Research Article

Environment friendly butyl ester biodiesel production from mahua oil: optimization and characterization

R. Sathish Kumar[1] · A. Krupa Vara Prasad[1]

Abstract
An attempt was made to optimize the key parameters of acid catalyst esterification and base catalyst transesterification processes for higher biodiesel yield from mahua oil with butanol using traditional optimization procedure. In esterification stage quantity of acid catalyst and molar ratio of butanol to mahua oil were considered for optimization of lowest value of free fatty acid (FFA). In transesterification stage molar ratio of butanol to mahua oil, quantity of alkaline catalyst, process temperature, process time and stirring speed were considered for maximum yield of mahua oil butyl ester. From the results, 6:1 molar ratio of butanol to mahua oil, 2% (w/w) concentrated sulphuric acid (H_2SO_4) were identified as optimized values with 1.1% FFA value which was reduced from 19.8%. In the second stage results, 6:1 molar ratio of butanol to oil, 1.5% (w/w) potassium hydroxide (KOH), 80 °C process temperature, 90 min process time and 500 rpm stirring speed were recorded as optimized values with 94.8% yield of mahua oil butyl ester. The butyl ester produced with optimum values of transesterification process parameters has been characterized and ensured the satisfaction of EN14214 biodiesel standards requirements.

Graphic abstract

Keywords Mahua oil · Esterification · Transesterification · Optimization · Butyl ester

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1 Introduction

Increased population, globalization, industrial revolution are the major reasons for increased power consumption and transportation in developing countries such as India. Due to high power consumption and increased transportation, two major threats faced by the globe for the past few decades are fossil fuel depletion and production of dangerous pollutants from fossil fuel combustion [1–3]. Hence, identification of alternative to fossil fuel, particularly to diesel has become most important nowadays. Biodiesel is such an auxiliary resource for fossil diesel in diesel engine applications. Mono alkyl esters of any fatty acid from triglycerides of vegetable oils or animal fats are known as biodiesel. Transesterification is one of the economic, robust, fast and simple chemical processes to produce biodiesel from triglyceride [4–7].

Characteristics and amount of alcohol and catalyst, process time and temperature, stirring or agitation speed, and moisture content in the raw materials are major parameters affecting the quality and amount of yield of biodiesel [8, 9]. Out of these parameters, the class of alcohol used affects the amount of yield and quality of biodiesel. The optimum level of all other parameters are also significantly affected by the class of alcohol used. For example, for the same oil and same catalyst with different alcohols will have different levels of other parameters for maximum yield of biodiesel [10, 11]. Methanol is widely used alcohol to produce biodiesel and ethanol to some extent. Other higher molecular weight alcohols like pentanol, propanol and butanol are very rarely utilized for biodiesel synthesis. Among them butanol has some interesting advantages such has higher miscibility with lipid resources, higher contribution to initial mass transfer in reaction, higher boiling point of butanol permits conduction of reaction process at higher temperature to achieve faster rate of reaction [12–15].

The value of acid number or quantity of free FFA content of raw oil is one of the key parameters to decide the suitable transesterification process. Higher FFA content of raw oil (2.5% and above) requires pre-treatment prior to transesterification process. In pre-treatment step esterification process is carried out with acid catalyst to reduce the acid value to required amount. If the value of FFA is not reduced less than 2% in the first step, further esterification also can be repeated. Once the amount of FFA is reduced considerably, transesterification process will be carried with base catalyst [7, 16–23].

The quality of the biodiesel is drastically affected by total count of carbon in the structure, amount of carbon double bond in the structure and quantity of unsaturated fatty acid of the resource oil. The relationship between physicochemical properties of biodiesel and chemical composition of the raw oil is very important to select the desirable resource for good quality biodiesel [24–30]. The characteristics of flow of the biodiesel at low temperatures are improved when the raw oil contains low level of saturated fatty acids. Raw oil with low unsaturated fatty acid group will improve the oxidation stability and cetane number of the biodiesel. The viscosity and density are two controversial properties. The degree of saturation and chain length of the raw oil are directly proportional to the viscosity and inversely proportional to the density. At the outset raw oils with high mono unsaturated and low in both saturated and poly unsaturated fatty acid will produce good quality biodiesel [31–38].

