



## Use of experimental design to investigate biodiesel production by multiple-stage Ultra-Shear reactor

Nívea De Lima Da Silva\*, Julian A.G. Garnica, César B. Batistella, Maria Regina Wolf Maciel, Rubens Maciel Filho

School of Chemical Engineer, State University of Campinas (UNICAMP), P.O. Box 6066, 13081-970 Campinas, SP, Brazil

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### ABSTRACT

This work presents biodiesel production from soybean oil and bioethanol by multiple-stage Ultra-Shear reactor (USR). The experiments were carried out in the following conditions: reaction time from 6 to 12 min; catalyst concentration from 0.5% to 1.5% by weight of soybean oil; ethanol: soybean oil molar ratio from 6:1 to 10:1. The experimental design was used to investigate the influence of process variables on the conversion in biodiesel. The best ethyl ester conversion obtained was 99.26 wt.%, with ethanol:soybean oil molar ratio of 6:1, catalyst concentration of 1.35% and with 12 min of reaction time.

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### 1. Introduction

Biofuels have an important role to play in future energy production. Researches in this area are showing that they are competitive due to the global demand for energy and to increasing environmental concerns. It is an attractive fuel alternative to the depleting fossil fuel resources. Most of the world energy demands are supplied by petrochemical sources, coal, and natural gases which are not sustainable and at current usage rates will be consumed shortly. The growth in biofuel production has largely been driven by policy targets and by the way in which they are produced and managed, which can determine if they can be beneficial to society, to economy and to the environment. In the coming years, international trade in biofuels is expected to grow as biofuel consumption increases in the USA, EU, Brazil and China (Science for Environment Policy, 2010; Meher et al., 2006a and Kralj, 2008).

Biodiesel is a fuel made from vegetable oils, animal fats or microbial oils (algae, bacteria and fungi). The raw materials are converted to biodiesel through a transesterification reaction involving alcohol and catalyst (Krawczyk, 1996; Ma and Hanna, 1999; Li et al., 2008).

Biodiesel has various advantages such as: it is a complement or substitute to petroleum based fuel; it is a renewable fuel; it has a favorable energy balance; it presents lesser harmful emissions

and it is a non-toxic fuel, which make it very attractive (Meher et al., 2006a; Ito et al., 2005; Vyas et al., 2010).

Transesterification reaction can be catalyzed by homogeneous (alkaline and acid) and heterogeneous catalysts or without catalyst using supercritical conditions. A continuous stirred tank reactor (CSTR) is commonly used in industrial biodiesel production. This equipment is generally modeled as there is no spiral variation in concentration, temperature, or reaction rate throughout the vessel (Fogler, 1999; Chongkhong et al., 2007). The use of batch stirred tank reactor (BSTR) is also common in this process (De Paola et al., 2009; Vicente et al., 2007). Others less conventional equipments have been studied in order to increase the biodiesel conversion and the reaction speed, while decreasing the process cost (Behzadi and Farid, 2009). These are micro-reactors (Wen et al., 2009), static mixers (Thompson and He, 2007), microwave reactor (Perin et al., 2008; Carucci et al., 2009), rotational packed bed (Chen et al., 2010), reactive distillation or heat integration system (Da Silva et al., 2010; He et al., 2006; Kralj, 2008), ultrasound reactor (Georgogianni et al., 2008), and gas–liquid reactor (Behzadi and Farid, 2009).

Mixing is an important variable in the transesterification reaction because the vegetable oils or fats are immiscible with catalyst–alcohol–solution. Once two phase are mixed and the reaction is started, stirring is no longer needed (Meher et al., 2006a). The insolubility of methanol in the oil phase hinders the progress of the transesterification reaction, so vigorous mixing is essential to create sufficient contact between the two immiscible phases. As methyl esters are formed, they act as a mutual solvent for the reactants and a single-phase system is formed. The mixing effect is

\* Corresponding author. Tel.: +55 1935213971.

