Research Article

Biodiesel synthesis from *Jatropha curcas* L. oil and ethanol in a continuous centrifugal contactor separator

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The synthesis of fatty acid ethyl esters (FAEE) from *Jatropha curcas* L. oil was studied in a batch reactor and a continuous centrifugal contactor separator (CCCS) using sodium ethoxide as the catalyst. The effect of relevant process variables like rotational speed, temperature, catalyst concentration, and molar ratio of ethanol to oil was investigated. Maximum yield of FAEE was 98 mol% for both the batch (70°C, 600 rpm, 0.8% w/w of sodium ethoxide) and CCCS reactor configuration (60°C, 2100 rpm, 1% w/w of sodium ethoxide, oil feed 28 mL/min). The volumetric production rate of FAEE in the CCCS at optimum conditions was 112 $kg_{FAEE}/m^3_{liquid} \cdot min$.

Practical applications: The *Jatropha curcas* L. shrub produces a non-edible oil in yields up to 1.5 ton/(ha. y), which is known to be suitable for biodiesel synthesis. We here report an experimental study on the synthesis of jatropha biodiesel using ethanol instead of methanol as the alcohol source. Ethanol is accessible from biomass by fermentation and as such a green alternative for methanol. In addition, it is more readily available in developing countries than methanol. Biodiesel synthesis was performed in a CSSS, a continuous device that integrates reaction and separation. The device has potential to be applied in small scale mobile biodiesel technology due to its compact size, robustness, flexibility in operation, and high volumetric productivities.

1 Introduction

Biodiesel is a very attractive biofuel that is produced from various virgin plant oils as well as waste cooking oils [1–4]. In 2009, the EU passed the Renewable Energy Directive (RED) which effectively established that renewable energy should account for 10% of the energy consumed in the transportation sector by 2020 [5]. This has stimulated the production of biofuels in Europe and for instance the current biodiesel production is estimated at 9.57 million tons/year (2010), 5.5% higher than 2009 [6].

Conventional biodiesel production involves trans-esterification of a plant oil with methanol in combination with a suitable catalyst [7–12]. Various studies have been conducted to gain insights in the process parameters affecting the transesterification reaction [13–18]. New reactor and process configurations have been developed recently such as the continuous centrifugal contactor separator (CCCS), a device that integrates reaction and separation of liquid–liquid systems in a single device [19–28].

The CCCS (Fig. 1) basically consists of a hollow rotating centrifuge in a static reactor house. The immiscible liquids enter the device in the annular zone between the static housing and the rotating centrifuge, where they are intensely mixed. The formed dispersion is then transferred into the hollow centrifuge, through a hole in the bottom. Here the phases are separated by centrifugal forces up to 900*g*, allowing excellent separation of the fluids [26–29]. We have

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Abbreviations: CCCS, continuous centrifugal contactor Separator; $F_{ethanol}$, ethanol flow rate [mL/min]; **FAEE,** fatty acid ethyl esters; **FAME,** fatty acid methyl esters; F_{oil}, jatropha oil flow rate [mL/min]; *N*, rotational speed [rpm]; *T*, temperature [°C]
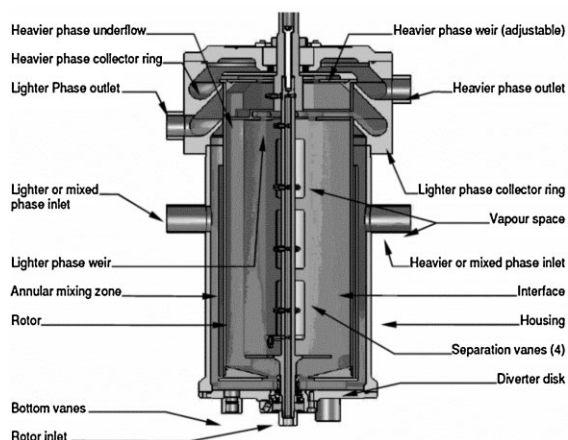
Figure 1. Schematic cross-sectional view of the CCCS (courtesy of Auxill, The Netherlands).

previously demonstrated the potential of the CCCS (type CINC V02) to produce fatty acid methyl esters (FAME) from sunflower oil and methanol with a reproducible yield of 96 mol% [29].

In this study, the application of *Jatropha curcas* L. (jatropha) oil for biodiesel synthesis with ethanol as the alcohol source is explored. Jatropha oil has received high interest in the last decade, as the oil is non-edible and has potential for a high oil productivity per ha per year. [30–35]. Ethanol was selected instead of conventional methanol as it has better solvent properties and lower toxicity [36] and the ethyl esters are reported to have a higher heat content and cetane number than the corresponding methyl esters [37]. Furthermore, ethanol is readily accessible from renewable resources like sugarcane and allows for the synthesis of a 100% renewable biofuel [38–39].

