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Optimization of biodiesel production process from low cost high FFA Polanga (Calophyllum Inophyllum L.) oil.

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ABSTRACT: Polanga biodiesel is an alternative fuel consisting of alkaline monoesters made from a low cost non edible vegetable oil. As it contains large amount of free fatty acids (FFA), it cannot be converted into biodiesel using only alkaline catalyst. In this paper, a catalyzed pretreatment technique has been discussed to reduce the FFA before trans esterifying the oil. Varying parameter such as time, molar-ratio, amount of catalyst, alcohol etc. has been studied. On the basis of study best strategy has been determined to reduce FFA. With this strategy FFA of the Polanga oil was reduced to less than 2% which was ideal for trans esterification to produce biodiesel with appropriate physico-chemical properties and maximum yield.

Keywords: Alternate fuel, Biodiesel, Pretreatment, Polanga

Introduction

As diesel engine fuel is a great subject for research & development from long, many researchers have been working on different vegetable oils to produce diesel like engine fuel. This is also becoming important due to depleting oil reserves and environmental consciousness. Many studies have been done on environmental friendly and wide availability of alternative fuels. Biodiesel being a biodegradable, non-toxic and renewable fuel can be used as alternative fuel for diesel engines.

The biodiesel stands for oil derived from vegetables or animal fat which consist of long chain alkyl esters. It is typically made by trans esterification of the triglyceride with methanol and a basic catalyst. Other alcohols are also used to make esters e.g. ethyl, propyl and butyl esters. Figure 1 shows the structure of triglycerides.

Figure: 1

CH_2OOR				CH_2OOH
|				|
CH_2OOR	+ $3CH_3OH$	→	$3CH_3OOCR$ +	CH_2OOH
|				|
CH_2OOR				CH_2OOH
Triglyceride	Methanol		Methyl ester	Glycerol

"Rudolf diesel" demonstrated his first diesel engine with vegetable oil at the world exhibition at Paris in 1990. He used peanut oil as fuel. So using vegetable oil is not a new concept but the readily available inexpensive petroleum fuels provided little seriousness on use of vegetable oils. With crisis of petroleum fuels many researchers have started experimenting on alternative fuels seriously. Now days several researches is being done on different vegetable oils to produce diesel like fuel which many be used as possible substitute.

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Many researchers have worked and concluded that vegetable oil based biodiesel hold good as alternate fuel for diesel engine. This also holds good in minimizing environmental degradation. The viscosity and low volatility of SVO can be minimized by trans esterification. The problem of engine deposit, piston ring sticking and injector coking can also be reduced or eliminated by using biodiesel. In this study the biodiesel produced by trans esterification is found close to diesel fuel.

The properties of the biodiesel depend on the feed stock and the transesterification process. This is also proved to be non-toxic, biodegradable and renewable fuel for diesel engine. Use of biodiesel also reduces emissions as it has higher cetane no than diesel (mittle back at al. 1992, peters on et al 1992). Also it has no aromatics, almost no sulfur, and contains more oxygen. As a result CO, HC and particulate matter (PM) gets reduced.

In the earlier articles, biodiesel from edible oil is not economical. Polanga oil being non-edible is a low-cost Vegetable oil. But the FFA is very high in a range of 20-22%. The excess amount of FFA level is undesirable as it results in loss of feed stock as well as deleterious effect of soap on glycerin separation. Formation of stable emulsions prevents separation of biodiesel from glycerin during processing. Acid catalyst is too slow for converting triglycerides to biodiesel (canakci and an Gerpen, 1999). An acid-catalyzed pretreatment step is required to convert FFAs to esters followed by an alkali-catalyzed step. One patent literature has shown that this has better effect on making biodiesel from high FFA (ka-wahara et al 1979; lepper et. Al 1986).

As per the literature, for alkaline-catalyzed trans esterification, the oil should not have more than 1-2% FFA.

Production of Polanga oil

Polanga oil, Calophyllum Inophyllum L., falls in a group of coastal family and now-a-days planted throughout the tropics. Its origin is uncertain but believed to be indigenous to India, (P.K.Sahoo et al, 2007), Malaysia , Indonesia and the Philippines. It grows in humid tropics with 1000-5000 mm rain at altitude from 0-200m. It grows on Sandy beaches and to a river margins. It is tolerant of wind and Salt which makes it suitable for dune stabilization. It is a Medium sized tree, ranging 25-35m tall with diameter up to 150cm. its fruit reported to be little toxic. The tree flower throughout the year in Indian condition.

