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Short communication

Optimization of scum oil biodiesel production by using response surface methodology

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ABSTRACT

The response surface methodology (RSM) was used to determine the optimal conditions for the biodiesel production from scum oil by using central composite design. Four process variables were assessed at five levels (2^4 experimental design). A total of 30 experiments had been designed and conducted to study the effect of methanol to oil molar ratio, reaction time, catalyst concentration (potassium hydroxide) and temperature on the biodiesel yield. An yield of 93% scum oil methyl ester (SOME/biodiesel) was obtained at different optimum conditions: 4.5:1 molar ratio of methanol to oil, 75 min reaction time, 1.20% catalyst concentration and 62 °C temperature. A linear relationship between the experimental yield and predicted values of biodiesel yield developed. The biodiesel product was characterized by Fourier transform infrared spectroscopy (FTIR). The fuel properties of the biodiesel such as kinematic viscosity, density, flash point, copper corrosion, calorific value, cloud point, pour point, ash content and carbon residue were determined.

1. Introduction

Fossil fuels are getting exhausted all over the world very fast due to increasing demand. Many countries in the world are in search of alternative sources of fuel for their energy needs. In this regard, biodiesel is an alternative to diesel fuel due to several benefits such as it is a renewable source that undergoes complete combustion, biodegradable, non-toxic, and less emissions of CO, SO_2, particulates and hydrocarbons compare to conventional diesel (Yoosuk et al., 2010; Kafuku et al., 2010). Biodiesel can be produced by the transesterification of triglycerides with alcohol (commonly methanol) in the presence of acid or base catalyst into fatty acid methyl ester (FAME) (Dussan et al., 2010; TuThanh et al., 2010). Primarily, the selection of suitable oil is to be made for the production of biodiesel considering the parameters as cost, availability, stability and manufacturing process. In present days, the edible oils are used for biodiesel production and these oils have high cost and negative impact on food chain (Kalam et al., 2008). Non-edible oils from seeds such as Karanja (*Pongamia pinnata*), Jatropha (*Jatropha curcas*), Rubber (*Hevca brasiliensis*), Mahua (*Madhuca indica*), Neem (*Azadirachta indica*), etc. are being used as source for biodiesel production as they are sufficiently available (Demirbas, 2009; Vedharaj et al., 2013). Here, another potential source from dairy industry i.e., the scum oil is used for biodiesel production (Sivakumar et al., 2011). A huge dairy produces large amount of scum per day which is difficult to dispose. Scum oil consists of fat, proteins, lipid, unsolicited materials, etc. A large quantity of water is used for washing the equipment and housekeeping. In this process, a large amount

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of dairy scum is collected from the effluent plant and disposed as waste (Yathish et al., 2013). From this dairy waste scum, scum oil is obtained by removing the unwanted material, water and suspended particles. Studying the optimization process is crucial for development of mass production of scum oil. Conservatively, the optimization of biodiesel production process was achieved with the variation of one factor at a time and the response is a function of a single parameter which is time overwhelming and excessive in cost (Bezerra et al., 2008). This technique does not include interactive effects among the variables and it does not depict the complete effect of the parameters on the process (Baş and Boyacı, 2007). However, application of response surface methodology (RSM) technique in multivariable system offers a research strategy in studying the interaction of the parameters using statistical methods. The experiment model of biodiesel synthesis which is developed using response surface methodology is able to simulate the reaction under various transesterification conditions with good estimations of errors. This is helpful when mass production of the biodiesel is needed. Rashid et al. used RSM to optimize the process parameters in base catalytic methanolysis of sunflower seed oil for biodiesel production (Rashid et al., 2009). In another study, Bojan et al. applied the same method for biodiesel production from high free fatty acid *J. curcas* oil (Bojan et al., 2011). Response surface methodology was applied to optimize transesterification conditions for biodiesel production using acid oil and jajoba oil (Bouaid et al., 2007; Chen et al., 2008). In the present work, efforts were made to optimize the process conditions for transesterification reaction to increase the yield of biodiesel from scum oil. The influence of the variables such as catalyst concentration, alcohol to oil molar ratio, reaction temperature and reaction time on transesterification was studied. The quality tests of the scum oil methyl esters were also reported.