Various plant oils have been tested for the trans-esterification with ethanol and an overview is given in Table 1. Most

of the studies were carried out in batch set-ups at laboratory scale using potassium hydroxide and sodium methoxide catalysts. For jatropha oil, four exploratory studies have been reported with KOH, NaOH, and NaOMe as the catalysts yielding the ethyl esters in 88–99+ mol% yield. Best results were obtained at 30°C, giving >99.5% yield, though reaction times were long and exceeded 2 h. Higher temperatures (70–75°C) lead to shorter reaction times (30 min) though the yields were considerably lower. However, systematic studies on jatropha oil synthesis with ethanol are lacking. We here report systematic studies on the ethanolysis of jatropha oil in a batch and a continuous CCCS device. The effects of process variables on FAEE yield were investigated for both reactors to optimize the biodiesel yield.

2 Materials and methods

2.1 Materials

The *Jatropha curcas* L. oil was obtained by pressing seeds (Cape Verde origin) with a mechanical screw press (DansihBT50). Ethanol (absolute, pro analysis) was obtained from Emsure. Sodium ethoxide solution (21%) in ethanol and $CDCl_3$ (99.8%) were obtained from Sigma–Aldrich.

2.2 Synthesis of FAEE in a batch reactor

The synthesis of FAEE was performed in a 250 mL glass batch reactor. The reactor was equipped with a double wall heating/cooling jacket connected to a thermostated water bath. Stirring was performed with a six-blade Rushton turbine with an impeller of 1.4 cm diameter, placed 0.5 cm from the bottom. The temperature and rotational speed were varied between 30–70°C and 400–800 rpm, respectively. A sampling port was provided for sample collection during the reaction. Baffles were inserted to enhance mixing. Sampling was performed at fixed intervals. The samples were quenched

Table 1. Overview of trans-esterification reactions of plant oils with ethanol

Oil source	Catalyst	Conditions	Yield	References
Cyanara cardunculus oil (12:1 molar ratio ethanol:oil)	1% w/w NaOH	Agitated batch, 75°C	94.5% after 10 min	[36]
Triolein (6:1 molar ratio ethanol:oil)	1% w/w KOH	Agitated batch, 25°C, 1800 rpm	70% after 5 min	[40]
Rapeseed oil (5.9:1 molar ratio ethanol:oil)	2% mol/mol KOH	Agitated batch, 60 °C, 500 rpm	94% after 120 min	[41]
Palm kernel oil (2:10 volume ratio ethanol:oil)	1% w/w KOH	Agitated batch, 60°C, stirring speed unknown	96% after 120 min	[42]
Sunflower oil (7.3:1 molar ratio ethanol:oil)	2% w/w KOH	Agitated batch, 80°C and 600 rpm	88% after 60 min	[43]
Castor oil (16:1 molar ratio ethanol:oil)	1% w/w $NaOCH_2CH_3$	Agitated batch, 30°C, 400 rpm	99% after 30 min	[44]
Jatropha oil (excess ethanol)	KOH, distilled water, H_2SO_4	Refluxed, 70°C	93% after 60 min	[45]
Jatropha oil (6:1 molar ratio ethanol:oil)	3% w/w KOH	Agitated batch, 75°C	83% after 90 min	[46]
Jatropha oil	2.0% w/w CH_3ONa	Agitated batch, 30°C, 600 rpm	99.98% after 120 min	[47]
Jatropha oil (7.5 v/w of ethanol:oil)	0.675% w/w NaOH	Agitated batch, 30°C, 300 rpm	99.53% after 150 min	[48]

with 0.1 M HCl in water and analyzed with ^{1}H NMR (*vide infra*).

2.3 Synthesis of FAEE in a CCCS

The synthesis of FAEE was performed in a CCCS type CINC V02 equipped with a heating/cooling jacket and a high-mix bottom plate. The annular zone was extended to 45 mL. A weir size of 0.875″ was used for all experiments. The jatropha oil and ethanol solution containing the appropriate amount of the sodium ethoxide catalyst were preheated to 70°C, while the jacket temperature was maintained at 60°C. The rotor (30–45 Hz) and the oil feed pump (12–36 mL/min) were started. As soon as the oil started to exit the heavy phase outlet, the reaction was started by feeding the sodium ethoxide in ethanol solution (0.75–1.5% w/w NaOEt with regards to the oil) at a flow rate of 4.4–40.5 mL/min. During a run, samples were taken from the crude FAEE exit flow and the samples were analyzed using ^{1}H NMR (*vide infra*).

