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Synthesis of fatty acid methyl ester from rice bran oil

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Abstract
Increase in urbanization, standard of living of people and expanding population are the key factors which contribute to the growth in energy demand. India ranks sixth in the world in terms of energy demand. India's demand for diesel fuel is increasing rapidly and much of the increase has to be met with expensive oil import. The known world wide reserves of petroleum are predicted to last for about 40 years, hence future availability of petroleum is uncertain. Alternative fuels have to be considered in order to undertake energy security and import substitution for diesel crude imports by 5% in 2015 and 10% in 2020. In this circumstances non-edible oil source play a vital role to meet the diesel requirement. In the present study, biodiesel has been prepared from Rice Bran oil by transesterification method.

Keywords: Biodiesel, Transesterification, Kinematic viscosity, Cetane number.

INTRODUCTION

Bio-fuels produced from renewable energy sources are gaining importance in the light of rising fossil fuel prices, depleting oils reserves and increasing 'green house effect'. Increasing threat to environment from exhaust emissions, global warming and threat of supply instabilities has led to a growing concern for it throught the world, more so in the petroleum importing countries like India (Gupta *et al.,* 2007). The global annual requirement of petroleum is approximately 17000 MT out of which, the estimated requirement is 120MT in India. Our domestic production of crude oil and natural gas will remain about 34MT during 2006/2007. Mostly, biodiesel is prepared from oils like Soybean, Rapeseed, Sunflower, Safflower etc throught the world (Lang *et al*., 2001). These oils are essentially edible in nature. Biodiesel produced from vegetable oils (Srivastava *et al*., 2000) forms a promising solution to the threat caused to the environment. Attempts have been made for producing bio diesel with non-edible oils such as jatropha and Pongamia especially in India (Kaul *et al.,* 2003). *Rice bran*, the most valuable by product of rice milling industry produce about 7,00,000 tons of oil per annum (Annual report-Ministry of Science and Technology, 2007), which forms a promising feedstock for transesterification process. The present work reports the transesterification of rice bran oil to fatty acid methyl ester and its fuel properties.

MATERIALS AND METHODS
Collection of Sample

Rice bran oil was collected from the Vaigai industry, Madurai. Determination of Acid Value Free fatty acid in the oil was estimated as detailed by Sadasivam *et al*. (1996). The acid value is defined as the mg of KOH necessary to neutralize the free fatty acids present in 1g of lipid.

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Fatty Acid Methyl Ester Preparation by Acid and Alkali Catalyzed Transesterification (Two-Stage Method)

In order to avoid the problem of saponification, the two stage method was used for the isolation of biodiesel from *rice bran oil*.

Oil sample was poured in to a round bottomed flask equipped with a reflux condenser and heated to the reaction temperature. 1% (v/v) H_2SO_4 in methanol (8% v/v) was added to the flask. After the reaction, the mixture was allowed to settle in a separating funnel over night.

To the pretreated oil 0.35% (w/v) of sodium hydroxide in 12% (v/v) methanol was added to the reaction flask. The methanol to oil molar ratio was 6:1. The reaction was allowed to proceed until completion. The lower glycerol layer was drawn off. The reaction time depended on the type of feedstock used (Fig.1).

Fuel properties measurement

The physical and chemical properties of Rice bran oil were measured and tabulated in Table 1. Acid value was determined by the titremetric method of Pearson (1970). A.O.C.S. Ca.14 – 56 methods (Firestone, 1997) was used to determine the total glycerol content of test sample. Free glycerol content of sample was determined using A.O.C.S Ca 14 – 56 method (Firestone, 1997). Ash content is measured as detailed in Indian Standard Method: 1448 (1992). The kinematic viscosity of the sample was determined by Redwood viscometer using American Standard Test Methods (ASTM) 445-88 (1994c). The flash point, by Pensky Martens closed-cup method (Indian Standard Method: 1448, 1992).The cloud point, by (ASTM D 2500, 1994h). The determination of cetane index was carried out in the present study following the method, as detailed by Krishnangkura (1986). Heat of combustion of FAME was estimated as detailed by Krishnangkura (1991).

RESULT AND DISCUSSION

Transesterification has proven to be the most significant step towards making biodiesel available as alternative to petroleum derived diesel fuel (Meka *et al.,* 2007). A catalyst is used to improve the reaction rate and yield. Enzymes, alkalis or acids can catalyze the reaction, ie lipases, NaOH and sulphuric acid, respectively.

High FFA (i.e., high acid value) in the feed stock would result in soap formation when alkali chemicals are used as catalyst; hence the FFA has to be neutralized (Vicente *et al.,* 2004). Hence, it has been proposed that a combined process of acid catalyzed pretreatment prior to alkali catalyzed transesterification is followed to overcome the problem (Gerpen and Knothe, 2005; Wang *et al.,* 2007). First step in this process is to esterify the FFA by acid catalyst and the next step is the transesterification of the preheated product using base catalyst.