Biodiesel feedstock supply chain in India facing two major illnesses such as lack of cultivation and seed processing. The government policy on the utilization of waste land for cultivation of biodiesel feedstocks like Jatropha and other tree born feed stocks and investment policy of private sectors for cultivation should be jointly derived to overcome the above mentioned illnesses. The high quality planting seeds, latest cultivation technology, increased marketing opportunity, subsidy for cultivation and guaranteed profit for the formers are some strategies to be implemented for increase the supply chain of biodiesel feedstock.

The first generation biodiesel feedstocks are edible vegetable oils. Due to the increased population, the demand for edible crops has been increased many folds. Hence, the utilization of edible feed stocks for energy production is not encouraged. At the same time, the increase in greenhouse gas emission in the globe has been increased to the alarming level due to fossil fuel combustion. This overshooting of CO_2 level in the earth should be suppressed by replacing fossil fuel by renewable fuel. Hence, it is mandatory to produce non-edible feedstocks for bioenergy production and also it is important to share the available land for cultivating food products, livestock production and crops for bioenergy production equally.

Mahua (*Madhuca indica*) which belongs to sapotaceae family is tree born seed oil available widely in north and southern parts of India. From the literature it is found that mahua oil is rich in mono unsaturated and poor in both saturated and polyunsaturated fatty acid group, which is well proved best suitable to biodiesel production. But no literature is contributed for butyl ester production from mahua oil. Hence, an attempt was made to optimize the butyl ester production from mahua oil and this research

article is reporting the results of optimization of esterification and transesterification process parameters of butyl ester production from mahua oil.

2 Materials and method

2.1 Materials and experimental setup

The well ripened mahua seeds were collected from the local villages in and around Chennai, India. 98% pure concentrated sulphuric acid, pellet form laboratory grade potassium hydroxide with 85% purity and 99% pure butanol were used in this experimental work. The experimental setup consists of a 500 ml capacity borosilicate glass reactor with three necks, speed governable mechanical stirrer with Remi stirrer motor carrying glass shaft and Teflon blades, heating mantle with temperature regulator and a thermometer of 0–100 °C range. The schematic diagram of the experimental setup is shown in Fig. 1.

2.2 Oil extraction and biodiesel preparation procedure

The collected mahua seeds were dried in the sunlight for about 48 h and the shells were removed manually. Then the dried kernels were crushed in a mechanical oil crusher and raw oil was collected. The collected oil was filtered using fine mesh cloth filter for removing the solid contaminants and heated in a beaker up to nearer the boiling point to evaporate the water content present in the oil and cooled down to room temperature. The flow chart showing the steps involved in mahua oil butyl ester production is given in Fig. 2.

In the esterification process, two key process parameters such as butanol to oil molar ratio and quantity of acid catalyst were taken for optimization and other parameters like process temperature, process time and stirring speed were kept constant as 60 °C, 1 h and 500 rpm respectively. 100 (± 0.1) grams of pre-treated oil was placed in the reactor and gradually heated up to the necessary temperature under the fixed stirring speed. Meanwhile the essential amount of butanol and concentrated H_2SO_4 were exactly measured and mixed thoroughly and added to the oil in the reactor once the required temperature was attained. This time was noted as the commencement time of the reaction. Once the decided time was attained the reactants were transferred to settling beaker and settled for 12 h. The top layer of butanol and water emulsion was removed and the bottom esterified product was used for testing the FFA value. Until the FFA value is reduced less than 2%, the process parameters were optimized.

In Transesterification process, five foremost process parameters such as molar ratio of butanol to mahua oil, quantity of KOH, process temperature, process time and stirring speed were considered for optimization. 100 (± 0.1) grams of esterified product was taken in the reactor and heated up to the necessary temperature under the prefixed stirring speed. The required quantity of butanol and KOH pellets were exactly measured and mixed thoroughly and added to the oil in the reactor once the required temperature was attained. This time was noted as the commencement time of the transesterification reaction. Once the decided time was attained the reactants were transferred to settling beaker and undisturbed for 24 h.

