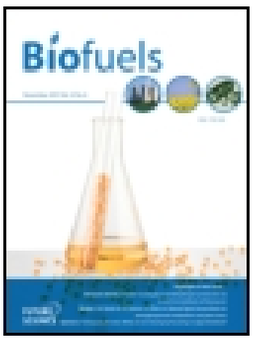

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RESEARCH ARTICLE

Microwave-assisted batch synthesis of *Pongamia* biodiesel

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Venkatesh Kamath[1], I Regupathi[†1] & MB Saidutta[1]

Background: The major bottleneck of biodiesel synthesis is its cost and this is mainly attributed to the feedstock material. *Pongamia pinnata* oil is a nonedible oil that is available in plenty in India and has negligible applications. Several methods of synthesis have been established, each having their own advantages and disadvantages. **Results & discussion:** Biodiesel from high-free fatty acid, nonedible, *Pongamia* oil was synthesized under microwave irradiation with single- and two-step methods. Experimental investigations showed that although the single-step method had a high yield (80%), the acid value of biodiesel was quite high. Hence, the two-step method seems to be a better approach as it yielded 90%, with 1:10 oil:methanol molar ratio and 1 wt% KOH. Along with a decrease in the reaction time to 4–5 min, separation time was also decreased by at least 90%. A conventional heating method was employed to compare the effects of microwave irradiation on biodiesel synthesis. **Conclusion:** The results indicate significant improvement in the yield, reaction time and processing time of biodiesel under microwave irradiation. The synthesis of *Pongamia* biodiesel under microwave irradiation could perhaps lead to cost effective and faster technology in countries such as India.

Biodiesel (fatty acid methyl or ethyl ester [FAME or FAEE]) synthesized from edible or nonedible oil through acid- or base-catalyzed **transesterification** has been the core of biofuels research for the past few years. Biodiesel production from edible oil has placed a strain on food production, price and availability. Developing nations, such as India, are importing their edible oil requirements. Hence, the search for additional regional biodiesel feedstock is an important objective. Azam *et al.* have discussed available trees, shrubs and herbs in India that could be exploited for biodiesel production [1]. Nonedible oils, such as ***Pongamia pinnata*** and *Jatropha*, are available in the Asian subcontinent in large quantities. In addition, these trees can be cultivated in waste land with variable saline levels. *Pongamia pinnata* (Karanja or Honge) is a medium-sized (18 m) deciduous tree that grows fast in humid and subtropical environments and matures after 4 to 7 years to provide fruit that contains one to two kidney-shaped kernels [1,2]. The oil content of Karanja kernels ranges between 30 and 40 wt%. The primary fatty acid found in Karanja oil is oleic acid (~50 wt%), followed by linoleic, palmitic and stearic acids [1,2].

In a typical method of biodiesel synthesis, the oil is reacted with an alcohol in the presence of a catalyst. Alkaline and acidic catalysts are the most widely used catalysts for homogeneous transesterification reactions. Schuchardt *et al.* have discussed the mechanism of base- and acid-catalyzed reactions in detail [3]. Base-catalyzed reactions are faster than acid-catalyzed reactions and high yields can be obtained with 1:6 to 1:12 oil:alcohol molar ratio [2,4–6]. **Free fatty acid** (FFA) content of oil is one of the major factors that affect biodiesel yields. In base-catalyzed transesterification of oil containing high FFA (>3%), due to the presence of hydroxyl ions the FFA react with the base to form soap, eventually reducing the yield of FAME or FAEE [3,7]. There are two simple, and most commonly

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Key terms

Biodiesel: Mono-alkyl or ethyl esters of long-chain fatty acids derived from edible or nonedible oil of plant, animal or microbial origin.

Transesterification: Chemical process in which an alcohol reacts with triglyceride esters to produce glycerol and the alcohol esters that constitute biodiesel or fatty acid methyl ester.

Pongamia: A nonedible, high-free fatty acid oil having less usage and available in plenty in Asian countries, also known as karanja oil in India.

Free fatty acids: Long-chain carboxylic acids present in oil owing to oxidative decomposition of triglycerides; and determine the quality of oil.

