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Review Article

Extraction, transesterification and process control in biodiesel production from *Jatropha curcas*

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Biodiesel has gained worldwide popularity as an alternative energy source due to its renewable, non-toxic, biodegradable and non-flammable properties. It also has low emission profiles and is environmentally beneficial. Biodiesel can be used either in pure form or blended with conventional petrodiesel in automobiles without any major engine modifications. Various non-edible and edible oils can be used for the preparation of biodiesel. With no competition with food uses, the use of non-edible oils as alternative source for engine fuel will be important. Among the non-edible oils, such as *Pongamia*, *Argemone* and *Castor*, *Jatropha curcas* has tremendous potential for biodiesel production. *J. curcas*, growing mainly in tropical and sub-tropical climates across the developing world, is a multipurpose species with many attributes and considerable potentials. In this article, we review the oil extraction and characterization, the role of different catalysts on transesterification, the current state-of-the-art in biodiesel production, the process control and future potential improvement of biodiesel production from *J. curcas*.

Keywords: Biodiesel production / *Jatropha curcas* / Oil extraction / Process control / Transesterification

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1 Introduction

For many years, the ready availability of inexpensive petroleum fuels gave little inducement for experimenting with alternative, renewable fuels for diesel engines [1–4]. However, due to the increasing price of petroleum as well as environmental concerns over pollution caused by the internal combustion gases, alternative fuels have been developed [5, 6]. Biodiesel is considered as one important alternative fuels for diesel engines [7].

Biodiesel is defined as the monoalkyl esters of long-chain fatty acids that are derived from renewable feedstocks such as vegetable oil or animal fats. It is used in the compression ignition engine [3]. It has been found that biodiesel is a clean-burning fuel, biodegradable, non-toxic and has low emission profiles. So, it is environmentally beneficial. The use of biodiesel has the potential to decrease the level of pollutants and of potential or probable carcinogens [8–10].

Edible oils, such as soybean, rapeseed oil and palm oil, are being used for the production of biodiesel for the compression ignition engine [11]. However, the use of these oils for engine fuel will compete with the food product. Consequently, the use of non-edible oils as alternative source for engine fuel will be important.

There are quite a few non-edible oil seed species that could be used as source for oil production. *Jatropha curcas*, which mainly grows in tropical and sub-tropical climates across the developing world, is one of them. This multipurpose species with many attributes and potentials [11, 12] can be grown in low- to high-rainfall areas, either on the farms as a commercial crop or on the boundaries as a hedge to protect the fields from

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grazing animals and to prevent erosion [13]. Its hardness, rapid growth, easy propagation and wide-ranging usefulness have resulted in its spread far beyond its original distribution [14].

There are several technical challenges that need to be addressed to make biodiesel profitable. First, the high cost of virgin vegetable oil as the source of triglycerides plays a large role in process profitability. To reduce the production costs and make it competitive with petroleum diesel, low-cost feedstocks, such as waste frying oils, and animal fats and non-edible oils could be used as raw materials. Among the non-edible oils, *J. curcas* has tremendous potential for biodiesel production. However, the relatively higher amounts of free fatty acids (FFA) and water in this feedstock result in the production of soap in the presence of alkali catalysts. Thus, it is necessary to have additional steps to remove any water and either the FFA or the soap from the reaction mixture.

Although publications of research on biodiesel production from *Jatropha* oil are available elsewhere, a systematic review on this topic is very limited. This paper provides comprehensive information on biodiesel production from *Jatropha* oil, including its oil extraction technique and composition, the role of different catalysts in its transesterification, the current state-of-the-art in biodiesel production, process control and future potential improvement of biodiesel production from *J. curcas*.

2 *Jatropha* oil as feedstock of biodiesel production

The growing demands for lower-cost, non-food, non-rainforest-based feedstocks for biodiesel are producing new opportunities and are stimulating fresh investment in the production of lower-cost, alternative feedstocks such as *Jatropha*. The governments in South Asia and Africa have identified between 20–50 million hectares of suitable land for *Jatropha* cultivation. Indonesia has identified nearly 23 million hectares of *Jatropha* land potential. One hectare of *Jatropha* can produce between 1.5 and 2.5 t of seed oil. Since the expansion of commercial-scale *Jatropha* production from India to Africa, Southeast Asia and Latin America, and due to pilot programs and larger-scale ventures in China, Central Asia, South/Central America, and southern parts of the USA, *Jatropha* is now becoming one of the prime contenders for biodiesel feedstock supply in the years ahead [15].

It is reported that *Jatropha* oil is present in the range of 40–60 g oil/100 g *Jatropha* seed kernels [16, 17]. A yield of 43.80% oil was obtained by extracting with hexane for 3 h at room temperature; increasing the extraction time did not affect the extraction efficiency [18]. Shah *et al.* [19] found that *Jatropha* seed kernel gave a yield of 44 g oil/100 g *Jatropha* seed kernels. The yield of oil obtained from the extraction of *J. curcas* with petroleum ether (b.p. 40–60 °C) in a Soxhlet apparatus was 46–48.6% [20]. According to Su *et al.* [21], extraction of *Jatropha* seed oil with *n*-hexane gave a yield of 54.90%, methyl acetate gave 55.92%, and ethyl acetate 56.65%.

2.1 Oil extraction

Various kinds of equipment are available to obtain oil from the seeds. The oil can be extracted mechanically with a press (ram, hydraulic or screw) or chemically with organic solvents or water [22–24], by three-phase partitioning extraction method [25, 26] and supercritical extraction method [27].

2.1.1 Mechanical press

In Indonesia, *Jatropha* oil is usually extracted by hydraulic press and screw press with 60 °C heat treatment. The yield of *Jatropha* oil using the hydraulic press method at a maximum pressure of 20 t is 47.2% and the oil extraction is done twice [28].

In Tanzania, presently, the *Jatropha* oil is obtained only mechanically with a ram press or a screw press, which is a small hand-press [29]. With the ram press method, the seeds are poured by applying pressure to the seeds. The oil is extracted and then dripped into a container. The extraction rate of this press is quite low as the seedcake, which is left after the pressing, still contains part of the oil. To obtain 1 L of oil, about 5 kg of seed is needed and the capacity is about 1.5 L/h. Larger expellers and screw presses that are run by an engine can also be used. The screw, which turns continuously, transports the seeds from one side of the press to the other while squeezing out the oil. The extraction rate of this press is higher because more oil is extracted from the seeds; the cake residue is also much dryer. The capacity of this screw is higher than that of the ram press. For example, the Sayari oil expeller, which is used in Tanzania, has a capacity of about 20 L/h (60 kg/h) and can extract 1 L of oil from 3 kg of seeds. The larger screw expellers, like the Chinese expellers, can extract about 50 L/h (150 kg/h). After the oil is expelled, it is filtered by letting it stand for some time or pouring it through a cloth [29].

2.1.2 Aqueous and solvent extraction

Aqueous oil extraction (AOE) is a method in which *Jatropha* seeds were cracked and the shells were carefully removed. The kernels obtained were used for oil extraction. The suspension was prepared with powdered (obtained by using a homogenizer) *Jatropha* seed kernels in distilled water, and then incubated at the desired temperature with constant shaking at 100 rpm for a specified time period. The upper oil phase was collected after centrifugation at 10,000 × g for 20 min. Enzyme-assisted AOE was performed similar to AOE; the difference is that the preparations, *i.e.* Protizymee, Cellulase, Pectinex Ultra SP-L, Promozyme as well as a mixture of all these enzymes were added after the pH of the suspension was adjusted. The amounts of oil obtained were calculated as percentages of the total oil present in *J. curcas* seed kernels [19].

