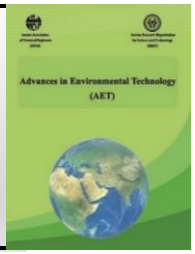
Advances in Environmental Technology 2 (2020) 83-89

Advances in Environmental Technology

journal homepage: http://aet.irost.ir



A novel method for the synthesis of biodiesel as an eco-friendly and sustainable fuel

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ARTICLE INFO

Article history:
Received 24 October 2020
Received in revised form
4 April 2021
Accepted 10 April 2021

Keywords:
Reactive Red 120
Ultrasonic transducer's network
Optimization
Response surface methodology
Artificial neural network

ABSTRACT

Biodiesel is a green diesel fuel that is synthesized via the transesterification reaction of plant oils or animal fats with light alcohols, mainly methanol, in the presence of commonly homogeneous alkaline catalysts. One way to make the biodiesel synthesis process more eco-friendly is the use of heterogeneous catalysts as they are reusable. But with these catalysts, the transesterification reaction mixture will be a three-phase mixture containing methanol, oil and catalyst that leads to long reaction times and high process costs. In this study, the ultrasound waves were used to synthesis of the biodiesel from the Jatropha oil in the presence of CaO as a heterogeneous catalyst. The effect of two different co-solvents (acetone & hexane) on improving the homogeneity of the reaction mixture and therefore to enhance the conversion yield of the oil to biodiesel was considered. Experiments were carried out by a probe type ultrasonic instrument at an output power of 200 W and frequency of 20 kHz. The results showed that both of the considered co-solvents had positive effect on developing the conversion yield and the acetone was the more efficient one due to its moderate polarity, which could improve the miscibility of the oil with the methanol. A biodiesel sample with a conversion yield of 90.33% was synthesized under optimal operating conditions including 25% v/v acetone to methanol, 12:1 methanol to oil molar ratio, 4% w/w catalyst to oil, 55°C reaction temperature and 60 min reaction time. Hnmr and densitometry were used to determine the percentage of the synthesized biodiesel samples. Also, some of the physicochemical properties of the synthesized biodiesel were analyzed, which were in accordance with the ASTM and EN standards.

1. Introduction

Biodiesel is a green and renewable alternative to the petroleum diesel fuel that can be used in diesel engines. It is defined as mono-alkyl esters of long chain fatty acids in the triglyceride oils, which are synthesized via the transesterification of oils with light alcohols, mainly methanol, in the presence of commonly alkaline base catalysts. Vegetable oils are the most interesting triglyceride sources for the synthesis of biodiesel. Plant oils have high viscosities, and their direct use in diesel engines as fuel, causes some drawbacks such as deposition of the oil on different parts of the engine. Transesterification is one of the best ways to reduce the viscosity of these oils for use in diesel engines [1]. One of the main reasons for the rising world tendency toward biodiesel is their minimal adverse effects on the environment compared to the fossil fuels. Generally, biodiesel is an eco-friendly energy source that can alleviate many current and future concerns relating to the environment [2]. Physicochemical and fuel properties of the biodiesel such as viscosity, cetane number, and energy content are close to the petroleum diesel fuel; therefore, it can easily be used in diesel engines without making any basic modifications to diesel engine structure [1,2]. Many studies and researches have been sought to find cheap and suitable bio-oil sources as biodiesel feedstock. Jatropha curcas L. is a poisonous and flowering species oil plant from the Euphorbiaceaea family [3]. All of the different Jatropha parts are extremely toxic and have no edible use for humans

