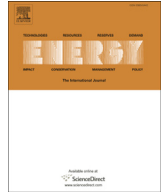




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Process parameter optimization of low temperature transesterification of algae-Jatropha Curcas oil blend

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ABSTRACT

Biodiesel, as a substitute for diesel has been getting the attention of researchers/scientists of all over the world. The R & D has indicated that up to B20, there is no need of modification and little work is available related to suitability and sustainability of biodiesel production from Jatropha and algae as non-edible oil sources. The objective of the present study was to optimize the process parameters for transesterification of low free fatty acid (FFA) Jatropha and algae oil blend. A low temperature transesterification process was selected to make the transesterification process more energy efficient. A model was developed to correlate the biodiesel yield with process parameters viz methanol/oil volumetric ratio, Catalyst concentration and reaction time. A biodiesel yield of 81.98% was achieved with methanol/oil volumetric ratio (3:5) using KOH as catalyst (0.9% w/w) in 180 min time at 50 °C temperature. It was observed that catalyst concentration, reaction time and methanol/oil volumetric ratio had a significant effect on blend yield. It is found out that this model can be used in the industry to improve the efficiency of biodiesel production from blend of Jatropha and algae oil thereby, saving time and cost of the process in optimizing the process parameters.

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1. Introduction

Conventional transport fuels derived from crude oil such as petrol, diesel and jet fuel comprises the large majority of fuel sales. MIT technological review on Indian energy crisis predicts that India will be having 20% of the world population by 2050 and dependency on fossil fuels such as coal and transportation petroleum fuel to develop economy and elevate living standards will result into climate catastrophe. However, alternative transport fuels such as biofuels, gaseous fuels and synthetic fuels have potential to improve environmental outcomes. Alternative transportation fuel such as biodiesel could be produced from any type of oil such as soybean, corn oil, palm or algae oil. However, algae oil is more attractive because of the algae capacity to yield more oil without requiring large area of arable lands, scope for better strain improvement and the capacity to enhance the value through co products [1]. But algae based biofuels requires a lot of energy and water and may lead to more GHG emissions than crop based biofuels over its complete life-cycle [2] On the other hand, the

preparation of biodiesel from Jatropha Curcas oil is a promising alternative in the present situation due to its higher oil content and non-edible nature [3]. Indian government has emphasized on Jatropha Curcas oil production under national biodiesel mission since 11th five year plan. The oil can be converted to biodiesel using transesterification, but the type of transesterification to be adopted depends on the free fatty acid (FFA) content of the oil. For the conversion of high FFA JCO, two-step acid-base catalyzed method has been-developed which consists of acid-catalyzed pretreatment/esterification step to reduce the FFA to less than 1% using H₂SO₄ as acid catalyst and transesterification of pretreated oil to biodiesel using alkali catalyst. This process consume extra time also [4]. Above problems of pretreatment of high FFA oil can be reduced by making a blend of both algae oil and Jatropha curcas oil, since it will not only reduce the amount of algae oil used but also the acid catalyzed esterification of Jatropha curcas oil is not required. Transesterification of the blend involves large number of parameters affecting the reaction, therefore optimization of these reaction factors needs a large number of experiments which is time consuming and uneconomical.

Researchers have applied various experimental techniques to optimize the process variables for biodiesel production. Gaurav et al. [5] implemented the Box–Behnken response surface

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methodology (RSM) for maximizing the biodiesel yield to 98.4% from Pongamia oil by optimizing the four process variables. Siddiqua et al. [6] used Box–Behnken response surface methodology to optimize three variables for predicting the best performing conditions (calorific value and yield) of algae biodiesel. Bandhu et al. [7] used RSM to Statistical design and optimize the single cell oil production from sugarcane bagasse hydrolysate by an oleaginous yeast *Rhodotorula* sp. IIP-33. Dasgupta et al. [8] used RSM to design and optimize the ethanol production from bagasse pith hydrolysate by thermotolerant yeast *Kluyveromyces* sp. IIEP453. RSM was also used by Sukjit and Punsuvon [9] for maximizing the production of the *Jatropha* biodiesel to 93.55% using CaO–MgO mixed oxide catalyst in transesterification process. Lee et al. [10] used RSM for maximizing the production of the *Jatropha* biodiesel to 96.57%. Vicente et al. [11] applied RSM to optimize the biodiesel production from refined sunflower oil. Carvalho et al. [12] optimized the process variables using RSM for maximizing the production of biodiesel from cotton seed oil to 96.79%. Renita et al. [13] produced biodiesel from the macroalgae *Caulerpa peltata* using central composite design to optimize the parameters like oil: alcohol ratio, catalyst amount, time and temperature. Xinyu et al. [14] used RSM and solid catalyst Cs₂O/c–Al₂O₃ for production of biodiesel from animal fat, the biodiesel yield was found to be 95.5%. Betiku and Adepoju [15] used RSM to investigate the biodiesel production from sesame oil with, highest conversion yield of 99.71% obtained under the optimized conditions. Aworanti et al. [16] investigated the effects of methanol-to-oil molar ratio, catalyst amount and reaction time on the transesterification of waste cooking oil (WCO) to biodiesel using RSM. Orthai et al. [17] used box Behnken design for RSM for optimizing the operating condition to reduce COD, O&G, and SS by 55.43%, 98.42%, and 96.59%, respectively. Junhua Zhang et al. [18] used box Behnken design for RSM to optimize the conditions for ZSO biodiesel production using CaO as a catalyst at this optimum condition, the conversion to biodiesel reached above 96%.

