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Effect of microwave-assisted system on transesterification of castor oil with ethanol

Gina M. Hincapié¹, Sabine Valange², Joël Barrault², Jorge A. Moreno¹, Diana P. López¹✉

Abstract

A systematic study of microwave-assisted transesterification of castor oil was conducted in the presence of ethanol and of potassium hydroxide as catalyst. Effects of various reaction parameters such as reaction time, catalyst concentration, reaction temperature and ethanol-oil molar ratio were analyzed. Ethyl esters were successfully produced by microwave-assisted transesterification. The maximum yield was 80.1% at 60°C, 10:1 alcohol: oil molar ratio, 1.5% potassium hydroxide and 10 min. The results show that there was a reduction in reaction time for microwave-assisted transesterification as compared to conventional heating, yields were slightly affected by temperature from 40-70°C; this indicates a significant effect of microwaves even at low temperatures.

Keywords: Microwave radiation; castor oil; biodiesel; transesterification.

Introduction

Due to the increasing energy demand and the environmental problems related with the use of fossil fuels, it has become necessary to develop alternative fuels. Among these, biodiesel is an interesting option because it is biodegradable, non-toxic and it is produced from renewable sources (Ma & Hanna 1999, Karmakar et al. 2010). Biodiesel is mainly produced by transesterification of vegetable oils or animal fats with short chain alcohols such as methanol or ethanol (Figure 1), with glycerol as a co-product (Fukuda et al. 2001, Pinto et al. 2005). The use of methanol is more common than ethanol because of its low cost and high reactivity but there is an increasing interest in the use of ethanol because it is renewable, non-toxic and it is safer to handle (Pisarello et al. 2010, Brunschwig et al. 2012).

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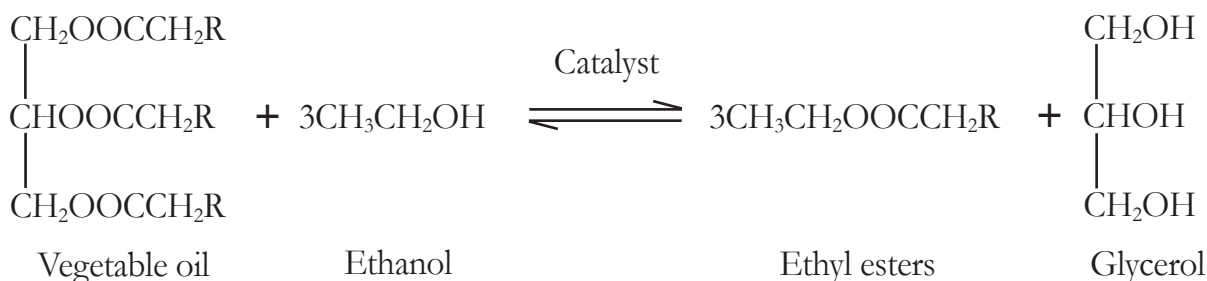


Fig. 1. Scheme of transesterification reaction

Edible oils like soybean, sunflower and palm oil have been used for biodiesel production. However, aiming to reduce biodiesel production costs, the use of less expensive feedstock such as non-edible oils, animal fats and waste cooking oils has gained interest (Berchmans & Hirata 2008). Among these feedstocks, castor oil, obtained from *Ricinus communis* L. seeds has been identified as an important non-edible raw material (Meneghetti et al. 2006, Lavanya et al. 2012, Madankar et al. 2013). Opposed to other vegetable oils, it is characterized by its indigestibility, solubility in alcohols, high hygroscopicity and high viscosity (Scholz & Nogueira da Silva 2008). It is mainly constituted of triglycerides of ricinoleic acid (12-hydroxy-*cis*-octadec-9-enoic acid) in which the presence of a hydroxyl group at C-12 imparts several unique chemical and physical properties that are exploited in the pharmaceutical and chemical industry (Meneghetti et al. 2006).

