

Selective Parameter Variables for Biodiesel Production from Acid Catalysed Transesterification of *Jatropha curcus* Oil

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Abstract : Worldwide, the concern for environment, reduction in dependence on oil import and energy security are found to be three driving forces for shaping the pattern of technology development and searching for alternate energy sources. Biodiesel extracted from vegetable oil is one such renewable alternative under consideration. The objective of this study was to investigate the effect of process variables on acid catalysed transesterification of *Jatropha curcus* oil. In acid catalysed transesterification, different variations of p-toluene sulphonic acid (1.0, 2.0 and 3.0 %) and reaction time (12, 16 and 18 h) were adopted with constant molar ratio of 24:1 methanol to oil at constant temperature of 60°C. The methyl ester yield varied widely in the range of 20 to 64 percent for the process variables adopted. It was found that acid catalysis can provide high conversion rates but much longer times are required than for alkaline catalysts.

Key words : Acid catalysed transesterification, *Jatropha curcus* and Biodiesel.

Introduction

India is looking for alternative fuel sources to reduce its dependence on foreign for oil. Biodiesel extracted from vegetable oil is one such renewable alternative under consideration.. The production of biodiesel would be cheap as it could be extracted from non-edible oil sources and from certain species that are common in many parts of India. *Jatropha curcus* (Linnaeus), a non-edible oil bearing and drought hardy shrub with ecological advantages belonging to Euphorbiaceae family was found to be the most appropriate renewable alternative source of biodiesel. The seeds of this perennial shrub are used to extract oil with 30 to 40 per cent recovery (Kandpal *et al.*, 1995).

Materials and Methods

The laboratory scale biodiesel reactor of one litre capacity was developed for the production of biodiesel from *Jatropha curcus* oil by acid catalysed transesterification method. The *Jatropha curcus* oil used for the study was obtained from M/S Renulakshmi Agro Industries (India) Ltd.,

Ganapathy, Coimbatore, Tamil Nadu, India. First the chemical properties like free fatty acid and acid value (AOCS Official Method Ca Sa-40) were determined for raw oil according to standard test methods.

Experimental conditions for acid catalysed transesterification :

The most commonly preferred acid catalysts were sulphuric, sulphonic and hydrochloric acids. Among these catalysts, sulphonic acid was more suitable compared to sulphuric acid and hydrochloric acids, since hydrochloric acid tends to form chlorohydrins and for kinetic studies, sulphonic acid was preferred compared to sulphuric acid. (Sridharan and Mathai, 1974).

Thus the chemicals used in the present study are methanol and p-toluene sulphonic acid ($\text{CH}_3\text{C}_6\text{H}_4\text{SO}_3\cdot\text{H}\cdot\text{H}_2\text{O}$) as an acid catalyst.

The *Jatropha curcus* oil of 200 g was taken in three-necked round bottom flask. Water-cooled condenser and thermometer with cork were

connected to the side openings on either side of the round bottom flask. The acid catalyst p-toluene sulphonic acid was added to methanol at room temperature. Mean while, the oil was warmed by placing the round bottom flask in the water bath maintained at the selected temperature mentioned above. The acid catalyst solution was added into the oil for vigorous mixing by means of mechanical stirrer fixed into the flask. The required temperature was maintained through out the reaction time and the reacted mixture was poured into the separating funnel. The mixture was allowed to separate and settle overnight by gravity settling into biodiesel on the top with the glycerol at the bottom. The next day, the glycerol was drained off from separating funnel leaving the biodiesel/ester at the top. The biodiesel was collected and water washed and weighed. The emulsion that comes out during washing and the glycerol were also measured on weight basis.

A statistical tool, Completely Randomised Design (AGRES) with single factor analysis was used to predict optimal conditions for maximum biodiesel production from acid-catalysed transesterification of *Jatropha curcus* oil. The parameters considered to find out higher biodiesel production were catalyst amount and reaction time. The significance was tested using Least Square Difference (LSD) both at 5 and 1 per cent levels. The sample size taken for the study was 200 g and the data were converted to 100 g basis for analysis. The experiment was replicated three times and average value of methyl ester yield was calculated and reported.

Results and Discussion

The preliminary studies to determine the optimum quantity of p-toluene sulphonic acid and reaction time required for transesterification of *Jatropha* oil were conducted by varying the concentration of sulphonic acid from 1.0 to 3.0 per cent and the reaction time from 12 to 18 h.

Effect of acid concentration and reaction time :

The different concentration of p-toluene sulphonic acid used in this study was 1.0, 2.0 and 3.0 per cent and the reaction time duration was 12, 16 and 18 h. The constant molar ratio of 24:1 of methanol to oil and constant temperature of 60°C was used in this study. The ester yield percentage increased with increase in sulphonic acid concentration and also increased with increase in reaction time.

