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Process optimisation of base catalysed transesterification of Karanja oil for biodiesel production

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Abstract: Biodiesel has been receiving increasing attention due to its renewable nature, lower pollution potential and green house gas benefit compared to mineral diesel. There are numerous factors, which affect the yield of biodiesel in a transesterification process such as reaction temperature, molar ratio of alcohol to oil, catalyst concentration, stirring speed, reaction time, etc. These variables are different for different vegetable oils and they are also affected by the free fatty acid content of the oil. In the present investigation, an attempt has been made to optimise the production process of biodiesel from Karanja oil, which is essentially a non-edible oil. This oil is tree-borne and has a great potential to produce good quality biodiesel in large quantities. In the present investigation, a large array of experiments were carried out on transesterification of Karanja oil with methanol to produce biodiesel under different reaction conditions and an attempt has been made to analyse the effect of various reaction conditions on biodiesel yield. The optimised yield of methyl esters from Karanja oil under the optimal condition was found to be 98–100% (w/w_{oil}). [Received: February 21, 2009; Accepted: April 30, 2009]

Keywords: biodiesel; process optimisation; Karanja oil; transesterification; catalysis.

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1 Introduction

World's energy needs are largely met by use of non-renewable resources such as petrochemicals, natural gas and coal. Increasing energy demands, environmental concerns and depletion of fossil fuel reserves has led to the search for alternate fuels. An alternative fuel must be technically feasible, economically competitive, environmentally acceptable and readily available. Renewable sources such as vegetable oils and their derivatives have gained importance as alternative fuels for diesel engines.

Although short-term engine tests using straight vegetable oils as fuel in unmodified engines have demonstrated promising results, longer-duration tests encountered issues such as injector coking, ring sticking, thickening of the lubricating oil, etc. (Peterson et al., 1983; Velleguth, 1983). Vegetable oils are inherently more viscous compared to mineral diesel and cannot be burned efficiently in modern engines/vehicles without pre-treatment. There is a need to modify the vegetable oils in order to use them as fuels; hence several methods are devised to convert vegetable oils into diesel-like fuels. Various techniques are employed to bring the physical, chemical and thermal properties of vegetable oils closer to mineral diesel namely blending, thermal cracking (pyrolysis), micro-emulsification, hydrogenation and transesterification (Peterson et al., 1983; Agarwal, 2007; Srivastava and Prasad, 2000). Among the several ways available to convert straight vegetable oils into a diesel-like fuel, the transesterification process is found to be the most feasible (Agarwal, 1998; Agarwal and Das, 2001; Meher et al., 2006; Dorado et al., 2003; Scholl and Serenson, 1993; Al-Widyan and Al-Shyoukh, 2002; Antolin et al., 2002; Altin et al., 2001). Biodiesel i.e., methyl esters of fatty acids emerge as promising and potential diesel fuel substitutes (Canacki and Gerpen, 1999; Diasokou et al., 1998; Muniyappa et al., 1996). The properties of vegetable oils and biodiesel vis-à-vis mineral diesel are given in Table 1.

Biodiesel offers various advantages e.g., it is biodegradable and non-toxic, assuring safe handling, transportation, storage and has no impact on surface and ground water; it does not contain sulphur or aromatic compounds, thus it contributes to the reduction in exhaust emissions; it comes from renewable sources (such as vegetable oils, animal fat, etc.); it can be produced domestically/locally, thus reducing dependence on foreign/imported fuel from long distances; given that all the organic carbon present is of photosynthesis-origin, it does not contribute to a net rise in the level of carbon dioxide in the atmosphere, and consequently to the greenhouse effect; and minor modifications of diesel engines can allow them to perform very efficiently when fuelled with biodiesel blends or even pure biodiesel (Canacki and Gerpen, 1999). Use of biodiesel can improve urban air quality, create employment, especially in rural areas, and help reduce the dependence on limited global petroleum resources. Biodiesel can also extend engine's operational life because of its inherent lubricating properties (Agarwal, 2007). Physical wear of various vital moving parts, and carbon deposits on piston, ring grooves, cylinder head and injectors were found to be substantially lower in biodiesel blend (B20) fuelled engine vis-à-vis mineral diesel fuelled engine (Agarwal et al., 2003).