The optimal value of a particular parameter in the esterification and transesterification process was obtained by conducting a series of experiments with different values of that particular parameter and keeping the other

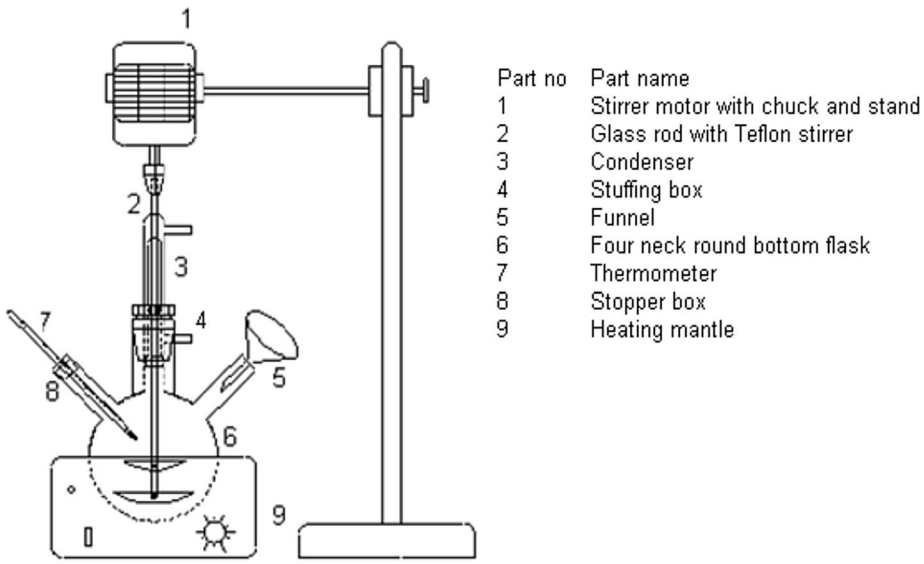

Fig. 1 Experimental setup for the batch type transesterification process

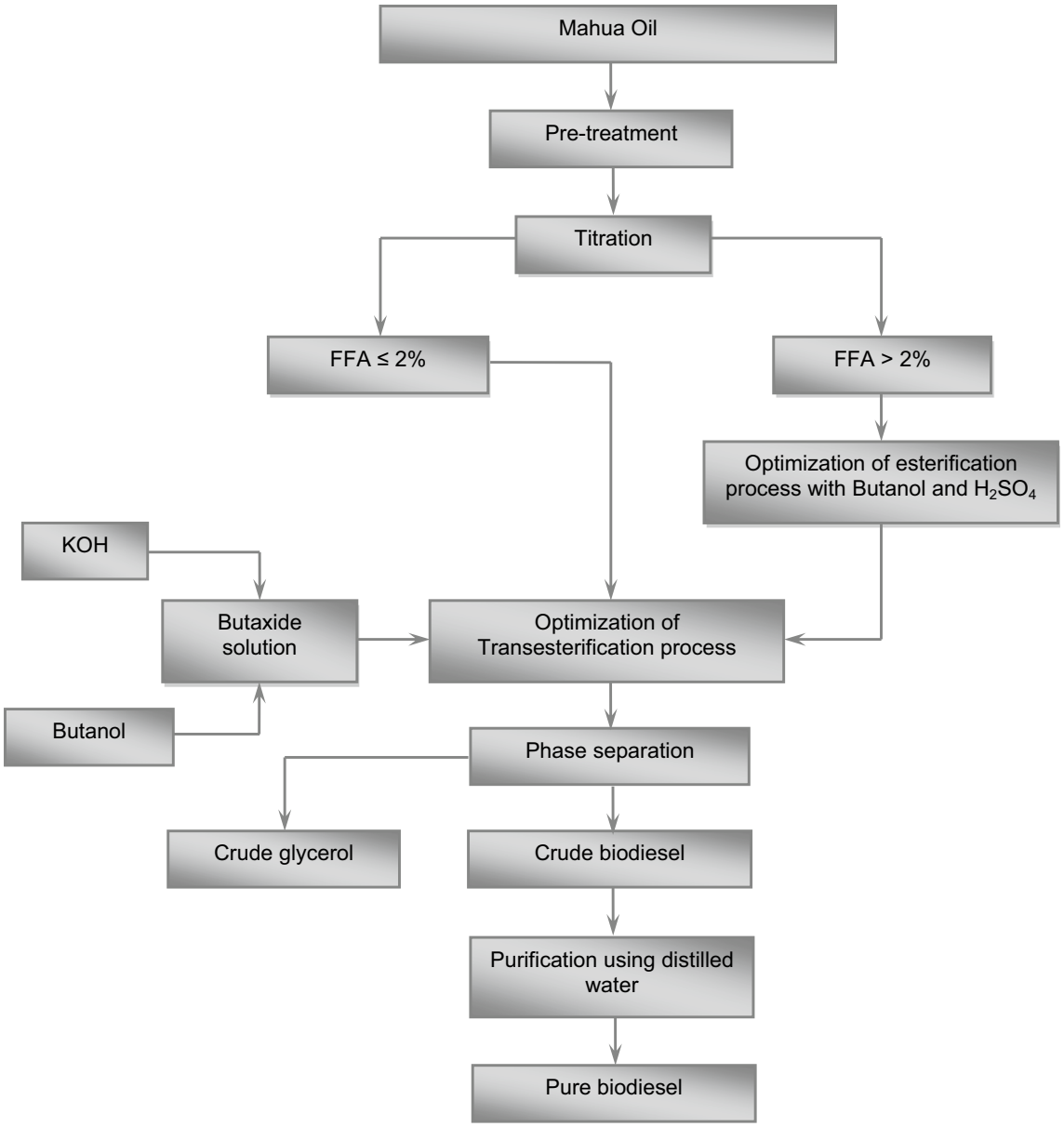

Fig. 2 Process flow chart of biodiesel production from mahua oil

parameters constant at any random value [13]. Once the optimal value was attained for that particular parameter, that value was fixed constant for the optimization of the other parameters [13].

2.3 Oil and biodiesel characterization

Fatty acid profile of the raw mahua oil was estimated using a Perkin-Elmer Clarus 500 Auto System XL with elite series PE-5 capillary column equipped with mass spectrometer. Kinematic viscosity was measured at 40 °C using a Brookfield DV-II Proviscometer as per the procedure of ASTM D 445. The pour point and the cloud point were simultaneously estimated in accordance with ASTM D 5949 and ASTM D 5773 respectively. Flash point was measured using Pensky Martene open cup apparatus. Heating value was determined with the use of Parr—6772 bomb calorimeter. Density at 15 °C was measured using a Rudolph DDM 2909 