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DOI: 10.22104/AET.2021.4468.1247

and animals. Jatropha seeds contain approximately 25 to 45% w/w oil by weight of the kernel seed depending on the several factors: geographic location, genotype, age of the plant, and growing conditions [4,5,6]. As a non-edible oil, Jatropha oil is a promising feedstock to produce biodiesel due to its good physicochemical properties such as good oxidative stability, low viscosity and good cold properties [7] In recent years, many studies and efforts have been focused on optimizing the biodiesel production to increase its quality, lower its price, and promote its production process in environmental terms by using different catalysts and different modern methods [8-14]. Transesterification of the oils with methanol in the presence of homogeneous base catalysts, especially NaOH and KOH, gives higher efficiency than the heterogeneous ones. It is for the reason that with the use of heterogeneous catalysts, the reaction mixture is a three phase mixture including oil and methanol that are non-miscible liquids and a solid catalyst. But one of the main disadvantages of the homogeneous catalysts is that they are not reusable and enter the waste effluents through the washing and purification of the biodiesel with water. So, this makes the biodiesel production an expensive, non-sustainable and non eco-friend process. In contrast, heterogeneous catalysts can be easily separated from the mixture and reused over and over again. One way to overcome the multiphase problem in the synthesis of biodiesel, especially in the presence of a heterogeneous catalyst, is to use co-solvents. The use of a co-solvent with a proper polarity can enhance the mass transfer and miscibility between the oil and methanol, and consequently, improve the reaction's conversion rate [15,16]. Roschat et al. used CaO as the heterogeneous catalyst and THF as the co-solvent to synthesize biodiesel from the palm oil. They reported a yield of 98.5% in the synthesis of biodiesel [17]. Singh et al. reported the synthesis of biodiesel from waste oils with a yield of 97.98% % using calcium aluminum oxide as the heterogeneous catalyst and in the presence of acetone as the co-solvent [18]. Shi et al. reported a conversion yield of 93% in the synthesis of biodiesel from the Jatropha oil with an eggshell-derived catalyst in the presence of acetone as the co-solvent [19].

Another solution to increase the contact between the reactants in the synthesis of biodiesel, especially in the present of heterogeneous catalysts, is the use of the ultrasonic waves. In the ultrasound-assisted synthesis method, acoustic cavitation is the main phenomenon responsible to well mixing and homogenization of the immiscible reagents to promote the efficiency and speed up the reaction. Acoustic cavitation is the formation and subsequent violent collapse of small vacuum bubbles in a liquid medium irradiated by intense ultrasound waves [20]. Deng et al. applied an ultrasonic bath at power of 210 W in the synthesis of Jatropha biodiesel and reported a 96.4% yield [21]. To our knowledge, there are no papers published about the ultrasound-assisted synthesis of biodiesel from the Jatropha oil with a heterogeneous catalyst in the presence of a co-solvent. This work considers the effect of two different co-solvents (acetone and hexane) on improving the reaction efficiency in the ultrasound-assisted synthesis of biodiesel from the Jatropha oil in the presence of CaO as a heterogeneous catalyst. Also, some reaction parameters were optimized: methanol to oil molar ratio, volume percentage of co-solvent to methanol, weight percentage of catalyst to oil, time and temperature. Finally, some of the physicochemical properties of the synthesized biodiesel were analyzed and compared with the standards.

2. Materials and methods

2 . 1. Materials and instruments

The crude Jatropha oil was provided from a local market and heated at 110°C for 1 hour to reduce its water content to below 0.5% w/w based on the oil's weight. Hexane, methanol and acetone were obtained from Fluka. All other reagents were purchased from Merck. A glass densitometer equipped with thermometer, a classic glass viscometer and an optic refractometer (Carl Zeiss JENA) were used to determine the related physical properties of the oil and biodiesel. A probe type ultrasound instrument (UP 200H) with an output power of 200 W and frequency of 24 kHz was used for the ultrasound-assisted synthesis. Some of the physical properties of the solvent and co-solvents used in this study are given in Table 1.

2.2. Experimental procedure

2.2.1. Acid catalyzed pre-esterification step

The Jatropha oil was esterified under a reflux system at optimal operating conditions to reduce its high FFAs content with regard to the literature [22,23]. The conditions included a methanol to oil molar ratio of 9:1 and 1% w/w sulfuric acid based on the oil weight under a reflux system at 65°C for 2 h accompanied with stirring at a speed of 300 rpm. After that, the mixture was transferred to a separating funnel and allowed to settle for 4 h. Then, the watery layer was removed and the esterified oil was washed in triplicate with warm distilled water to remove impurities. Finally, to completely refine the oil from the water and methanol, it was heated on a heater equipped with a magnetic stirrer at temperature of 110°C and stirrer speed of 300 rpm for 1 h; then, it was used for the synthesis of biodiesel.

