



Optimization of biodiesel production from animal fat residue in wastewater using response surface methodology

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HIGHLIGHTS

- Biodiesel was obtained from waste water animal fat residue.
- Two-step acid-catalyzed transesterification process was employed.
- Operating parameters were optimized by response surface methodology.

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ABSTRACT

Animal fat residues (AFR) from waste water were used as feedstock to produce biodiesel by a two-step acid-catalyzed process. Treatment of the AFRs with 5.4% (w/w) of 17 M H₂SO₄ at a methanol/AFR ratio of 13:1 (50% w/w) at 60 °C converted more than 95% of the triglycerides into fatty acid methyl esters (FAMES) with an acid value (AV) of 1.3 mg_{KOH}/g_{biodiesel}. Response surface methodology indicated that a lower AV cannot be reached using a one-step acid catalyzed process. Thus a two-step acid catalyzed process was employed using 3.6% catalyst and 30% methanol for 5 h for the first step and 1.8% catalyst and 10% methanol for 1 h in the second step, resulting in a yield higher than 98% and an AV of 0.3 mg_{KOH}/g_{biodiesel}. The product thus conforms to the European norm EN14214 concerning biodiesel.

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1. Introduction

Biodiesel is a promising biofuel because its physical and chemical properties are similar to those of diesel fuel (Demirbas, 2008; Balat and Balat, 2008) and because its oxygen content enhances combustion and reduces hydrocarbon and particulate matter emissions (Demirbas, 2005; Rahimi et al., 2009; Basha et al., 2009).

Most of the produced biodiesel in the world is from vegetable oils, namely rapeseed oil in Europe and Canada and soybean oil in USA (Karmakar et al., 2010). Since biodiesel production from these sources can lead to competition with food production, other feedstock for biodiesel need to be explored. Animal fat residues (AFR) collected from fat traps are a cheap source of lipids that can be used to produce biodiesel. Andersen and Weinbach (2010) have estimated that in Norway 2.5 tons of AFR per 1000 households per year could be collected. Assuming that the European Union has the same potential as Norway, with 501 million households, the EU-27 has a potential to collect

1.25 million tons of AFR that could be transformed into 1250 million liters of biodiesel. The problem with AFRs is their high content of free fatty acids (FFA). Using an alkali catalyzed process, the FFA will be transformed immediately into soap. An acid-catalyzed reaction will be 4000 times slower than an alkali-catalyzed process and requires higher amounts of alcohol and catalyst and higher temperatures (Sharma and Singh, 2009; Lam et al., 2010; Agarwal, 2007). In order to solve this problem, an acid-catalyzed pre-esterification step is required to decrease the FFA level to 1% (West et al., 2008; Tiwari et al., 2007; Dias et al., 2009). This process is usually followed by an alkali-catalyzed process due to its reaction speed. Optimization of these reactions requires a large number of experiments and a mathematical tool that can predict the effect of each parameter of the reaction and their interactions. Response surface methodology has been successfully applied to the optimization of biodiesel production from different raw materials using different types of catalysts (Yuan et al., 2008; Bautista et al., 2009; Tiwari et al., 2007; Ghadge and Raheman, 2005).

In the present study, biodiesel production from AFR using a two-step acid catalyzed reaction was studied. The first step of reaction was optimized using response surface methodology. The produced biodiesel was analyzed and compared to European norm EN14214.

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2. Methods

2.1. Raw materials

Animal fat residues (AFR) were collected and pre-treated in a wastewater treatment centre, CTMV-CTMA, in Lussac near to Bordeaux, France. In this centre waste water was collected from an area of 2800 km² with 250,000 households and a number of food and meat processing companies. The AFR were separated from the wastewater as detailed in [Supplementary information \(Appendix A\)](#). Sulfuric acid (17 M) and methanol with a purity of 98% were purchased from Sigma Aldrich, France. The duck fat was a co-product of the duck meat processing industry. Waste cooking oil (WCO) was obtained from the University's restaurant and was composed of 45% palm, 45% sunflower, and 10% rapeseed oil. Sunflower oil was purchased from a supermarket. The three types of oils and fats were used to compare their viscosities to that of AFR in order to demonstrate the effect of degradation on the latter.

2.2. Experimental work

2.2.1. AFR analyses

Elemental composition analysis was performed using a CHNS-O Analyzer FLASH 1112 series E. The heating value was measured using an isoperibolic PARR 6200CLEF calorimeter. Acid values were determined by titrimetry. Viscosities of AFR, vegetable oils and duck fat were measured using a vibro viscometer with an error margin of 3%. Samples were heated to 80 °C and introduced into the viscometer equipped with a thermometer. The viscosity was measured at different temperatures while the liquid was cooling down until its temperature reached 30 °C. Density was measured with a densimeter having a precision of 0.01 kg/l. Water content was measured using the xylene vapor method (ASTM D 4006, ISO 9029).