2.4 Analytical methods

The FAEE yield was determined using ^{1}H NMR. A 0.5 mL sample of the crude FAEE phase was directly quenched by adding 0.5 mL of a 0.1 M HCl solution in water to neutralize the remaining sodium ethoxide. The dispersion was centrifuged for 10 min. A few drops were taken from the top layer and dissolved in CDCl$_3$. The samples were then analysed using a 200 MHz Varian NMR. The FAEE yield was determined by comparing the intensity of the characteristic quartet signal of the CH$_2$ group of the ester end group (δ 4.1 ppm) with respect to the signal of the methyl end group of the fatty acids (δ 0.9 ppm). However, one of the resonances of the hydrogen atoms of the CH$_2$ group of glycerol attached to the remaining triglycerides (δ 4.1 and 4.3 ppm) overlaps with the ester end-group and a correction was made to compensate for this effect [49].

The fatty acid composition of the oil was analyzed by gas chromatography–mass spectrometry (GC–MS) using a Hewlett-Packard (HP) 5890 series II Plus device in combination with a with HP chemstation G1701BA using the B0100/NIST library software. A fused-silica column (HP-5 column: 30 m length × 0.25 mm internal diameter and 0.25 μm film thickness) was used. Helium was used as the gas carrier at a column pressure of 49 kPa. The oven temperature was initially set at 40°C for 4 min. Next, the temperature was increased to 180°C at a rate of 15°C/min. The temperature was then increased at 5°C/min to 280°C and held for 10 min at this temperature. A split ratio of 1:100 was used, the injector and detector temperature were set at 280°C whereas the gas flow rate was set to 1 mL/min.

The water content in the samples was measured by Karl Fischer (KF) titration using a 702SM Titrino titrator. One

milliliter of sample was weighed and injected into the reaction vessel. The amount of water present in the sample was calculated based on the amount of KF reagent (Hydranal Solvent) consumed in the titration.

The acid value of the samples was measured by an acid–base titration using phenolphthalein as the indicator. The sample (3 g) was dissolved in 20 mL of an ethanol/ether solvent mixture (1:1). A few drops of phenolphthalein were added and the solution was titrated with KOH (0.1 N) in ethanol until a faint color persisted. The acid value is the mass of KOH (in milligrams) required to neutralize 1 g of sample. The normality of the KOH solution was determined by a titration with an oxalic acid solution (0.2 N).

The phosphorus and Na content of the jatropha oil and biodiesel products was performed at ASG Analytik-Service GmbH, Neusass, Germany according to the methods described in EN 14107 and EN 14108, respectively.

The flash point of the samples was measured according to the methods described in ASTM D 6450 using a MINIFLASH FLP/H/L.

Cloud- (CP) and pour point (PP) were measured using a Tanaka Scientific Limited Type MPC-102 L. The CP and PP of the samples were measured according to methods described in ASTM D 6749 and ASTM D 2500.

3 Results and discussion

3.1 Experiments in a batch reactor

Exploratory experiments were performed in a batch reactor to obtain the proof of principle for the ethanolysis of jatropha oil using sodium ethoxide as the catalyst and to gain insights in typical reaction rates for FAEE synthesis. The experiments were carried out with an oil obtained from the seeds from Cape Verde. The fatty acid content was determined (GC) and shown to consist mainly of palmitic acid (13% w), linoleic acid (35.5% w), oleic acid (45.7% w), and stearic acid (5.8% w). The measured fatty acid content is within the range reported in the literature viz.; 12–15% w for palimitic acid, 35–54% w for linoleic acid, 14–46% w for oleic acid and 5–19% w for stearic acid [32, 35]. The acid value was 1.6 mg KOH/g oil, corresponding with an FFA of 2.5%. The phosphorus and water content of the oil were 17.7 and 404 mg/kg, respectively. All values are well below the standards for plant oils, and therefore the oil was not purified prior to a trans-esterification reaction.

Operating conditions and particularly the molar ratio of ethanol to oil (6:1–24:1), catalyst concentration (0.6–1.2% w/w on oil), temperature (30–70°C), and rotational speed (400–800 rpm) were varied systematically to determine optimum conditions for highest FAEE yield. The results of the optimization studies are illustrated in Figs. 2–4.