It has been found that the use of ultrasonication as a pretreatment before AOE and aqueous enzymatic oil extraction is useful in the case of extraction of oil from the seeds of *J. curcas* L. [19]. The use of ultrasonication for a period of 10 min at pH 9.0 followed by AOE resulted in a yield of 67% of available oil. The maximum yield of 74% was obtained by ultrasonication for 5 min followed by aqueous enzymatic oil extraction using an alkaline protease at pH 9.0 (44 g oil/100 g *Jatropha* seed kernels was taken as 100% recovery). Use of ultrasonication can also reduce the process time from 18 to 6 h [19].

Optimized conditions for extraction of *Jatropha* using microwaves are achieved by using petroleum ether as the solvent with a ratio of seed powder to solvent of 1 : 3. It is done at a microwave power of 810 W for a total radiation time of 5 min [27]. The extraction rate was 31.49%, with the oil product having an acid number of 5.22 mg/g and a peroxide value of 8.78 meq/g. For the ultrasonic method, hexane was used as the solvent and the ratio of seed powder to solvent was 1 : 7; the soaking time applied was 18 h and sonication was performed for 0.5 h. The extraction rate was 37.37% with an acid number of 5.91 mg/g and a peroxide value of 8.37 meq/g in the final oil product [27].

Alkyl acetates, especially methyl acetate and ethyl acetate, are important chemicals and suitable solvents for seed oil extractions that are assisted by Novozym 435 [21]. The results were compared to those obtained by extraction with *n*-hexane. Ground seeds were mixed with methyl acetate or ethyl acetate in screw-capped glass vials. Then, 30 wt-% of Novozym 435 based on theoretical oil content was added. The reactions were carried out at 50 °C and 180 rpm for 6 h in a shaker fitted with a thermostat. After filtration, the ground seed mixture was mixed with another solvent and then extracted under the same conditions for another 2 h. The two filtrates were pooled and centrifuged at 17,400 × g for 10 min; the supernatant was collected in a round-bottom flask and the solvent was evaporated using a rotary evaporator. The oil content in g/100 g was 54.90% (*n*-hexane), 55.92% (methyl acetate) or 56.65% (ethyl acetate) [21].

2.1.3 Three-phase partitioning extraction method

Three-phase partitioning is a method that is carried out by cracking the *Jatropha* seeds and removing the shells; the kernels obtained are used for slurry preparation [25]. The slurry, with the pH adjusted to the desired value with 0.1 N NaOH or 0.1 N HCl, is prepared by grinding the seed kernels in distilled water. Ammonium sulfate in an appropriate amount is added and vortexed gently; an appropriate amount of *t*-butanol is added. Then, the slurry is incubated at 25 °C for 1 h for the formation of the three phases. The three phases are then separated by centrifugation at 2000 × g for 10 min. To obtain the oil, the upper organic layer is collected and evaporated in a rotary evaporator (under reduced pressure at 50 °C, for 5 min) [25].

Combining a recently developed technique of three-phase partitioning with enzyme pretreatment and sonication may constitute an economical or efficient procedure for obtaining oil from *Jatropha* seed kernels [30]. This method only takes about 2 h. Three-phase partitioning has been evaluated for extraction of oil from *Jatropha* seeds. This process consisted of simultaneous addition of *t*-butanol (1 : 1, vol/vol) and 30% (wt/vol) ammonium sulfate to the slurry prepared from *Jatropha* seed kernels. Combination of sonication and enzyme treatment with a commercial preparation of fungal proteases at pH 9 resulted in 97% oil yield within 2 h [30].

2.1.4 Supercritical carbon dioxide extraction method

Supercritical carbon dioxide extraction (SCE) is a process for the production of oil with high yields that does not use organic solvents. In this process, the oil is dissolved in CO_2 and extracted from the plant material [31]. The SCE method developed by Yan *et al.* [27] resulted in an actual extraction rate of 37.45%; the final oil product contained KOH 0.79 mg/g and peroxide 3.63 meq/g. Here, seeds of *Jatropha curcas* were collected and powdered. The extraction pressure was 43 MPa, the temperature for the extraction was 45 °C, the flow rate of CO_2 was 20 kg/h, and the extraction time was 80 min. Even though the cost of the supercritical extraction method was higher, the oil quality was the best and refining was not needed [27].

2.1.5 Gas-assisted mechanical expression

Gas-assisted mechanical expression (GAME) is another potential alternative process for the production of oil with high yields that does not use organic solvents. In this process, CO_2 is dissolved in the oil contained in the seeds before pressing them, as shown in Fig. 1 [31]. It was found that at the same effective mechanical pressure (absolute mechanical pressure minus the actual CO_2 pressure), the liquid content is the same in both the conventional and the GAME press cake. The liquid in the GAME press cake is saturated with CO_2 (typically 20–50 wt-%), reducing the oil content compared to the conventional cake by the same amount. The contribution of this effect increases with increasing solubility of the CO_2 in the oil. Furthermore, the dissolved CO_2 reduces the viscosity of the oil, which could increase the oil yield [32]. CO_2 is easily removed from the cake and oil by depressurization, while some additional oil is removed by entrainment in the gas flow.

GAME has some advantages compared with conventional pressing. The first advantage of GAME is the increased yield at lower mechanical pressure. Compared with supercritical extraction, the amount of CO_2 that has to be recycled is reduced by two orders of magnitude, from typically 1 kg/kg [32] to 100 kg of CO_2 per kilogram [33] of seeds. Therefore,

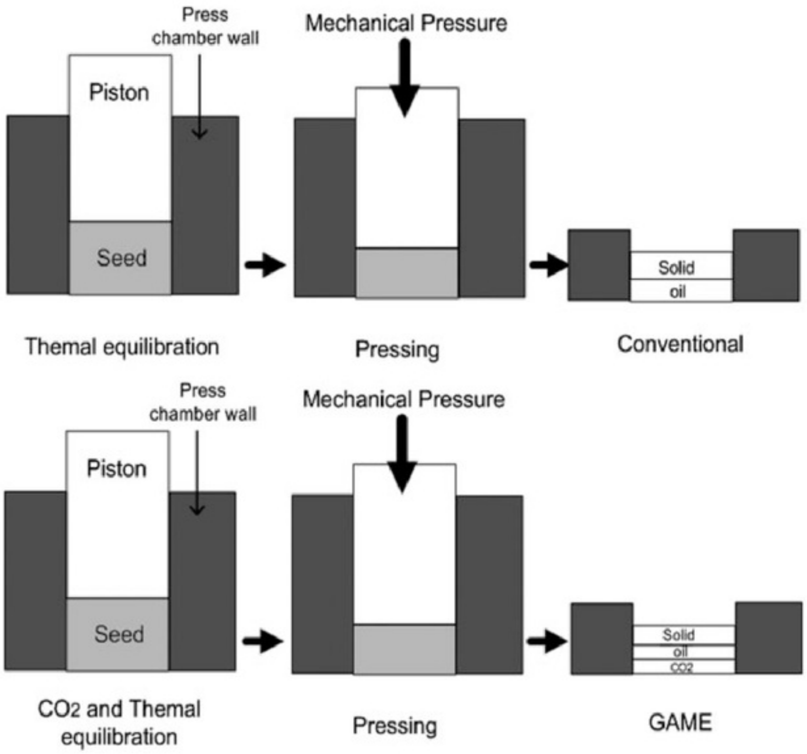

Figure 1. Principle of (a) conventional mechanical expression and (b) GAME. Adopted from [31].

energy and equipment cost for solvent recycling can be reduced. Compared with SCE, the second advantage of GAME is that the required CO_2 pressure is low, which is approximately 10 MPa. In contrast, for SCE extraction, pressures of 40–70 MPa are not unusual [33, 34]. These two effects provide a significant reduction in the energy requirements for recycling and repressurising the CO_2. Additionally, some reports in the literature suggest that the use of CO_2 at 7–20 MPa has a sterilizing effect on the substrates [35, 36]; this may be a beneficial side effect of the GAME process. Fig. 2 shows a schematic representation of the hydraulic press used in GAME.

