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FACTOR AFFECTING THE PREPARATION OF BIODIESEL FROM SORGHUM OIL

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Abstract

Biodiesel has been gaining worldwide popularity as an alternative energy source because it is non toxic, biodegradable, non flammable and environmental friendly fuel. These biodiesel can be prepared from edible and non-edible oils by transesterification with methanol or ethanol. The transesterification can be carried out chemically or enzymatically. In the present work the factor affecting the preparation of biodiesel from crude sorghum oil. In addition, the optimum condition for producing biodiesel using sodium hydroxide as the catalyst in batch reactor is provided. The optimization was carried out based on variables namely, mass ratio of ethanol to oil, catalyst concentration, reaction temperature and reaction time. Using at 2^4 factorial experimental designs, four variables were studied at both high and low levels. Thin layer chromatography (TLC) and gas chromatography (GC) was used to analyze the purity of biodiesel. The production yield and purity of biodiesel were used to verify the optimization. Results suggested that the catalyst concentration was the most important factor affecting the ethyl ester yield. Room temperature was considered to be the optimum temperature for the preparation of biodiesel from crude sorghum oil with 1% sodium hydroxide catalyst, 1:3 mass ratio of ethanol to oil, 120 minutes reaction time which gave 91.00% production yield and 98.29% biodiesel concentration .

Keyword: Biodiesel; Transesterification; Sorghum oil; environmentally friendly fuel.

1. Introduction

Biodiesel is a renewable fuel that can be synthesized from edible, non-edible and waste oils. Due to diminishing petroleum reserves, vegetable oils have attracted attention as a potential renewable source for the production of alternatives to petroleum-based diesel fuel. A number of processes have been developed for biodiesel production involving chemical or enzyme catalysis or supercritical alcohol treatment [1-4]. The most common process for producing biodiesel is known as transesterification reaction. During this process, the edible and non-edible oil reacted with alcohol such as methanol or ethanol in the presence of base or acid catalyst, resulted in biodiesel and glycerin as valuable by product. The of factor affecting the production of biodiesel in terms of production yield and purity of biodiesel include reactant purity, mixing time, reaction temperature, catalyst type and concentration, and mass ratio of methanol to oil. Operating condition used in biodiesel production and property of biodiesel produced depend upon the feedstock source. Numerous feedstock sources have been used for producing biodiesel. In the Indian context, the bulk of the efforts have been directed toward obtaining biodiesel by chemical transesterification of jatropha curcas oil [5] and other Many starting materials such as soybean oil [6, 7], sunflower oil [8, 9], cotton seed oil [10], rapeseed oil [11], palm oil [12, 13] and restaurant kitchen wastes [14] have been evaluated for preparation of biodiesel by the base catalytic route. However, it is felt that alternative starting oils also need to be studied [15]. Sorghum constitutes one such potential alternative source for biofuel. Sorghum (sorghum bicolor) belongs to Poaceae family and popularly called as jowar, jondhri, jundi, chari, is most extensively grown creal grain in country. The crop is environmentally-friendly as it is water-efficient, requires little or no fertilizer or pesticides and biodegradable [6]. The sorghum grown in two major seasons, viz kharif and rabi. The decline in area is mostly in kharif and at present, area of both kharif and rabi is more or less equal. As far as the productivity is concerned, the kharif crop yield is higher when compared with rabi crop[17].

Sorghum crops are genus comparing many species growing in tropical and subtropical countries; eight species are reported to occur in India. Sorghum grain is crop plant, which grown in several parts of India. The food, feed and fodder needs of farmers will not be affected, as the oil extraction. The seed kernel of sorghum is comprised of 30-50% oil [10].The fatty composition of sorghum oil consist of palmitic acid 10-14%, stearic acid 3-6%, oleic acid 3-47%, lenoliec acid 40-55% and lenolenic acid 0-1% [18], while composition of the oil is similar to other oil, which are edible and non-edible oil purpose. Thus it is good choice as the starting oil for the production of biodiesel.

The present paper reports the studies of factor affecting the preparation of biodiesel from crude sorghum oil; can be the potential raw material for biodiesel production in India.