The ethanol to oil ratio within the experimental range (6:1–24:1) did not have a significant effect on the FAEE yield (figure not shown), and a maximum FAEE yield of 98 mol%
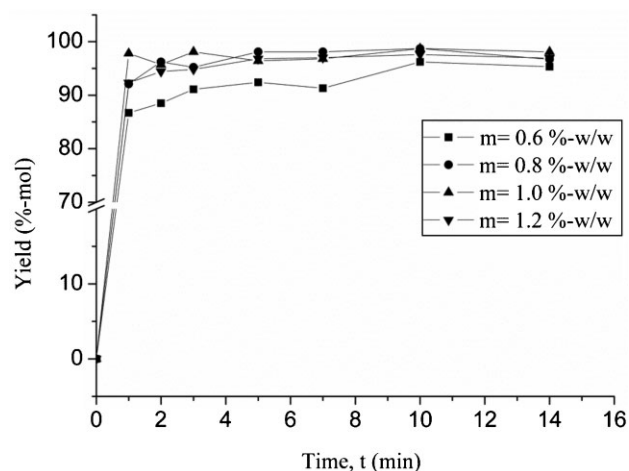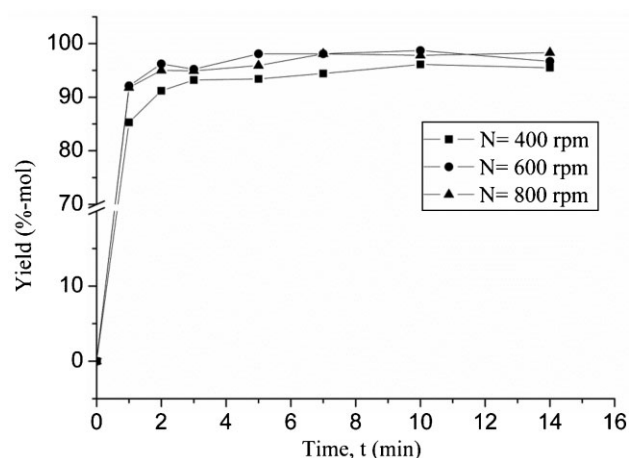
Figure 2. Effect of catalyst concentration (6:1 molar ratio of ethanol to oil, 70°C, 600 rpm) for jatropha oil ethanolysis in batch.

Figure 3. Effect of rotational speed (6:1 molar ratio of ethanol to oil, 0.8% w/w of catalyst concentration, 70°C) for jatropha oil ethanolysis in batch.

was obtained after about 5 min, irrespective of the ratio. Apparently, a 6–1 excess is sufficient to drive the trans-esterification reaction to a yield >96% [15]. It is also of particular interest to observe that the reaction is very fast, and essentially complete within 5 min, which is much faster than the literature data reported in Table 1.

The effect of catalyst concentration on the FAEE yield versus the reaction time is given in Fig. 2. A minimum catalyst concentration of 0.8% w/w is required to achieve a FAEE yield of 98 mol% after 5 min. Higher catalyst concentration did not lead to higher yields. As such, a catalyst intake of 0.8% w/w seems preferred, both from an economic point of view, as well as to prevent the formation of soap at higher catalyst loadings. Soap formation by saponification is undesirable as it partially consumes the catalyst and complicates subsequent separation and purification steps [9].

Figure 3 shows the effect of rotational speed on the FAEE yield versus time. Though some scatter in the data is present, the reaction rate at 400 rpm is lower than at 600 and 800 rpm. A possible explanation is the occurrence of mass transfer limitations. It is well known for trans-esterifications of plant oils with methanol that the reaction mixture is a liquid–liquid system at low conversions, consisting of a methanol rich and plant oil rich phase [53–55]. In this situation, mass transfer of reactants may have an effect on the overall reaction rate and this apparently is also the case for ethanol.

The effect of reaction temperature on the FAEE yield is presented in Fig. 4. Higher reaction temperatures enhance the rate of the esterification reaction as well as the FAEE yield. Temperature effects are reported to be pronounced for the rates of trans-esterification reactions [7, 53–55]. Of interest is also the temperature dependence of the maximum FAEE yields. The yield was highest at 70°C (98 mol%), and reduced to 93 and 91 mol% for the reactions at 50

and 30°C, respectively. These data suggest that the equilibrium position of the trans-esterification reaction shifts to the right at higher temperatures, implying that the trans-esterification reaction is slightly endothermic. Similar temperature dependencies of the equilibrium position were observed by Narvaez et al. [53] for palm oil methanolysis. Here, the maximum (equilibrium) conversion increased from 90.6% at 50°C to 94.3% at 60°C.

