants*

Impurities in the oil affect the conversion level considerably. It is reported that about 65% to 84% conversion into esters using crude vegetable oils has been obtained as compared to 94% to 97% yields refined oil under the same reaction conditions. The free fatty acids in the crude oils have been found to interfere with the catalyst. This problem can be solved if the reaction is carried out under high temperature and pressure conditions.

2.6 *Transesterification of crude oil from *Jatropha curcas* and *Pongamia pinnata**

First 1000 ml of crude jatropha oil were transesterified with a solution of known amount of KOH and NaOH in 150 ml methanol separately. The reaction was carried out at 30°C,

40°C, 45°C, and 50°C with continuous stirring at 200 rpm was maintained as desired. First, the oils were mixed with the methanolic KOH solution and methanolic NaOH and the reaction mixture was stirred for fixed time interval before it was transferred to a separation funnel. The alkaline mixture is neutralised/sweetening using required amt of phosphoric acid and kept for settling. After 5 hrs the glycerol layer was separated from the lower layer. The esters were washed three times with 250 ml of warm water each (50°C) to remove traces of soap and glycerol and dried over calcium oxide and then passed through charcoal column to obtain pure methyl esters.

2.7 Transesterification of crude oil from *Jatropha curcas* using NaOH and KOH

1,000 ml of crude pongamia oil, a known amount of catalyst NaOH and KOH, dissolved in the required amount of methanol was added. The temperature 50°C was maintained as desired with a continuous stirring at 200 rpm for 6 hrs. After this mixture is which is already alkaline is neutralised/sweetening using required amt of phosphoric acid and kept for settling. After 5 hrs the glycerol layer was separated from the lower layer. The esters were washed with water to remove unreacted base, trace amount of glycerol and soap and dried over calcium oxide and then passed through charcoal column to obtain pure methyl esters. Various stages for the production of biodiesel have been shown in Figure 3.

Figure 3 Various stages in the production procedure of biodiesel, (a) crude oil (b) oil after addition of methoxide observe the murkiness (c) mixture after completion of reaction and neutralisation with orthophosphoric acid (d) methyl ester after settling (e) glycerine layer (dark brown colour) (see online version for colours)

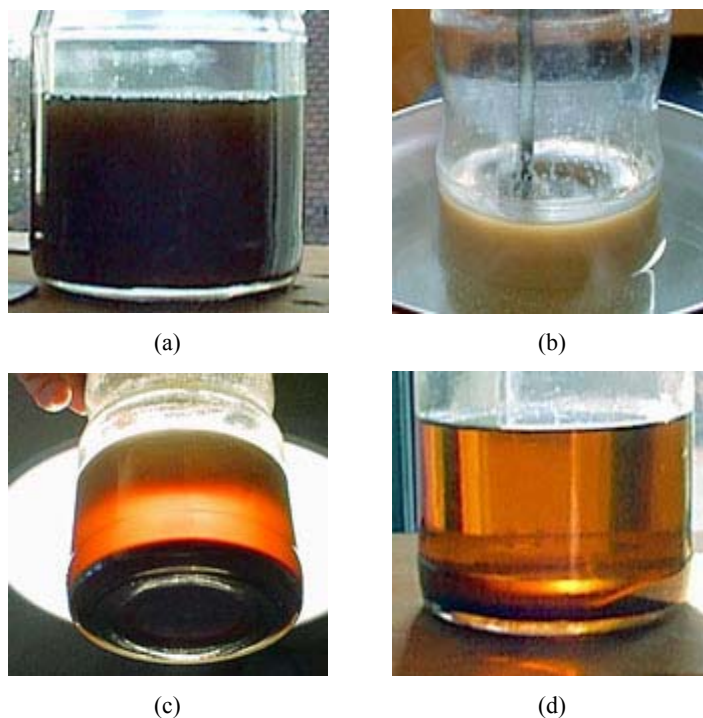


Figure 3 Various stages in the production procedure of biodiesel, (a) crude oil (b) oil after addition of methoxide observe the murkiness (c) mixture after completion of reaction and neutralisation with orthophosphoric acid (d) methyl ester after settling (e) glycerine layer (dark brown colour) (continued) (see online version for colours)



(e)

2.8 Physical and chemical analysis

Physical and chemical analysis was performed to authenticate whether the fuel actually produced is biodiesel at Central Lab of Indian Railways in collaboration with Research Designs and Standards Organization (R.D.S.O), Ministry of Railways, Government of India. Table 6 contains all the technical fuel parameters analysed such as pour point, cloud point, viscosity, cetane number, distillation recovery, flash point, specific gravity etc. The protocol followed was as per American Society for Testing and Materials (ASTM) standards.

3 Results and discussion

3.1 Effect of temperature and reaction time on transesterification of *Jatropha curcas* oil and *Pongamia pinnata*

Transesterification can occur at different temperatures, depending on the oil used. For the transesterification of *Jatropha curcas* oil with methanol (15% of oil) and 12 g NaOH/litre, the reaction was studied with different temperatures. At 45°C, purified ester yields were 95/125 (v/v), i.e., 76% and 100/125 (v/v), i.e., 80% for 1 hr and 2 hr runs, respectively. At 50°C, purified ester yields were 2165/2765(v/v), i.e., 79% and 92/120 (v/v), i.e., 76.75% for 4 hr and 6 hr runs, respectively (Table 2).