2. Experimental

2.1 Experimental design

A 2^4 factorial experimental design was used to determine the optimum conditions, four variables were studied at both high and low levels. The response is ethyl ester yield. The low level of ethanol: oil mass ratio was 1/5 and the high level was 1/2. The low level of catalyst concentration chosen was 0.5 % and the high level was 1.5 % sodium hydroxide catalyst by weight of sorghum oil. The low level of temperature was chosen as room temperature (30°C) and the high level was chosen at 60°C. The reaction time chosen for the lower level was 30 minutes and 120 minutes for the higher level. Additional experiments were conducted to study the effect of catalyst concentration and mass ratio of ethanol to oil at 1 % sodium hydroxide and 1:3 mass ratios [19].

3. Materials and method

Sorghum oil was purchased at the local market of Moga, Punjab in India was used in the experimental study. Physical and chemical properties of crude sorghum oil are presented in table 1. Sodium hydroxide and ethanol A.R. Grade of Merck were used.

Transesterification reactions were carried out in 500-ml flask equipped with a reflux condenser. The reactor was filled with 200-g crude sorghum oil. The catalyst, sodium hydroxide was dissolved in ethanol and then added to the reactor. The mixture was heated to selected temperature. After the end of the reaction, the mixture was cooled to room temperature and transferred to a separating funnel. The two layers were separated by sedimentation. The ethyl ester phase was washed with hot distilled water. The excess ethanol was removed on a rotary evaporator at atmospheric pressure. Dried the solution over anhydrous sodium sulfate and filtered. The biodiesel product was analyzed by carrying out thin layer chromatography (TLC) and gas chromatography (GC), after measuring the production weight. The ethyl ester was defined as fallows
Ethyl ester (biodiesel) yield (%) = Production yield % X Ethyl ester concentration (wt/wt)

Table 1 Physical and chemical properties of crude sorghum oil [18]

Properties of crude sorghum oil	value
Free Fatty Acid (mg KOH/g)	0.864
Acid value (mg KOH/g)	0.434
Saponification value (mg KOH/g)	181-191
Iodine value (gI-2/g)	108-122
Density (g/mL)	0.9099
Specific gravity (g/mL)	0.9153
Viscosity 34°C (mm^2/S)	3.14
Flash point (°C)	225
Fatty acid composition (wt %)	
Linoleic acid	40-55
Oleic acid	3.0-47
Palmitic acid	6.0-10
Stearic acid	3.0-6.0
Linolenic acid	0-0.1
Myristic acid	0-0.1

4. Results and Discussion

The experimental results were obtained from 16 runs and according to the experimental design, which are shown in Table 2.The results were analyzed using normal probability method. It indicated that the catalyst concentration was the most important factor affecting the ethyl ester yield. Temperature, mass ratio of ethanol to oil and reaction time had because slightly significant effect on the ethyl ester yield

Table 2 Reaction conditions, production yield, ethyl ester concentration and ethyl ester yield

EtOH:Oil Mass ratio	NaOH (wt %)	Temperature. (°C)	Retention time (min)	Production Yield (wt%)	Ethyl ester Concentration (wt%)	Ethyl ester Yield (wt%)
1:2	1.5	60	120	73.85	99.45	73.45
1:5	1.5	60	120	79.95	97.87	78.25
1:2	0.5	60	120	88.70	99.43	88.20
1:5	0.5	60	120	92.90	94.51	87.80
1:2	1.5	30	120	90.20	98.66	89.00
1:5	1.5	30	120	87.00	95.97	83.50
1:2	0.5	30	120	91.60	98.36	90.10
1:5	0.5	30	120	95.70	85.57	81.90
1:2	1.5	60	30	81.55	98.52	80.35
1:5	1.5	60	30	73.48	93.19	68.48
1:2	0.5	60	30	86.90	98.38	85.50
1:5	0.5	60	30	92.80	88.68	82.30
1:2	1.5	30	30	91.75	98.63	90.50
1:5	1.5	30	30	88.30	98.41	82.30
1:2	0.5	30	30	93.60	96.26	86.90
1:5	0.5	30	30	92.50	84.05	77.75

4.1 Effect of reaction time

The production yield which is nearly independent of reaction time but the methyl ester concentration increases with increased reaction time. Due to the increasing of mixing and dispersion of methanol in oil phase increased with reaction time [20].