E-mail address: [niveadelimadasilva@gmail.com](mailto:niveadelimadasilva@gmail.com) (N. De Lima Da Silva).

most significant during the slow rate region of the reaction (Chen et al., 2010; Ma and Hanna, 1999; Srivastava and Prasad, 2000). Within this concept, in this work, a multiple stage high-speed mix was used for biodiesel production. This equipment has up to three sets of rotors and stators that convert mechanical energy to high tip speed, high shear stress and high shear-frequencies. The reaction takes place in the high-energy shear zone, with a small droplet size and a large surface area, helping the catalyst reaction to occur faster. This drastically reduces production time and increases production volume (Ika guide, 2010).

The experimental design procedure was used to optimize the following process variables: reaction time, catalyst content and soybean oil: ethanol molar ratio. The effects of these variables on biodiesel conversion were investigated through two experimental designs. The first experimental set was carried out using a central composite design (CCD). The CCD was used to reduce the number of observations while giving the desired information, enabling the selection of the significant variables. The second experimental set was a (CCD) plus axial points, generating a second order model and the results were optimized using response surface methodology (RSM).

## 2. Methods

### 2.1. Materials

The experiments were carried out with refined soybean oil obtained from a supermarket (Brazil). Sodium hydroxide (Synth-Brazil) was used as catalyst. Anhydrous ethanol was purchased from Synth (Brazil). All the standards were supplied by Sigma-Aldrich (St. Louis, MO), polytetrafluorethylene filter (PTFE filter) was supplied by Millipore (US), and HPLC-grade THF (Tetrahydrofuran) was from B&J/ACS (US).

### 2.2. Equipment description

The transesterification reaction was carried out in an Ultra-Shear reactor (USR). This equipment is a rotor–stator mixing with

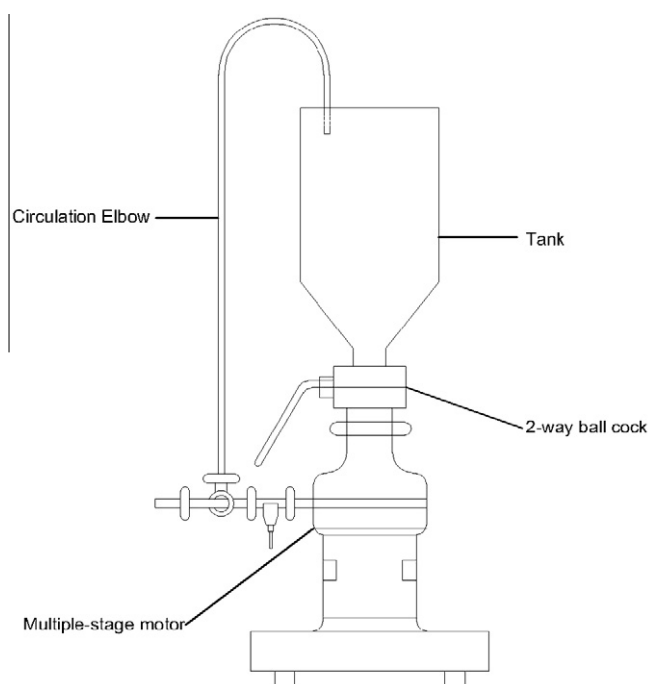


Fig. 1. Ultra-Shear reactor.

high speed and intense shear frequency (PROCESS PILOT 2000/4 of IKA WORKS Inc., USA), Fig. 1. The agitation was kept constant at 7900 rpm.

The Ultra-Shear reactor has a modular design. This machine can be operated as basic device or can be turned into different operating devices using modules. The basic device is a single-stage high-performance instrument for continuous dispersion of liquids. In this work, three modules (multiple-stage) were used. The dispersing action is based on the rotor–stator principle, which means that a high-speed rotor with very narrow slots rotates in a stator. This produces high shearing energies between rotor and stator. The system consisting of rotor and stator is also called generator. In this system, a standard three-phase motor drives at 3000 rpm, and a transmission ratio of the belt drive increases the speed of the rotor shaft to 7900 rpm. A circulating elbow was used to increase the reaction mixture.