The general applicability of the GAME process to enhance the oil recovery from oilseeds was shown by pressing experiments with sesame, linseed, rapeseed, *Jatropha* and palm kernel by Willems *et al.* [31]. It was proven that GAME is capable of reaching yields that are up to 30 wt-% higher than with conventional expression under the same conditions. Despite the lower yields for hulled seeds in conventional expression, GAME yields for hulled and dehulled seeds are very similar. The oil yields obtained by GAME increased with increasing effective mechanical pressure; the yields were the highest at a temperature of 100 °C. These effects are similar to conventional expression. With CO_2 pressures up to 10 MPa, the oil yield increased significantly. However, increasing the CO_2 pressure above 10 MPa did not significantly increase the oil yield.

2.2 Characterization of *Jatropha curcas* oil

Table 1 shows the fatty acid composition of *Jatropha* oil. It consists of 23.6% saturated fatty acids containing mainly palmitic, stearic and myristic acids, and 76.4% unsaturated fatty acids comprising mainly oleic, linoleic and palmitoleic acids. Saturated fatty acid methyl esters increase the cloud point and cetane number, and improve the stability, whereas unsaturated fatty acid methyl esters reduce cloud point, cetane number and stability [40]. Variation of type and percentage of fatty acids in *Jatropha* oil are influenced by the variety and the growing conditions of the plant. The physicochemical properties of *Jatropha* biodiesel are shown in Table 2. The properties are observed to meet all specifications of the American Society for Testing Materials (ASTM) and the Indian Standard (IS), but fail in the oxidation stability test.

In many cases, improper handling and inappropriate storage conditions that increase the water content lead to gradual *Jatropha* oil deterioration. In addition, exposing the oil to open air and sunlight for long duration would affect the concentration of FFA [7]. *Jatropha* oil contains about 14% FFA; it is far beyond the limit of the 1% FFA level that can be converted into biodiesel by transesterification using an alkaline catalyst [7, 37].

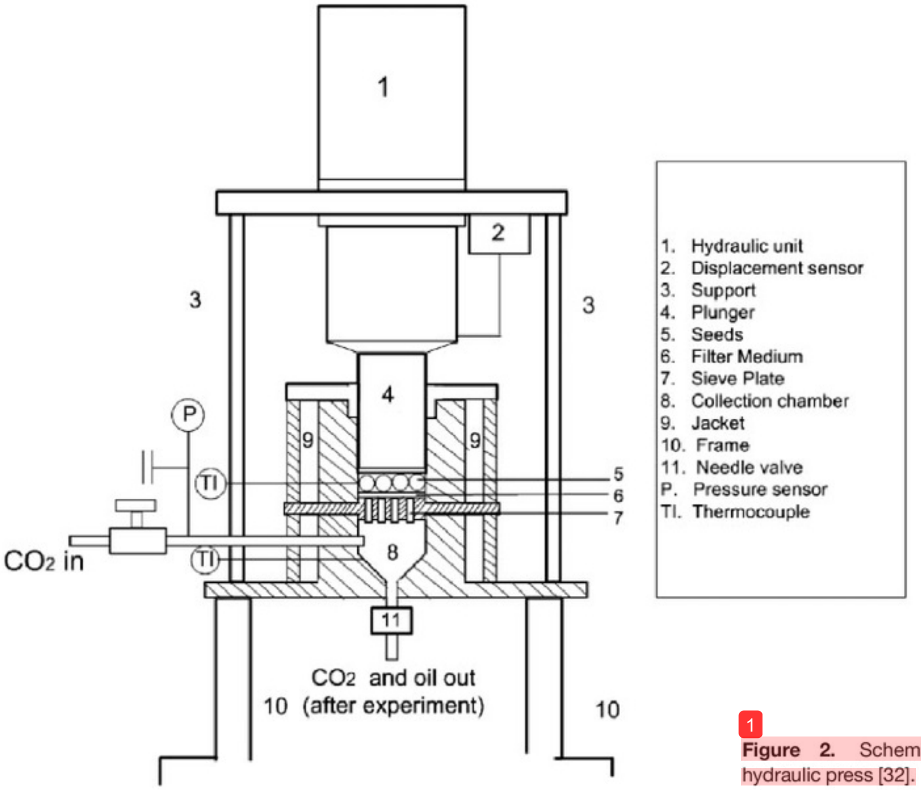

Figure 2. Schematic representation of the hydraulic press [32].

Table 1. Different fatty acids present in *Jatropha curcas* oil.

Acid, common name	Acid, IUPAC name	Structure[§]	Weight [%]					
			A[$]	B	C	D	E	Avg.
Capric	Decanoic acid	C10:0	0.1	0.1				
Lauric	Dodecanoic acid	C12:0						
Myristic	Tetradecanoic acid	C14:0	0.1	0.1	0–0.1		1.4	
Palmitic	Hexadecanoic acid	C16:0	15.1	13.6	14.1–15.3	14.2	15.6	
Stearic	Octadecanoic acid	C18:0	7.1	7.4	3.7–9.8	6.9	9.7	
Arachidic	Eicosanoic acid	C20:0	0.2	0.3	0–0.3	–	0.4	
Behenic	Docosanoic acid	C22:0	0.2	–	0–0.3	–	–	
Palmitoleic	*Cis*-9-hexadecenoic acid	C16:1	0.9	0.8	0–1.3	1.4	–	
Oleic	*Cis*-9-octadecenoic acid	C18:1	44.7	34.3	34.3–45.8	43.1	40.8	
Linoleic	*Cis*-9,*cis*-12-octadecadienoic acid	C18:2	31.4	43.2	29.0–44.2	34.4	32.1	
Linolenic	*Cis*-9,*cis*-12,*cis*-15-octadecatrienoic acid	C18:3	0.2	–	0–0.3	–		
Saturated			22.8	21.7	22.6	23.7	27.1	23.6
Unsaturated			77.2	78.3	77.4	76.3	72.9	76.4

References: A and B [17]; C [37]; D [38]; E [39].

[§] Number of carbons in chain:number of double bonds.

[$] Variety Caboverde.

[#] Variety Nicaragua.

3 Production of biodiesel

Table 3 shows a comparison of the different technologies to produce biodiesel. The method commonly used for the production of biodiesel is the transesterification of vegetable oils with methanol, using alkali, acid, or enzyme catalysts. Transesterification, also called alcoholysis, is the reaction of triglycerides with alcohols to generate, for example, methyl or ethyl esters, and glycerol as by-product. Usually, a catalyst is used to improve the reaction rate and yield. The reaction requires an excess of alcohols to improve the efficiency of the transesterification process [3].

The transesterification reaction consists of three reactions, which are equivalent, consecutive and reversible. The triglyceride is converted stepwise to diglyceride, monoglyceride and finally glycerol. At every reaction step, one molecule of methyl or ethyl ester is produced for each molecule of methanol or ethanol consumed.

There are important variables that affect the yield of biodiesel from transesterification; they are: reaction temperature, molar ratio of alcohol and oil, catalyst, reaction time, presence of moisture and free fatty acids (FFA), and mixing intensity [43–46]. The rate of reaction is strongly determined by the reaction temperature. However, giving enough time will help the reaction proceed to near completion even at room temperature. The reaction is commonly conducted close to the boiling point of the alcohol at atmospheric pressure [43].