Table 1 Comparison of vegetable oil and biodiesel with diesel

Vegetable oil	*Biodiesel*	*Mineral diesel*
Triglycerides of fatty acid (molecular wt. 700–1000).	Alkyl esters of fatty acid (molecular wt.~ 260 to 300).	Saturated chain hydrocarbon (molecular wt. ~200 to 300).
~10% lower heating value than diesel because it contains oxygen.	~10% lower heating value than diesel because it contains oxygen.	Mainly comprise of hydrogen and carbon.
Kinematic viscosity is higher.	Kinematic viscosity is in the same range as that of diesel.	Kinematic viscosity is lower (≈ 4 cSt at 40°C).
Less volatility.	Less volatile than diesel.	-

Source: Demirbas (2003)

Vegetable oils and animal fats are mainly triglyceride molecules, in which three fatty acid groups are attached to one glycerol molecule. The biodiesel can be produced from these triglycerides by process of transesterification. Transesterification is the chemical process of transforming the large, branched, triglyceride molecules of vegetable oils and fats into smaller, straight chain molecules, similar in size to the constituents of diesel (Muniyappa et al., 1996). The process uses vegetable oil and alcohol in presence of alkaline or acidic catalyst. A catalyst is usually used to improve the reaction rate and yield. Alcohol such as ethanol, methanol, or butanol can be used (Ma and Hanna, 1999; Canacki and Gerpen, 1999). A dense, liquid phase rich in glycerol is the by-product of this process and the fatty esters have cetane number and heating value closer to diesel.

Relatively few studies have directly dealt with the optimisation of the transesterification process (Freedman et al., 1986; Boocock et al., 1996; Noureddini and Zhu, 1997; Ma et al., 1999; Darnoko and Cheryan, 2000).

Currently used technologies for producing biodiesel can be classified into three categories:

- base catalysed transesterification with refined oils
- base catalysed transesterification with low fatty acid greases and fats
- acid esterification followed by transesterification of lower or high free fatty acid (FFA), fat and oils.

The majority of biodiesel is produced today through the base catalysed transesterification reaction because it involves low temperature and pressure processing, high conversion rates, no intermediate steps, and lower costs of processing materials. If a methanol molecules contact a fatty acid molecule, they will bond and form biodiesel molecule. The hydroxyl group from the catalyst stabilises the glycerine. The product esters named ethyl, methyl or butyl esters respectively have significantly lower viscosity and increased volatility relative to the triglycerides present in vegetable oils (Ma and Hanna, 1999; Canacki and Gerpen, 1999).

Figure 1 The transesterification reactions of vegetable oils

$$1.\quad \text{Triglyceride}\left(\text{TG}\right) + \text{R'OH} \underset{k_4}{\overset{k_1}{\Leftrightarrow}} \text{Diglyceride}\left(\text{DG}\right) + \text{R'COOR}_1$$

$$2.\quad \text{Diglyceride}\left(\text{DG}\right) + \text{R'OH} \underset{k_5}{\overset{k_2}{\Leftrightarrow}} \text{Monoglyceride}\left(\text{MG}\right) + \text{R'COOR}_2$$

$$3.\quad \text{Monoglyceride}\left(\text{MG}\right) + \text{R'OH} \underset{k_6}{\overset{k_3}{\Leftrightarrow}} \text{Glycerol}\left(\text{GL}\right) + \text{R'COOR}_3$$

Source: Ma and Hanna (1999)

Table 2 Various biodiesel standards applicable in different countries

Property	*BIS (IS 15670)*	*ASTM D6751*	*EN 14214*
Density at 15°C, kg/m^3	860–900	-	860–900
Kinematic viscosity at 40°C, cSt	2.5 to 6.0	1.9 to 6.0	3.5 to 5.0
Flash point (PMCC) °C, min	120	93 (closed cup)	Above 101
Sulphur, ppm (max)	50	15	10
Carbon residue, % by mass	0.05	0.05% mass (max)	0.3
Water content and sediment	500 mg/kg	0.05% vol (max)	500 mg/kg
Total contamination, mg/kg	24 ppm (max)	24 ppm (max)	24 ppm (max)
Sulphated ash content % (m/m)	0.02	200 ppm (max)	0.02
Copper strip corrosion (3 hours at 50°C)	Class 1 (max)	Class 3	Class 1 (max)
Cetane no., min	51 (min)	47 (min)	51 (min)
Acid value, mg KOH/g	0.5	0.50	0.5
Iodine value	NA	NA	120
Free gylcerol by % mass	0.02 % mass (max)	0.020 % mass (max)	0.02 % mass (max)
Total glycerol, by % mass	0.25 % mass (max)	0.240 % mass (max)	0.25 % mass (max)
Oxidation stability, 110°C (hr)	6 hours at 110°C (max)	3 hours at 110°C (max)	6 hours at 110°C (max)
Phosphorous, mg/kg	10 mg/kg	10 ppm (max)	10 mg/kg
Alkaline metals	NA	< 5 ppm Group I (Na, K) < 5 ppm Group II (Ca, Mg)	< 5 mg/kg Group I(Na, K) < 5 mg/kg Group II (Ca, Mg)