Generally, transesterification reaction is performed under conventional heating, nevertheless, over the past few years, microwave irradiation has gained interest as an alternative heating system for this type of reactions (Hernando et al. 2007, Azcan & Yilmaz 2013, Yadav & Kadam 2013, Yuan & Shu 2013). The changing electrical field component of microwave radiation interacts with the dipoles of the molecules increasing their rotation and as a consequence heat is generated due to molecular friction. Hence, microwave irradiation increases the reaction rate and higher yields are generally obtained in short times, as compared to conventional heating (Azcan & Danisman 2007, Manco et al. 2012).

Some recent studies on the transesterification of castor oil have been carried out under microwave irradiation. Koberg and Gendanken (2012) reported the transesterification of castor oil with methanol and SrO as a catalyst under a two-stage (extraction and transesterification) and a one-stage method (*in situ* transesterification) using three techniques: conventional heating, sonochemistry and microwave irradiation. For the two-stage method, the authors found the highest yield of FAMEs as 52.7% and a conversion of 99.5% using microwave irradiation as a heating source. On the other hand, 57.2% yield of FAMEs and a conversion of 99.9% were achieved in a one-stage method lasting 5 min using microwave irradiation. Yuan et al. (2011) used acid catalysts ($\text{NaHSO}_4 \cdot \text{H}_2\text{O}$ and AlCl_3) and a base catalyst (Na_2CO_3) to evaluate transesterification of castor oil under microwaves. Best results were obtained at 65°C, 18:1 molar ratio of methanol to castor oil, 7.5 wt% of catalyst with respect to castor oil, 200W microwave radiation power and 120 min of reaction time, at these reaction conditions, they obtained yields of 74, 73, 90% using $\text{NaHSO}_4 \cdot \text{H}_2\text{O}$, AlCl_3 and Na_2CO_3 respectively. They also found that microwave heating consumes less energy than conventional heating to achieve the same amount of FAME.

There is a diversity of scientific literature on transesterification of castor oil with ethanol (de Oliveira et al. 2005, Meneghetti et al. 2006, Plentz Meneghetti et al. 2006, Varma & Madras 2007, Albuquerque et al. 2009; de Lima da Silva et al. 2009, Cavalcante et al. 2010, Efeovbokhan et al. 2012, Martínez Arias et al. 2012, Thomas et al. 2013). In a study developed by our group (Hincapié et al. 2011), conventional biodiesel

production from crude castor oil using ethanol was conducted. The content of ethyl esters was determined by ^1H NMR. The higher conversions (around 95%) were found after esterification (reaction time 1 h) and transesterification (reaction time 1h) using an alcohol-oil molar ratio of 60 and a temperature of 60°C.

However, little information is available on transesterification of castor oil with ethanol assisted by microwaves (Perin et al. 2008) and, as far as the authors are aware, no systematic studies have been carried out modifying reaction parameters to optimize this transesterification with homogeneous catalysts. Thus, we have conducted a study analyzing the effect of microwave irradiation on biodiesel production from castor oil using ethanol. Variables such as reaction time, catalyst concentration, reaction temperature and ethanol-oil molar ratio were considered.

Methodology

Materials: Analytical grade isopropanol (>99.5%), potassium hydroxide (85%), sulfuric acid (96%), anhydrous sodium sulfate (99%) and tetradecane (99.5%), were used as purchased without any further purification. Absolute ethanol (99.7%) was used for transesterification reactions. Ethyl ricinoleate (87%) was purchased from TCI Europe. Refined castor oil (CODEX) and ethyl esters mixture for gas chromatography (GC) quantifications, were provided by VALAGRO Carbone Renouvelable Poitou-Charentes.

Procedures:

Microwave assisted transesterification: A MicroSYNTH (Microwave synthesis labstation, Milestone Inc.) microwave unit with a power output range from 0 to 1000 W was used to perform transesterification reactions. The system was equipped with a reflux condenser, a magnetic stirrer bar and a fiber optic temperature sensor placed in a thermowell (PTFE).