Table 1. Physical properties of methanol, hexane and acetone.

Solvent	Dipole moment (D) at 25°C	Dielectric constant (ε) at 25°C	Density at 25°C ($g.cm^{-3}$)	Boiling point (°C)
Hexane	0.08	1.88	0.659	68.7
Acetone	2.88	20.7	0.791	56
Methanol	1.70	32.6	0.791	65

2.2.2. Conventional synthesis of biodiesel

In this study, conventional synthesis of biodiesel from the esterified Jatropha oil was performed to obtain a bench line for the conversion yield (CY%) of the oil to biodiesel and also to have a standard biodiesel sample with a high content of methyl ester to use in the next steps. Conventional synthesis was done by a reflux system at optimal operating conditions with regard to the literature [22, 23] including methanol to oil molar ratio of 7:1, NaOH of 1.4% w/w per weight of the oil, temperature of 65°C and time of 1 h. The reaction mixture was stirred magnetically at 400 rpm. The separation of the synthesized biodiesel from the reaction mixture and its purification was performed the same as the method described for the esterification step in the previous section of this paper. The CY% of the synthesized biodiesel was determined by Hnmr [24] that was 92.31%.

2.2.3. Ultrasound-assisted synthesis of biodiesel

In the ultrasound-assisted synthesis of biodiesel from the esterified Jatropha oil, a probe type ultrasound instrument was used at constant amplitude and pulse mode of 70% and 0.7 s respectively. The experiments were carried out in a jacketed glass cell with a volume of 200 ml that was equipped with a condenser to prevent the evaporation of the solvent. The temperature of the cell was set via the circulating water at the desired temperature from a bath equipped with a thermostat with an accuracy of ±1°C. Firstly, a certain concentration of co-solvent in methanol was added to the oil inside the cell and mixed for 30 s. Next, a certain amount of the CaO powder as catalyst was added to the mixture and the reaction was performed by propagation of the ultrasonic waves into the mixture for a certain time. After that, the catalyst was removed from the mixture by simple filter paper. The rest of the steps to work up the synthesized biodiesel were similar to those of the esterification process described in this paper.

2.2.4. Analysis methods

The fatty acids composition of the Jatropha oil was determined by Gas chromatography (GC) in a GC 6890N gas chromatograph equipped with a flame ionization detector (FID). An HP88 capillary column with 100 m length, 0.25 µm thicknesses and 250 µm internal diameter was used. The detector's temperature was programmed at 250°C. The H_2, air and He at flow rates of 30, 300 and 25ml/min respectively were used as auxiliary gas for the FID detector. Helium was used as carrier gas at constant flow rate of 0.5 ml/min. The injector's temperature was set at 220°C and 1 µl of the sample was injected with split inlet mode at split ratio of 100:1. The following oven temperature ramp program was used: Initial temperature of 180°C, maintained for 5 min, followed by an increase of 1°C/min up to 190°C, maintained for 20 min and then 1°C/min up to 200°C, maintained for 37 min. A calibration curve of the CY% vs. density (Figure 1) was used to determine the methyl ester content in the synthesized biodiesel samples. The analysis of the biodiesel samples was performed after complete washing and purification with distilled water. In order to arrange the calibration curve, a number of the Jatropha biodiesel samples with different methyl ester content were prepared by mixing the Jatropha biodiesel synthesized by reflux in this study, with different amounts of the Jatropha oil. The CY% of these samples was determined by Hnmr spectroscopy analysis [24], and their densities were measured by a pycnometer at temperature of 25°C. The equation line for this calibration curve with R^2 of 0.9961 was obtained as follow:

$$Y = -3563.6X + 3206.7 \quad (1)$$

Some of the physicochemical properties of the Jatropha oil and its biodiesel were measured according to the standers and compared with the literature [25-27].