Simplified transesterification reaction is shown as below:

$$\text{Triglycerides} + \text{Free fatty acids} (< 4\%) + \text{Alcohol} \rightarrow \text{Alkyl esters} + \text{Glycerine}$$

Oil feedstocks containing more than 4% FFAs have to go through an acid esterification process step in order to increase the biodiesel yield. These feedstocks are filtered and preprocessed to remove water and contaminants, and then fed to the acid esterification process. The catalyst (sulphuric acid) is dissolved in methanol and then mixed with the pretreated oil. Fatty acid composition has the highest impact on the fuel properties like freezing point, oxidative stability, cetane number, NO_x emissions, etc.

Technically, biodiesel is a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fat, which meets prevailing standard specifications. Various countries have now evolved their own biodiesel standards in due course of time. Table 2 shows Indian, American, and European biodiesel standards.

1.1 Karanja oil

Karanja (*Pongamia pinnata*) is an oil seed bearing tree, and the oil is non-edible in nature and does not find any major suitable application. Only 6% is being utilised out of 200 million tons seeds available per annum in India (Biswas, 2002). The oil yield from the seeds is typically between 25–30% (w/w). Karanja is a native of humid and subtropical environments having annual rainfall ranging from 500–2,500 mm in its natural habitat. The maximum temperature ranges from 27–38°C and the minimum 1–16°C. It can grow on most soil types ranging from stony to sandy to clay, including verticals. It does not do well in dry sands. It is highly tolerant to salinity. It can be propagated either by seeds or by root suckers (Duke, 1983). The yield of kernels per tree is between 8 and 24 kg per year (Bringi and Mukerjee, 1987). The seed yield is approx 9–12 tons per hectare. Karanja is a native of the Western Ghats and is mainly found along the banks of streams and rivers or near the sea on beaches and tidal forests. It also grows in dry places far in the interior and up to an elevation of 1,000 m. It grows all over the country, from the coastline to the hill slopes. It has rich leathery evergreen foliage that can be used as green manure. After five years of age, the plant is expected to give economic yields and it may continue through to its life span of 100 years. When in bloom, the *Pongamia* trees can be used for bee keeping and honey production.

Various chemical and physical properties of Karanja oil is given in Table 3. Fatty acid composition of the oil is essential for determining the quantity of reactants and catalyst. FFA can be determined from the acid value. Dorado et al. (2002) found that the transesterification process would not occur if oils have FFAs content more than 3%.

Various attempts have been made to investigate the conversion of Karanja oil to fatty acid methyl ester i.e., biodiesel (De and Bhattacharyya, 1999; Karmee et al., 2004). A recent study by Vivek and Gupta (2004) suggested that the maximum yield of methyl ester from Karanja oil was up to 89% with molar ratio of methanol to oil (8–10: 1), with catalyst KOH (1.5% w/w_{oil}) while the reaction was conducted for 40 minutes at 68–70°C temperature. In the present study, the optimal reaction conditions and chemical kinetics were investigated to increase the yield of methyl ester from Karanja oil.

Table 3 Typical properties of Karanja oil

Colour	Dark brown
Odour	Repulsive
Refractive index at 40°C	1.4734–1.4790
Specific gravity at 30°C	0.925–0.940
Iodine value	80–96
Saponification value	117–195
Unsaponifiable matter	0.9–4.2%
Fatty acids	*Percentage*
Palmitic acid	3.7–7.9
Stearic acid	2.4–8.9
Arachidic acid	2.2–4.7
Oleic acid	44.5–71.3
Linoleic acid	10.8–18.3
Lignoceric	1.1–3.5
Eicosenoic	9.5–12.4
Behenic	4.2–5.3

2 Experimental procedure

In the present research, base catalysed transesterification (catalyst: KOH, NaOH) is used to prepare biodiesel from Karanja oil. Methyl alcohol of 99.5% purity (density: 0.792–0.792 kg/l) was used in the process.