Automatic Density Meter. The values of iodine number and cetane number were calculated as per the standards of ASTM. The acid value was determined using a suitable titration with standardized KOH solution with phenolphthalein as the indicator [34].

Table 1 Fatty acid composition of mahua oil

Fatty acids	Molecular formula	Saturated/unsaturated	Content (%)	Molecular weight (g/mol)
Palmitic acid (C16:0)	$C_{16}H_{32}O_2$	Saturated	20.4	256.40
Stearic acid (C18:0)	$C_{18}H_{36}O_2$	Saturated	13.6	284.48
Oleic acid (C18:1)	$C_{18}H_{34}O_2$	Monounsaturated	46.5	282.46
Linoleic acid (C18:2)	$C_{18}H_{32}O_2$	Polyunsaturated	17.8	280.45
Linolenic acid (C18:3)	$C_{18}H_{30}O_2$	Polyunsaturated	0.8	278.43
Arachidic acid (C20:0)	$C_{20}H_{40}O_2$	Saturated	0.9	312.53

Table 2 Physicochemical properties of mahua oil

Parameters	Values
Density at 15 °C (kg/m^3)	932
Specific gravity	0.932
Kinematic viscosity at 40 °C (mm^2/s)	42.32
Free fatty acid (% FFA as oleic acid)	19.8
Iodine value (g Iodine/100 g)	74
Acid value (mg KOH/g)	39.6
Cetane number	56
Color	Greenish yellow
Peroxide value (g/kg O_2)	188.64
Molecular weight (g/mol)	869.95
Physical state at room temperature	Liquid
pH	4.1
Cloud point (°C)	14
Pour point (°C)	9
Flash point (°C)	228
Fire point (°C)	244
Calorific value (kJ/kg)	37,980