The aim of the present paper, therefore, is to analyse the effect of process parameters on transesterification process and yield of biodiesel. A model was formulated and validated for predicting the response. It is found out that this model can be used in the industry to improve the efficiency of biodiesel production from the blend thereby, saving time and cost of the process in optimizing the process parameters.

2. Materials and method

The paper reports the results of the optimization of three process variables viz. catalyst (KOH) concentration (0–2% w/w), reaction time (60–180 min) and methanol/oil ratio (v/v) (20–60%) for the transesterification process of blend of algae oil and *Jatropha curcas* oil at reaction temperature of 50 °C using RSM based Box–Behnken Design in 15 experimental runs with the help of Design Expert 9.0.6.2 software.

JCO was procured from *Jatropha* Vikas Sansthan, New Delhi, India. All chemicals like KOH, methanol, and NaOH were of analytical reagent grade and 99% pure. KOH in pellet form was used as a base catalyst. The fuel properties of *Jatropha Curcas* Oil and algae oil after refining were determined as per standard methods.

FFA content of *Jatropha curcas* oil was calculated very high as 22% while that of algae oil was 0.5% therefore these two oils were blended (1:42 (v/v)) to get the final FFA of 1%. Table 1 shows that a FFA content of the blended oil is as 1.0%. Owing to low FFA content, here we have used base catalyzed transesterification processes.

3. Experimental design

A Box–Behnken experimental design, with three variables, was used to study the response pattern and to determine the optimum combination of variables. The effect of the C (ratio of methanol to oil, (v/v)) A (reaction time (min)), and B (catalyst amount (w/w%)), at three variables levels in the reaction process is shown in Table 2. A total of 17 experiments were conducted separately for getting the experimental response of yield. The methanol/oil ratio (v/v) (C), reaction time (A) (min), and catalyst concentration (B) (%) were the independent variables selected for optimization. The coded and uncoded levels of the independent variables used for the transesterification of *Jatropha Curcas* Oil are given in Table 2.

3.1. Statistical analysis

The Design Expert 9.0.6.2 software is used for the regression and graphical analysis of the data. The maximum values of blend (*Jatropha Curcas* + algae) yield were taken as the response of the design experiment for transesterification process. The experimental data obtained by the above procedure was analyzed by the response surface regression using the polynomial Equation (1).

$$Y = \beta_0 + \sum_{j=1}^K \beta_j X_j + \sum_{j=1}^K \beta_{jj} X_j^2 + \sum_{i=1}^{j-1} \sum_{k=2}^k \beta_{ijk} X_i X_j X_k + \varepsilon \quad (1)$$

where Y is the response, i and j are the linear and quadratic coefficients respectively, x_i and x_j are the uncoded independent variables, is the regression coefficient, k is the number of factors studied and optimized in the experiment. Equation was also validated by carrying out confirmatory experiments.

3.2. Transesterification

Blend of *Jatropha Curcas* oil and algae oils was transesterified by using methanol and KOH as base catalyst for the production of biodiesel. The methyl ester layer was separated and processed according to Dwivedi and Sharma [5]. Transesterification of oil blend has been optimized using RSM for the maximization of biodiesel yield.

3.3. GC analysis

The optimized sample was analyzed for fatty acid composition by Gas Chromatograph (Netal make) equipped with a flame ionization detector and a capillary column for injecting the sample. The GC oven was kept at 230 °C (5 °C/min) and a total analytical time was 30 min. Nitrogen was used as carrier gas.

Quantitative analysis of % ME was done using European standard EN 14103:2003 [19]. The % ME yield was calculated using Eq. (2). Free fatty acids in the samples were determined using stock solution (Methyl heptadecanoate and n-heptane).

$$\% \text{ of ME} = \frac{\Sigma A - A_{EI}}{A_{EI}} \times \frac{C_{EI} - V_{EI}}{m} \times 100 \quad (2)$$

- ΣA Total peak area from the methyl ester in C14 to that in C24:1;
- A_{EI} Peak area corresponding to methyl heptadecanoate;
- C_{EI} Concentration of the methyl heptadecanoate solution (mg/ml);
- V_{EI} Volume of the methyl heptadecanoate solution (ml);
- m Mass of the sample (mg).

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