Table 2 Comparison for 100 ml jatropha oil for effect of reaction time and temperature

NaOH	MeOH	Time	Temp.	H ₃ PO ₃	Biodiesel	Glycerine
1.2 g	15 ml	1 hr	45°C	15 ml	95 ml	25 ml
1.2 g	15 ml	2 hr	45°C	15 ml	100 ml	25 ml
1.2 g	15 ml	4 hr	50°C	10.7 ml	110 ml	16.6 ml
1.2 g	15 ml	6 hr	50°C	17.2 ml	100 ml	24.4 ml

Note: There is some vol. of FFAs formation.

For the pongamia oil temperature is kept higher since at lower temperature reaction mixture become semi-solid which prevents uniform mixing necessary for reaction to proceed. Thus temperature clearly influenced the reaction rate and yield of esters (Table 3).

Table 3 Comparison for 100 ml pongamia oil for effect of reaction time

<i>NaOH</i>	<i>MeOH</i>	<i>Time</i>	<i>Temp.</i>	<i>H₃PO₃</i>	<i>Biodiesel</i>	<i>Glycerine</i>
1.2 g	15 ml	6 hr	50°C	20 ml	95 ml	25 ml
1.2 g	15 ml	6 hr	50°C	15 ml	100 ml	28 ml
1.2 g	15 ml	4 hr	50°C	17 ml	98 ml	26 ml

Note: There is some vol. of FFAs formation.

3.2 Comparison of NaOH catalysed and KOH catalysed transesterification

As catalysts in the process of alkaline methanolysis, both sodium hydroxide and potassium hydroxide have been used. Crude oils with 15% either sodium methoxide or potassium methoxide catalyst resulted successful conversion to methyl ester (Tables 4 and 5).

Table 4 Comparison for jatropha oil on treatment with different alkali at 50°C

<i>Oil quantity</i>	<i>Alkali</i>	<i>MeOH</i>	<i>Time</i>	<i>H₃PO₃</i>	<i>Biodiesel</i>	<i>Glycerine</i>
100 ml	0.5 g NaOH	15 ml	6 hr	17 ml	92 ml	34 ml
100 ml	1.2 g KOH	15 ml	6 hr	15 ml	92 ml	28 ml

Table 5 Comparison for Karanj oil on treatment with different alkali at 50°C

<i>Oil quantity</i>	<i>Alkali</i>	<i>MeOH</i>	<i>Time</i>	<i>H₃PO₃</i>	<i>Biodiesel</i>	<i>Glycerine</i>
100 ml	0.5 g NaOH	15 ml	6 hr	20 ml	96 ml	32 ml
100 ml	1.2 g KOH	15 ml	6 hr	15 ml	93 ml	30 ml

Methanolysis of jatropha and pongamia oil with the catalyst potassium hydroxide (12 g/litre) has given the best yields and viscosities of the esters but it is prohibitively expensive. Best grades of potassium hydroxide have 14% to 15% water, which can not be removed. It should be low in carbonate, because the carbonate is not an efficient catalyst and may cause cloudiness in the final ester. Sodium hydroxide pellets have given very good results. Because quantity of catalyst used is quite less, good quality catalyst (in spite of high cost) can be used.

3.3 Fuel properties of fatty acid methyl esters in comparison with high speed diesel

When the engine is operated on biodiesel, soot emissions (insoluble) are dramatically reduced, but the proportion of emissions composed of fuel derived hydrocarbons (fuel soluble), condensed on the soot, is much higher. This implies that the biodiesel may not

burn to completion as readily as diesel fuel. It should, however, be noted that gaseous HC emissions were reduced with biodiesel in tests.

Issariyakul et al. (2007) reported cetane index is used to evaluate combustion quality of esters. The higher the cetane index indicates the better quality of esters as diesel fuel. On the contrary, vegetable oils and biodiesels are very good Diesel engine fuels due to their reasonably high cetane number (Hansen et al., 2005; Li et al., 2005). Fuel properties of fatty acid methyl esters in comparison with high speed diesel (HSD) among jatropha methyl ester, pongamia methyl ester and diesel fuel has been indicated in Table 6. Moreover heating value is another important property of ester as it is aimed for use as diesel fuel substitute. On the other hand Cloud point is the temperature at which a cloud of wax crystals first appears in the oil when it is cooled. Pour point is the lowest temperature at which the oil specimen can still be moved. Cloud point and pour point are used to measure the cold temperature usability of ester as fuel. Cloud point of esters was in the range. The properties in make of biodiesel by jatropha methyl ester, pongamia methyl ester may also include water content, distillation, viscosity and flash point. These factors are important steps to make of purified biodiesel.

Table 6 Comparison of jatropha and pongamia methyl ester with diesel fuel

<i>Fuel property</i>	<i>Jatropha methyl ester</i>	<i>Pongamia methyl ester</i>	<i>Diesel fuel</i>
Formula	C ₁₈ to C ₁₉	C ₁₈ to C ₁₉	C ₈ to C ₂₅
Water content	Nil	Nil	Nil
Specific gravity	0.905	0.926	0.81
Pour point (°C)	-5°C	-7°C	-23°C
Cloud Point	3°C	-1.5°C	-5°C to +5°C
Viscosity mPa-s at 20°C	3.6	3.6	2.6-4.1
Lower heating value KJ/lit	32	37	35-37
Flash point °C	202°C	220°C	74
Cetane number	52	62	40-55
Distillation	90% at 308°C	80% at 280°C	98% at 350°C

4 Conclusions

Thus, our designed methodology can well be the solution for developing an alternative fuel production strategy taking into consideration the depleting reserves of conventional fuels which in turn will reduce our dependency on conventional sources. Producing fuel from green sources definitely reduces the CO_x and NO_x emissions which are today's major contributors of air pollution. Designing a cost effective method which can be easily scaled up is the gist of this work. The only thing which should be taken care off is the reaction temperature for maximum yield and kinematic viscosity for better fuel performance.

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