Microwave: Electromagnetic radiation in the frequency range of 300 MHz to 30 GHz (1–0.01 m wavelength).

used, strategies cited in the literature to overcome this problem. Either a single-step process, where base catalyst is added in excess or a two-step process in which an acid pretreatment step is followed by a base-catalyzed transesterification step [6,8–10]. Reduction in FFA by acid pretreatment followed by base catalysis gives a greater yield compared with single-step base transesterification when the FFA content of oil is very high [9,10].

Various biodiesel synthesis methods, such as lipase-catalyzed methods, supercritical methanol, ultrasonic methods and **microwave** irradiation have been used [11–16]. In the most commonly employed conventional method of transesterification, heat is supplied through conduction, convection and radiation to the reacting system necessitating a medium of heat transfer that consumes large amounts of energy [17]. Microwave-assisted synthesis is faster, takes less than 5–6 min, gives higher yields, and produces fewer byproducts [15,16,18]. Separation of the glycerol layer is easy and fast [15].

Microwaves are electromagnetic radiation in the frequency range of 300 MHz to 30 GHz (1–0.01 m wavelength). Domestic microwave ovens operate at 2450 MHz. Microwaves are nonionizing radiation influencing molecular motions (ion migration or dipole rotation) without altering molecular structure [19,20]. An applied electromagnetic field forces the dipoles to rotate and ions to migrate [21]. Rapid reversal of electric fields (at a rate of 2450 MHz) cause the orientation and re-orientation of these molecules, which results in heating effects. Polar solvents of low molecular weight and high dielectric constant (e.g., lower alcohols) under microwave irradiation, show a rapid rise in temperature, reaching their boiling point quickly [15,19]. In contrast to conventional heating, in microwave dielectric heating the microwave energy is introduced into the chemical reactor remotely, that is, only the reactants get heated and not the reactor. The temperature of a sample can be maintained uniform throughout the system and this eventually reduces the possible side reactions [21].

In the present investigation, *Pongamia* methyl ester or *Pongamia* biodiesel were synthesized under microwave irradiation. Biodiesel was synthesized using homogeneously catalyzed transesterification of crude and pretreated *Pongamia* oil. KOH was used as catalyst for the transesterification reaction. Pretreatment of crude *Pongamia* oil was carried out through microwave-assisted esterification of free fatty acid content of oil with sulfuric acid as catalyst. Studies were carried out at various oil to methanol molar ratios, microwave power and irradiation time. The influence of microwave irradiation over *Pongamia* biodiesel synthesis is reported and compared with the conventional heating technique. The quality of biodiesel was analyzed using thermogravimetric technique.

Experimental

Materials

Commercial grade methanol was purchased from Rankem (Mohali, India). KOH pellets and anhydrous sulfuric acid were purchased from Merck (Mumbai, India). *Pongamia* oil was purchased from a local department store. Reference standards of analytical grade, such as oleic acid, methyl ester of oleic acid, and mono-, di- and triglyceride mix, were purchased from Merck. All other chemicals were of reagent grade and used without any further modification.

Microwave apparatus

A domestic microwave oven (Samsung M183DN) 800 W, 2450 MHz was used with modification as shown in Figure 1 for all microwave-mediated experiments. Teflon base was provided as support by replacing the carousel plate in such a way that the carousel axis

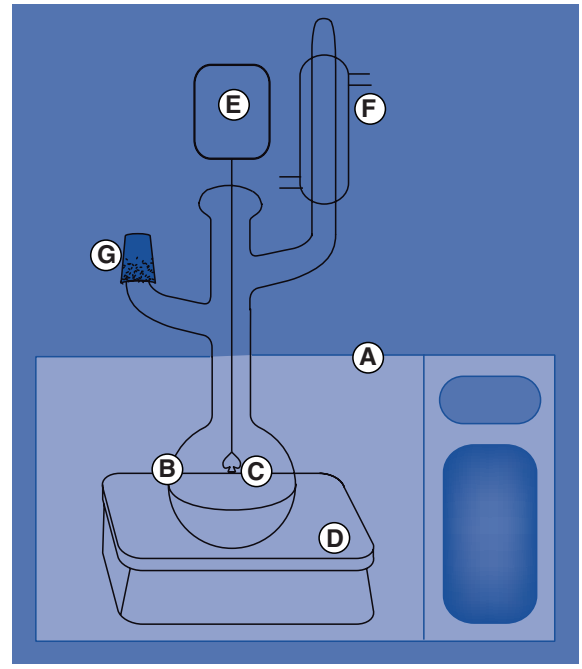

Figure 1. Domestic microwave oven modified for batch experiments. **(A)** Microwave oven; **(B)** Round-bottom flask; **(C)** Teflon stirrer; **(D)** Base; **(E)** Motor; **(F)** Condenser; **(G)** Feed neck.