4.2 Effect of catalyst concentration

Sodium hydroxide was used as a catalyst for transesterification reaction in this work because of its economic reason. The effects of sodium hydroxide concentration on the production yield and ethyl ester concentration are presented in figure 1 and figure 2. It can be observed that the production yield decreases with increased sodium hydroxide concentration from 0.5 to 1.5 % by oil weight, because of soap formation from the reaction of oil and excessive amount of catalyst used. The rise in soap formation made the ethyl ester dissolution in glycerol layer greater [21]. The ethyl ester concentration increases with increased catalyst concentration at lower EtOH: oil mass ratio. It is probably due to the lag of ethyl ester production because the mass transfer limitation at the lower mass ratio of reactants [22,23]. However, catalyst concentration had no detectable effect on ethyl ester concentration at higher EtOH: oil mass ratio.

4.3 Effect of mass ratio methanol to oil

Higher mass ratio of reactant increases the contact between the ethanol and oil molecules so the ethyl ester concentration increases with increasing mass ratio of ethanol to oil. But the production yield decreases with increased mass ratio of reactant. These results agree with those obtained [24] who indicated that an excess of alcohol will increase the ester conversion by shifting the equilibrium to the right, but higher amount of alcohol interferes the separation of glycerin because there is an increase in solubility.

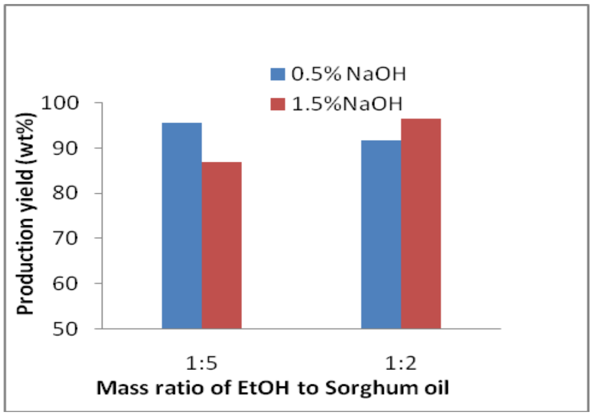

Fig. 1 Effect of mass ratio of ethanol to sorghum oil and catalyst concentration on the production yield at 30°C and 120 minutes: 0.5 and 1.5% NaOH

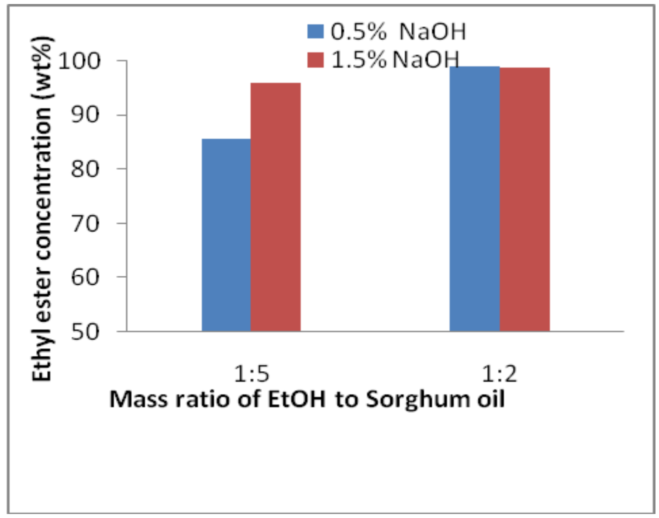

Fig. 2 Effect of mass ratio of ethanol to sorghum oil and catalyst concentration on the ethyl ester concentration at 30°C and 120 minutes: 0.5% and 1.5% NaOH.

4.4 Effect of temperature

To date, most of the research has focused on the transesterification at near boiling point of alcohol used. A few works reported the reaction at room temperature [24], studied the synthesis of ethyl ester of *Cynara cardunculus* L. oil in batch reactor and reported 91.6 % conversion at room temperature. In this work, the effects of reaction temperature on the production yield and ethyl ester concentration are presented in figure 3 and figure 4. The productions yield decreases with increased temperature because the higher solubility of reactant at higher temperature reduced the separation between ethyl ester and glycerol phase. The temperature had slight effect on ethyl ester concentration. As results above, room temperature is considered to be the optimum temperature.