First, the effect of reaction time and catalyst concentration was analyzed by placing a castor oil sample (10 g) into a three-neck round-bottom reaction flask. Initially, the catalyst (KOH) was dissolved in the desired amount of ethanol and the resulting solution was then added to the oil. The catalyst was used at three different concentrations 0.5%, 1.0% and 1.5%

and we evaluated four different reaction times (3, 5, 7 or 10 min); temperature was maintained at 60°C and the ethanol:oil molar ratio was 10:1. The mixture was irradiated at a maximum of 250 W. At the end of the reaction, the mixture was cooled at room temperature and H_2SO_4 was added to neutralize the KOH catalyst. Centrifugation was used to separate the glycerol phase while ethanol was removed from biodiesel phase by vacuum distillation and further purified with hot water washes and dried over anhydrous Na_2SO_4 . The effect of temperature (40, 50, 60, 70°C) and the effect of ethanol:oil (4:1, 6:1 or 10:1) molar ratio were evaluated, with a reaction time of 10 min and a catalyst concentration of 1.5%. Each experiment was performed in duplicate.

Analysis: The acid value of the oil was determined by an acid-base titration technique (modified ASTM D974, 2006) using a standard solution of KOH 0.5 N. Saponification and hydroxyl indexes were determined under the NF ISO 3657 and NF T60-213 regulations respectively.

A GC Varian CP-3380 instrument equipped with a FID detector and a capillary column BPX-70 (25 m x 0.32 mm ID x 0.25 μm) was used to analyze the products using tetradecane as internal standard.

Results and discussion

The acid value of the oil was 2.05 mg KOH/g sample. Saponification and hydroxyl values were 180.2 mg KOH/g sample and 179.8 mg KOH/g sample, respectively; these results are in agreement with previous reports (Benavides et al. 2007). Fatty acid composition for castor oil CODEX is reported in **Table 1** (data provided by supplier). Molecular weight of the oil was estimated at 926 g/mol using this composition.

Table 1. Fatty acid composition of castor oil CODEX (provided by supplier)

Fatty acid	Composition / %wt.
Ricinoleic acid	85-92
Linoleic acid	2.5-7.0
Oleic acid	2.5-6.0
Palmitic acid	≤ 2.0
Estearic acid	≤ 2.5
Linolenic acid	≤ 1.0

Results of transesterification for different reaction times and catalyst concentrations (**Table 2**) indicated that for a blank reaction performed under the same experimental conditions (molar ratio ethanol:oil 10:1, $T=60^{\circ}\text{C}$, 2h) a yield of 0.74% was obtained. Also, biodiesel yield increases with catalyst concentration, obtaining the highest yield with 1.5% of KOH. Table 2 also shows that, for the same catalyst concentration, biodiesel yield does not change significantly with reaction time, except for 10 min, where the yields increased. Taking into account that the yields are not significantly increased for transesterification at 30 min, we decided to continue the work with 10 min as reaction time; therefore, further experiments were performed using 1.5% KOH and 10 min. To compare, a 79% yield was obtained using a conventional heating system for 1h, which shows that microwave heating strongly increases transesterification rate.

In accordance with the Arrhenius equation Eq(1), the rate of a chemical reaction depends on the pre-exponential factor (A) and the activation energy (E_a) (Lidström et al. 2001). From experiments involving microwaves, some authors have found evidence of the modification of the pre-exponential factor A (Binner et al. 1995, Polshettiwar & Varma 2010). This factor describes the molecular mobility and depends on the frequency of collisions at the reaction interface (Lidström et al. 2001).

$$k = Ae^{-E_a/RT} \quad (1)$$

It is also necessary to consider that polar solvents are required for microwave heating, and the ability of a solvent to absorb microwave energy and to convert this energy into heat is related with the loss angle, δ , which is usually expressed in the form of a tangent (Lidström et al. 2001) Eq. (2):