Noureddini and Zhu [54] reported that the maximum conversion for the trans-esterification of soybean oil at 70°C (90%) is considerably higher than at 50°C (80%). In addition, Vicente et al. [7] reported that the maximum conversion at 65°C (99%) is higher than at 25°C (94%) for sunflower oil methanolysis.
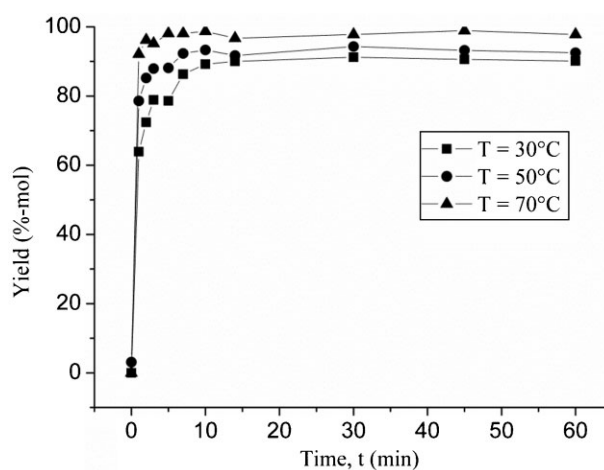
Figure 4. Effect of temperature (6:1 molar ratio of ethanol to oil, 0.8% w/w of catalyst concentration, 600 rpm) for jatropha oil ethanolysis in batch.

Thus, on the basis of this batch study, we can conclude that the synthesis of FAEE is a fast reaction and at optimum conditions (6:1 molar ratio of ethanol to oil, 0.8% w/w of catalyst concentration, 600 rpm and 70°C) a reproducible FAEE yield of 98 mol% was achieved after approximately 5 min reaction time. Our reaction rates are much faster than reported for jatropha oil ethanolysis in the literature [45–48], for which more than 60 min were required to achieve a maximum yield of 93% w/w (Table 1). A possible explanation for these differences is a higher FFA and/or water level in the jatropha oil feeds used in these studies, leading to a loss in catalytic activity. For instance, the jatropha oil used by Shah et al. [45] was obtained by mechanical pressing and was used as such without any pretreatment. Another explanation for the observed lower reactivity are the low temperature (30°C) and low stirring speed (300 rpm) at which these experiments were performed [48].
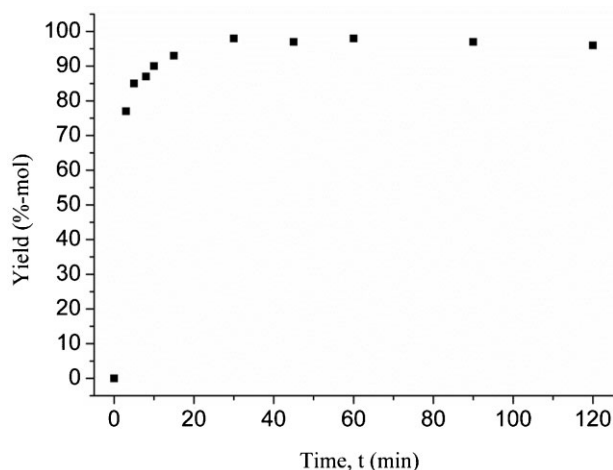
3.2 Synthesis of FAEE from jatropha oil in a CCCS

The production of FAEE was performed with sodium ethoxide as the catalyst in a CCCS type CINC V02 using the same jatropha oil feed as in the batch studies. The effect of the flow rate of the oil (12–36 mL/min), ethanol (4.4–13.2 mL/min), catalyst concentration (0.75–1.5% w/w with regard to the oil) and rotational speed (1800–2700 rpm) on the FAEE yield were assessed. The operating temperature was set at 60°C for all experiments. Experiments at 70°C, the optimum temperature determined for batch experiments (*vide supra*), were also performed but this lead to reduced separation performance, likely due to ethanol evaporation in the hotter parts of the device. The ethanol flow rate (containing the sodium ethoxide catalyst) was coupled to the oil flow rate to ensure the desired ethanol to oil molar ratio. An overview of ranges of operational variables and a base case is given in Table 2.

A typical profile of the FAEE yield versus time is given in Fig. 5 (refer to Table 3 for the operating conditions). After about 30 min, steady state was achieved with, in this particular experiment, an FAEE yield of 98% mol. Typically, steady state is achieved within four to five residence times for continuous reactors [51]. When considering a total flow rate of

Figure 5. FAEE yield (%-mol) versus time in the CCCS (6:1 molar ratio of ethanol to oil, 1.0% w/w of catalyst concentration, 60°C, 2100 rpm).