It is observed that the stoichiometry of the transesterification reaction needs 3 mols of alcohol per mol of triglyceride to yield 3 mols of fatty esters and 1 mol of glycerol. To shift the transesterification reaction to the right, it is necessary to use a large excess of alcohol or to continuously remove one of the products from the reaction mixture. Wherever feasible, the second option is preferred because it can drive the reaction toward completion. The reaction rate is at its highest if 100% excess methanol is used. In industrial processes, a molar ratio of 6 : 1 is normally used to obtain methyl ester yields higher than 98% by weight [43].

Catalysts are categorized as alkali, acid, enzyme or heterogeneous catalysts. Among these, alkali catalysts such as sodium hydroxide, sodium methoxide, potassium hydroxide, or potassium methoxide are more effective [45]. It is found

Table 2. Physicochemical properties of *Jatropha* biodiesel [41].

Property	ASTM 6751 test method	ASTM 6751 limits	IS 15607 test method	IS 15607 limits	*Jatropha* methyl ester
Flash point [°C]	D-93	min. 130	IS 1448 P:21	min. 120	163
Vicosity at 40 °C [cSt]	D-445	1.9–6.0	IS 1448 P:25	2.5–6.0	4.40
Sulfated ash [% mass]	D-874	max. 0.02	IS 1448 P:4	max. 0.02	0.002
Sulfur [% mass]	D-5453	max. 0.05	ASTM D5453	max. 0.05	0.004
Cloud point [°C]	D-2500	N.A.	IS 1448 P:10	N.A.	4
Copper corrosion	D-130	max. 3	IS 1448 P:15	max. 1	1
Cetane number	D-613	min. 47	IS 1448 P:9	min. 51	57.1
Water and sediment [vol-%]	D-2709	max. 0.05	D-2709	max. 0.05	0.05
CCR 100% [% mass]	D-4530	max. 0.05	D-4530	max. 0.05	<0.01
Neutralization value [mg KOH/g]	D-664	max. 0.80	IS 1448 P:1/Sec.1	max. 0.50	0.48
Free glycerin [% mass]	D-6584	max. 0.02	D-6584	max. 0.02	0.01
Total glycerin [% mass]	D-6584	max. 0.24	D-6584	max. 0.25	0.02
Phosphorus [% mass]	D-4951	max. 0.001	D-4951	<0.001	<0.001
Distillation temperature	D-1160	90% at 360 °C	Not under spec.	90%	90%
Oxidation stability [h]	N.A.	N.A.	EN 14112	min. 6 h	3.23

Table 3. Comparison of different technologies to produce biodiesel [42].

No.	Variable	Alkali catalyst	Lipase catalyst	Supercritical alcohol	Acid catalyst
1	Reaction temperature [K]	333–343	303–313	512–658	328–353
2	FFA in raw material	Saponified products	Methyl esters	Esters	Esters
3	Water in raw material	Interference with reaction	No influence	[illegible]	Interference with reaction
4	Yield of methyl esters	Normal	Higher	Good	Normal
5	Recovery of glycerol	Difficult	Easy	–	Difficult
6	Purification of methyl esters	Repeated washing	None	–	Repeated washing
7	Production cost of catalyst	Cheap	Relatively expensive	Medium	Cheap

that alkali-catalyzed transesterification is much faster than the acid-catalyzed reaction. However, the acid-catalyzed transesterification reaction is more suitable if a vegetable oil has a high FFA and water content. Most commercial transesterification reactions are conducted with alkaline catalysts. This is due partly to the faster esterification and partly to the fact that alkaline catalysts are less corrosive to industrial equipment than acidic catalysts. It has been observed that sodium methoxide is more effective than sodium hydroxide. Sodium alkoxides are among the most efficient catalysts used for this purpose. However, due to its low cost, NaOH has been widely used in large-scale transesterification [45].

The effects of FFA and water on transesterification with methanol were examined by Ma *et al.* [45]. The results showed that in order to get the best conversion, the water content should be kept below 0.06 wt-% and the FFA content should be kept below 0.5 wt-%. In the transesterification, water content was a more critical process variable than FFA content; the presence of water may cause ester saponification. FFA can react with the alkali catalyst to produce soap and water [45].

In the transeterification reaction, the reactants first form a two-phase liquid system [41]. The reaction is diffusion controlled; poor diffusion between the phases results in a slow rate. When methyl esters are formed, they act as a mutual solvent for the reactants and a single-phase system is formed. The mixing effect is most significant during the slow-rate region of the reaction. As the single phase is established, mixing becomes insignificant [41].

In base-catalyzed transesterification, the glycerides and alcohol must be substantially anhydrous because water causes the reaction to partially change to saponification, which produces soap. The soap makes the yield of esters low and causes the separation of ester and glycerol and difficult water washing. In addition, the catalyst recovery is slow and expensive; this increases the operational cost [11].

Acid catalysts can be used for transesterification. However, they are generally considered to be too slow for industrial processing. Besides, they have high alcohol/triglycerides requirements (20 : 1 and more). Acid catalysts are more commonly used for the esterification of FFA [47]. Despite its insensitivity to FFA in the feedstock, acid-catalyzed transesterification has been largely disregarded due to its relatively slow reaction rate [9].

Mittlebach [48] found that transesterification can also be catalyzed by lipases. He found that the lipase from *Pseudomonas fluorescens* was superior to those from *Candida* sp. and *Mucor miehei* for sunflower oil alcoholysis. The alcoholysis was carried out both in the presence of solvent (petroleum ether) and under solvent-free conditions using five homologous alcohols with or without the addition of water. Subsequent studies have focused on different lipases, different triglyceride feedstocks, different alcohols and different experimental conditions such as temperature, water content, stoichiometric ratio between reagents, enzyme concentration, solvent use *etc.* The commercial use of enzymes is limited because of their high cost and slow reaction rate. Besides, yields to methyl esters are usually less than the 99.7% needed for fuel-grade biodiesel [47].

The transesterification reaction can be promoted by using low-frequency ultrasound (40 kHz) [49–51]. The effects of ultrasonic irradiation in the medium are heating, mass transfer, and cavitation. The ultrasonic assistant is successfully used in the production of biodiesel using a chemical method. Low-frequency ultrasonic irradiation is a useful tool for the emulsification of immiscible liquids. The collapse of the cavitation bubbles disturbs the phase boundary and causes emulsification by ultrasonic jets that impinge from one liquid to another. The reaction time was shortened and the amount of alkali catalyst was reduced by 2–3 times compared with the conventional method [51, 52].

Microwave heating can also promote the transesterification reaction. Different from conventional heating where large amounts of energy are used to heat the media, microwave radiation, on the other hand, delivers energy directly to the reactants. Therefore, the preheating step is eliminated. The heat transfer is more effective than for conventional heat. Consequently, transesterification with microwave radiation can be completed in much shorter time. The results indicated that microwave heating effectively increased the biodiesel yield and decreased the reaction time [53–55].

The supercritical alcohol process is a non-catalyzed transesterification [56]. The transesterification reaction using supercritical methanol can form a single phase, in contrast to the two-phase nature of an oil/methanol mixture under ambient condition. This is due to the decrease in the dielectric constant of methanol in the supercritical state. The reaction was completed in a very short time, *i.e.* within 2–4 min. Due to the non-catalytic process, pretreatment or post-reaction processes became much simpler compared with the conventional alkali catalyst process [57–59]. So, since there is no wastewater produced by pretreatment or washing processes, this process is environmentally friendly. However, because of the high temperature and pressure in the supercritical state, high investments and energy costs are probably required. It would be economically more feasible if the usage of methanol could be reduced [60].

It is observed that the transesterification process can be monitored by analytical procedures for quality parameters specific to FAME fuels or for parameters applicable to both biodiesel and fossil diesel fuel. The quality parameters specific to FAME fuels are minimum percentage of ester content, maximum amount of free glycerol, total glycerol, methanol content, acid value, iodine number, phosphorous content, alkali and alkaline-earth metals. The parameters applicable to both biodiesel and fossil diesel fuel are density, kinematic viscosity, flash point, sulfur point, cetane number, ash content, water content, total contamination, fuel stability, and cold-temperature properties [61].