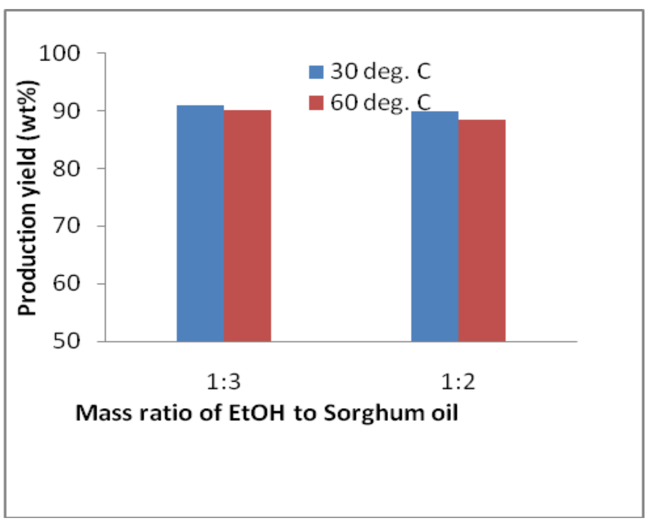

Fig. 3 Effect of reaction temperature on the production yield at 30°C and 60°C, 1.0% NaOH and 120 minutes

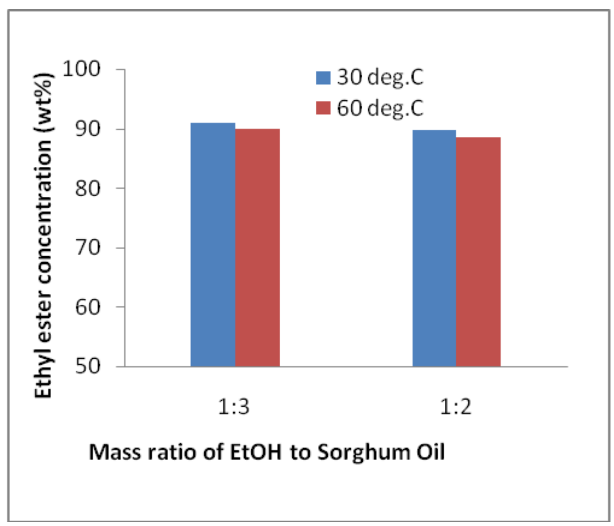

Fig. 4 Effect of reaction temperature on the ethyl ester concentration at 30°C and 60°C, 1.0% NaOH and 120 minutes

5. Conclusions

The study showed that the optimum temperature for the synthesis of biodiesel from crude sorghum oil was at room temperature, 1% NaOH catalyst, 1:3 mass ratio of ethanol to oil, 120 minute reaction time which gave 91.00% production yield and 98.29% ethyl ester concentration.

References

[1] Fukuda, H., Kondo, A., Noda H.: Biodiesel fuel production by transesterification of oils. J. Biosci Bioeng., (2001), 92:405-416.

[2] Zhang, Y., Dube, M. A., McLean, D.D., Kates M.: Biodiesel production from waste cooking oil. 1. Process design and technological assessment. Bioresour Technol, 2003, 89:1-16.

[3] Kusdiana, D., Saka, S.: Effects of water on biodiesel fuel production by supercritical methanol treatment. Bioresour Technol, 2004, 91:289-295.

[4] Warabi, Y., Kusdiana, D., Saka, S.: Reactivity of triglycerides and fatty acids of rapeseed oil in supercritical alcohols. BioresourTechnol 2004, 91:283-287.

[5] Francis, G., Edinger, R., and Becker, K.: A concept for simultaneous wasteland reclamation, fuel production, and socio-economic development in degraded areas in India: Need, potential and perspectives of Jatropha plantations. Nat. Resource Forum 2005, 29, 12-24.

[6] Schwab,A.W., Dykstra, G.J., Selke, E., Sorenson, S.C., Pryde, E.H.: Diesel fuel from thermal decomposition of soybean oil. J Am Oil Chem Soc 1988, 65:1781-1786.