Table 3. Optimum conditions for the ethanolysis of jatropha oil in a CCCS and a batch reactor

Parameter	CCCS	Batch reactor
Molar ratio of ethanol:oil	6:1	6:1
Catalyst concentration (% w/w)	1.0	0.8
Oil flow rate (mL/min)	28	n/a
Ethanol flow rate (mL/min)	10.3	n/a
N (rpm)	2100	600
T (°C)	60	70
FAEE yield (% mol)	98	98
Productivity ($kg_{FAEE}/m^3_{liquid} \cdot min$)	112	176

Table 2. Base case and ranges of variables for the ethanolysis of jatropha oil in the CCCS

Parameter	Base case	Range
Molar ratio of ethanol:oil	6:1	6:1–24:1
Catalyst concentration (% w/w)	1.0	0.75–1.5
Oil flow rate (mL/min)	12	12–36
Ethanol flow rate (mL/min)	4.4	4.4–40.5
N (rpm)	2100	1800–2700
T (°C)	60	n/a

about 30 mL/min and a geometric volume of the CCCS of about 220 mL, the residence time in case of a high liquid hold-up is in the order of 7 min. Thus, the observed time to reach steady state is well within the values predicted by theory.

The reported FAEE yield for each experiment is the average yield after the system reached steady state. The results of the optimization of each parameter with respect to the FAEE yield are illustrated in Figs. 6–9 and will be discussed in the following sections.

3.2.1 Effect of catalyst concentration

The catalyst concentration was varied from 0.75 to 1.5% w/w with respect to the oil phase, while other parameters were kept constant at base case conditions (Table 2). As shown in Fig. 6, an optimum FAEE yield of 94 mol% was obtained for a catalyst concentration of 1.0% w/w. This result resembles
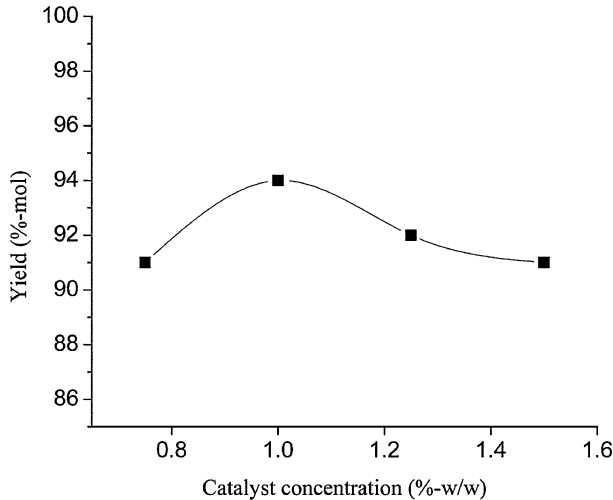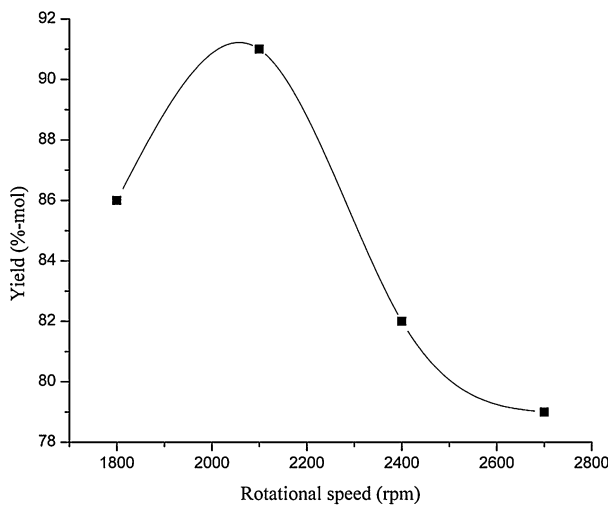
Figure 6. FAEE yield (%-mol) versus time in the CCCS as a function of the catalyst concentration (6:1 molar ratio of ethanol to oil, F_{oil}: 12 mL/min, $F_{ethanol}$: 4.4 mL/min, 60°C, 2100 rpm).

the results of a previous study on sunflower oil methanolysis in a CCCS [29], where lower yields at higher catalyst intakes were related to saponification.