For transesterification, one litre of Karanja oil was heated unto 65°C in round bottom flask. The FFA content of the oil was 2%. The catalyst is sodium hydroxide or potassium hydroxide and it dissolves in alcohol and then it is poured into the round bottom flask containing heated Karanja oil, while stirring the mixture continuously. Temperature was maintained for one hour and then reaction products were allowed to settle under gravity for six hours in a separating funnel. After esterification, the colour of Karanja oil changed from deep brown to reddish yellow. The products of transesterification process, i.e., Karanja oil methyl ester (biodiesel) and glycerol from upper and lower layers respectively. The glycerine is much denser than biodiesel. The catalysts and unused methanol were in the lower glycerol layer whereas fewer amounts of catalysts, methanol and glycerol were in the upper biodiesel layer (Meher et al., 2006). Once separated from the glycerine, the biodiesel goes through a clean-up or purification step to remove excess alcohol, residual catalyst and soaps. The biodiesel after separation was washed using same amount of hot distilled water (10% v/v at 60°C). The mixture was allowed to settle under gravity for six hours and lower layer of water containing impurities was removed. Washing was observed for both the catalyst with different rate of agitation.

This step is repeated thrice to completely remove catalysts, KOH and glycerol, if any, present in it. It is then dried and sent to storage. Washing of biodiesel is essential for the removal of the impurities and the residual catalyst, which may be harmful for the engines, when used as a fuel. In the experiments, transesterification and washing of biodiesel was thoroughly studied.

The glycerine by-product contains unreacted catalyst and soap that are neutralised with an acid. Water and alcohol are removed to produce 70–80% purity crude glycerine. The remaining contaminants include unreacted fats and oils. Schematic of experimental setup for the process of transesterification is shown in Figure 2.

Figure 2 Schematic of experimental setup

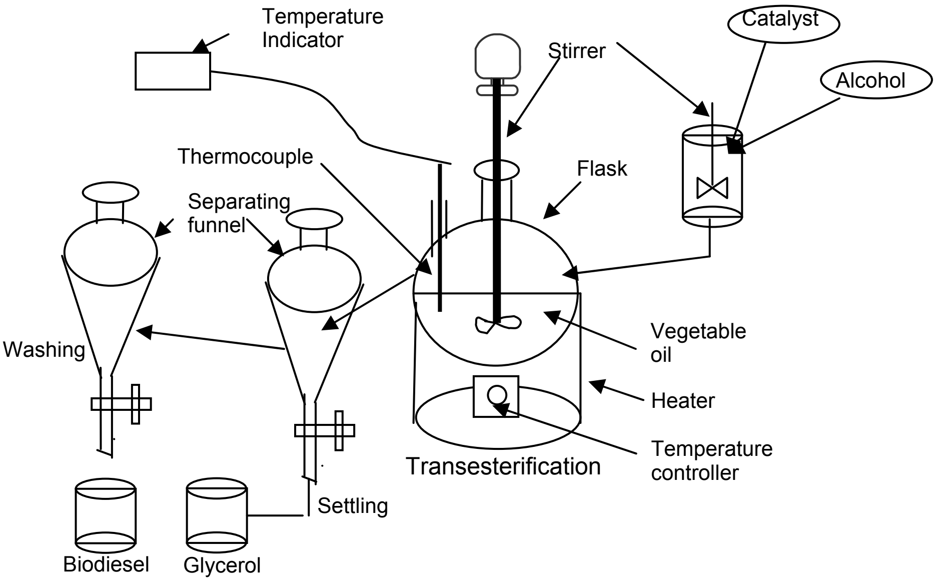


Figure 3 Biodiesel and Karanja oil (see online version for colours)

Process yield investigations were conducted with NaOH and KOH as a catalyst to study the effect of different parameters like reaction temperature, reaction time, stirring speed, molar ratio of alcohol to oil and amount of catalyst on ester yield as well as the extent of conversion (represented by the viscosity of ester). The reaction temperature was varied from room temperature to 70°C, molar ratio from 4.5:1 to 15:1, process time varied from 30 minutes to 120 minutes and the catalyst amount was varied from 0.25% to 1.25% (w/w_{oil}). Experiments for each set of variable on the ester yield were recorded, while keeping other factors constant. Ester yield results were related to the weight of oil at the starting. These four parameters were optimised for the transesterification of Karanja oil.