rotated freely. A round-bottom (RB) flask with a Teflon agitator connected to a motor was used as the batch reactor. A three neck adapter was connected externally through a hole (10 mm diameter) made in the top of oven. A condenser was provided to aid the refluxing of methanol vapors.

▪ Single-step biodiesel synthesis under microwave irradiation

The FFA content of *Pongamia* oil was measured by standard titrimetry method using phenolphthalein indicator [22]. The acid value of crude *Pongamia* oil was 17.5 mgKOH/g. Catalyst KOH concentration of 3 wt% catalyst was used. KOH added was in excess so as to neutralize the free acids and also to catalyze the reaction [4]. Anhydrous KOH (0.75 g) was dissolved in 6.2 ml of methanol (1:5 molar ratio of oil:methanol). KOH pellets were dissolved in preweighed methanol. Average molecular weight of *Pongamia* oil was taken as 820 [23]. *Pongamia* oil (25 g) in RB flask was reacted with a KOH–methanol mixture in a microwave oven for 2 min at 180 W. The reaction mixture was mechanically agitated at 300 rpm. Similar experiments were carried out to study parameters such as time of irradiation and molar ratios of oil:methanol at various microwave powers.

▪ Two-step biodiesel synthesis under microwave irradiation

In the two-step method, first the FFA of oil was reduced with anhydrous sulfuric acid-catalyzed esterification reaction followed by a KOH-catalyzed transesterification reaction. For the acid-catalyzed esterification reaction, H_2SO_4–methanol mixture (2 wt% or 0.5g H_2SO_4; 1:6 molar ratio or 7.4 ml methanol) was reacted with *Pongamia* oil (25 g) under microwave irradiation. The reaction mixture was agitated at 300 rpm and 300 W microwave power for 2 min. After separating, the lower layer was dried in an oven at 100°C, until a constant weight, to remove traces of water. The acid value of pretreated *Pongamia* oil was 3.2 mg KOH/g and FFA content of 1.6%. For the transesterification reaction, KOH catalyst concentration of 1 wt% was used, which includes the amount of KOH required to neutralize the free acids [3,8]. Anhydrous KOH (0.25 g) was dissolved in 6.2 ml methanol (1:5 oil–methanol molar ratio). Methanolic KOH was mixed with pretreated oil from the first step. The reaction was carried out at 180 W for 2 min irradiation time with mechanical mixing at a rate of 300 rpm. Furthermore, similar experiments were carried out at various oil–methanol ratio and microwave powers for different irradiation durations.

▪ Biodiesel synthesis through conventional heating

The conventional method of heating was used for analyzing the advantages of microwave heating over biodiesel synthesis [19]. For the single-step method, 3 wt% KOH and 1:10 oil–methanol molar ratio was used. For the two-step method, 2 wt% H_2SO_4 and 1% KOH was used for esterification and transesterification reactions, respectively. Oil–methanol molar ratios of 1:6 and 1:10 were used for esterification and transesterification reactions, respectively. The reactions were carried out under reflux at 60°C using a heating mantle for 1 h [6,10]. The reactants were agitated with a magnetic stirrer.

▪ Separation & purification of biodiesel

The reaction mixture was added with a few drops of methanolic oxalic acid solution to neutralize and arrest the reaction [24]. The reaction mixture was kept for separation; the separated upper layer was warmed to remove methanol. Biodiesel was washed with warm water (three to five times with 50ml). The washed biodiesel was dried over anhydrous sodium sulfate and filtered. The samples were then tested for complete removal of glycerin using the ceric ammonium nitrate test and analyzed for purity using thermogravimetric technique [25]. The biodiesel yield was calculated as the % weight ratio of ester content in purified biodiesel phase to mass of crude oil.