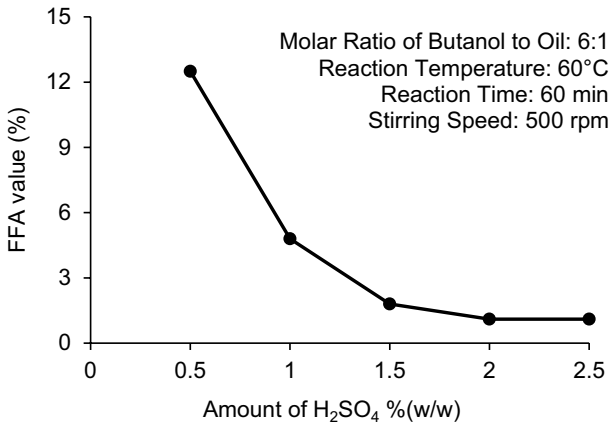

Fig. 3 Effect of amount of H_2SO_4 on FFA value

3 Result and discussion

3.1 Fatty acid profile and physicochemical properties of mahua oil

The fatty acid profile and their weight content in percentage of mahua oil were presented in Table 1. From Table 1 it was found that oleic acid was the dominating fatty acid which shares 46.5% in the total weight of mahua oil. The total amount of saturated fatty acid content was 34.9%, total amount of monounsaturated fatty acid content was 46.5% and the total amount of polyunsaturated fatty acid content was 18.6%. From the above results it was perceived that mahua oil is rich in monounsaturated fatty acid, hence, suitable for high quality biodiesel production. Table 2 shows the physicochemical properties of raw mahua oil are at par with other vegetable oils. A crucial property to decide the suitability of raw oil as fuel for diesel engine is kinematic viscosity and its value for mahua oil at 40 °C was 42.32 (mm^2/s). It was approximately 15 times greater than the fossil diesel, therefore raw mahua oil is not suitable for diesel engine application as fuel. Hence it is to be converted as biodiesel through two step transesterification process because the FFA content of the oil was 19.8 (% FFA as oleic acid).

3.2 Optimization of esterification process parameters

3.2.1 Effect of amount of acid catalyst

The variation of the FFA with the respective amount of acid catalyst is shown in Fig. 3. In this study the amount of H_2SO_4 was varied from 0.5% (w/w) to 2.5% (w/w) in the incremental step of 0.5%. While other parameters were kept constant, a 6:1 molar ratio of butanol to oil, at 60 °C, with 60 min of reaction time and 500 rpm stirring speed was used during the esterification. From the Fig. 3 it was perceived that the conversion rate of FFA to esters was greatly influenced by the variation in amount of H_2SO_4. It was identified that there was no appreciable reduction

in FFA at 0.5% catalyst concentration and around 75% of FFA were converted into esters at 1% catalyst concentration. At 0.5% catalyst concentration the rate of conversion was very low because of inadequate amount of catalyst to speed up the process. The optimum level of acid catalyst amount was identified as 2% with corresponding optimum FFA value of 1.1% and further increase in amount of catalyst had no effect in FFA conversion, because the maximum conversion has already happened and process has been stagnated therefore no improvement in the conversion.

3.2.2 Effect of butanol to oil molar ratio

Figure 4 shows the effect of butanol to oil molar ratio on the conversion of FFA into esters. In this set of experiments the molar ratio of butanol to oil was varied from 3:1 to 7:1. The amount of acid catalyst was set to its optimal level obtained in previous sections and other parameters were set as same as in the previous section. From the Fig. 4 it was identified that the rate of reduction of FFA value was greatly affected by the amount of butanol. At the molar ratio levels 3:1 and 4:1 there were no significant reduction in FFA value, this is due to the inability of lower amount of butanol to hold the faster rate of reaction. The rate of conversion of FFA to ester at the molar ratio levels 5:1 and 6:1, was very high and the optimum level was identified as 6:1. The value of free fatty acid at molar ratio 6:1 was recorded as 1.1%. Beyond 6:1 molar ratio the further increase in amount of butanol had no effect on FFA conversion, because the higher amount of alcohol present in the esterification process will lead to water formation during reaction.

3.3 Optimization of transesterification process parameters

3.3.1 Effect of butanol to oil molar ratio on butyl ester yield

The amount of alcohol plays a major role and has greater influence on biodiesel yield in transesterification process. The stoichiometric molar ratio of alcohol to any triglyceride for transesterification is 3:1, but the transesterification process is highly reversible in nature. Therefore excess alcohol is needed to maintain the rate of forward reaction. At the same time, very high amount of alcohol in the process will emulsify the reaction due to the water formation during reaction. Hence, identification of optimum amount of alcohol is very much important. The effect of amount of butanol on butyl ester yield was shown in Fig. 5. In this study the butanol amount was varied from molar ratio 4:1 to 12:1 in the interval of 2. While other parameters were kept constant, a 1% KOH, at 60 °C, with 60 min of reaction time and 500 rpm stirring speed was used during the transesterification. It was clearly identified from the Fig. 5 that highest biodiesel yield was attained as 92.8% at 8:1 molar ratio. Since butanol is less reactive compared to methanol, higher amount of butanol was consumed in this process. If the molar ratio is increased beyond 8:1, slight drop in yield of biodiesel was noticed. This is due to the water formation of excess alcohol during the reaction which will reduce the rate of conversion. Even though 8:1 molar ratio recorded high percentage of butyl ester yield, 6:1 was considered as optimized level due to the reason that it produced result at par with 8:1.