The oil from Polanga seed is used here as feed stock for biodiesel production. The properties of the Polanga oil along with other common non-edible oil are given in the table 1.

Table 1. Properties of Polanga seed oil in comparison with other oils

Product	Fatty acid by weight (%)					Acid Value (mg KOH/gm)	Reference
	Palmitic acid	Stearic acid	Oleic acid	Linoleic acid	Linolenic acid		
Polanga	12.01	12.95	34.09	38.26	0.3	40-44	Sahoo, 2007
Jatropha	16.9	6.5	43.5	34.4	0.80	3.8	Sahoo, 2007
Karanja	11.65	7.5	51.59	16.49	2.65	5.06	Sahoo, 2007

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Rapeseed	3.49	0.85	64.40	22.30	8.23	4.34	Goering, 1982
Tallow	3-6	20-25	37-43	2-3	-	47-43	Canakci, 2001

The FFA content of the Polanga oil is comparatively high which can be reduced by pretreatment of the oil with acid catalyst. The FFA in this oil found to be 20%. The acid value of 40 mg KOH/g is determined by titrimetric method.

Chemical composition

Polanga oil is a triglyceride of various fatty acids consisting of approximately 12% Palmitic acid, 38.26% Linoleic acid, 34% Oleic acid, and 12.95% stearic acid (P.K. Sahoo et al, 2009). The very high viscosity of Polanga oil is to be addressed while making the biodiesel.

It has very high proportion of saturated fatty acids. The main reason of high FFA is high Oleic (HO), Linoleic and Linolenic acids.

Physical Properties

This provides viscous, non–drying, and odorless oil. The refined one is initially dark green in color and unpleasant taste. Later in processed state it becomes clear. It is soluble in alcohol, very high viscosity and high hygroscopicity. High cetane number and high calorific value is an advantage of being used as feed stock to biodiesel production. The main disadvantage is the high viscosity which may cause problem in extraction and injection. Another problem is the water content of the oil which may cause filtration and corrosion problems. It has good flash point which is 221^0C, far above the diesel fuel. The cloud point and pour point is higher than that of diesel fuel. The remaining proprieties are fairly close to diesel fuel. The different physical properties in comparison with other are given in table 2.

Table 2. Physical properties Polanga along with other non-edible oils.

Product	Kinematic viscosity (mm^2/sec)	Heating Value (MJ/ kg)	Flash point (^{0}C)	Density (Kg/l)
Polanga	27.40	39.5	221	0.941
Jatropha	52.76	38.2	210	0.933
Karanja	27.84	34.0	205	0.912
Rapeseed	37.00	39.7	246	0.912
Tallow	39.6	37.5	271	0.946

Experimental Procedure

As presence of moisture and organic materials pose difficulty in biodiesel production they have been removed by heating the oil for 1 hour at around 70^0C and chemical pretreatment. Then we experimented two steps to remove high FFA level to make it suitable for transsterification and biodiesel production.

1. In first technique the high FFA level of 20% has been reduce to 1-2% with acid-catalyst
2. An optimized alkali-based transesterification was used to produce diesel like biodiesel.

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The experiments were conducted in a 1 liter capacity biodiesel reactor (Figure 2). This setup had a reactor with controlled temp heating, controller stirring and cooling facility. This had a water cooled condenser to send back the vaporized methanol to the reaction mixture. The same reactor was used for both pretreatment and transesterification process.

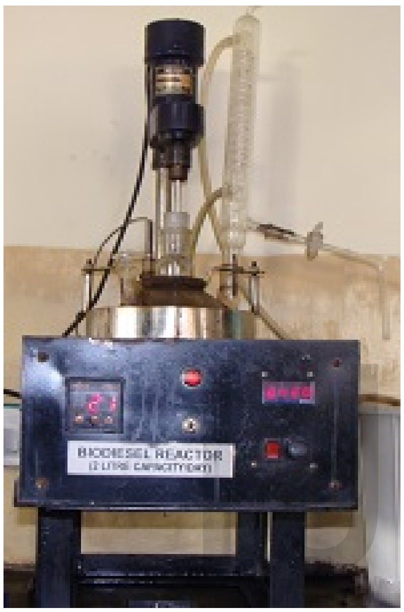

Figure 2. Biodiesel Reactor 1 liter capacity per day

Pretreatment process

As suggested by published papers the amount of FFA of an oil must be around 1-2% for alkali-based catalysis. The first target for pretreatment was to reduce it at this level to have satisfactory result.