### 2.3. Biodiesel production

The USR was loaded with 400 g of soybean oil, preheated to the desired temperature (78 °C) and then agitation starts. Previous experiments showed that high agitation speed of USR leads ethanol to reach boiling point temperature, so that a water batch was used to maintain the temperature of the reactor at 78 °C. The catalyst was dissolved in ethanol and the alcoholic solution was added to the vegetable oil.

During the reaction, samples were collected through the dilution of 0.1 g of the reaction products in 10 ml of tetrahydrofuran (THF); this large dilution quickly stops the reaction course. Then, the samples were filtered using a PTFE filter and analyzed using HPSEC (high-performance size-exclusion chromatography). After the reaction, the excess of ethanol was evaporated under vacuum using a roto-evaporator. Then, the ester and the glycerol layers were separated in a funnel, and the ethyl ester layer was purified. In order to remove the residue from raw materials and the catalyst, the ester layer was washed with water plus phosphoric acid, until neutralization. After that, the mixture was dried with sodium sulfate.

### 2.4. Methods of analyses

The raw material composition was determined by gas chromatography (GC) equipped with a flame ionization detector and with a DB 23 column. The soybean oil composition was obtained using the methodology of Hartman and Lago (1973). The soybean oil molar mass ( $Mw_{tri}$ ) was calculated according to Eq. (1) and the ethanol quantities were determined according to the soybean oil molar mass ( $Mw_{acid}$  is the fatty acid molar mass,  $Mw_{glycerol}$  is the glycerol molar mass and  $Mw_{water}$  is the water molar mass).

$$Mw_{tri} = 3 \times (Mw_{acid}) + Mw_{glycerol} - 3 \times Mw_{water} \quad (1)$$

The following soybean oil properties were determined in order to characterize this raw material: the free fatty acid content was determined according to the AOCS official method Ca 5a-40 as oleic acid; the iodine index (IV) was determined according to the AOCS Cd 1c-85; the moisture was determined by Karl Fischer (Metler Toledo); the density and viscosity were determined by Stabinger Viscometer SVM 3000 (Anton Paar) whose properties were measured at 40 °C according to ASTM D-445.

The transesterification reaction composition was determined by high-performance size-exclusion chromatography (HPSEC) supplied by Waters (US). The Schoenfelder (2003) methodology is specific for analyses of triacylglycerols (TG), diacylglycerols (DG), monoacylglycerols (MG), and glycerol (GL), nevertheless, this methodology was adapted for the analyses of esters (EE) because the ester peak appeared between the MG and the GL peaks. The

mobile phase was THF. The chromatographic system consisted of an isocratic HPLC pump (model 515; Waters, Milford, MA), a differential refractometer detector (model 2410; Waters), and of an oven in order to maintain the columns at 40 °C by a temperature control module (Waters). Two HPSEC columns, Phenogel 50 Å and 100 Å (Phenomenex), with dimensions of 7.8 × 300 mm and particle size of 5 µm, were connected in series. The relative percentage of each component ( $X_i$ ) was given through HPSEC and it was determined through Eq. (2), where  $X_{EE}$  was calculated dividing the peak area of the ester ( $A_{EE}$ ) by the sum of the peak areas of all components. The  $X_{EE}$  is the ester concentration and these results were used in the experimental design. The ester conversion ( $Y$ ) was determined according to Eq. (3).  $C_0$  is the raw material (TG, MG, and DG) concentration (wt.%) at  $t = 0$  min, and  $C_i$  is the raw material concentration at the end of the reaction. The identification of the (TG), (DG), (MG), (EE), and (GL) is based on reference standards (Sigma–Aldrich).

$$X_{EE} = \left( \frac{A_{EE}}{A_{TG} + A_{DG} + A_{MG} + A_{EE} + A_{GL}} \right) \quad (2)$$

$$Y = \left( \frac{C_0 - C_i}{C_0} \right) \quad (3)$$

### 2.5. Experimental design

The effects of process variables (catalyst concentration, ethanol:soybean oil molar ratio and reaction time) on biodiesel conversion ( $Y$ ) were verified using experimental design. The experiments were carried out and optimized following CCD and response surface methodology (RMS). The software Statistica (Statsoft, v.7) was used to analyze the results.