▪ Determination of properties of biodiesel

Percent FFA, acid value, saponification value and iodine value of biodiesel and oil were determined as per the International Union of Pure and Applied Chemistry methods [22]. Viscosity of oil and biodiesel were measured at 40°C using Redwood and Ostwald viscometers, respectively. Cetane Index (CI), a measure of ignition delay, was calculated using **Equation 1** [26].

$$CI = 46.3 + 5458/SN - 0.225 \times IV \quad \textbf{(Equation 1)}$$

where, SN is saponification and IV is iodine number.

Results & discussion

▪ Characterization of oil

The quality of oil is characterized in terms of its physicochemical properties, such as acid value, % FFA, iodine value and saponification number. The acid value, % FFA, iodine value and saponification number of *Pongamia* oil were 17.5 mg KOH/g, 8.8%, 85 g/100 g and 210 mg KOH/g, respectively. The density and kinematic viscosity of oil were 935 kg/m^3 and 41 mm^2/s, respectively. Azam *et al.* have reported the fatty acid profile of *Pongamia* oil, which shows that oleic acid (>50%) is the major component [1].

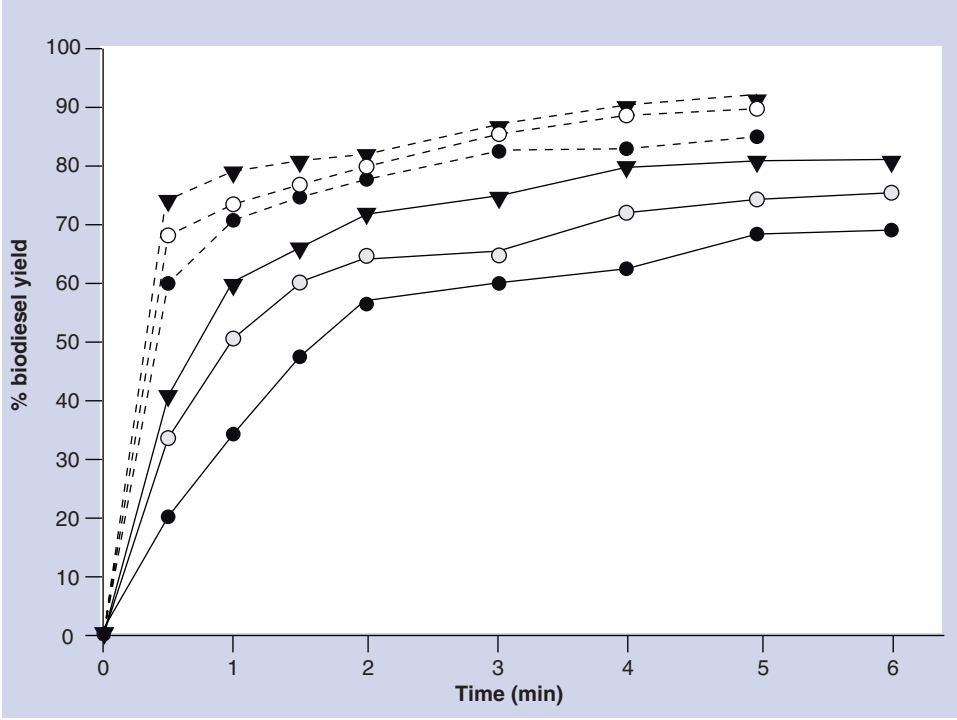

Figure 2. Influence of microwave power and irradiation time on % fatty acid methyl ester yield in single-step method (—) and two-step method (- - -) at microwave powers 100 W (●), 180 W (○), and 300 W (▼).

▪ Single-step microwave-assisted biodiesel synthesis

The biodiesel was prepared through a single-step conversion of triglycerides to FAME by microwave irradiation. The biodiesel yield was analyzed as a function of process parameters, such as irradiation time and oil–methanol ratio. Figure 2 shows the FAME yield versus irradiation time at various power levels of microwave. The yield reached a maximum of 81% at 300 W (5 min), 74% at 180 W (5 min) and 69% at 100 W (6 min). Within the first 2 min, the FAME yields increased rapidly with the transesterification time. After 4 min, the yield increased slightly, indicating that FAME almost reached an equilibrium distribution. Since the transesterification is an equilibrium reaction, a large excess of alcohol is required to drive the transesterification reaction in the forward direction. Figure 3 shows the effect of methanol ratio on FAME yield. Here the FAME yield increased with the alcohol ratio up to 1:10 and then decreased. Literature results show that a large excess of methanol decreases the yield owing to the increased solubility of glycerine in methanol, which drives the equilibrium in the backward direction [27]. Therefore, an optimum ratio of methanol could be used to obtain a higher yield of FAME. Patil and Deng reported that FAME yield remains constant beyond a molar ratio of 9:1 [6]. In the present investigation, the FAME yield reached a maximum at 1:10 molar ratio beyond which there was no remarkable improvement in the yield.