3.3.2 Effect of catalyst amount on butyl ester yield

Figure 6 depicts the effect of variation of catalyst amount on the variation of butyl ester yield. Investigations were made by varying the catalyst concentration from 0.5 to

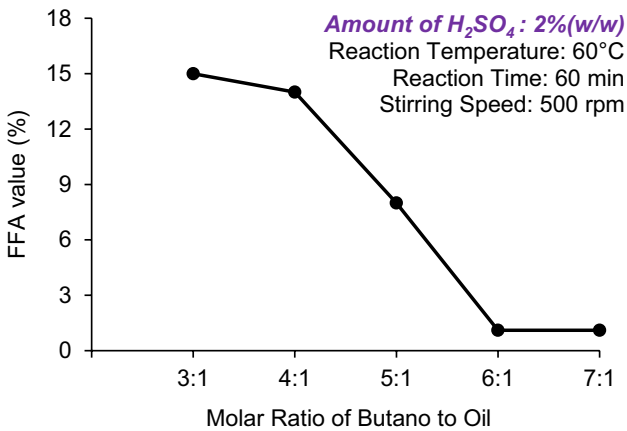

Fig. 4 Effect of molar ratio of butanol to oil on FFA value

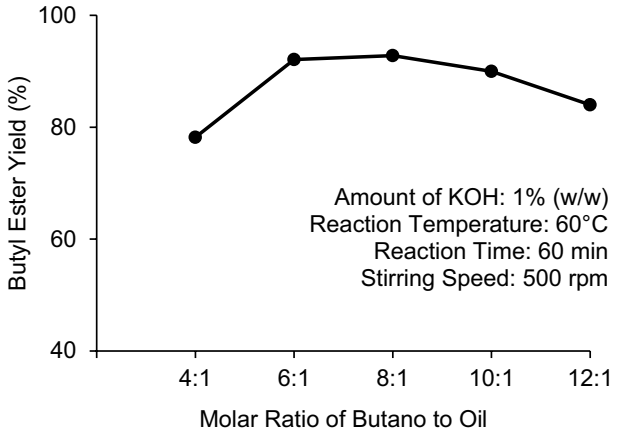

Fig. 5 Effect of molar ratio of butanol to oil on butyl ester yield

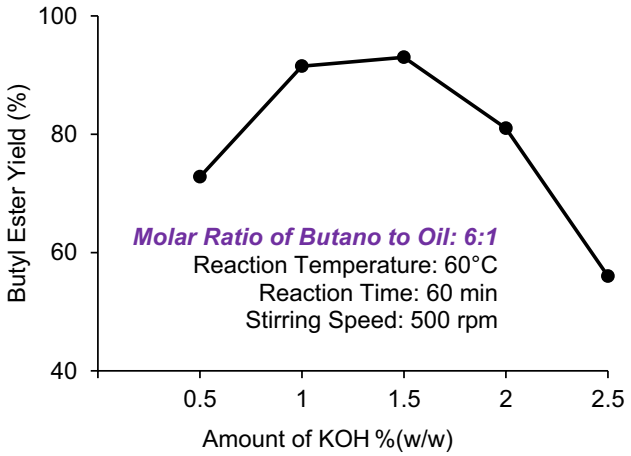

Fig. 6 Effect of amount of KOH on butyl ester yield

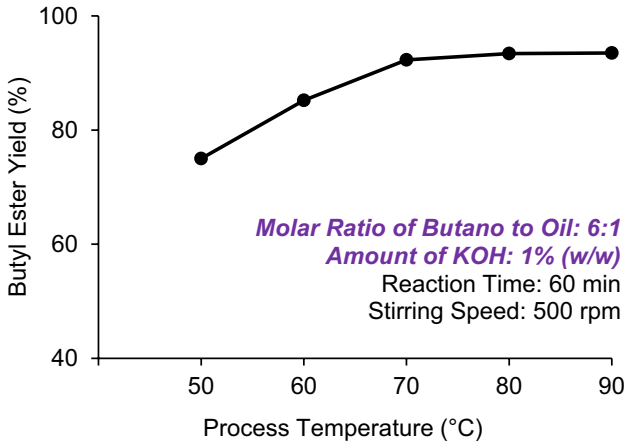

Fig. 7 Effect of process temperature on butyl ester yield

2.5% (w/w). The optimum value of butanol to oil molar ratio as 6:1 was set and other parameters were kept constant as same as previous section. Catalyst concentration had high influence than that of all other parameters which was evident from Fig. 6. This is due to the rate of change of biodiesel yield with respect to variation of catalyst concentration was very high. 0.5% catalyst amount was inadequate to aggravate the reaction process and hence, only 72.8% yield was produced. The maximum biodiesel yield was recorded as 93% for the catalyst concentration 1.5%. Further increase in catalyst amount had negative effect on reaction and the butyl ester yield was significantly reduced and also higher amount of soap formation was noted. This is due to the higher rate of saponification reaction of excess KOH with triglycerides.