The RSM is a set of mathematical and statistical techniques which are useful for modeling and analyzing problems in which a response of interest is influenced by several variables and the objective is to optimize this response (Montgomery, 2001; Hameed et al., 2009). The first step of RMS was to derive the polynomial equation that describes the response (ester conversion) as a function of the independent variables (catalyst content, molar ratio and reaction time). The RSM equation takes into account only the significant coefficients, Eq. (4). Coefficient  $b_0$  is the outcome (response) at the central point, and the other coefficients measure the main effects and the interactions of the coded variables  $X_i$  on the response  $Y$ .

$$Y = b_0 + \sum b_i X_i + \sum b_{ii} X_i^2 + \sum b_{ij} X_i X_j \quad (\text{where } i \neq j) \quad (4)$$

The second step was to determine the quality of the fitted model. It was evaluated by the analysis of variance (ANOVA), based on  $F$ -test (Box and Hunter, 1978).

## 3. Results and discussion

### 3.1. Raw material characterization

The iodine value of soybean oil was 131.32, the free fatty acid content was 0.3%, the moisture was 0.054wt.%, the density was 0.8752 g/cm<sup>3</sup> and the viscosity was 40.26 mm<sup>2</sup>/s. The low free fatty acid and moisture content permitted the use of basic catalysis because both excess as well as insufficient amount of catalyst may cause soap formation (Meher et al., 2006a). The iodine value is a description of vegetable oil unsaturations. Unsaturation can lead to deposit formation and storage stability problems with fuels (Graboski and McCormick, 1998). Table 1 shows the composition of soybean oil fatty acid. The soybean oil molar mass was determined according to Eq. (1).

**Table 1**  
Gas chromatographic analysis of soybean ethyl esters.

Fatty acid	Palmitic (16:0)	Stearic (18:0)	Oleic (18:1)	Linoleic (18:2)	Linolenic (18:3)
(wt.%)	11.20	3.57	23.79	55.84	5.60
Mw (g/mol)	256.4	284.5	282.5	280.5	278.5

$MW_{tri} = 872.92$  g/mol.

**Table 2**  
First experimental Design.

Run	Time (min)	Molar ratio			Catalyst (wt.%)		Ester (wt.%)
1	–1	6	–1	6	–1	0.5	82.58
2	1	12	–1	6	–1	0.5	95.48
3	–1	6	1	10	–1	0.5	90.07
4	1	12	1	10	–1	0.5	91.10
5	–1	6	–1	6	1	1.5	92.64
6	1	12	–1	6	1	1.5	97.28
7	–1	6	1	10	1	1.5	96.66
8	1	12	1	10	1	1.5	97.88
9	0	9	0	8	0	1	96.12
10	0	9	0	8	0	1	96.33
11	0	9	0	8	0	1	97.95

### 3.2. Experimental design results

Two experimental designs were carried out in order to optimize the process variables. The first part of the experiments was a 2<sup>3</sup> plus three central points (CCD). The second one was a 2<sup>2</sup> CCD plus four axial points. The axial points are also called star points (Box and Hunter, 1978). The limits of the first experimental design were: ethanol: soybean oil molar ratio from 6:1 to 10:1; catalyst concentration level from 0.5% to 1.5% and reaction time from 6 to 12 min. Table 2 shows the results of the first experimental design. Biodiesel conversions higher than 97 wt.% were obtained in the experimental design. Fig. 2 presents the Pareto graph showing that the molar ratio effect, MR, the interaction between the catalyst and time ( $t^*C$ ), and the interaction between the molar ratio and catalyst ( $MR^*C$ ) were not significant, because these effects were on the left side of the  $p$ -value. This value was used as a tool to check the significance of each effect. Good conversion was obtained using the ethanol: soybean oil molar ratio of 6:1. Other research has shown that the use of molar ratio of alcohol: vegetable oil of 6:1 leads to higher ethanolysis conversion. Fillieres et al. (1995) analyzed the effect of the ethanol quantity on biodiesel production from rapeseed oil and the best result was obtained with a 6:1 ratio. Therefore, the molar ratio of ethanol:soybean oil was fixed at a minimum level and the second experimental design considers only the catalyst content and the reaction time as process variables.