Table 2. Effect of reaction time and catalyst concentration on biodiesel yield. Molar ratio ethanol-oil 10:1, $T=60^{\circ}\text{C}$

Reaction time / min	Catalyst concentration / %w / w	Biodiesel yield / %
3	0.5	26.5
	1.0	57.2
	1.5	64.6
5	0.5	29.5
	1.0	56.0
	1.5	61.6
7	0.5	29.0
	1.0	54.2
	1.5	66.3
10	0.5	34.3
	1.0	70.3
	1.5	80.1
30	0.5	13.9
	1.0	68.0
	1.5	83.0
120 (Blank under microwaves)	0.0	0.74
60 (conventional heating experiment)	1.5	79

$$\tan \delta = \frac{\epsilon''}{\epsilon'} \quad (2)$$

Where ϵ' is the dielectric constant and represents the ability of a dielectric material to store electrical potential energy under the influence of an electric field. The loss factor, ϵ'' , quantifies the efficiency with which the absorbed energy is converted into heat (Lidström et al. 2001).

Ethanol has a higher loss tangent (0.941) compared with other solvents as methanol (0.659) (Dressen 2009). Due to its loss tangent, ethanol couples better than methanol with microwave irradiation which induces dipolar polarization as a consequence of dipole-dipole interactions of polar molecules with the electromagnetic field (Perreuz & Loupy 2006). This phenomenon results in a rapid heating leading to an efficient intermolecular mixing and agitation (i.e. increase in effective collisions), therefore, increasing the reaction rate compared to conventional heating. Nevertheless, non-thermal effects related with the change on pre-exponential factor A should not be discarded (Polshettiwar & Varma 2010) and could be the subject of further investigations.

The effects of temperature and ethanol:oil molar ratio were also evaluated and the results are shown in **Figure 2a and 2b**.

As seen in Figure 2a, there are two temperature ranges in which there is not a marked increase on biodiesel yield as temperature increases. In the first one (40 – 50°C), biodiesel yield is around 72%, which is an encouraging result considering that transesterification is generally conducted nearly to the boiling point of the solvent (78°C for ethanol). In the second temperature range (60 – 70°C), biodiesel yield is around 80%. These results suggest that the phenomenon of dipolar polarization generated by microwave irradiation is effective even at very lower temperatures compared to the boiling point of the solvent, condition that is strictly necessary in conventional heating.

When the ethanol:oil molar ratio is reduced from 10 to 4 (Figure 2b), biodiesel yield decreases from 80.1 to 48.3%. This observation is in agreement with the above-mentioned positive effect of microwave irradiation, the higher the amount of ethanol, which is the polar reagent sensitive to microwaves, the higher the number of dipole-dipole interactions.

Conclusions

Microwave heating was a useful technique to obtain high biodiesel yields from castor oil in short reaction times, achieving better performances at lower times