3 Results and discussion

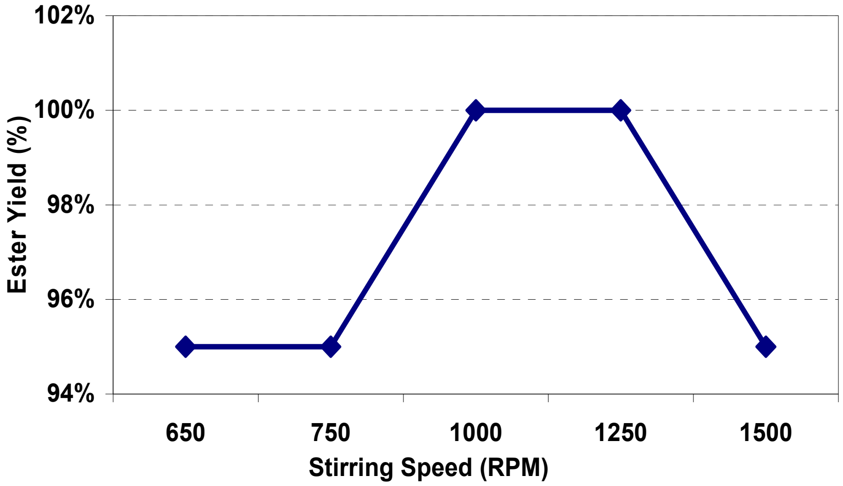
3.1 Transesterification parameters optimisation

KOH and NaOH were selected as the catalyst for the base catalysed transesterification of Karanja oil for further investigation. Reactions were performed under atmospheric pressure. Results of experiment conducted are shown graphically below.

3.1.1 Effect of stirring speed

The reaction was carried out at a methanol to oil molar ratio of 9:1, at a reaction temperature of 55ºC, 0.75% catalyst (KOH) and a reaction time of 60 minutes. The stirring speed of the mixture was varied from 650 rpm to 1,500 rpm. Figure 4 reflects the effect of stirring speed on the biodiesel yield, which varies between 95–100% (v/v_{oil}). Based on the above observation, it can be seen the 1,250 rpm stirring speed gives maximum product output, therefore, this is chosen as the optimum stirring speed for the remaining experiment.

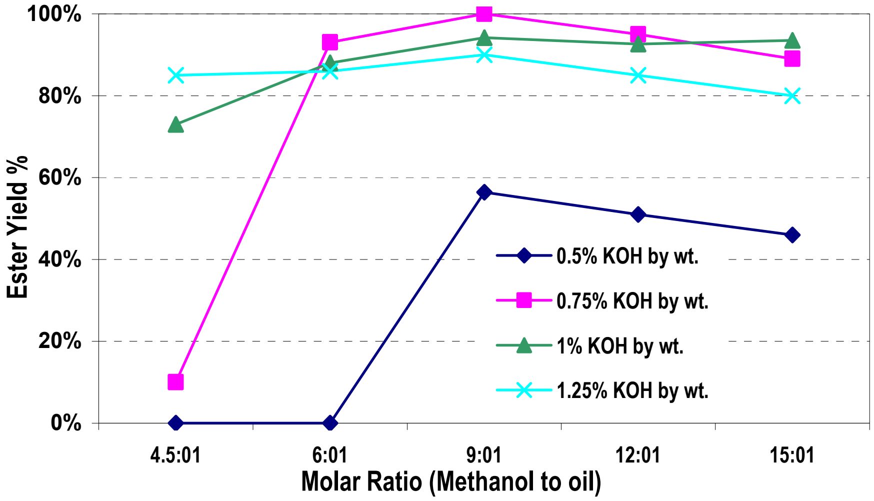
Figure 4 The effect of stirring speed on the biodiesel yield (see online version for colours)



3.1.2 *Effect of molar ratio*

The effect of molar ratio of methanol to oil is in the range of 4.5:1 to 15:1 was investigated keeping other process parameters fixed. The reaction temperature was kept at 55°C and the reaction was performed for one hour. The reaction was performed with different concentration of KOH ranging from 0.5% to 1.25% (w/w_{oil}) (Figure 5). It was found that ester yield increases with increase in molar ratio of methanol up until ratio 9:1 and for lower molar ratios; yield is sensitive to concentration of catalyst. When the amount of KOH was less (0.5% w/w_{oil}) then the yield is observed to be negligible, this is probably due to incomplete reaction of Karanja oil at such a low catalyst concentration.

