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Bio-Diesel Production from Crude Cotton Seed Oil

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Abstract:

Biodiesel, known as fatty acid methyl ester (FAME), was produced from crude cottonseed oil (triglycerides) by transesterification with methanol in the presence of sodium hydroxide. This process was optimized by applying factorial design and response surface methodology (RSM) with SAS and PSIPLOT programs. A second-order mathematical model was obtained to predict the yield as a function of methanol/oil molar ratio, catalyst concentration, reaction temperature, and rate of mixing. Based on ridge max analysis and RSM, as well as economic cost consideration, the practical optimal condition for the production of biodiesel was found to be: methanol/oil molar ratio, 7.9; temperature, 53 °C; time, 45 min; catalyst concentration, 1.0%; and rate of mixing, 268 rpm. The optimized condition was validated with the actual biodiesel yield of 97%. Furthermore, the biodiesel was confirmed by HPLC analyses that triglycerides of cottonseed oil were almost completely converted to FAME.

Keywords: Biodiesel, Cottonseed oil, Methanolysis, renewable energy, Transesterification

1. Introduction

A method of production of bio-diesel is trans-esterification or alcohplysis. Generally this method is done by mixing the reactant-cotton seed oil(fatty acids),alcohol, catalyst. The by-product glycerol obtained from this process cab be used as a subtract for food industry and it can also be as substract for anaerobic digestion. These hydroxide are dry Flakes or pellet and must be dissolved in methanol, which produce “methoxide” concentrated in methanol. cotton seeds are sent to water bath shaker . The catalyst is dissolved in methanol very slowly to prevent splashing and sudden temperature rise. The practical optimum condition for synthesis of biodiesel was found to be methanol/ oil molar ratio-7:9; temperature -53 degree Celsius; time- 45 minutes: catalyst concentration – 1% and the rate of mixing 268 rpm. The biodiesel obtained from transesterification is bio degradable, renewable and non-toxic, less emission of gaseous and particulate pollutants with higher cetane number than normal diesel transesterification is a sequential process than usually starts at one reasonable operating condition, and then requires three stages to achieve a set of ” better” conditions as rapidly and efficiently as possible. The first stage is to conduct several experiments to determine the direction so as to take next new towards the optimal value. The

sw second stage is to perform several ends along the direction as indicated by first stage until an optimal value was approached. The last step is to deduce a mathematical model(equation) and profile the response surface to determine the optimal condition, which should be validated by the actual process.

2. Materials and Methodology:-

2.1.Materials

Methanol and sodium hydroxide were purchased from Fisher Scientific (Suwanee, GA, USA). Crude cottonseed oil derived from expeller, i.e. screw pressed cottonseed, was obtained from the Elgin Cotton Oil Mill, Inc. (Elgin, TX, USA). The Gyrotory water bath shaker was purchased from New Brunswick Scientific Co. Inc. (NJ, USA).

2.2. Characterization of Crude Cottonseed Oil

An aliquot of about 10 mg of oil was weighed and mixed with 2 ml of hexane, then 0.2 ml of 2 M methanolic KOH was added for transesterification. The mixture was vortexed for 2 min at room temperature, and centrifuged, then an aliquot (2 microliters) of the hexane layer was collected for GC analysis. Shimadzu's GC-FID system, used for the qualitative and quantitative analyses of fatty acids of the crude cottonseed oil and biodiesel consists of a GC-17A, a flame ionization detector, and a DB-WAX capillary column (60 m0.25 mm, thickness=0.25 m; J&W Scientific). The initial temperature for oven was set at 180 °C and held for 2 min. Then the temperature increased from 180 °C to 250 °C at the ramp of 5 °C/min and held at 250 °C for 30 min. The injector and detector were maintained at 200 °C and 220 °C, respectively. Helium was used as a carrier gas, and its flow rate was kept at 1.5 ml/min. Free fatty acid content of the cottonseed oil was measured according to the A.O.C.S. Official Method Ca 5a-40 [11]

2.3. Transesterification of Crude Cottonseed Oil

The crude cottonseed oil reacted with methanol in the presence of sodium hydroxide to produce methyl esters of fatty acids (biodiesel) and glycerol . To optimize the above transesterification process, a three-level-five-factor (25) fractional factorial experimental design was employed. The crude cottonseed oil was precisely quantitatively transferred into an Erlenmeyer flask immersed in the Gyrotory water bath shaker. Then specific amount of sodium hydroxide (by weight of crude cottonseed oil) dissolved in the required amount of methanol was added. The reaction flask was kept in the water bath under constant temperature with defined agitation throughout the reaction. At the defined time, sample was taken out, cooled, and the biodiesel (i.e. the methyl ester in the upper layer) was separated from the by-product (i.e., the glycerol in the lower layer) by settlement overnight under ambient condition. The percentage of the biodiesel yield was determined by comparing the weight of up layer biodiesel with the weight of crude cottonseed oil added

2.4.Purification of Methyl Ester Phase

Since the remaining unreacted methanol in the biodiesel has safety risks and can corrode engine components, the residual catalyst (sodium hydroxide) can damage engine components, and soap in the biodiesel can reduce fuel lubricity and cause injector coking and other deposits [12], the methyl ester layer (biodiesel) was washed by mist washing with 1:1 volume of hot distilled water (about 60 °C) using a misting nozzle to make a fine, gentle mist, which was allowed to float over the surface of the biodiesel. After removing the unreacted methanol, the remaining catalyst, and soap, the washed biodiesel was placed into an oven at 55 °C to evaporate the water residue and then dried with sodium sulphate so as to minimize the undesired biological growth.