3.2.2 Effect of rotational speed

The effect of rotational speed on FAEE yield was assessed in the range 1800–2700 rpm while keeping the other parameters constant at base case values (Table 2) and the result are given in Fig. 7. Clearly, the yield is a function of

the rotational speed and actually an optimum value at about 2100 rpm was observed. Similar effects were recently demonstrated for sunflower oil methanolysis in a CCCS, with a maximum yield in the range of 1800–2400 rpm. [29].

It is well possible that at lower rotational speeds (< 2100 rpm), mass transfer effects play a role and reduce the overall reaction rate. However, when mass transfer limitation is negligible, as expected at higher stirring rates, a constant FAEE yield is expected, corresponding with the intrinsic reaction rate [29, 51]. This is clearly not the case here and actually the yield decreases at rotational speeds beyond 2100 rpm. A possible explanation may be related to variations in hold-ups of both liquids in the CCCS at different rotor speeds, leading to changes in the residence times of the liquids in the device. When the liquid hold ups are lowered, the residence times will reduce and as a consequence, the yields are expected to drop. Hydrodynamic investigations are in progress to determine the liquid hold-ups as a function of the rotational speeds and will be reported in due course.

3.2.3 Effect of oil flow rate

The oil flow rate was varied in the range of 12–36 mL/min while the other parameters were kept constant at base case conditions (Table 2). The ethanol flow rate was coupled to the oil flow rate to ensure a fixed sixfold molar excess of ethanol over jatropha oil [15]. Figure 8 shows the effect of oil flow rates at different rotational speeds with regards to the FAEE yield. An optimum yield of 98 mol% was achieved at a rotational speed of 2100 rpm and an oil flow rate of 28 mL/min.
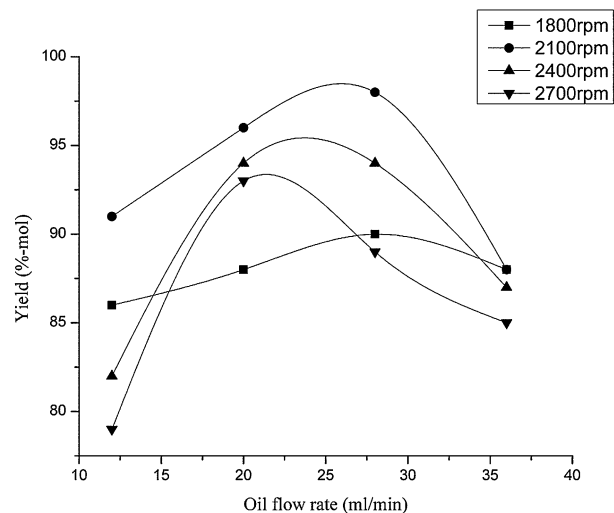

Figure 7. FAEE yield (%-mol) versus time in the CCCS as a function of rotational speed (6:1 molar ratio of ethanol to oil, 1.0% w/w of catalyst concentration F_{oil}: 12 mL/min, $F_{ethanol}$: 4.4 mL/min, 60°C).
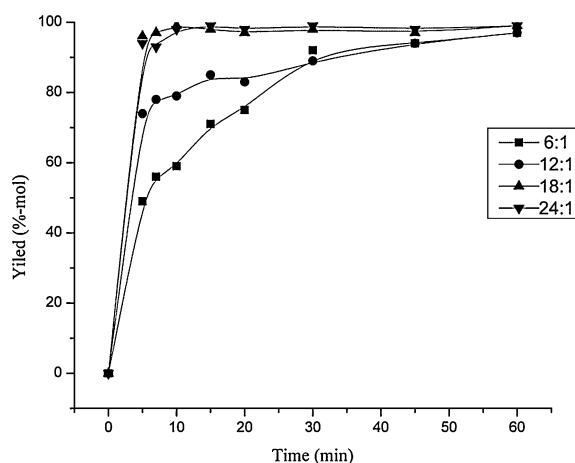

Figure 8. FAEE yield (%-mol) versus time in the CCCS as a function of flow rate and rotational speed (6:1 molar ratio of ethanol to oil, 1.0% w/w of catalyst concentration F_{oil}: 12–36 mL/min, $F_{ethanol}$: 4.4–13.3 mL/min, 60°C).

This optimized oil flow rate is considerably higher than our previous results on sunflower oil methanolysis (16 mL/min) in a similar CCCS. As a result, the liquid throughput may be considerably increased, leading to a significantly larger volumetric productivity of 112 $kg_{FAEE}/m^3_{liquid} \cdot min$ as compared to 67 $kg_{FAME}/m^3_{liquid} \cdot min$ in the case of continuous synthesis of sunflower oil methanolysis in the CCCS.