In order to optimize the significant variables, a second experimental design was done with reaction time from 6 to 12 min, catalyst concentration level from 0.5 to 1.5%, and ethanol: soybean oil molar ratio of 6:1. Table 3 shows the results of the second experimental design. Fig. 3 presents the Pareto graph. The effects of the reaction time ( $t$ ) and the catalyst content ( $C$ ) were significant with confidence level of 95%. The catalyst concentration ( $C$ ) is the most important variable, as shown in Fig. 3. Increasing catalyst concentration from 0.5% to 1.5%, runs 7 and 8 (Table 3), increases the ester conversion from 76.89 to 98.92 wt.%.

A coded quadratic model was obtained using factorial design, and this model describes the influence of reaction time ( $t$ ), and of catalyst concentration ( $C$ ) on the ethyl ester conversion ( $E$ ), Eq. (5). The ANOVA demonstrates that the model is significant, as evidenced from the  $F_{test}$ . According to  $F_{test}$ , the model has statistical

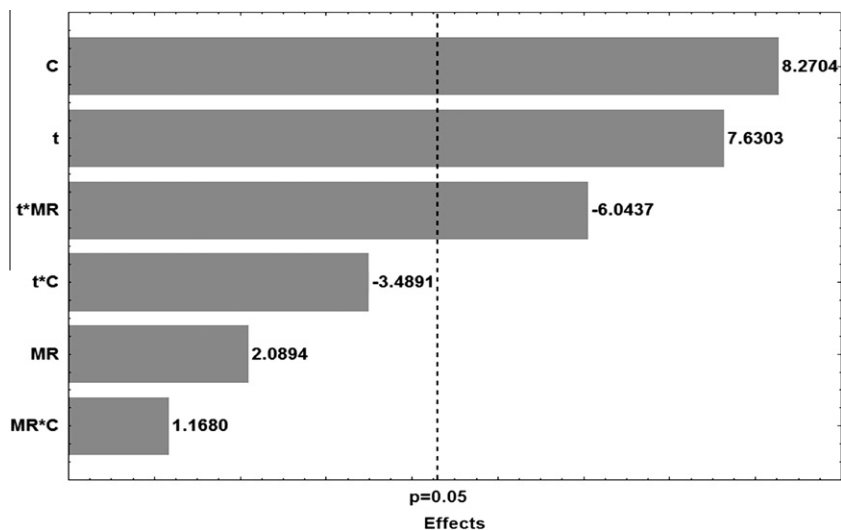


Fig. 2. Effects of the process variables on the soybean ethyl ester conversion: First experimental design.

Table 3  
Second experimental design.

Run	Time (min)	C (wt.%)		Ester (wt.%)	
1	-1	7	-1	0.64	94.34
2	1	11	-1	0.64	94.60
3	-1	7	1	1.35	99.03
4	1	11	1	1.35	99.26
5	-1.414	6	0	1	98.01
6	+1.414	12	0	1	99.18
7	0	9	-1.414	0.5	76.89
8	0	9	+1.414	1.5	98.92
9	0	9	0	1	98.07
10	0	9	0	1	98.09
11	0	9	0	1	98.02

significance when the  $F_{\text{calculated}}$  is higher than the  $F_{\text{listed}}$  value. Hence, a satisfactory adjustment of the experimental results was achieved. Fig. 4 shows the response surface of ester conversion as a function of the reaction time and the catalyst concentration obtained from Eq. (5). Both process variables are important. Increasing reaction time and catalyst content, the ethyl ester con-

version increases. According to Fig. 4, ethyl ester conversion of 99.26 wt.% can be obtained with catalyst concentration from 1% to 1.4%, and a reaction time up to 12 min. The use of USR reduces the reaction time because the high speed of mixture promotes a large ester conversion in a shorter time.