3.1 Production of biodiesel from *Jatropha curcas*

Table 4 shows works on *Jatropha* biodiesel production under various conditions. Type and amount of variables such as alcohol, FFA, molar ratio, catalyst, reaction temperature, reaction time, rate and mode of stirring affect the yield and conversion of *Jatropha* biodiesel. Different processes for the transesterification of vegetable oils for the production of *J. curcas* biodiesel are currently available. Among them are homogenous catalysis treatment [7, 16, 28, 41, 62, 63, 68], heterogeneous catalyst [57] or enzyme catalysis alcohol treatment [21, 64, 65, 67, 70, 71], supercritical alcohol treatments [65, 69] and lipase-catalyzed *in situ* reactive extraction [21].

3.1.1 Biodiesel production using homogeneous chemical catalysts

Foidl *et al.* [16] reported a technical process for processing seed oil and production of methyl esters and ethyl esters from the oil of *Jatropha* seeds. The fuel properties were also determined. Production of biodiesel used two-step transesterification: alkali-alkali transesterification for methyl esters and alkali-acid transesterification for ethyl esters.

Chitra *et al.* [62] found that a methyl ester yield of 98% was obtained using 20 wt-% methanol and 1.0% NaOH at 60 °C. The maximum reaction time needed for a maximum ester yield was 90 min. Total biodiesel of 96% was obtained from experimental studies on large-scale production (reactor capacity of 75 kg). An esterification-transesterification reaction for *Jatropha* biodiesel was done by Sudradjat *et al.* [27, 63], but the methyl ester yield was not reported.

Tiwari *et al.* [68] and Berchmans and Hirata [7] have developed a technique to produce biodiesel from *Jatropha* with high FFA contents (15% FFA). They selected a two-stage transesterification process to improve the methyl ester yield. The first stage involved an acid pretreatment process to reduce the FFA level of crude *Jatropha* seed oil to less than 1%. The second stage was the alkali base-catalyzed transesterification process resulting in 90% methyl ester yield. Tiwari *et al.* [68] found that the optimum combination to reduce the FFA content of *Jatropha* oil from 14% to less than 1% was 1.43% vol/vol H_2SO_4 acid catalyst, 0.28 vol/vol methanol-to-oil ratio, and 88 min reaction time at a reaction temperature of 60 °C. This process produced a yield of biodiesel of more than 99%.

Berchmans and Hirata [7] reduced the high FFA level of *Jatropha* oil to less than 1% by a two-step process. The first step was carried out with a 0.60 wt/wt methanol-to-oil ratio in the presence of 1 wt-% H_2SO_4 as an acid catalyst in 1 h of reaction at 50 °C. The second step was transesterification using 0.24 wt/wt methanol-to-oil ratio and 1.4 wt-% NaOH to oil as alkaline catalyst to produce biodiesel at 65 °C. The final yield of methyl esters of fatty acids was ca. 90% in 2 h.

3.1.2 Biodiesel production using a heterogeneous solid super base catalyst

An environmentally friendly process was developed for the production of biodiesel from *Jatropha* oil using a heterogeneous solid super base catalyst and calcium oxide. The results revealed that, under the optimum conditions of catalyst calcination, a temperature of 900 °C, a reaction temperature of 70 °C, a reaction time of 2.5 h, a catalyst dosage of 1.5%, and a methanol/oil molar ratio of 9 : 1, the oil conversion was 93% [66].

3.1.3 Biodiesel production using a heterogeneous enzyme catalyst

The interest in using lipases as enzymatic catalysts for the production of alkyl fatty acid esters grows continuously. Some people work on the triglycerides by converting them to methyl esters, while some work on the fatty acids. One of the main obstacles to the bio-catalytic production of biodiesel is the high cost of the enzyme; enzyme recycling might be the solution to this problem.

It was found that *Pseudomonas fluorescens* lipase immobilized on kaolinite lost one third of its activity when it was used for the second time, but no further decrease was observed in successive applications. The initial decrease in activity was put down to enzyme desorption from the solid support that was not observed after repeated (10 times) use [73]. Repeated batch reactions revealed that *Mucor miehei* lipase had high stability, retaining about 70% of its initial conversion after 8 cycles (24 h each cycle). Meanwhile, under the same experimental conditions, *Thermomyces lanuginosa* retained only 35% of the initial conversion. This difference was credited to factors such as inactivation of the biocatalyst in the oil phase, the type of carrier that is used for the immobilization, or enzyme sensitivity to long-term methanol exposure [74].

It is observed that various substances can slow down lipase activity (methanol, glycerol, phospholipids); however, a number of ways have been proposed to overcome these problems [75]. Commonly, a biocatalytic process does not produce soaps or other by-products. If the reaction runs to completion, only esters and glycerol are produced. This makes the purification steps simpler and consequentially lowers the plant costs. Once immobilized, lipases can be used many times or even in continuous processes. This resolves the major disadvantage related to high cost. In conclusion, if obtained by biocatalysis, biodiesel is an environmentally friendly fuel that will contribute to reducing negative impacts on the environment. Thus, it may be reasonable to rename it 'bio-biodiesel' [75].

Modi *et al.* [64] used propan-2-ol as an acyl acceptor for immobilized lipase-catalyzed preparation of biodiesel. The optimum conditions set for the transesterification of crude *Jatropha* oil were 10% Novozym 435 (immobilized *Candida antarctica* lipase B) based on oil weight, alcohol-to-oil molar

Table 4. Biodiesel production *J. curcas* from various conditions.