To the best of our knowledge systematic studies in the literature to compare the reactivity of methanolysis versus ethanolysis for plant oils in general and jatropha oil in particular have not been reported. In fact, the scattered results are conflicting [36, 37, 56], and some authors have reported that ethanol is less reactive than ethanol. These conflicting results are likely due to the fact that the reaction is a reactive liquid–liquid system, for which the overall kinetics are not only determined by intrinsic kinetics of the reaction but also by mass transport of reactive components between the L–L interface. Regarding mass transfer limitations, these are expected to be less of importance than ethanol as the solubility of the plant oil in ethanol is better than in methanol [36, 50].

3.2.4 Effect of the ethanol to oil molar ratio

The effect of the ethanol to oil molar ratio was studied in the range of 6:1–24:1 while other parameters were kept constant. The oil flow rate was set at 28 mL/min and the ethanol flow rate was set accordingly to achieve the desired ethanol to oil molar ratio. Other parameters were as given in Table 2. When increasing the ethanol to oil molar ratio from 6:1 to 18:1 the time to reach steady state was considerably reduced, an implication for a considerably higher reaction rate (Fig. 9). However, a further increase to 24:1 ratio did not lead to further rate enhancements. Eventually, after reaching the

Figure 9. FAEE yield (%-mol) versus time in the CCCS as a function of molar ratio ethanol to oil (1.0% w/w of catalyst concentration F_{oil}: 28 mL/min, $F_{ethanol}$: 10.3–40.5 mL/min, 60°C, 2100 rpm).

steady state, the FAEE yield for all ethanol to oil molar ratios was approximately similar and reached 98 mol%.

3.3 Volumetric production rates of FAEE in batch and the CCCS

The volumetric production rate of FAEE in the CCCS at optimized setting (see Table 3) was estimated to be 112 $kg_{FAEE}/m^3_{liquid} \cdot min$. This is slightly lower than the estimated productivity for a batch process (176 $kg_{FAEE}/m^3_{liquid} \cdot min$). It implies that further improvements of jatroha oil ethanolysis in the CCCS device are possible by further process optimization and modeling studies. Nevertheless, the unique advantage of a CCCS is that it eliminates the need for a settling tank after reaction to separate the glycerol layer from the FAEE, which may take up to 2 h to obtain complete phase separation [52]. Furthermore, the CCCS also offers all the benefits of continuous processes (product consistency, higher time on stream), making a CCCS an attractive device for the synthesis of biodiesel.

The estimated productivity of 112 $kg_{FAEE}/m^3_{liquid} \cdot min$ corresponds to an annual production volume of about 10.1 ton/year. When using a commercially available CCCS type CINC V20 with a maximum flow throughput of 757 L/min, the production volume for this larger CCCS device may be as high as 4.04 kton/year. In this calculation, it is assumed that (i) the volumetric production rate in the CINC V20 is equal to that in the smaller CCCS and (ii) the volumetric ratio for both devices is about 400 [29].

3.4 Properties of FAEE

Relevant properties of the FAEE after being washed with reverse osmosis water and drying with compressed air are shown in Table 4. When possible, the properties were compared to the biodiesel standard set in EN 14214. The water, sodium, phosphorous, acid number, and the flash point are well within the specification.

Table 4. Properties of FAEE produced at optimum conditions in the CCS

Property	Refined FAEE	Specification limit EN 14214[a]
Water content	327 mg/kg	500 mg/kg max
Acid value	0.39 mg KOH/g	0.5 mg KOH/g
Ethanol content	n.d.[b]	
Cloud point	2°C	–
Pour point	0°C	–
Sodium content	< 0.5 mg/kg	5 mg/kg max
Phosphorus content	< 0.5 mg/kg	10 mg/kg max
Flash point	162°C	120°C min

[a] For biodiesel using methanol as the alcohol source.
[b] n.d, not detected based on ^1H NMR measurements.

4 Conclusions and outlook

Jatropha oil ethanolysis has been studied in a batch and continuous reactor. At optimum conditions, a reproducible FAEE yield of 98 mol% was obtained for both the batch and continuous reactor configurations. The experimental volumetric productivity for the CCCS is slightly lower than for the batch system. However, as the CCCS is very compact, robust, and flexible in operation, it has high potential to be used in small scale mobile biodiesel units. Actually, such a mobile unit may consist of a cascade of two CCCS in series, one for biodiesel synthesis and a second for a refining step with water/acid, followed by ethanol removal in a stripper. The design of such a unit and experimental validation is currently in progress.

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