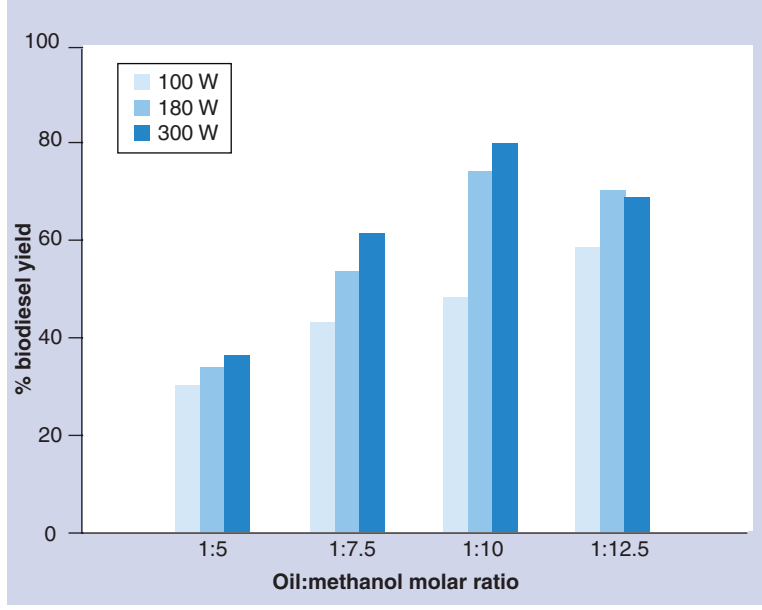

Figure 3. Effect of oil:methanol molar ratio on % biodiesel yield at different microwave powers in single-step method.

▪ Two-step microwave-assisted biodiesel synthesis

Although the one-step process yielded a high % of biodiesel, acid value was not reduced below the American standards for testing materials standard. Hence, an additional step was included to reduce the acid value of the product. In the two-step method, first the FFA content of *Pongamia* oil was reduced through an acid-catalyzed esterification reaction followed by a base-catalyzed transesterification reaction. Concentrated sulfuric acid (2 wt%) was used as acid catalyst with methanol (1:6 oil–methanol molar ratio) to reduce the FFA of *Pongamia* oil, with

an initial level of 8.8%. A number of experiments were performed to reduce the FFA level below 3% [3,28] by optimizing the process variables such as irradiation power and time. The optimized result was obtained at 300 W (2 min irradiation), which reduced FFAs by 86% to a final level of 1.25% FFA. The oil with reduced FFA content was used for the next step, namely base-catalyzed transesterification.

The *Pongamia* oil, with reduced FFA, was reacted with methanol (1:10 oil:methanol molar ratio) in the presence of base catalyst 1 wt% KOH under microwave irradiation for different time intervals. The reported literature results on two-step methods show that the optimal alkali catalyst concentration is within the range of 0.5 to 1.2% [2,6,10]. The results as shown in Figure 2 indicate that % FAME yield increases rapidly within 1 min and reaches maximum yield after 4 min of irradiation for all levels of power tested. The maximum yield, 90%, was obtained at 180 W (5 min irradiation). The yield did not improve much upon increasing the power to 300 W, where a maximum yield of 92% was achieved upon irradiating for 5 min. This indicates that, for the two-step method, power beyond 180 W has less significance over FAME yield. In a domestic microwave oven, microwave radiation is ejected as a pulse. At higher microwave power, the methanol vaporizes faster and before it is condensed the next pulse of radiation issues vapors rapidly. As the methanol in vapor form is unavailable for reaction the yield of FAME is decreased. To study the influence of alcohol ratio on base-catalyzed transesterification, methanol ratio was varied with 1 wt% KOH under microwave irradiation. The results, as shown in Figure 4, indicate that there is little improvement in yield beyond 1:10 molar ratio of oil to methanol for all three levels of microwave powers. FAME yield drastically increased compared with single-step method, at 1:10 molar ratio. In the two-step method for almost all molar ratios, microwave power seems to have negligible effect. Since the reaction system under microwave heating involves different moieties and several simultaneous reactions occurring, it requires detailed analysis to explain these effects.