3. Results and discussion

3.1. Physicochemical properties of the Jatropha oil

As shown in Table 1, the Jatropha oil had a high viscosity value, so it was not suitable for direct use in a diesel engine as fuel. The upper limit of fuel viscosity for use in diesel engines specified by ASTM (D7751-07) is 6 mm^2/s [27]; Higher values of viscosity are associated with some drawbacks such as sedimentation of fuel on different parts of the engine, increased fuel consumption and decreased engine efficiency. The Iodine value is a measure of the degree of un-saturation of the triacylglycerol oils. This parameter is used to assess the susceptibility of the oils against oxidation. A higher iodine value means a higher degree of un-saturation and therefore a higher potential against oxidation. In accordance with the EN (14214) standard, biodiesel must have an iodine value below of 120 g I_2/100 g [27]. So the oils with an iodine value higher than 120 g I_2/100 g are not suitable to produce biodiesel. As shown in Table 1, the Jatropha oil used in this study had appropriate iodine value to produce of biodiesel. As given in Table 2, The FFAs content of the Jatropha oil was very

high, therefore it was reduced to below 0.5 mg NaOH/g via esterification with methanol and an acid catalyst. A high content of FFAs in the oil causes soup formation during the base-catalyzed transesterification, which decreases the reaction conversion yield [27,28].

As given in Table 3, accordance to the results of the GC analysis, the oleic and linoleic acid were the dominant fatty acids in the Jatropha oil that was in agreement with the literature [7,29].

3.2. Effect of the co-solvent on the conversion yield of biodiesel

To investigate the innate effect of the co-solvents (acetone and hexane) and their amounts on improving the conversion yield of the Jatropha oil to biodiesel, first, experiments were done under mild operation conditions including methanol to oil molar ratio of 10:1, 4% w/w CaO based on the oil weight, reaction time of 30 min and without heating from an outer source. As shown in Figure 1, both of the considered co-solvents had positive effect on improving the CY% when applied at appropriate concentrations [30,31]. The optimal amounts of the acetone and hexane for maximum effectiveness on the CY% were found to be 25 and 20% v/v based on the methanol volume, respectively. In the lower amounts, this positive effect was less due to the insufficient concentration of the co-solvents into the mixture; conversely, at the higher values, this effect was negative due to a decrease in concentration of the reactants into the reaction mixture. According to our results, acetone was more effective co-solvent than hexane. It is possibly due to its polarity that is partially between the oil and methanol, which in turn can significantly enhance the oil-methanol miscibility [22]. In regard to these results, the optimization of the other operating parameters in this study was performed at the optimal concentration of the acetone (25% v/v) as the co-solvent.

3.3. Effect of the catalyst amount on the conversion yield of biodiesel

To optimize the amount of the CaO as catalyst, the experiments were done with 25% v/v acetone, 10:1 methanol to oil molar ratio, 30 min reaction time and without heating from an external source. As shown in Figure 2, the optimal amount of the CaO based on the oil weight was 4% w/w and an excess amounts led to a reduction of the CY%. This may be due to the solidity of the catalyst, which increases the viscosity of the solution and as a result, makes it difficult to mix and transfer mass between the reactants [15,32].

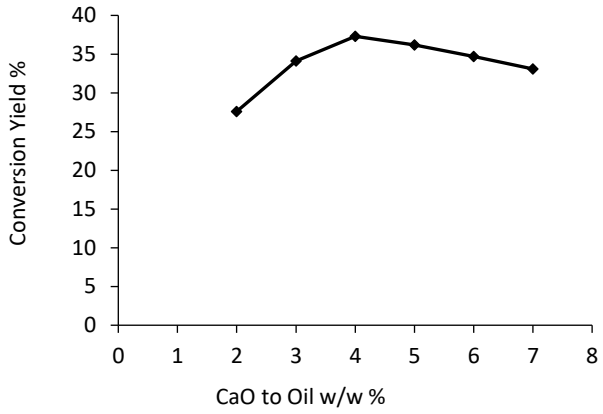

Fig. 2. Optimization of the catalyst to oil (w/w %) .