3.3.3 Effect of process temperature on butyl ester yield

The temperature of the transesterification reaction is one of the key parameters to improve the biodiesel conversion. Higher the temperature higher the yield in general, because higher temperature leads to easy braking of cohesive bonding between triglyceride molecules and make them active in transesterification conversion. At the same time if the temperature is set greater than the boiling point of any reactant will lead to evaporation of that reactant from the process and further reduces the yield of biodiesel. The higher boiling point of butanol helps in conducting the experiments with high temperature. Figure 7 shows the variation of butyl ester yield with respect to variation of temperature. Five different temperatures were set between 50 and 90 °C with 10 °C interval. The optimal values of molar ratio and catalyst concentration obtained from previous sections were adopted and other factors were kept constant. The optimal value of biodiesel yield as 93.5% at 90 °C. At the same time the biodiesel yield for 80 °C was 93.4%. The difference between the biodiesel yield at temperature 80 °C and 90 °C is 0.1% which is very meager. The energy required for raising 10 °C temperature is very significant than the increase in the yield, hence the optimal value of temperature is considered as 80 °C.

3.3.4 Effect of process time on butyl ester yield

The rate of transesterification reaction in the beginning of the process is high. More than 50% of ester conversion will happen in first 10 to 15 min of reaction. On the other hand, the optimum yield will be attained very late due to hydrolysis reaction of oil and alcohol in later stage. Hence, the identification of optimal reaction time is very important for biodiesel yield. The amount of butyl ester yield at various process times are given in Fig. 8. Except stirring speed all other parameters were set to their optimal values and stirring speed was set to 500 rpm. The experiments were conducted at five different reactions times.

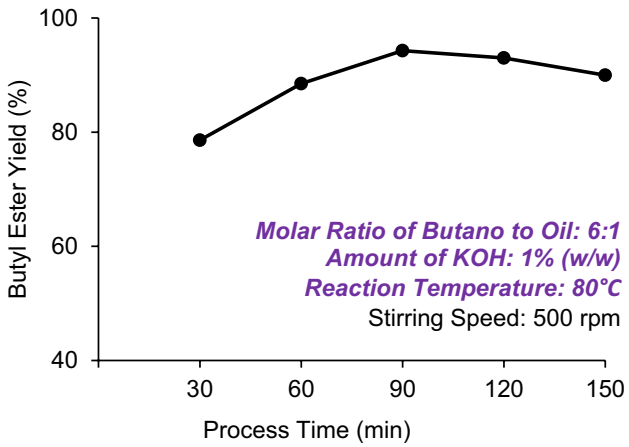

Fig. 8 Effect of process time on butyl ester yield

The optimal value of yield of butyl ester was obtained at 90 min reaction time. Long reaction time beyond 90 min recorded lower biodiesel yield. This is due to the initiation of hydrolysis reaction by the additional residence time of reactants. The backward reaction was initiated at longer reaction times and more soap formation was also identified at the end of the reaction.

3.3.5 Effect of stirring speed on butyl ester yield

Triglyceride and alcohol are two immiscible liquids taking part in the transesterification process. Therefore the area of contact between the oil and alcohol is very limited. The area of contact between alcohol and oil will be increased by continuous stirring or agitating the reactants at optimal speed. The change in butyl ester yield with respect to the change in stirring speed is shown in Fig. 9. The stirring speed in this set of experiments was varied from 300 to 700 rpm with 100 rpm incremental step. All other parameters were set to their optimal levels. At lower speeds such as 300 rpm and 400 rpm, the yield value of biodiesel was around 80 to 85% only. This is due to the reason that at lower speeds the level of heterogeneity of the oil alcohol mixture was not reduced reasonably and the level of homogeneity of the mixture was not appreciably improved. At the same time further increase in stirring speed beyond 500 rpm had negative effect. There was reduction in yield of biodiesel at speeds greater than 500 rpm. This is due to that during the experiments with higher stirring speeds, splashing of reactants all around the reactor was noticed and hence, the improper interface between the oil and alcohol had pulled down the yield value. The results showed that the optimal value of butyl ester yield was recorded as 94.8% for 500 rpm. This is because at optimal speed of mixing the mass transfer of triglyceride from oil to interface with alcohol is appreciably increased and also the kinetics of transesterification process was altered and better results were attained.