$$E = 98.0593 + 0.2681t + 1.1580t^2 + 5.0633C - 4.1887C^2 \quad (5)$$

### 3.3. Influence of process variables on BSTR and USR reactors

Fig. 5 shows the effect of molar ratio on ethyl ester conversion. An excess of alcohol increases the ester conversion by shifting the equilibrium to the right (Le Chatelier’s principle). The use of ethanol: soybean oil molar ratio of 10:1 promoted an increase in ester conversion in the first minutes of the reaction; however, after 10 min, the conversion is almost molar ratio of 6:1.

As can be observed with the ethanol: soybean oil molar ratio of 6:1, the conversion of ester was almost 60 wt.% after 1 min, but the conversion increases to 80 wt.% with a molar ratio of 10:1 (Fig. 5a). The same behavior was observed with 1.5% of catalyst (Fig. 5b).

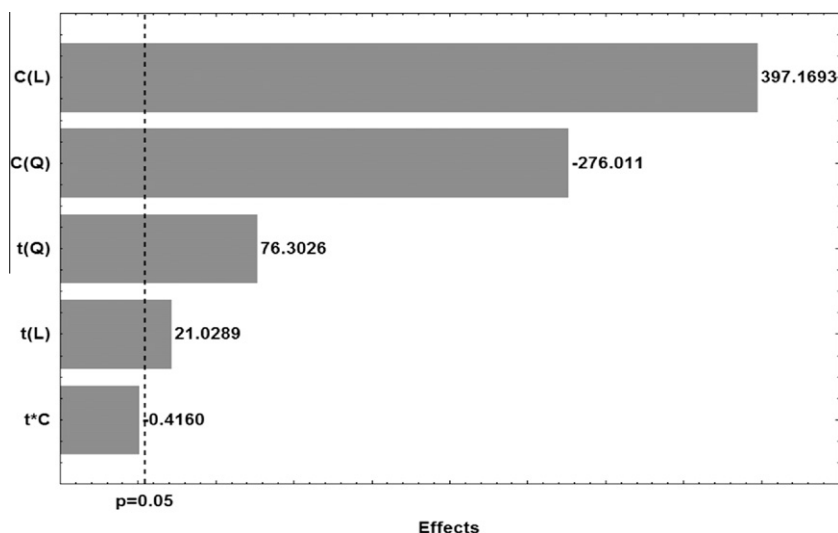


Fig. 3. Effects of the process variables on the soybean ethyl ester conversion: Second experimental design.

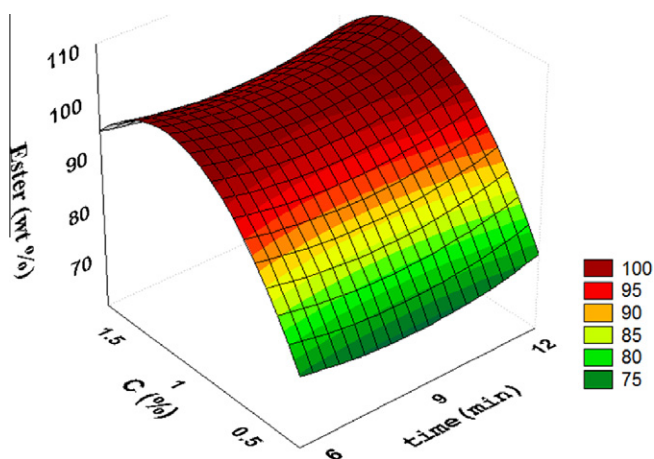


Fig. 4. Response surface of ethyl ester conversion as function of reaction time and catalyst content.