Figure 5 Effect of molar ratio (methanol to oil) ester yield at different KOH concentration (see online version for colours)



3.1.3 *Effect of catalyst concentration*

The effect of KOH concentration was studied in the range of 0.25–1.5% (w/w_{oil}). The reaction temperature was kept constant at 55°C and reaction was performed for one hour. Ester yield is maximum when 0.75% of catalyst is used but decreases rapidly with the increasing weight concentration of KOH (Figure 6). This lower yield at high KOH concentration is probably due to soap formation. Excess KOH reduces the yield and also lead to undesirable extra processing cost because it is necessary to remove it from the reaction products at the end. With the increase in the concentration of catalyst, there was decrease in the yield of methyl esters. This was in accordance with the result obtained by Dorado et al. (2002) and was due to the formation of soap in presence of high amount of catalysts, which increased the viscosity of the reactants and lowered the yield.

The above figure shows the effect of yield on varying the concentration of NaOH (used as catalyst). Maximum yield is obtained at 0.75% of NAOH (Figure 7). The biodiesel obtained from NaOH has better properties like more clarity and less viscosity.

Figure 6 Effect of KOH concentration on ester yield for different molar ratio of alcohol to oil (see online version for colours)

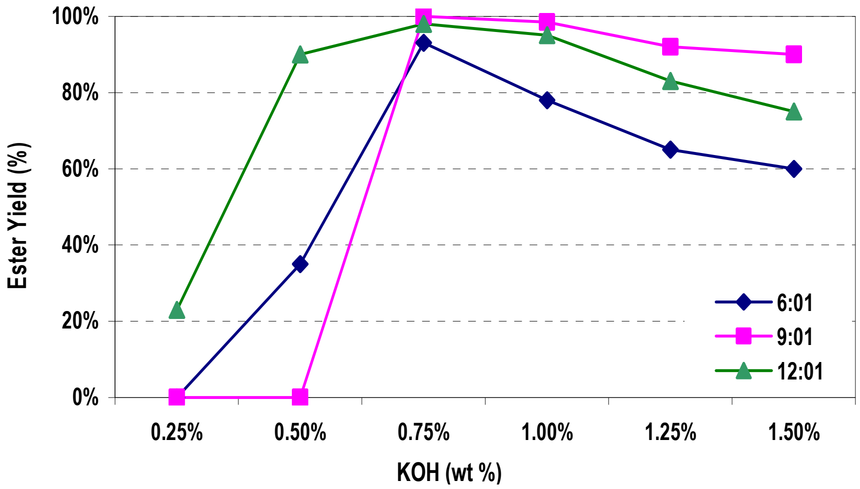
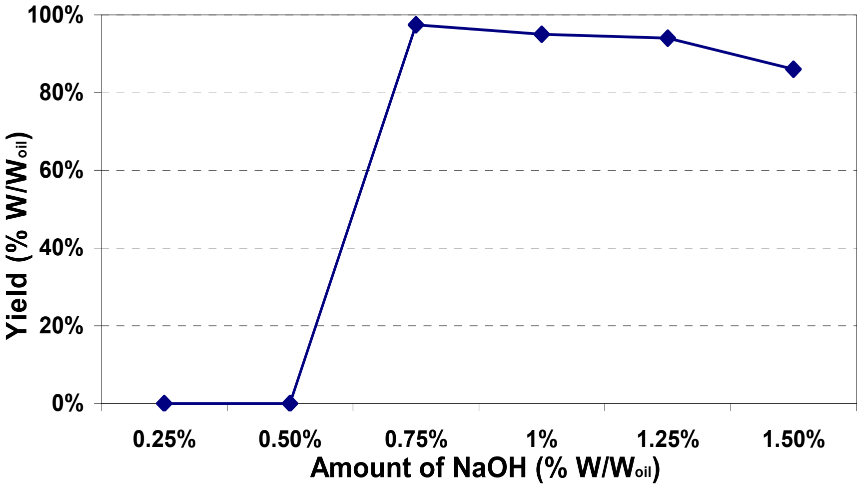