The maximum ester yield of 64 per cent was obtained by using 3.0 per cent acid concentration at reaction time and temperature of 18 hours and 60°C respectively, for 24:1 molar ratio of methanol to oil.

The acid catalysed process did not give any satisfactory ester yield even at 18 h of reaction time with increased acid catalyst concentration compared to alkali-catalysed transesterification.

In the study of transesterification of 30:1 molar ratio of methanol to soybean oil using 1.0 per cent concentrated H_2SO_4 at temperature of 65°C, required 69 h to obtain more than 90 per cent conversion to methyl esters (Freedman *et al.*, 1984). He reported that alkali catalysed transesterification was much faster than acid catalysed transesterification.

The ester formation increased from 72.7 to 95.0 per cent as the H_2SO_4 catalyst was increased from 1 to 5 per cent (Canakei *et al.*, 1999). The alkaline catalyst had higher rate of conversion from triglycerides to methyl esters especially if the acidity index is higher than 0.5 percent (Alacantara *et al.*, 2000).

The acid catalysed transesterification is technically feasible but it is not economically feasible because it requires higher reaction time and more concentration of catalyst and methanol. This would increase to the processing cost and time consuming.

Table 1. Ana

Source
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Trt
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R
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RC
ERR

R - reaction ti
Best performi
Grand mean : 4

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18 h of reac
64.2 per ce
sulphonic ac
oil at 60°C o
time of 18 h

References

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Canakei, M
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The free fatty acid and acid value for *Jatropha curcus* oil was found to be 3.095 per cent and 6.16 mg of KOH/g of the sample. The acid catalyst would be much effective than the alkaline catalyst if the vegetable oil contains high free fatty acid (Freedman *et al.*, 1984 and Liu, 1994).

The maximum ester yield predicted by using Completely Randomised Design is given in the form of ANOVA in Table 1. The significance was tested by Least Square Difference (LSD) at both 5 percent and 1 percent levels.

Table 1. Analysis of variance for: Biodiesel yield from acid transesterification

Source	Degrees of freedom	Sum of squares	Mean sum of squares	F value	Prob
TOT	26	3940.259	151.548	122.332	
Trt	8	3917.960	489.745	395.330	0.851 NS
ERR	18	22.299	1.239	1.000	
R	2	2358.842	1179.421	952.048	0.000**
C	2	1437.409	728.705	588.222	0.000**
RC	4	101.709	25.427	20.525	0.006**
ERR	18	22.299	1.239	1.000	

R - reaction time; C - sulphonic acid concentration

Best performing treatment : R3C3

Grand mean : 43.1574 CV%:2.89 CD : 1.90931 (0.05); 2.61630 (0.01)

The following conclusions could be drawn from the study of acid catalysed transesterification of *Jatropha curcus* oil. Acid catalysed transesterification was found to be much slower than alkali catalysed and a higher molar ratio was required in acid catalysed transesterification. The completeness of methyl ester formation increased with increased catalyst amount. The acid catalysed process did not give any satisfactory yield even at 18 h of reaction time. The maximum ester yield of 64.2 per cent was obtained using 3.0 per cent sulphonic acid and 24:1 molar ratio of methanol to oil at 60°C of reaction temperature after the reaction time of 18 h.

References

Alacantara, R., J. Amores, L. Canoira, E. Fidalgo, M.J. Franco and A. Navarro (2000). Catalytic production of biodiesel from soybean oil, used frying oil and tallow, **Biomass and Bioenergy**: 515-527.

Canakei, M. and J. Van Gerpen. (1999). Biodiesel Production via Acid Catalysis, **American**

Society of Agricultural Engineers 42(5): 1203-1210.

Foidl, N., G. Foidl, M. Sanchez, M. Mittelbach and S. Hackel. (1996). *Jatropha curcas* L. as a source for the Production of Biofuel in Nicaragua, **Bioresource Technology 58**: 77-82.

Freedman, B., E.H. Pyrd and T.L. Mounts. (1984). Variables affecting the yields of fatty esters from transesterified vegetable oils, **JAOCS 61**: 1638-1643.

Kandpal, J.B. and Mira Madan. (1995). *Jatropha curcus* : a renewable source of energy for meeting future energy needs, **Renewable Energy 6(2)** : 159-160.

Liu, K. (1994). Preparation of fatty acid methyl esters for gas chromatographic analysis of lipids in biological materials, **JAOCS 71(11)**: 1179-1187.

Sridharan, R. and I.M. Mathai; (1974). Transesterification Reactions, **J. SCIENT. IND. Research 33**: 178-186.

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