2.5.HPLC Methods

Reverse phase HPLC was used to qualitatively and quantitatively analyze the conversion of triglyceride into biodiesel. The Shimadzu HPLC system consisted of an evaporative light scattering detector (ELSD) with a Phenomenex Gemini C18 column . HPLC grade acetonitrile (A) and dichloromethane (B) were selected as the mobile phase. The gradient program was as follows: Time: (0, 5, 30, 32, 35 min) for solvent B: (0, 15, 70, 70, 0%). The flow rate of the mobile phase was 1.0 ml/min. Twenty microliters of the diluted biodiesel sample was injected via autosampler.

3. Biodiesel characterization

Determination of Specific gravity A clean and dry bottle of 25ml capacity was weighed and then filled with the biodiesel sample, stopper inserted and reweighed to give (W1). The sample was substituted with water after washing and drying the bottle and weighed to give (W2). The specific gravity was determined by (W1-W0) / (W2-W0).

3.1 Determination of viscosity

Viscosity is a measure of the resistance of a fluid which is being deformed by either shear stress or tensile stress.15ml of water was sucked through suction pipe till it crossed the upper mark of the viscometer, and then the time required by the water to flow from upper-mark to lower-mark was noted down with the help of a stopwatch. Further 15ml of biodiesel sample was taken in a viscometer. It was sucked through suction pipe till the sample crossed the upper mark of the viscometer. Then, the time required by the sample to flow from upper-mark to lower- mark was noted. Relative Viscosity was determined by the equation To/Tw, Where To = Time taken for biodiesel to travel from upper mark to lower mark, Tw = Time taken for water to travel from upper mark to lower mark.

3.2 Determination of Flash Point

Flash point was measured using Pensky Marten's apparatus. The cup was rinsed, cleaned and dried before starting the test. The cup was filled up to the mark with the biodiesel sample and covered with the lid. Thermometer was inserted such that, the bulb got immersed in the sample and care was taken that stirrer would not touch the thermometer. The initial temperature of the sample was noted down. Heater was started and the power level was set such that temperature of sample rises at the rate of 3o C/min. The stirrer rotated at 2 rev/ sec. Test flame was applied by operating shooter. For every 2ºC rise in temperature, the test flame was brought near cup surface for observing the phenomenon. When flash appeared on the surface of cup, the temperature was noted down and taken as Flash point.

4. Results and Discussion

Determination of % FFA content Higher amount of free fatty acids (>1% w/w) in the feedstock can directly react with the alkaline catalyst to form soaps, which are subject to form stable emulsions and thus prevent separation of the biodiesel from the glycerol fraction and decrease the yield, it is better to select reactant oils with low FFA content or to remove FFA from the oil to an acceptable level before the reaction [12]. The below table shows the %FFA of cottonseed oil to be equal to 0.357 % which is below 2.5 %. So the %FFA content of the oil taken for the research is within the limits.

5. Conclusion

Several parameters affecting biodiesel production from Cottonseed oil were studied. The free fatty acid of the cottonseed oil was determined and found to be below 2.5%.The optimum values of different parameters affecting the biodiesel production were as follows: 0.75% NaOH (w/woil) concentration, 1:1 methanol to oil molar ratio and 150 minutes reaction time. The results showed that NaOH was the best catalyst for this reaction condition. It was found that excessive catalyst concentration results in formation of soap and cause emulsion formation during purification of biodiesel which results in decreased ester yield. Increasing the alcohol to oil molar ratio decreased the ester yield, because of the presence of glycerin in the solution. The experimental values of different properties were found to meet the international standards. Overall results showed that it was effective to produce good quality biodiesel from Cottonseed oil which could be used for diesel engine.

References :-

[1] Xiaohu fan, Xwang, feng chen. The open fuels and energy science journal, 2011, 4: 1-8,

[2] Singh ABH, Thompson J, Van Gerpen J. Appl. Eng. Agric. 2006, 22 (4):597-600.

[3] De Oliveira D, Di Luccio M, Faccio C, Dalla Rosa C, Bender JP, Lipke N, Amroginski C, Dariva C, De Oliveira JV. Appl. Biochem. Biotech. 2005, 122 (1-3): 553-560.

[4] Jeong GT, Park DH. Biotechn. Appl. Bioc. 1999,131 (1-3): 668-679.

[5] Vicente G, Martinez M, Aracil J. Bioresource Tech. 2004, 92 (3):297-305.

[6] Meka PK, Tripathi V, Singh RP. J. Oleo Sci. 2007, 56 (1): 9-12.
[7] Darnoko D, Cheryman M. J. Am. Oil Chem.Soc. 2000, 77 (12):1263-1267.
[8] El Mashad HM, Zhang R, Roberto J. American Society of Agricultural Engineers Annual Meeting, 066144.
[9] Canakei M, Van Gerpen J.Transactions of the ASAE, 2001,44(6):1429-1436.
[10] Hossain ABMS, Nasrulhaq BA, Salleh A, Chandran S. African Journal of Biotechnology. 2010, 9(27): 4233-4240.
[11] Akpan UG, Jimoh A, Mohammed AD. Leonardo Juornal of Sciences, 2006, 8: 43-52.
[12] Demirbas A. Energ. Convers. Manage. 2009, 50 (4):923-927.
[13] Ashish K, Naveen K, Hasan MM, Rajeev C, Arshad NS, Zahid AK. Iranica Journal of Energy & Environment. 2010, 1(4): 352-358.