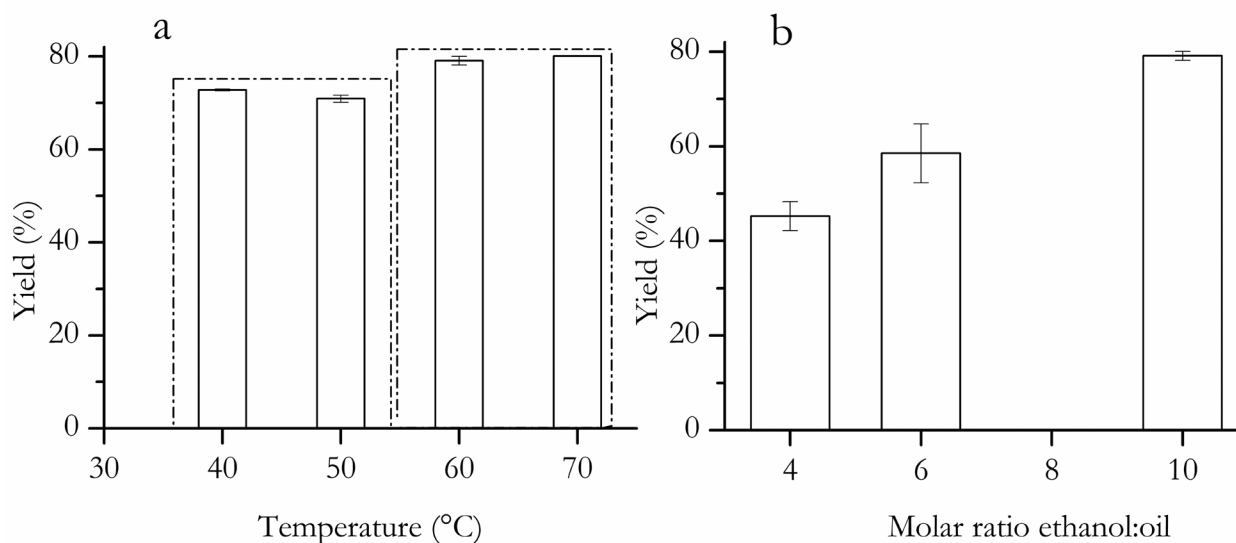


Fig. 2. a. Effect of temperature on biodiesel yield (KOH 1.5%, 10 min, molar ratio ethanol-oil 10:1). **b.** Effect of molar ratio ethanol-oil on biodiesel yield (KOH 1.5%, 10 min, 60°C)

compared with conventional heating. Moreover, temperature has low effect on yield, which confirms that the influence of microwaves is effective even at low temperatures, contrary to that observed with conventional method. These results are encouraging for the future analysis of transesterification assisted by microwaves using ethanol derived from biomass or using low-quality oils.

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Conflict of interest

There are no conflicts of interest with funding sources or institutions.

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Efecto de microondas en la transesterificación de aceite de higuera

Resumen. Se realizó un estudio sistemático del efecto de la radiación microondas en la transesterificación de aceite de higuera con etanol en presencia de hidróxido de potasio como catalizador. Diversos parámetros fueron analizados, entre ellos el tiempo de reacción, concentración de catalizador, temperatura y relación molar etanol-aceite. De acuerdo con los resultados, la transesterificación asistida por microondas resultó útil para la producción de ésteres obteniéndose un rendimiento máximo de 80.1% a 60°C, una relación molar alcohol: aceite de 10:1, 1.5% de hidróxido de potasio y 10 min de reacción. Se observó una reducción en el tiempo de reacción para la transesterificación asistida por microondas comparada con el calentamiento convencional. Los resultados muestran que los rendimientos son ligeramente afectados por la temperatura en el rango 40-70°C lo que indica un efecto importante de las microondas incluso a bajas temperaturas.

Palabras clave: Radiación microondas; aceite de higuera; biodiesel; transesterificación.

Efeito microondas em transesterificação do óleo de rícino

Resumo. O presente artigo, foi realizado um estudo sistemático da transesterificação assistida por irradiação de microondas com óleo de rícino na presença de etanol e de hidróxido de potássio como catalizador. Analisou-se os efeitos da variação alguns parâmetros da reação, tais como, tempo de reação, concentração de catalisador, temperatura de reação e proporção molar de óleo de etanol. De acordo com os resultados, os ésteres etílicos foram produzidos com êxito por transesterificação assistida por microondas, obtendo um rendimento máximo de 80.1% a 60 °C, razão molar de 10:1 álcool: óleo, 1.5% de hidróxido de potássio em 10 min. Houve uma redução no tempo de reação de transesterificação assistida por microondas, em relação ao aquecimento convencional. Os resultados mostram que os rendimentos são ligeiramente afetados pela temperatura, no intervalo de 40-70 °C, indicando um efeito significativo de microondas mesmo a baixas temperaturas.

Palavras-chave: Radiação de microondas; óleo de rícino; biodiesel; transesterificação.