The pretreatment was carried out with following four variables

1. Catalyst amount
2. Volume of methanol
3. Temperature
4. Time

The catalyst used for the pretreatment was H_2SO_4 in amount ranging 5%, 10%, 20% and 25% of FFA. As FFA is found to be 20% I.e. 200 ml for 1 liter oil the H_2SO_4 taken was 10 ml, 20 ml, 30ml, 40 ml and 50 ml.

Table.3 Shows the FFA reduction when quantity of H_2SO_4 is varied keeping methanol 105 ml and time of reaction 1 hr. at 60^0C.

Table 3. Reduction of FFA with increase in Acid Catalyst (H_2SO_4)

H_2SO_4	FFA (mg KOH)	FFA (%)
5%	18mg KoH	9%
10%	10 mg KoH	5%
15%	6 mg KoH	3%
20%	3.4 mg KoH	1.7%

After 1 hr. reaction the mixture was allowed to settle (Figure 4) and the separated water and methanol mixture at the bottom was taken out. After the mixture is separated the acid values were recorded and found to be reducing with increase in % of H_2SO_4 (acid-catalyst). The target of 2% FFA was reached with 20% of H_2SO_4.

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Figure 3. Feed stock in two layers after pretreatment

Figure 5. Biodiesel after transesterification

Transesterification

The alkali-based transesterification was conducted after reducing FFA less than 2% by pretreatment. The reaction was again conducted in the same reactor with different amount of methanol and KOH (alkali catalyst). The amount of methanol varied from 90 ml to 150 ml per liter of oil the KOH also varied from 10gm to 16gm per liter of vegetable oil. It has been found experimentally that 120 ml of methanol with 12 gm of KOH at 60^{0}C gave maximum yield 90% of the biodiesel with good viscosity level near to diesel (Figure 5).

The fuel properties of Polanga based biodiesel in comparison with other biodiesel and petro diesel is shown in table 3.

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Table 3. Properties of Polanga oil methyl ester along with other biodiesel and Petro- Diesel.

Biodiesel	Density (kg/l)	Calorific value (MJ/kg)	Viscosity (cSt)	Flash point (^{0}C)	Cloud point (^{0}C)	Pour point (^{0}C)
Polanga	0.872	40.8	4.1	129	12.9	3.9
Jatropha	0.873	42.67	4.23	148	10.2	4.8
Karanja	0.883	42.13	4.37	163	14.6	5.1
Rapeseed	0.882	41.55	4.2	80	13.2	3.8
DieselHSD	0.823	44.00	3.21	75	6.3	-3.3

Conclusions

This study was carried out to produce biodiesel from non-edible high FFA Polanga oil. With traditional transesterification it was not possible to convert it into biodiesel as Alkaline catalysts form soap when they react with the FFAs. Due to soap formation the separation of glycerine and ester becomes difficult. One more step has been introduced to reduce the FFA before transesterification. In this step the Feedstock is reacted with acid catalyst H_2SO_4 for 1 Hr until the FFA level reduced below 2%., allowing the subsequent use of alkaline catalysts to convert the triglycerides. The amount of methanol molar ratio, acid catalyst, time of reaction and temp were studied in order to reduce high FFA level of 20 % of Polanga oil to less than 2%. This showed that the reduction below 2% can be done by pretreatment. Two step pretreatment also studied but that also gave same result in terms of FFA reduction. So one step pretreatment was considered as economical and less time consuming. It can be concluded from this part of study that

1.The pretreatment reaction using acid based catalyst reaction reduced the acid value of the high FFA low cost Polanga feedstock to less than 2% by one step pretreatment. With one step acid catalyzed pretreatment, the transesterification of polanga oil to produce methyl ester could be completed in much less time and the yield also increased. The amount of Methanol used also got reduced compared to other authors showed in their study.

2. The study also shows that the increasing amount of acid in the pretreatment process decrease the FFA % to the required level of less than 2% to be suitable for transesterification.

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