No.	Oil	Transesterification steps	Alcohol	Molar ratio (alcohol/oil)	Catalyst	Reaction temp. [K]	Duration	Stirring	Yield [wt-%]	Ref.
1	FFA 0.29–1.27%	Two steps: Alkali catalyst Alkali catalyst	Methanol Methanol	4.50 : 1 mol (two parts) (one part)	KOH 1.5 wt-% (two parts) (one part)	333 333	30 min 30 min	Stirring Stirring	92%	[16] [16]
	FFA 0.29–1.27%	Two steps Alkali catalyzed Acid catalyzed	Ethanol Ethanol	6.9 : 1.14 mol	KOH 1.5 wt-% H_2SO_4 2 wt-%	348 353	90 min 6 h	Stirring Stirring	88.4%	
2	FFA 44.15%	Two steps: Acid catalyzed Alkali catalyzed	Methanol Methanol	20% vol/vol 40% vol/vol	H_2SO_4 2% KOH 0.3%	333 333	90 min 90 min			[28]
3	FFA 3.09%	Single step	Methanol	20 wt-%	NaOH 1 wt-%	333	90 min	stirring	98%	[62]
4		Two steps: Acid catalyzed Alkali catalyzed	Methanol Methanol	20% vol/vol 10% vol/vol	HCl 1% NaOH 0.5%	333 333	42 min 30 min			[63]
5		Single step	Ethyl acetate	Ethyl acetate to oil molar ratio of 11 : 1	10% of Novozym 435 (immobilized *Candida antarctica* lipase B)	323	12 h	150 rpm	91.3%	[64]
6		Single step	Propan-2-ol	4 : 1	30% *Candida antarctica* lipase B immobilized on macroporous acrylic resin	323	8 h	150 rpm	92.8%	[65]
7		Single step	Methanol	methanol/oil molar ratio of 9 : 1	1.5% CaO	343	2.5 h	Stirring	93%	[66]
8	*J. curcas* L seed, 4.62% water content	Single step: *In situ* reactive extraction	Methyl acetate Ethyl acetate	solvent/seed ratio of 7.5 : 1	30 wt-% of Novozym 435 (lipase B from *Candida antarctica*, immobilized on a macroporous acrylic resin	323	36 h	180 rpm	86.1% 87.2%	[21]
9		Single step	Methanol	3 : 1	NaOH/KOH (1 wt-% of oil)		2–4 h	Stirring		[41]
10	2.71% FFA	Single step: Biocatalyst	Ethanol	4 : 1	*Pseudomonas cepacia* lipase immobilized on celite	323	8 h	200 rpm	98%	[67]
11	FFA 14%	Two steps: Acid catalyzed Alkali catalyzed	Methanol Methanol	0.28 vol/vol 0.16 vol/vol	H_2SO_4 1.43% vol/vol KOH (3.5 + acid value wt/vol)	333 333	88 min 24 min		>99%	[68]
12	FFA 14.9% 14.9%	Single step: Alkali catalyzed Two steps: Acid catalyzed Alkali catalyzed	Methanol Methanol	70 wt-% 60 wt-% 24 wt-%	NaOH 3.3 wt-% H_2SO_4 1 wt-% NaOH 1.4 wt-%	338 323 323	2 h 1 h 2 h	400 rpm	55% 90%	[7]
13	Crude JCO	Single step: No catalyst	Methanol Ethanol	50 : 1 50 : 1		573 573	40 min 40 min		82.5% 85%	[65]
14	Crude JCO	Single step: Alkali catalyzed	Methanol	24 : 1	0.8 wt-‰	523	28 min	400 rpm	90.5%	[69]
15	FFA 8.7%	Single step	Methanol	4 : 1	Immobilized *P. flourescence* 6% wt/vol oil	313	48 h	150 oscillation/min shaking	72%	[70]
16	Water 1.5 wt-%	Single step	Methanol	3 : 1	Lipase 6 wt-% of oil	303	60 h	150 rpm	80%	[71]
17	RBD oil	Single step	Methanol Ethanol	9 : 1 9 : 1	$NaOCH_3$ 0.8% $NaOCH_3$ 0.8%	318 318	30 min 45 min	300 rpm 300 rpm	96.29% 96.29%	[11]

ratio of 4 : 1 at 50 °C for 8 h. The maximum conversion reached using propan-2-ol was 92.8% from crude *Jatropha* oil. Reusability of the lipase was preserved over 12 repeated cycles with propan-2-ol whereas it reached zero by the 7th cycle when methanol was utilized as the acyl acceptor, under standard reaction conditions [64].

Modi [76] explored ethyl acetate as an acyl acceptor for immobilized lipase-catalyzed preparation of biodiesel from the crude oil of *J. curcas*. The optimum reaction conditions set for interesterification of the oils with ethyl acetate were 10% of Novozym 435 (immobilized *Candida antarctica* lipase B) based on oil weight, an ethyl acetate-to-oil molar ratio of 11 : 1, and a reaction period of 12 h at 50 °C. Under the above optimum conditions, the maximum result of ethyl esters was 91.3%. Reusability of the lipase over repeated cycles in interesterification and ethanolysis was also examined under standard reaction conditions. The relative activity of the lipase could be well preserved over 12 repeated cycles with ethyl acetate whereas it reached zero by the 6th cycle when ethanol was used as the acyl acceptor.

Shah and Gupta [67] conducted a process of optimization of monoethyl esters of long-chain fatty acids (biodiesel) by alcoholysis of *Jatropha* oil using lipase. The process included (a) screening of various commercial lipase preparations, (b) pH tuning, (c) immobilization, (d) varying water content in the reaction media, (e) varying amount of enzyme used, and (f) varying temperature of the reaction. The best yield of 98 wt-% was obtained by using *Pseudomonas cepacia* lipase immobilized on celite at 50 °C in the presence of 4–5 wt-% water in 8 h. The yield was not affected if analytical-grade alcohol was replaced by commercial-grade alcohol. This biocatalyst could be applied four times without loss of any activity.

In order to lower the cost of biodiesel fuel production from *Jatropha*, Tamalampudi *et al.* [71] used lipase-producing whole cells of *Rhizopus oryzae* which were immobilized on biomass support particles. The activity of *Rhizopus oryzae* was compared with Novozym 435, the most effective lipase. Methanolysis of *Jatropha* oil progressed faster than other kinds of alcoholysis, regardless of the lipase used. The maximum methyl ester content in the reaction mixture reached 80 wt-% after 60 h using *Rhizopus oryzae*, but 76% after 90 h using Novozym 435. Both lipases could be used for repeated batches. They also exhibited more than 90% of their initial activities after five cycles. Whole-cell immobilized *Rhizopus oryzae* is a promising biocatalyst for producing biodiesel from oil [71]. *Jatropha* biodiesel production from various sources of lipase and acyl acceptors is shown in Table 5.

3.1.4 Biodiesel production using supercritical alcohols

The synthesis of biodiesel from *Jatropha* oil has been investigated in supercritical methanol and ethanol without using any catalysts, from 200 to 400 °C at 200 bar [67]. It is found that, for the synthesis of biodiesel in supercritical alcohols with an optimum molar ratio of 50 : 1, very high conversions (>80%) were obtained within 10 min and nearly complete conversions within 40 min. The conversion into ethyl esters is higher than that into methyl esters [65].

Tang *et al.* [69] studied the transesterification of the crude *Jatropha* oil catalyzed by micro-NaOH (0.2 to 0.5 to 0.8 wt-‰) in supercritical methanol. When the catalyst content, reaction temperature and molar ratio of methanol to oil were developed at 0.8 wt-‰ NaOH, 534 K, 7.0 MPa and 24 : 1, respectively, the methyl ester yield could reach 90.5% within 28 min.

3.1.5 Biodiesel production using lipase-catalyzed *in situ* reactive extraction

According to Su *et al.* [21], extraction and lipase-catalyzed transesterification with methyl acetate and ethyl acetate can be done under the same conditions. They can be simply combined to a two-step one-pot *in situ* reactive extraction. First, the alkyl acetates performs as the extraction solvent and afterwards as the transesterification agent. Then, by removing the catalyst, defatted plant material (by filtration) and the solvent (by evaporation), the methyl/ethyl esters are obtained.

The negative effects of glycerol and alcohol on the lipase can be reduced by substituting short-chain alkyl acetates for short-chain alcohols as acyl acceptors for fatty acid ester production. Short-chain alkyl acetates are also appropriate solvents for seed oil extraction. Therefore, Su *et al.* [21] adopted

Table 5. The use of raw material for *Jatropha* biodiesel production from various chemical catalysts.

No.	Alcohol	Ratio alcohol/oil	Alcohol volume [mL]	Oil volume [mL]	FFA	Catalyst	Ratio catalyst	Catalyst weight [g]	Yield [%]	Ref.
1	Methanol	4.50 : 1 mol	182	965	0.29–1.27%	KOH	1.5 wt-%	13.5	92.00	[16]
	Ethanol	6.9 : 1.14 mol	353		0.29–1.27%	KOH	1.5 wt-%	13.5	88.40	
						H_2SO_4	2 wt-%	18.0		
2	Methanol	20% wt/wt	299	965	3.09%	NaOH	1 wt-%	9.0	98	[62]
3	Methanol	0.28 vol/vol	424	965	14%	H_2SO_4	1.43%	12.87	>99	[68]
		0.16 vol/vol				KOH	0.55% wt/vol	5.30		
4	a. Methanol	70% wt/wt	796	965	14.9%	NaOH	3.3 wt-%	29.7	55	[7]
	b. Methanol	60% wt/wt	955		14.9%	H_2SO_4	1 wt-%	9.0	90	
		24% wt/wt				NaOH	1.4 wt-%	12.6		
5	a. Methanol	9 : 1	364	965	RBD oil	NaOH	0.8 wt-%	7.2	96.29	[11]
	b. Ethanol	9 : 1	525			NaOH	0.8 wt-%	7.2	96.29	

methyl acetate and ethyl acetate as extraction solvents and transesterification reagents at the same time for *in situ* reactive extraction of *J. curcas* L. seed. Fatty acid methyl esters and ethyl esters were respectively obtained with higher yields than those resulting from conventional two-step extraction/transesterification. The improvement varied from 5.3 to 22%. The key parameters such as solvent/seed ratio and water content were further examined to find their effects on the *in situ* reactive extraction. The highest *J. curcas* L. methyl/ethyl ester yields could achieve 86.1 and 87.2%, respectively, under the optimized conditions [21].