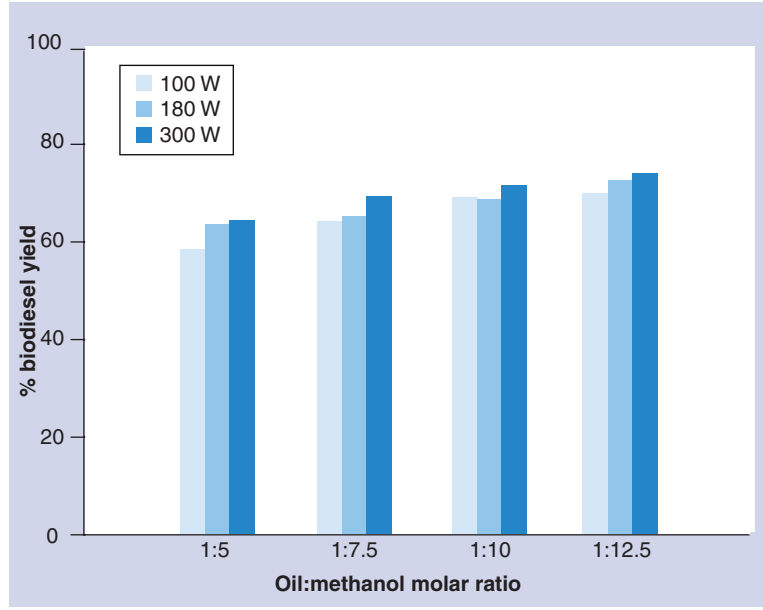

Figure 4. Effect of oil:methanol molar ratio on % biodiesel yield at different microwave powers in two-step method.

▪ Comparison with conventional method of synthesis

Pongamia biodiesel was synthesized under the conventional method in order to compare it with the microwave-assisted process. In the single-step method only a 30% biodiesel yield was obtained with 1:6 oil–methanol molar ratio and 3 wt% KOH at 60°C. In the two-step method, the FFA of oil was first reduced to 1.55% by a conventional heated esterification reaction with 1:6 wt% methanol and 2 wt% H_2SO_4 at a temperature of 60°C for 1 h. Furthermore, the oil with

Table 1. Comparison of biodiesel yield and reaction time with different techniques of synthesis.

Oil source	Method/process condition	Reaction time	Biodiesel yield (%)	Ref.
Jatropha oil (14–15% FFA)	CH: two step	30 min–2 h after pretreatment	90	[9,10]
Rapeseed oil (0.018% FFA)	CH: two step, 30 l reactor	30 min after pretreatment	98.5	[5]
Freshly extracted *Pongamia* oil (1.2% FFA)	CH: single step, 1% KOH, 1:6 molar ratio, 65°C.	15 min 2 h	85 98	[4]
Pongamia oil (18% FFA)	CH: two step	30 min after pretreatment	80	[6]
Soybean oil	US	30 min	>99	[13]
Vegetable oils	SCM: no catalyst	6–15 min	98	[12]
Vegetable oils (very low FFA)	MW: single step, CEM Explorer PLS oven	1 min	95	[14]
Pongamia oil (8.8% FFA)	MW: two step, domestic microwave oven	4 min	90	Discussed here

CH: Conventional heating; FFA: Free-fatty acid; MW: Microwave assisted; SCM: Supercritical methanol; US: Ultrasound assisted.

Table 2. Properties of *Pongamia* methyl esters synthesized by microwave and conventional methods.