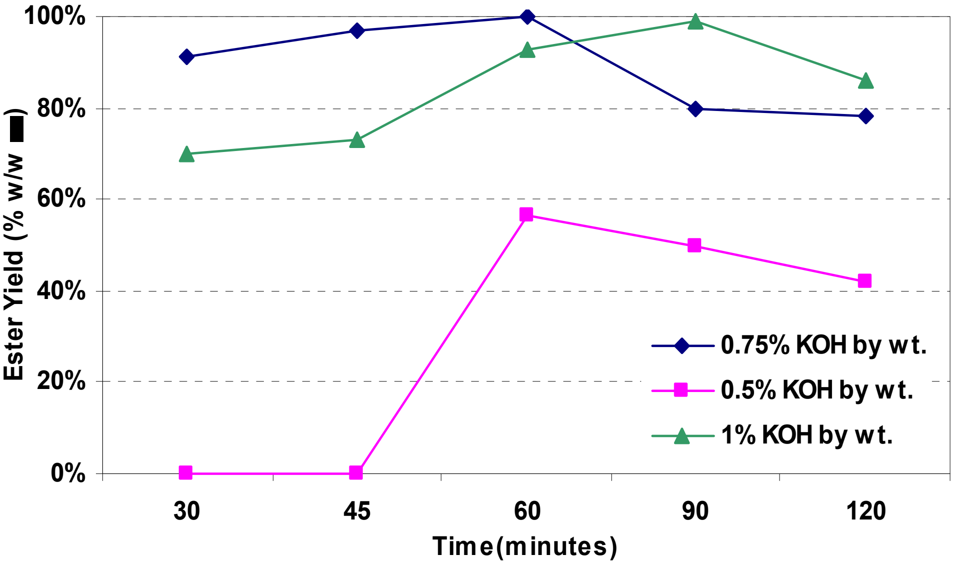
Figure 7 Effect of using NaOH as catalyst for 9:01 molar ratio (see online version for colours)



3.1.4 Effect of reaction time

Several investigators found that reaction starts very fast and almost 80% of conversion take place in the first five minutes. Thereafter, the reaction slows down significantly and after one hour, almost 93–98% conversion of triglycerides into ester takes place (Ma and Hanna, 1999; Srivastava and Prasad, 2000; Canacki and Gerpen, 1999; Alcantara et al., 2000). In the present work, effect of reaction time from 30–120 minutes on reaction yield was investigated. It was found that as the reaction time was increased beyond one hour, the ester yield decreases slightly (Figure 8).

Figure 8 Effect of reaction time on ester yield (see online version for colours)



3.1.5 Effect of reaction temperature

For studying the effect of temperature on the transesterification reaction, the reaction was performed at room temperature, 40, 50, 55, 60 and 70°C respectively. While the other parameters such as molar ratio of methanol to oil (9:1), reaction time (one hour), KOH (0.75% w/w_{oil}) were kept constant. The effect of reaction temperature on the ester yield is shown in above Figure 9.

Several researchers found that the temperature increase influences the reaction in the positive manner (Ma and Hanna, 1999; Srivastava and Prasad, 2000; Altin et al., 2001; Canacko and Gerpen, 1999). Dorado et al. (2002) found that ester yield slightly decreases above 50°C reaction temperature. It may be possibly due to the negative interaction between temperature and catalyst concentration. Due to side reaction of saponification, high process temperature tends to accelerate the saponification of the triglycerides by the alkaline catalyst before completion of the transesterification. In the present work, it was found that ester yield decreases as the reaction temperature increases above 55–60°C. It was found that 3–4 gentle washing with hot water (10% v/v) at 60°C removes all impurities and the biodiesel having pH of 7.0 can be produced for use in engines. It was found that ester has tendency to form emulsion on contact with water during catalyst removal process. Emulsion formation also depends on the level of agitation; this is the reason why prolonged and rigorous mixing is avoided. Loss of biodiesel due to formation of emulsion was found to be less when NaOH was used as catalyst.