This transesterification method reduces the risk of deactivation of the enzyme by short-chain alcohol and glycerol because in the reaction the short-chain alcohol is substituted by short-chain alkyl acetate and no glycerol is produced. Furthermore, such a route to fatty acid esters can decrease the expense associated with solvent extraction and oil cleanup. Due to its low-cost production, *in situ* reactive extraction would be very promising for fatty acid ester production [21].

3.2 Post-reaction processing

Other ester treatments and additization are required in order to fulfill the requirements of biodiesel standards such as ASTM D6751 or EN 14214 post-reaction processes, such as ester/glycerol separation, ester washing, ester drying. Ester washing and ester drying are needed due to the use of alkali catalysts. Ester washing is used to neutralize any residual catalyst and remove any soap formed, while ester drying removes the water used for washing [47].

As stated by Gerpen *et al.* [47], the ester/glycerol separation is usually the first step of product recovery in most biodiesel processes. This is done due to the fact that fatty acyl alcohol esters and glycerol are hardly mutually soluble. Besides, the ester (0.88 g/cm^3) and glycerol (1.05 g/cm^3) have a significant difference in density. The glycerol density is dependent on the amount of methanol, water, and catalyst in the glycerol. This density difference is sufficient for a simple gravity separation technique for the two phases [47]. The catalyst and unreacted alcohol were removed by hot-water washings [11]. To avoid the formation of an emulsion after the transesterification reaction, 0.1% aqueous citric acid was used as a washing solution to remove the catalyst. At last, the ester was dried in a conventional dryer vessel, under a nitrogen stream, for 1 h at 130 °C.

In order to remove any soaps formed during the esterification reaction, and to remove residual free glycerol and methanol, ester washing is used to neutralize any residual catalyst [47]. Three traditional methods for the refining step in biodiesel production were compared: (1) washing with distilled water, (2) washing with acid, and (3) dissolving and extracting in a solvent (hexane or petroleum ether) and then washing with distilled water. Although all three methods resulted in biodiesel with a high purity (97.5%), serious emulsification occurred during the refining processes, leading to high refining losses. So, a novel refining method was developed by using hollow-fiber membrane extraction, and polysulfone was chosen as the most suitable membrane. This process was effectively able to avoid emulsification during refining and to reduce the refining loss. The resulting purity of the biodiesel was about 99%, and other properties, such as density, kinematic viscosity, water content, and acid value, fulfilled the standards [77].

4 Discussion

Research on *Jatropha* biodiesel production has been performed extensively since the year 2000. The focus of the research has mainly been to find more effective and efficient technological processes for producing fuel-grade biodiesel from *Jatropha* oil. There are three important and correlated points of production that have to be considered: (1) oil preparation, (2) the transesterification reaction and (3) post-reaction processing. Oil preparation would affect the biodiesel processing, which subsequently affects post-reaction processing. A combination of oil preparation, transesterification reaction and post-reaction processing is engaged in producing a measurable output of fuel-grade biodiesel. A method by which the combination of these three processes can be controlled to produce fuel-grade biodiesel with consistent results needs to be developed in process control of biodiesel production.

4.1 Oil preparation

Jatropha oil is reportedly present in the range of 40–60 g oil/100 g *Jatropha* seed kernels [16, 17]. The oil content depends on the variety and growth conditions of the *Jatropha* plant. There are five oil extraction methods that have been developed: (a) mechanical extraction [28, 29]; (b) aqueous and solvent extraction, which comprise enzyme-assisted aqueous/solvent extraction, use of ultrasonication as a pretreatment prior to AOE and aqueous enzymatic oil extraction [21, 23, 27]; (c) the three-phase partitioning extraction method [25, 72]; (d) the SCE method [27] and (e) GAME [31, 32]. The objective of oil preparation is to find an efficient and effective method for extracting oil from *Jatropha* seed. The technique of three-phase partitioning with enzyme pretreatment and sonication constitutes an efficient procedure for obtaining oil from *Jatropha* seed kernels, which can extract 97% of oil within 2 h [72]. Extraction using ethyl acetate and methyl acetate is better than using hexane [21]. It was found that the GAME process for *Jatropha* oil extraction is capable of reaching yields up to 30 wt-% higher than conventional expression under the same conditions [31]. It is noted that the crude oil is subjected to acid degumming for removing hydratable and non-hydratable phosphatides, which is followed by alkali refining for removing the FFA [78].

Table 6. *Jatropha* biodiesel production from various sources of lipase and acyl acceptors (solvent free and batch reactor).

No.	Alcohols/ acyl acceptor	Lipase source	Amount of acyl acceptor[§]	Amount of enzyme[$]	Yield	Ref.
1	Ethyl acetate	*Candida antarctica*	873 mL		91.3%	[63]
2	Propan-2-ol	*Candida antarctica*			92.8%	[64]
3	Ethanol	*Pseudomonas cepacia* lipase immobilized on celite 10 wt-% oil	233 mL	90 g	98%	[66]
4	Methanol	Immobilized *P. flourescence* 6% wt/vol oil	160 mL	54 g	72%	[69]
5	Methyl acetate	*Candida antarctica*	595 mL		86.1%	[21]
	Ethyl acetate		735 mL		87.2%	
6	Methanol	*Rhizopus oryzae* immobilized onto biomass support particles	120 mL	54 g	80.2%	[70]
7	Ethanol	*Chromobacterium viscosum*			92%	[71]

[§] Based on volume.
[$] Weight of 1 mol of *Jatropha* oil.

4.2 Transesterification reaction

The five biodiesel processing methods developed for producing *Jatropha* oil are: (a) transesterification using a homogeneous chemical catalyst, (b) transesterification using a heterogeneous catalyst, (c) transesterification in supercritical alcohols, (d) transesterification using an enzyme catalyst, and (e) transesterification using lipase-catalyzed *in situ* reactive extraction. Lipases from various sources can be used to catalyze the transesterification process by immobilizing them on a suitable support. The advantage of immobilization is that the enzyme can be reused without a separation process [79]. The biodiesel yield of these methods is dependent on several factors, such as reaction temperature, molar ratio of alcohol to oil, catalyst, reaction time, presence of moisture and FFA, and mixing intensity [43–46].

Most of the commercial processes to produce biodiesel from plant oils use very effective homogeneous alkali catalysts such as NaOH, KOH or sodium methoxide. These catalysts can provide yields of the transesterification reaction close to 99% [80–83]. However, these alkali compounds have some disadvantages: the necessity of application of refined plant oils, problems related to the recovery of pure glycerol (the main by-product), and formation of soaps, mono- and diacylglycerols. Moreover, these compounds have to be neutralized, giving rise to wastewaters, and cannot be reutilized. The formation of stable emulsions makes the separation of methyl esters difficult, and the reaction becomes very sensitive to the presence of water and FFA. These problems can be solved by using a heterogeneous transesterification catalyst [84–86]. Heterogeneous catalysts are expected to be effective for producing biodiesel because of their low cost and minimal environmental impact, and their possibility of simplifying the production and purification process under mild conditions [87].