[7] Samukawa, T., Kaieda, M., Matsumoto, T., Ban, K., Kondo, A., Shimada, Y., Noda, H., Fukuda H.: Pretreatment of immobilized Candida Antarctica lipase for biodiesel fuel production from plant oil. J Biosci Bioeng 2000, 90:180-183.

[8] Belafi Bako, K., Kovacs, F., Gubicza, L., Hancsok, J.: Enzymatic biodiesel production from sunflower oil by Candida antarctica lipase in a solvent-free system. Biocatal Biotransform 2002, 20:437-439.

[9] Dossat, V., Combes, D., Marty, A.: Lipase-catalyzed transesterification of high oleic sunflower oil. Enzyme Microbiol Technol 2002, 30:90-94.

[10] Oznur, K., Tuter, M., Aksoy, H. A.: Immobilized Candida Antarctica lipase-catalyzed alcoholysis of cottonseed oil in a solventfree medium. Bioresour Technol 2002, 83:125-129.

[11] Nelson, L. A., Foglia, T. A., Marmer, W. N.: Lipase catalyzed production of biodiesel. J Am Oil Chem Soc 1996, 73:1191-1195.

[12] Abigor, R. D., Uadia, P. O., Foglia, T. A., Hass, M. J., Jones, K. C., Okpefa, E., Obibuzor, J. U., Bafor, M. E.: Lipase-catalyzed production of biodiesel fuel from some Nigerian lauric oils. Biochem Soc Trans 2000, 28:979-981.

[13] Crabbe, E., Nolasco-Hipolito, C., Kobayashi, G., Sonomoto, K., Ishizaki, A.: Biodiesel production from crude palm oil and evaluation of butanol extraction and fuel properties. Process Biochem 2001, 37:65-71.

[14] Hsu, A. F., Jones, K., Foglia, T. A., Marmer, W. N.: Immobilized lipase-catalyzed production of alkyl esters of restaurant grease as biodiesel. Biotechnol Appl Biochem 2002, 36:181-186.

[15] Srivastava, A., Prasad, R.: Triglycerides-based diesel fuels. Renewable and Sustainable Energy Reviews 2000, 4: 111-133.

[16] Schwab A, W., Dykstra G, J., Selke, E., Sorenson S, C., Pryde E, H.: Dieselfuel from thermal decomposition of soybean oil. *J Am Oil Chem Soc* 1988, 65:1781-1786.

[17] Samukawa, T., Kaieda, M., Matsumoto, T., Ban, K., Kondo, A., Shimada, Y., Noda, H., Fukuda, H.: Pretreatment of immobilized *Candida Antarctica* lipase for biodiesel fuel production from plant oil. *J Biosci Bioeng* 2000, 90:180-183.

[18] www.chempro.in/Fatty acid.htm

[19] Attanatho, L., Magmee, S., and Jenvanitpanjakul, P.: Factors Affecting the Synthesis of Biodiesel from Crude Palm Kernel Oil, The Joint International Conference on "Sustainable Energy and Environment (SEE)" 1-3 December 2004, Hua Hin, Thailand

[20] Freedman, B., Pryde, E.M. and Mounts, T, L.: Variables Affecting the Yield of Fatty Esters from Transesterified Vegetable Oils, *J. Am. Oil Chem. Soc.,* 1984, 61, pp. 1638-1643.

[21] Vincente, G., Coteron, A., Martinez M. and Aracil, J.: Application of the Factorial Design of Experiments and Response Surface Methodology to Optimize Biodiesel Production, Industrial Crops and Products 1998, 8, pp. 29-35.

[22] Boocock, G.B., Konar, S.K., Mao, V., Lee, C. and Eiiligan, S. Fast Formation of High Purity Methyl Esters from Vegetable Oils, *J. Am. Oil Chem. Soc*. 1998, 75, pp. 1167-1172.
[23] Darnoko, D. and Cheryan, M. Kinetic of Palm Oil Transesterification in a Batch Reactor, *J. Am. Oil Chem. Soc*. 2000, 77, pp. 1263-1267.
[24] Encicar, J.M., Gonzalez, J.F., Rodriguez, J.J. and Tejedor, A. Biodiesel Fuels from Vegetable oil; Transesterification of *Cynara cardunculus* L. Oils with Ethanol, *Energy & Fuels* 2001.