2. Materials and methods

2.1. Materials

The dairy scum was collected from Karnataka milk Federation (KMF), Tumkur Milk Union (TMU), Tumakuru, Karnataka. Fresh scum was collected to avoid increase in free fatty acid and biological action. Scum is semi solid and white in color. A known quantity of scum was heated at a temperature of 100 °C to convert to liquid form and allowed to settle for few minutes. After settling lower aqueous phase has to be removed and the top layer is separated and centrifuged to remove the solid wastes and suspended solids. Then the oil was filtered and used for transesterification reaction. The potassium hydroxide and methanol were purchased from Fisher Scientifics.

2.2. Production method

The transesterification reaction was carried out in a 500 ml three neck flask equipped with a reflex condenser, thermostat, mechanical stirrer, sampling outlet and mechanical stirrer set at 600 rpm. 100 g of scum oil was added to the flask and preheated the oil before the reaction started. A definite amount of KOH is added to the methanol and the mixture was stirred until the KOH dissolved completely. The resultant solution was added to the preheated oil and stirred at preferred time and temperature. After completion of the reaction, the reaction mixture was transferred into a separating funnel and allowed to cool and equilibrate, which resulted in the separation of two phases. The upper phase consisted of methyl ester and lower phase contained the glycerol, remained catalyst and excess methanol. After separation of two layers by sedimentation, the methyl ester was purified by distilling the remaining methanol at 70 °C, then methyl ester was washed using pH 7 water (50 °C) to remove the residual catalyst, soaps and entrained glycerol. Finally the residual water was removed by heating the methyl ester above 100 °C. The yield of methyl ester always expressed as

Table 1 – Experimental range and values for RSM.

Independent variables	Range and level				
	$-\alpha$	-1	0	1	α
Molar ratio	3:1	4.5:1	6:1	7.5:1	9:1
Reaction time (min)	30	45	60	75	90
Catalyst concentration (wt%)	0.6	0.8	1.0	1.2	1.4
Temperature (°C)	41	48	55	62	69

$$\text{Yield of methyl ester} = \frac{\text{Gram of ester produced (g)}}{\text{Gram of oil taken for reaction (g)}} \times 100 \quad (1)$$

2.3. Experimental design

The effects of four transesterification variables on the yield of scum oil methyl esters were evaluated using response surface methodology based on four factors and five level central composite rotatable designs (CCRD) consisting of 30 experiments ($=2^k + 2k + 6$, where k is the number of independent variables) including 6 replicates at the center point to estimate the pure error (Haaland, 1990). The design variables were molar ratio (P_1), reaction time (P_2, min), catalyst concentration (P_3, wt%) and temperature (P_4, °C) while the response variable was biodiesel yield (Y, %). The range and the levels of the independent variables chosen for the present study are presented in Table 1. Each experiment was performed in duplicates and the average yield of biodiesel was taken as the response variable Y.

2.4. Statistical analysis (ANOVA)

The polynomial equation raised to the order of two was then assigned to the obtained data by a multiple regression protocol. Thus complying an empirical model which gives the nature between responses measured to the independent variables of the experiment.

The empirical regression model equation for a four factor system was taken as

$$\begin{aligned} Y = {} & \beta0 + \beta1P_1 + \beta2P_2 + \beta3P_3 + \beta4P_4 + \beta11P_1^2 + \beta22P_2^2 + \beta33P_3^2 \\ & + \beta44P_4^2 + \beta12P_1P_2 + \beta23P_2P_3 + \beta13P_1P_3 + \beta34P_3P_4 \\ & + \beta14P_1P_4 + \beta24P_2P_4 \end{aligned} \quad (2)$$

where Y is the predicted response, $\beta0$ is the intercept, $\beta1$, $\beta2$, $\beta3$, $\beta4$ are linear coefficients, $\beta11$, $\beta22$, $\beta33$, $\beta44$ are squared coefficients, and $\beta12$, $\beta23$, $\beta13$, $\beta34$, $\beta14$, $\beta24$ are interaction coefficients and P_1 denoted molar ratio (i.e. methanol:oil ratio), P_2 was reaction time (min), P_3 was catalyst concentration (wt%) and P_4 was temperature (°C). The response of the CCRD design was fitted with a second-order polynomial