Table 2. Physicochemical properties of the Jatropha oil.

Parameter	Value
Kinematic Viscosity at 40°C (mm^2/s)	38.6
Density at 25°C ($g.cm^3$)	0.899
Water (w/w %)	2
Acidity (mg/g)	34.03
Saponification Value	197.02
Iodine Value (g I_2.$100g^{-1}$ oil)	100
Refract Index at 27˚C	1.466

Table 3. Fatty acids profile in the Jatropha oil.

Component	Composition (wt%)	Component	Composition (wt%)
Miristic acid (14:0)	0.07	Linolenic acid (18:3)	0.2
Palmitic acid (16:0)	15.06	Arachidic acid (20:0)	0.17
Palmitoleic acid (16:1)	0.86	(20:1)	0.06
Stearic acid (18:0)	6.4	Oleic acid (18:1)	41.8
Linoleic acid (18:2)	35.2	Total saturated fatty acids	21.53

3.4. Effect of the methanol to oil molar ratio on the conversion yield of biodiesel

To optimize the methanol to oil molar ratio, experiments were done with 25% v/v acetone, 4% w/w CaO, 30 min reaction time and without heating from an external source. As can be seen in Figure 3, the CY% enhanced with the increasing the methanol to oil molar ratio up to 12:1 and after that a further increase in this parameter had negative effect on the CY%. An excess amount of methanol in the reaction mixture leads to an increase in the solubility of the produced glycerol in the ester phase which promotes the reverse reaction and so reduces the CY% [15,33].

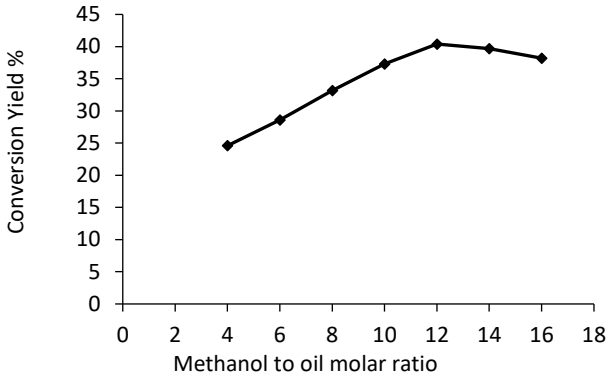

Fig. 3. Optimization of the methanol to oil molar ratio.

3.5. Effect of the reaction temperature on the conversion yield of biodiesel

The experiments to optimize the reaction temperature were done with 25% v/v acetone, 4% w/w CaO, 12:1 methanol to oil molar ratio and 30 min reaction time. As indicated in Figure 4, the optimal temperature for the reaction was 55°C and a further increase had negative effect on the CY%. This may be due to the evaporation of the acetone and the change in its concentration from the optimal amount in the reaction mixture [18].

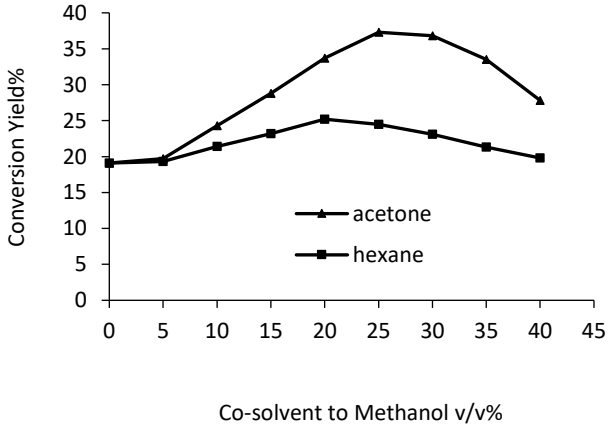

Fig. 1. Optimization of the co-solvents amount to methanol (v/v %).