2.2.2. FAME production

The experimental setup was composed of a 2-l double-necked flask immersed in an isothermal bath of water at 60 °C. A mechanical stirrer was introduced vertically through a rubber plug that sealed the flask's neck. The second neck was also plugged with rubber stopper and was used to introduce reagents at the beginning of the reaction and to take samples. Each experiment consisted of mixing 200 g of AFR with a mixture of methanol (CH₃OH) and sulfuric acid (H₂SO₄). The final H₂SO₄ and methanol concentrations were 1.8–5.4% and 30–50% by wt., respectively. Samples were taken after 1 h and 3 h and the reaction was stopped after 5 h. Before each sampling, the flask was removed from the water bath for 2 min, to minimize the amount of methanol vapors that could escape. The samples and the final products were separated from glycerol and impurities by decantation using a separatory funnel for 2 h at 30 °C, washed with distilled water, dried with calcium chloride and the acid values were measured. After the first-step reaction, the product was washed with distilled water, the phases were separated by centrifugation at a relative centrifugal force of 450g for 5 min and filtered through a layer of anhydrous calcium chloride. The second step was accomplished by reacting the filtered products with methanol via an acid-catalyzed process to eliminate the FFAs.

2.2.3. FAME characterization

Viscosity, density, water content, elemental composition, heating and acid value were determined as described in Section 2.2.1. PENSKEY MARTENS NPM440 device with a precision of 2 °C was used to determine the flash point of the biodiesel. A Perkin Elmer Turbo Mass Gold mass spectrometer coupled to a Clarus 500 gas

chromatograph was used to analyze the composition and to determine the amounts of fatty acid methyl esters (FAMES), mono-, di-, and triglycerides, free glycerol and methanol in the samples. A SBLTM-5 ms capillary column of 30 m length and 0.25 mm diameter with a 0.25-μm film thickness was used for this analysis. A FID Perkin Elmer Autosampler XL chromatograph and an FID Perkin Elmer Clarus 500 gas chromatograph were used to analyze the reaction products. In both chromatographs a Varian WCOT fused silica column, 30 m in length, 0.39 mm in diameter and with a 0.25-μm film thickness was used. Based on the spectrometry analysis of different samples, the predominant components were the methyl esters of myristic, palmitoleic, palmitic, stearic, oleic and linoleic acids. Thus the following standards were purchased from Sigma Aldrich laboratories and used to analyze the reaction products: AOCS N°6 analytical standard (FAME mixture), EN 14105: 2003 Monoglyceride stock solution and EN14105:2003 Standard kit (diglycerides, triglycerides and free glycerol mixture).

2.3. Statistical analysis

A three-level-three-factor design requiring 27 experimental combinations was employed in this optimization study. Each experiment was repeated three times and the mean acid value was calculated. The coded and uncoded levels of the independent variables are given in [Table 1](#). The experimental data obtained by the procedure described above were analyzed by a second order polynomial regression described in Eq. (1).

$$y = \beta_0 + \sum_{i=1}^3 \beta_i x_i + \sum_{i=1}^3 \beta_{ii} x_i^2 + \sum_{i=1}^2 \sum_{j=i+1}^3 \beta_{ij} x_i x_j \quad (1)$$

where y is the acid value of the first step reaction product, x_i and x_j are the independent coded variables, β_0 , β_i , β_{ii} , β_{ij} are the intercept, linear, quadratic and interaction coefficients, respectively. The regression analysis and the Analysis of Variance (ANOVA) were performed and the effects of independent variables on the reaction were evaluated using statistical tools like the Taylor test also called t -statistic test.

3. Results and discussion

3.1. AFR characteristics

The AFR had a very high acid value of 60 mg_{KOH}/g_{fat}, and a lower viscosity than usual animal fats and vegetable oils ([Fig. 1](#)). The elemental analysis indicated that the fat did not have apparent amounts of impurities and has almost same composition as non degraded fat (77% C, 13% H and 10% O). The density and the higher heating value of AFR were 870 g/l and 38 MJ/kg, respectively. No water was detected in the tested samples. The high acid value of the AFR required acid-catalyzed pretreatment.

Table 1
Coded and uncoded levels of the design.

Variable	Symbol	Coded levels		
		−1	0	1
Reaction's duration [h]	T	1	3	5
Catalyst amount [%]	C	1.8	3.6	5.4
Methanol amount [%]	M	30	40	50

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