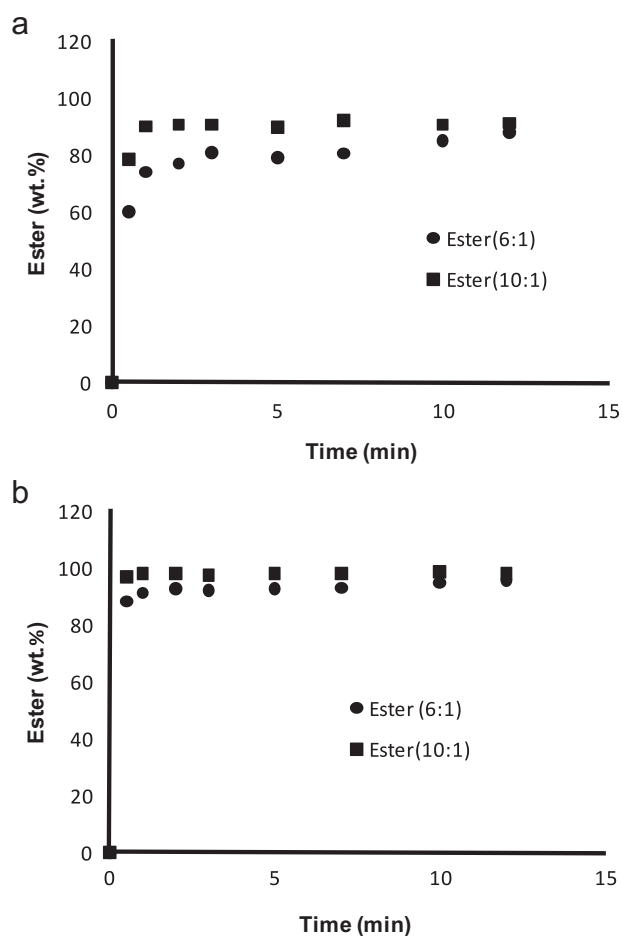


Fig. 5. Time evolution of ethyl ester conversion with catalyst content. (a) Catalyst content of 0.5 wt.%; (b) Catalyst content of 1.5 wt.%.

Nevertheless, after 10 min, similar conversions were obtained. This behavior is in agreement with the experimental design. Some researchers have shown the effect of molar ratio of alcohol to oil on biodiesel production using BSTR reactor (Encimar et al., 2007; Fillieres et al., 1995; Meher et al., 2006b) and the influence of the variables is similar to the USR reactor. Meher et al. (2006b) observed that a conversion of 97 wt.% of methyl esters from karanja oil was obtained using a molar ratio of methanol: karanja

oil of 6:1 after 3 h of reaction or using a molar ratio of 24:1 after a reaction time of 30 min.

The effect of the catalyst content on biodiesel production is also shown in Fig. 5. The reaction with a molar ratio of 6:1 and 0.5% catalyst achieved 60 wt.% of conversion after 1 min, while the use of 1.5% catalyst increased the conversion to 88.25 wt.%. The use of a molar ratio of 10:1 and catalyst contents of 0.5% and 1.5% led to an ethyl ester conversion of 74 and 97 wt.%, respectively. The ester conversions obtained after 12 min of reaction and 0.5% of catalyst to 6:1 and 10:1 molar ratios were 88 and 91 wt.%, respectively. The corresponding conversions using 1.5% of catalyst were 95 and 98 wt.%. These results are in agreement with studies using a BSTR reactor, but the highest conversions were obtained after 120 min (Fillieres et al., 1995; Meher et al., 2006b; Encimar et al., 2007).

The use of USR enabled the development of a fast process because the reaction takes place in the high-energetic shear zone of the mixer by reducing the droplet size of the immiscible liquids (soybean oil and ethanol).

#### 4. Conclusions

This work has presented the production of biodiesel using multiple-stage Ultra-Shear reactor. Different combinations of process variables were tested in the experimental design and the results show that a ethyl ester conversion of 99.26 wt.% can be obtained with a reaction of up to 12 min, with a catalyst content of 1.35 % and an ethanol:soybean oil molar ratio of 6:1. The use of this system reduces the reaction time and increases production (volume). This enables a continuous production process at a lower cost than the conventional process (BSTR reactor).

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