The rice bran oil used in this present study were tested for FFA content and showed 3.9% respectively. Due to the presence of high FFA content (above 3%) in the feed stock direct conversion of the above oils via alkaline transesterification was not possible. Hence, a combined process has been suggested as an effective and efficient method to convert high FFA feed stock to biodiesel. The conversion efficiency of *rice bran* oil was observed as 80.4% with a catalyst concentration of 0.9 wt% of oil (Fig 2). Similar studies have been reported in Yellow Grease (Canacki and Gerpen, 2001); *Madhuca indica* (Ghadge and Raheman, 2005); Rice bran oil (Zullaikah *et al.,* 2005).

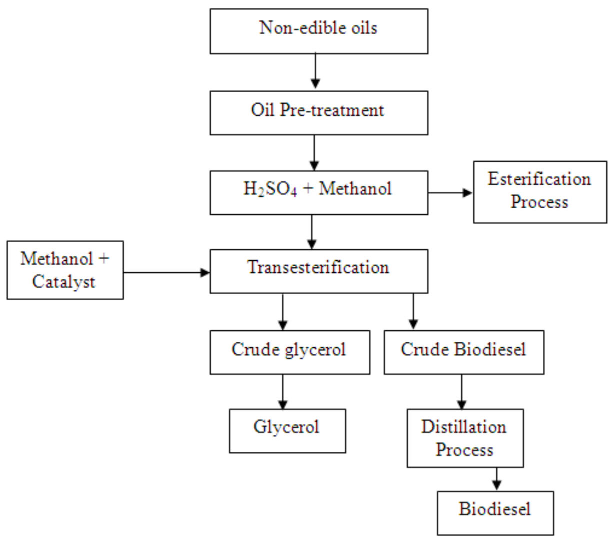

Fig 1. Acid and Alkali Catalyzed Transesterification (Two Step Process)

Table 1. Fuel Properties of Rice bran Methyl Ester

Property	Units	Sample value	ASTM Specification	Standard diesel value
Acid value	mg KOH/gm	0.44± 0.183	0.50max	NA
Total glycerol	%mass	0.321 ± 0.001	0.240 max.	NA
Free glycerol	%mass	0.010 ± 0.0005	0.020 max.	NA
Sulphated ash	%mass	0.01 ± 0	0.020 max.	0.02
Kinematic viscosity	mm²/s (cst)	4.07±0.03	1.92-6.0	2-4
Flash point	ºC	100.75 ± 0.5	130 ºC	65-88 ºC
Cloud point	ºC	18± 1.15	4-16 ºC	-10 to -15 ºC
Cetane index		52 ± 2.06	47 min	45-50
Heat of combustion	k-cal / mol	2971 ± 33.05	NA	NA

Fuel properties of biodiesel play a significant role in the combustion process. Acid value is a measure of the amount of free acids present in a given amount of oil sample. Acid value of rice bran oil was high ranging 7.8mg KOH/g but by using two-stage transesterification process the acid value of rice bran methyl ester was reduced substantially to 0.44 mgKOH/g and it was within the limit of the ASTM standard. Free glycerol is a by-product of the transesterification process and is separated from the ester. Total glycerol content (the sum of free and bound glycerol) is one of the main parameters indicating the final quality of biodiesel. ASTM has specified the standard limit of free and total glycerol in the biodiesel ranging between 0.005 to 0.05 mass% and 0.05 to 0.5 mass% respectively. The amount of total glycerol determined in the rice bran biodiesel was 0.321% and the free glycerol content is found to be 0.010%.The present results showed that free glycerol and total glycerol content was below the detection range of the test method. This result indicates efficient separation and sufficient methyl ester washing. Earlier studies in *Gossypium hirsutum* methyl ester showed a free glycerin value of 0.01 wt% and total glycerin value of 0.12 wt % (Qian and Yun, 2009).

The ash content determined in the biodiesel sample is 0.01% which is within the maximum limit specified by ASTM and diesel value. Kinematic viscosity is an important parameter regarding fuel atomization as well as fuel distribution (Mittelbach, 1988). The acceptable and prescribed range of viscosity at 40ºC for biodiesel by ASTM standard D- 6751 is 1.9 – 6.0 mm²/s. Rice bran methyl ester had viscosity of 4.08 mm²/s.

The flash point is the temperature at which the fuel will ignite when exposed to an ignition source. The flash point of Rice bran biodiesel was 100.75°C. Cetane number is the ability of fuel to ignite quickly after being injected. Higher its value the better the ignition quality of fuel (Azam *et al.,* 2005). Cetane number increases from 47.9 to 75.6 when the number of carbons in the fatty acids in biodiesel increases. A comparative study carried out by Azam *et al.* (2005) on the cetane number of FAME in 75 species showed that 42 species have the cetane number value higher than value higher than 51.In this experiment; the cetane number of rice bran methyl ester was determined to be 52.

CONCLUSION

The aim of this study was to evaluate rice bran oil as a potential raw material for biodiesel production. The biodiesel sample prepared in the present study showed better results and not deviating from ASTM standard except the free and total glycerol content which can be rectified by further suitable methods. Use of the biodiesel as a partial diesel substitute can boost the farm economy, reduce uncertainty of fuel availability and make farmers of fuel availability and make farmers more self-reliant. Also, this help in controlling air pollution to a great extent.

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