Properties	MW1	MW2	CH1	CH2	ASTM D6751:09†
Density (kg/m^3)	890	887	889	885	-
Kinematic viscosity (mm^2/s)	5.1	4.9	3.8	3.9	1.9–6.0
Acid value (mg KOH/g)	16.0	0.38	16.4	0.45	<0.5
Cetane index	57.3	56.0	56.7	55.0	>47

†Data from [29].
ASTM: American standards for testing materials; CH1/CH2: Conventional heated one-step method/two-step method; MW1/MW2: Microwave-assisted one-step method/two-step method.

reduced FFA was transesterified with 1:10 molar ratio of oil to methanol and 1 wt% KOH at 60°C for 1 h. A biodiesel/FAME yield of 78% was obtained in this process, which is far less compared with that of microwave-assisted synthesis (92%). It has been reported that for the two-step conventional method, the yield was up to 80% for *Pongamia* oil, with 18% initial FFA having been reported [6]. Higher yields have been reported for the single-step conventional method with optimum conditions of transesterification time and temperature, catalyst concentration and alcohol ratio [2]. Lertsathapornsuk *et al.* reported an 83% yield with 3% NaOH catalyst at 9:1 molar ratio for a microwave-assisted continuous process using waste frying oil, having an initial FFA of 4.5% [8]. From the present work, it was observed that the biodiesel yield and reaction rate were highly enhanced with microwave irradiation, when compared with conventional method. The comparative results of microwave-assisted synthesis and conventional methods are summarized in Table 1.

▪ Properties of *Pongamia* biodiesel

Properties of biodiesel, such as FFA, acid value, saponification number, iodine number, density, kinematic viscosity and cetane index, were analyzed for biodiesel synthesized through single- and two-step methods assisted by conventional and microwave heating, and are listed in Table 2. The properties were compared with the American standards for testing materials and were found to be in good agreement, except for acid value in the case of the single-step method [29]. Physical properties remained the same irrespective of the synthesis method followed; this shows that microwave irradiation accelerates the process without altering the physical properties of biodiesel. The quality of biodiesel was analyzed using thermogravimetry. All the biodiesel synthesized was found to have an ester content of over 90% (Figure 5). The biodiesel synthesized in the two-step method under microwave irradiation had esters in the range of 97 to 99.5%.

Table 3. The advantages of microwave-assisted synthesis over conventional method for the two-step method.

Parameter	Microwave	Conventional	Time improvements (%)
Time of reaction	4 min (180W)	1 h	93
Separation time	15–20 min	>6 h	96
Final % FFA after pretreatment (from initial 8.8%)	1.25 (2 min)	1.55 (45 min)	96
Biodiesel yield (%)	90	80	11

FFA: Free-fatty acids.

▪ Overall influence of microwave energy on *Pongamia* biodiesel synthesis

The specific microwave effect on chemical reactions is evaluated by comparing the time needed to obtain a given yield with respect to traditional heating [19]. In the present work, it is clearly established that microwave energy has significant influence over biodiesel synthesis. In the single-step method, compared with 30% yield in conventional method, microwave-assisted synthesis (81% yield) resulted in more than doubling the yield. In the two-step method, microwave-assisted synthesis gave a better yield of 92% with respect to the conventional method, which yielded 75% biodiesel. With microwave-assisted heating, although the yield in the single-step method is quite significant, the high acid value of biodiesel makes it unsuitable for use as a fuel [24]. Hence, the two-step method, where acid value is quite low, seems to be a better approach for microwave-assisted *Pongamia* FAME synthesis. Application of microwave energy also improves the product recovery from the reaction mixture. The static separation time required for separation of glycerol layer is approximately 6 h for the conventional method of heating but for microwave heating it takes only 15–20 min. Approximately a 93% reduction in static separation time is observed with microwave heating, which is in good agreement with available literature results [15]. The advantages of microwave-assisted synthesis over the conventional method are summarized in Table 3.