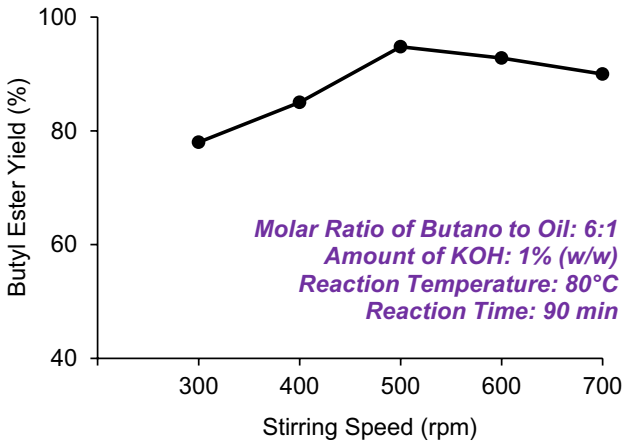

Fig. 9 Effect of stirring speed on butyl ester yield

3.4 Properties of mahua oil butyl ester

Key physicochemical properties of mahua oil butyl ester were estimated using ASTM standards and reported in Table 3. The comparison between estimated properties and EN 14214 biodiesel standards were also reported in Table 3. The major properties of the mahua oil butyl ester are comparable with other biodiesels [14–16]. From the results it was confirmed that the requirements of EN14214 biodiesel standards were met by all the properties of mahua oil butyl ester and hence, it can be a suitable alternate fuel to fossil diesel in diesel engine applications.

Table 3 Properties of mahua oil butyl ester (MOBE)

Properties	Units	EN 14214	MOBE	Diesel	Test method
Ester content	% (m/m)	Min 96.5	94.8	–	EN14103
Density at 15 °C	g/cm^3	0.86–0.90	0.894	0.861	ASTM D4052
Kinematic viscosity	mm^2/s	3.5–5	4.12	2.96	ASTM D445
Acid value	mg KOH/g	Max 0.50	0.81	0.18	ASTM D664
Iodine value	g iodine/100 g	Max 120	72	–	AOAC CD1-25
Cloud point	°C	Max 0	4	– 12	ASTM D97
Pour point	°C	Max 0	2		ASTM D97
Flash point	°C	Min 120	198	48	ASTM D93
Fire point	°C	Min 120	210		ASTM D93
Calorific value	MJ/kg	Min 35	38.86	44.8	ASTM D240
Cetane number	–	Min 51	52	51	ASTM D613
Sulphur content	mg/kg	Max 10	0	350	ASTM D5459

4 Conclusion

In this experimental analysis the oil extraction, fatty acid profile, physicochemical properties, and optimization of mahua oil butyl ester production where investigated and reported. The FFA value of the raw mahua oil was estimated as 19.8%, hence, two step transesterification process was adapted. In the esterification step two significant parameters such as butanol to oil molar ratio and amount of acid catalyst were optimized. In the second step, key transesterification process parameters such as molar ratio of butanol to oil, amount of alkaline catalyst, process temperature, process time and stirring speed were optimized. Based on the experimental results the following major conclusions were made.

- Oleic acid was the dominating fatty acid which shares 46.5% in the total weight of mahua oil.
- Mahua oil is rich in monounsaturated fatty acid hence, suitable for high quality biodiesel production.
- 6:1 molar ratio of butanol to mahua oil, 2% (w/w) concentrated H_2SO_4 were identified as optimized values with 1.1% FFA value which was reduced from 19.8%.
- 6:1 molar ratio of butanol to oil, 1.5% (w/w) KOH, 80 °C process temperature, 90 min process time and 500 rpm stirring speed were recorded as optimized values with 94.8% yield of mahua oil butyl ester.
- These optimized parameters are well suitable for batch type biodiesel production industries of small and medium scale capacity and will not be suitable for continuous production.
- The properties of the mahua oil butyl ester observed are in compliance with the EN 14214 biodiesel standard.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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