Table 4 represent the typical values of various important fuel properties of the biodiesel produced from Karanja oil. This table suggests that the biodiesel produced meets the specifications given in Table 2.

Figure 9 Effect of reaction temperature on ester yield (see online version for colours)

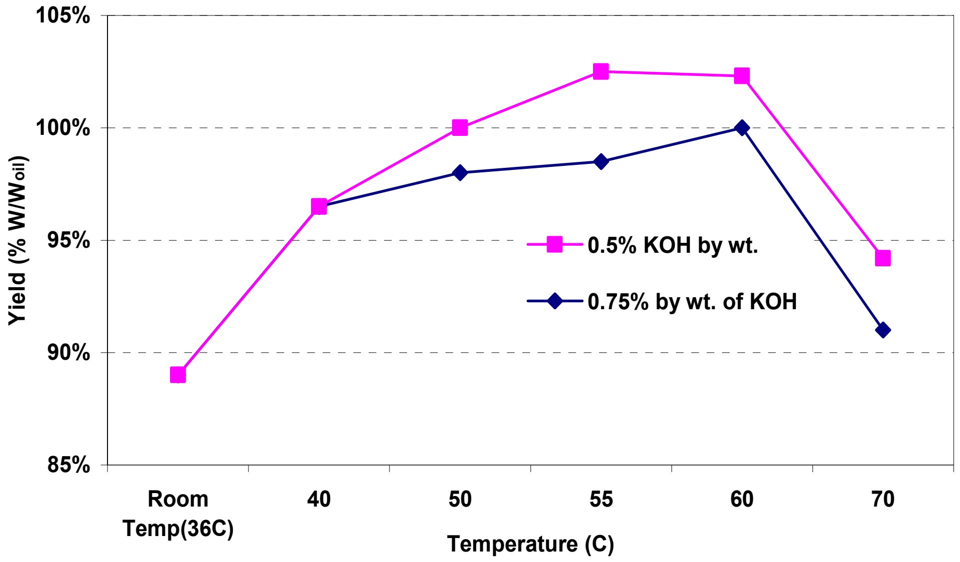


Table 4 Main properties of biodiesel produced from Karanja oil

Test	*Specifications*			*Results*
	Min	*Typical*	*Max*	
% Sulphated ash			0.020	0
Viscosity at 40ºC	1.9		6.0	4.2
Neutralisation value			0.80	0.35
Flash point (PMCC)	130			150
Appearance				Typical
% water and sediment			0.050	0

4 Conclusions

Base catalysed transesterification was studied with KOH and NaOH as catalyst for biodiesel production from Karanja oil. Transesterification as well as catalyst removal of biodiesel have been studied. Transesterification controls the yield of the process, while catalyst removal is required for purification of ester as fuel for diesel engine.

Effect of different parameters such as process temperature, reaction time, molar ratio of alcohol of oil and catalyst concentration on the ester yield was thoroughly investigated. It was found that 9:1 molar ratio of alcohol to oil is optimum for best biodiesel yield for all catalyst concentrations. Increasing the molar ratio beyond this value leads to reduction in biodiesel yield and may prove to be costly because the excess alcohol present in the product is removed by washing, leading to wastage of this expensive reactant. Catalyst in excess of 0.75% (w/w_{oil}) did not enhance the biodiesel conversion and it also led to a marked reduction in the biodiesel yield and added extra cost to the process of catalyst removal. Higher process temperature decreases the viscosities of the feedstock oils and has a positive influence on increasing the rate of transesterification and shortening the reaction time. However, higher temperature also induces a negative impact on the ester yield due to the acceleration of the saponification of triglycerides. Higher reaction temperature (i.e., > 60°C) had a negative impact on the product yield for Karanja oil. However, excess reaction time could not promote the conversion but favours the reverse reaction of transesterification (i.e., hydrolysis of esters), resulting in a reduction in the product yield. This also leads to increase in process energy input. Among all operational variables studied, catalyst amount and molar ratio of methanol to oil showed greater influences on the ester formation than reaction temperature and reaction time.

Therefore, it can be concluded that optimum biodiesel yield from Karanja oil (with FFA 2%) can be achieved at 9:1 molar ratio of methanol to oil, 0.75% (w/w_{oil}) KOH, 60°C reaction temperature and one hour reaction time followed by 3–4 times gentle washing of the biodiesel with 60°C distilled water (10% v/v).

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