The exact mechanism behind the acceleration of the reaction rate under microwave heating is too complex and not clear. The experimental results indicate that microwave energy has several advantages over conventional heating techniques. This is attributed to the rapid heating profile of the microwave achieved at a molecular level that cannot be accessed by any other type of heating system [19,21]. However, microwave heating is totally different from conventional heating. The accelerated reaction rate results from material–wave interactions

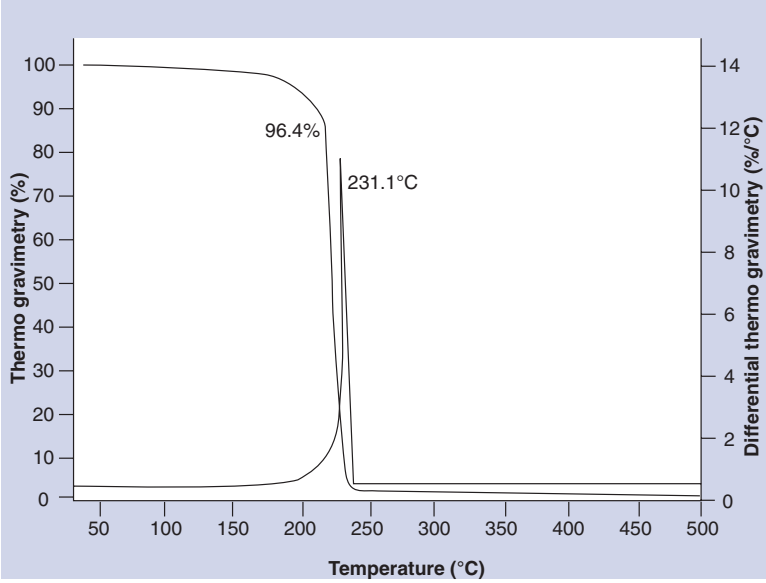

Figure 5. Thermogravimetric and differential thermogravimetric analysis traces of *Pongamia* biodiesel synthesized in microwave-assisted two-step method.

leading to thermal effects (connected to dipolar and charge space polarization) and specific (nonthermal) effects. Thermal effects are caused by the presence of polar molecules (alcohol) in the reaction system and their changing alignment under an oscillating electromagnetic field [20]. Nonthermal effects are attributed to the Arrhenius equation, where the reaction rate can be increased by either elevating the Arrhenius constant or lowering Gibb's free energy of activation [21]. Nonthermal effects can therefore originate by a lowering of the Gibbs energy of activation of the reactions through either enthalpy effect or entropic effect [19]. The microwave energy is being utilized in most of the organic reactions at laboratory scale. However, owing to the difficulty in controlling mechanisms, microwave reactors are not yet developed on a commercial scale. A solution to this bottleneck could improve the technology, as well as the economics, of biodiesel production.

Conclusion

Fatty acid methyl esters from *Pongamia* oil were synthesized with single- and two-step methods under microwave irradiation. The two-step method was better in comparison with the single-step method of FAME synthesis; which yielded 92% FAME under microwave-assisted synthesis. The results of microwave irradiation were compared with conventional heating and microwave irradiation was reported with several advantages. Reaction time and static separation time decreased significantly for microwave-assisted synthesis. The physical properties did not vary with the source of heating. Intense research in the field of microwave chemistry is required in order to exploit microwave-assisted synthesis on an industrial scale.

Future perspective

The demanding needs for energy and diminishing fossil fuels has led to pioneering works for alternative sources of energy. Biodiesel, being a promising present and future alternative energy source, has several disadvantages, including cost and inefficient process schemes. Use of microwave energy could reduce the power consumption for synthesis and processing, increase speed of production and also improves methanol recovery. The major concern is the requirement for safety guidelines and regulatory issues for microwave applications on an industrial scale. Use of nonedible oil, which is grown in plenty and has no major application, could be a cheaper feedstock for biodiesel. *Pongamia*, an example of such oil, could be a potential feedstock and in combination with microwave synthesis would drastically reduce the cost of biodiesel.

Financial & competing interests disclosure

The authors have no relevant affiliations or financial involvement with any organization or entity with a financial interest in or financial conflict with the subject matter or materials discussed in the manuscript. This includes employment, consultancies, honoraria, stock ownership or options, expert testimony, grants or patents received or pending, or royalties.

No writing assistance was utilized in the production of this manuscript.

Executive summary

- Microwave energy has a significant effect on the production of biodiesel, mainly reducing the time of synthesis by at least 90% and also the power requirement.
- The use of microwaves enhance the recovery/separation time and also reduces the power consumption.
- The fuel quality remains unchanged upon using microwave irradiation.
- Although the single-step method yields quite high biodiesel, due to its high acid value, two-step synthesis is the preferred method for high-free fatty acid oils such as *Pongamia*.

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