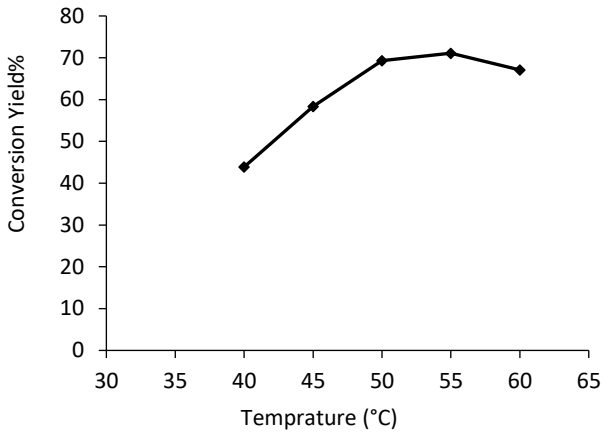

Fig. 4. Optimization of the reaction temperature.

3.6. Effect of the reaction time on the conversion yield of biodiesel

To optimize the reaction time, the experiments were carried out with 25% v/v acetone, 12:1 methanol to oil molar ratio, 4% w/w CaO and temperature of 55°C. As shown in Figure 5, the maximum CY% was obtained within 60 min and after that a further increase had negative effect on the CY%. Transesterification is a reversible reaction and it seems that a long reaction time causes the reverse reaction to prevail and thus the CY% decreases [15].

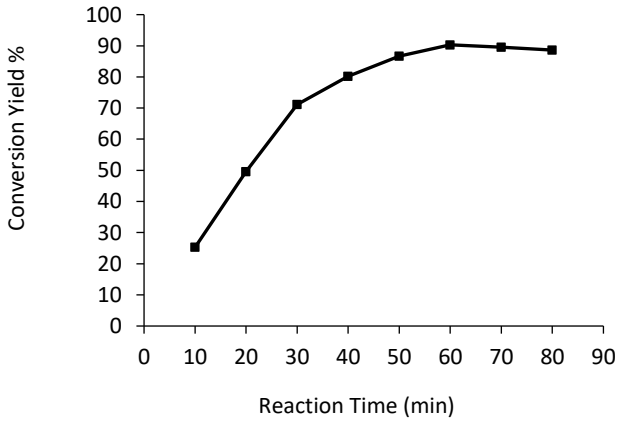

Fig. 5. Optimization of the reaction time.

According to the results, the maximum yield for conversion of the Jatropha oil to biodiesel was 90.33% which was obtained in the optimal operating conditions including methanol to oil molar ratio of 12:1, CaO of 4% w/w, acetone of 25% v/v, temperature of 55°C and time of 60 min. The physicochemical properties of this sample were in accordance with the ASTM and EN standards (table 4). The results of this paper regarding the positive effect of co-solvent to improve the conversion yield in the synthesis of biodiesel with heterogeneous catalyst are in agreement with other similar literature [17,34].

Table 4. Physicochemical properties of the Jatropha biodiesel.

Parameter	Value	US (ASTM D6751-07)	EU (EN14214)
Kinematic Viscosity at 40°C (mm^2/s)	4.6	3.5-5.0	1.9-6.0
Density at 25°C ($g.cm^3$)	0.8745	-	-
Water content (w/w %)	0.04	<0.05	<0.05
Iodine Value (g I_2.100g^{-1} oil)	100	-	<120
Refract Index at 27°C	1.450	-	-
Acidity (mg/g)	0.02	<0. 5	<0. 5

4. Conclusions

This study used CaO as the heterogeneous alkaline catalyst instead of homogeneous ones in the ultrasound-assisted synthesis of biodiesel from the Jatropha oil to improve the environmental terms in the biodiesel production. To overcome the multiphase problem in the reaction mixture, the effect of two different co-solvents, namely acetone and hexane were considered to increase the conversion yield. According to the results, both co-solvents had positive effect on increasing the CY% when applied in appropriate concentrations. Acetone was the more efficient co-solvent because of its moderate polarity, which increased the miscibility of the oil and methanol. Therefore, acetone was chosen as the co-solvent for the optimization process. Finally, a biodiesel sample with a 90.33% content of methyl ester was synthesized under mild operating conditions which had satisfactory physicochemical properties according to the standards.

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