Considerable research on heterogeneous catalyst-enhanced transesterification of vegetable oils shows good performance and seems promising for potential applications [65, 84, 87–90]. However, the application of heterogeneous catalysts on *Jatropha* biodiesel production needs specific experiments.

A novel process of biodiesel fuel production has been developed by using non-catalytic [65, 69] and micro-catalytic supercritical alcohols [69]. The supercritical alcohol process involves a much simpler purification of products, has a lower reaction time, requires lower energy use and is more environmentally friendly. However, the reaction requires higher temperatures of 525–675 K and pressures of 35–65 MPa [56, 60].

Biodiesel can also be obtained from enzymatic or biocatalytic transesterification methods [56]. The concept of using enzymes for biodiesel production has been well understood in general. On the technical level, enzyme approaches could offer better possibilities and simpler technology for low-quality material (plant, animal or waste oils) compared with chemical methods currently used in biodiesel production [91]. A very recent review discussing both technical and economical considerations for enzymatic biodiesel production concluded a reasonable cost profile, based on results from immobilized lipases such as Novozym 435 (*Candida antartica* B) and Lipozym TL IM (*Thermomyces lanuginosa*). It now seems possible that new immobilization technologies can reduce the enzyme costs to a level comparable to chemical catalyst costs, *i.e.* 25 USD/t biodiesel produced [92].

The replacement of chemical catalysts with enzymes can make this process even more acceptable. This is first due to the fact that ethanol derived from renewable materials can be used for enzymatic biodiesel synthesis. Second, certain additional steps crucial for chemical synthesis such as saponification or acid-catalyzed esterification of FFA abundant in worse-quality fat substrates, catalyst neutralization, removal of soaps and product purification are omitted. Third, raw oils (no need of refining) and waste fats can be used as lipid substrates. Fourth, the enzymatic process is carried out at much lower temperatures and consumes much less energy. Next, enzymatic preparations can be reused and/or applied in continuous biodiesel synthesis processes if their operational stability is sufficiently high. At last, biocatalysts are biodegradable and non-toxic unlike some chemical catalysts [86]. However, there is still work to be done with respect to the up-scaling of the process layout from laboratory or pilot scale to production [92].

The application of ultrasonic irradiation [49–51] and microwave heating [93–95] in order to promote the transesterification of *Jatropha* oil seems to be possible. The results indicated that ultrasonic irradiation and microwave heating effectively increased the biodiesel yield and decreased the reaction time. Detailed information concerning their performance on *Jatropha* oil transesterification, however, is not available.

4.3 Post-reaction processing

Post-reaction processes comprise ester/glycerol separation, ester washing, ester drying, alcohol recovery and glycerol refining. These steps are very important in order to produce fuel-grade biodiesel and to decrease biodiesel oil cost through alcohol recovery and glycerol refining. There are not yet any publications dealing with these post-reaction processes.

For best economy and pollution prevention, the alcohol must be fully recycled. Glycerol is an economical co-product that should be fully refined [47]. As by-product, 1 mol of glycerol is produced for every 3 mols of methyl esters, which is equivalent to approximately 10 wt-% of the total product. The glycerol markets have reacted strongly to the increasing availability of glycerol. Although the global production of biodiesel is still limited, the market price of glycerol has dropped rapidly [96]. Therefore, new uses for glycerol need to be developed and economical ways to utilize low-grade glycerol should be further explored.

As *Jatropha* oil possesses a significant amount of fatty acids with double bonds, oxidative stability is a concern, especially when storing biodiesel over an extended period of time. The storage problem is worsened by storage conditions like exposure to air and/or light, temperatures above ambient, and the presence of extraneous materials (contaminants) with catalytic effects on oxidation [97]. The presence of air or oxygen will hydrolyze the oil to alcohol and acid. While the presence of alcohol will lead to a reduction in flash point, the presence of acid will increase the total acid number. All these conditions make methyl esters relatively unstable during storage and cause damage to engine parts [98]. That is why oxidative stability is an important criterion for biodiesel production.

The stability of *Jatropha* biodiesel is very critical. Various strategies for the improvement of biodiesel fuel quality have been suggested. Biodiesel requires antioxidants to meet the storage requirements and to ensure fuel quality at all points along the distribution chain. In order to meet the EN 14112 specification, a concentration of antioxidant of around 200 ppm is required which needs much higher antioxidant concentrations than those required for petroleum diesel industry. To minimize the dosage of antioxidant, appropriate blends of *Jatropha* and palm biodiesel are made. The antioxidant dosage could be reduced by 80–90% if palm oil biodiesel is blended with *Jatropha* biodiesel at around 20–40% concentration [41]. An alternative strategy would be the addition of fuel additives used in the petrodiesel industry [61].

4.4 Process control of biodiesel processing

There are three critical processes of production and three outputs that have to be considered; they are: (1) oil preparation, (2) the transesterification reaction, and (3) post-reaction processing. In process control, we must ensure that both quantitative and qualitative product specifications and economic performance meet the health, safety and environmental regulations. The tasks of a control system are to ensure the stability of the process, to minimize the influence of disturbances and perturbations, and to optimize the overall performance [99]. Figure 3 shows a schematic diagram of a process control approach for *Jatropha* biodiesel processing.

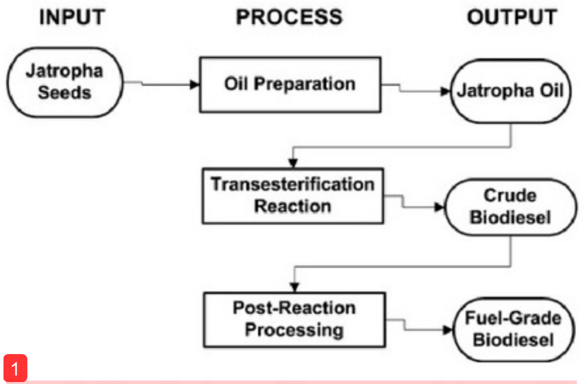

Figure 3. Schematic diagram of the process control approach for *Jatropha* biodiesel processing.

The following is a sequence of steps that could be employed to develop a process control procedure for best performance and cost-effective and environmentally friendly *Jatropha* biodiesel production:

(1) Constructing a process flow diagram which identifies the major process operations.

(2) Developing a strategy to improve the quality of *Jatropha* seed input. This strategy involves pre-harvest treatments and post-harvest handling and technology.

(3) Identifying the output characteristics that will be achieved.

(4) Determining the principle process that will be applied for every output characteristic.

(5) Identifying detection methods used to detect production problems and to prevent causes in the determined process.

(6) Evaluating and analyzing the cost feasibility of the process while always fulfilling health, safety and environmental regulations.

(7) Reviewing various possible actions for production system improvements.

5 Conclusions

High cost of biodiesel production is the major impediment to its large-scale commercialization. Methods to reduce the production cost of *Jatropha* biodiesel must be developed. There are still many future potential improvements of biodiesel production of *J. curcas*. These include (1) development of better and cheaper oil extraction and post-reaction processing methods; (2) development of better and cheaper catalysts; (3) improvements in the current technology for producing high-quality biodiesel with lower production costs; (4) development of technologies to use methanol/ethanol in *in situ* extraction and transesterification; (5) development of techniques to improve the fuel stability of *Jatropha* biodiesel; and (6) conversion of by-products, such as glycerol, to useful products, such as methanol and ethanol or glycerol *tert*-butyl ether (GTBE). For the time being, we have been working on *in situ* extraction and transesterification, developing heterogeneous catalysts for the esterification/transesterification reaction and developing heterogeneous and ionic liquid catalysts in GTBE synthesis from glycerol, the by-product of *Jatropha* biodiesel production.

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Conflict of interest statement

The authors have declared no conflict of interest.

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