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An Initial Investigation on Production of Biodiesel from Ayurvedic Waste Oil

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ABSTRACT: Biodiesel is considered as the best alternative peterodiesel for internal combustion engines. The simple and easy transesterification process of natural oils and fats is a widely accepted method for the production of biodiesel. But the main concern in usage of biodiesel is the economic viability of producing biodiesel where the cost of source plays a major role. The work proposes the idea of using the oil discharged after the treatment purpose from an ayurvedic hospital or a herbal treatment centre, as a biodiesel source. Preliminary experiments were performed to investigate the suitability of ayurvedic waste oil for biodiesel production. A two stage process involving acid esterification followed by alkali transesterification was adopted for the study. Biodiesel yield of 79% was achieved by the optimisation of alkali catalysed transesterification process. Ayurvedic waste oil was proved to be a potential feed stock for biodiesel preparation. The current work is an initial study towards the complete optimisation of biodiesel production from ayurvedic waste oil. The biodiesel produced from ayurvedic waste oil is a novel substitute for petroleum-based diesel and a green solution to energy and environmental hurdles.

KEYWORDS: Biodiesel, ayurvedic waste oil, acid esterification, alkali transesterification, optimisation

I. INTRODUCTION

The era of fossil fuel is gradually coming to an end, where oil and natural gas will be depleted first, followed eventually by depletion of coal. Active research programs have been considered worldwide to reduce reliance on fossil fuels by the use of bio-based alternative and sustainable fuel sources. One of the liquid fuels which has attracted most attention at present is biodiesel [2]. Biodiesel is mono-alkyl esters of long chain fatty acids of vegetable oils or animal fats, widely Fatty Acid Methyl Esters (FAME) derived from triglycerides by transesterification with methanol [3]. Biodiesel is considered as an alternative fuel for internal combustion engines and it emits far less regulated pollutants than the standard diesel fuel. The highlights of biodiesel are the following; simple and easy production, fuel properties similar to diesel fuel; can be used directly into compression ignition engine without any modification of engine, high energy yield; 280% greater than petroleum diesel, higher combustion efficiency and cetane number, renewable, biodegradable, environmentally safe and non-toxic; low sulphur and aromatic content, better quality exhaust gas emission; does not contribute to a rise in the level of carbon dioxide in the atmosphere and consequently to the green house effect, non-flammable and non-explosive with superior flash point of 423K for biodiesel as compared to 337K for petrodiesel, easy portability, ready availability, and yields value added by-products like seed cake and glycerine [1, 2].

However, one main concern in further usage of biodiesel is the economic viability of producing biodiesel [1]. The selection of feed stock material critically affects this matter. Use of edible oils to produce biodiesel is not feasible in view of a big gap in demand and supply of such oils. A suitable source to produce biodiesel should not be competent with other applications that rise prices. As much as possible the biodiesel source should fulfil two requirements: low production costs and large production scale. Refined oils have high production costs, but low production scale; on the other side, non-edible seeds, algae and sewerage have low production costs and are more available than refined or recycled oils [3]. There comes the idea of using expelled oil from ayurvedic hospitals and massage centres for the production of biodiesel. Since the herbal treatments are getting more and more popularity nowadays, the concerned

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firms are booming very rapidly. Very large amount of herbal oil is being used every day in such places. Actually the oil used for the treatment or massage of a person becomes a waste product after its one time use. The quite large amount of this discharged oil is a potentially problematic waste stream which requires to be properly managed. Properties of degraded used waste oil after it gets into sewage system are conducive to corrosion of metal and concrete elements. It also affects installations in waste water treatment plants. Thus, it adds to the cost of treating effluent and causes possible contamination of the water and land resources. These waste oils fulfil the requirements of biodiesel source as they are cheap and persistently available in large quantity. Therefore, the use of this kind of oil for the production of biodiesel would be a good option to handle waste oil disposal problems too. And the biodiesel thus produced can be used back for fulfilling energy requirements.

II. MATERIALS

About 5 litres of ayurvedic waste oil driven out from Oushadhi Panchakarma Hospital and Research Centre (a Kerala Government undertaking Ayurvedic Pharmaceutical Corporation) Thrissur, Kerala, India, was collected for the study. The oil was filtrated using muslin cloth to remove solid impurities. The major chemicals used include sulphuric acid (H_2SO_4), sodium hydroxide (NaOH) and methanol (CH_3OH).

III. PROPERTIES OF AYURVEDIC WASTE OIL

The crude oil properties such as density, kinematic viscosity, acid value, iodine value and saponification number were estimated according to the IUPAC procedures. Determination of density and kinematic viscosity were carried out using specific gravity bottle and Brookfield viscometer respectively. The acid value, saponification value and iodine value were determined by titrimetry [6]. The quality of raw oil was analysed and expressed in terms of these physico-chemical properties. Table 1 gives the properties of ayurvedic waste oil. The acid value and free fatty acid (FFA) content for the ayurvedic waste oil were found to be 6.172 mg KOH/g oil and 3.102 wt.% respectively. Since the FFA content of feed stock was beyond 3 wt.%, a two stage process was suggested.

Table 1: Properties of ayurvedic waste oil

Property	**Value**
Density (kg/m^3)	936.1
Kinematic viscosity (mm^2/s)	72.16
Acid value (mg KOH/g oil)	6.172
Free fatty acid (FFA) content (wt.%)	3.102
Saponification value (mg KOH/g oil)	162.6
Iodine value (g I_2/100g)	26.649

IV. TWO STAGE TRANSESTERIFICATION PROCESS

The problem with substituting triglycerides for diesel fuel is mostly associated with high viscosity, low volatility and polyunsaturated characters. Therefore, the direct use of vegetable oils and/or oil blends is generally considered to be unsatisfactory and impractical for both direct injection and indirect type diesel engines. The conversion of oils into biodiesel by transesterification is an effective way to overcome all the problems associated with the combustion of oils in engines. Transesterification (also called alcoholysis) is the reaction of a fat or oil with an alcohol to form esters and glycerol. A catalyst is usually used to improve the reaction rate and yield. Transesterification reaction is an equilibrium reaction. Excess alcohol is used to shift the equilibrium toward the product because of reversible nature of reaction. Alcohols employed in the transesterification are generally short chain alcohols such as methanol, ethanol, propanol, and butanol. Ethanol is good in transesterification reaction because it is derived from agricultural products, is renewable and biologically less objectionable in the environment. However, methanol is preferred because of its low cost and its physical and chemical advantages being polar shortest chain alcohol. Transesterification consist of a number of

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consecutive, reversible reactions. The triglycerides are converted step wise to diglycerides, monoglycerides and finally to glycerol. A mole of ester liberated at each step [2].

Biodiesel production normally incorporates use of conventional catalysts like acids and alkali catalysts. Choice of acid and alkali catalysts depends on the acid value (or FFA content: nonesterified fatty acids released by the hydrolysis of triglycerides) in the raw vegetable oil. Alkali catalysis is performed only when the FFA is low (< 3%). NaOH or KOH is used as the catalyst for this. The alkali transesterification occurs at relatively low temperature and has short reaction time and high performance. But a single step alkali transesterification is not sufficient for oils with FFA content higher than 3% because biodiesel yield is dropped off by the undesired soap formation reaction between the alkaline catalyst and FFA. Consequently, an acid esterification which turns FFAs to biodiesel using H_2SO_4 as catalyst, has to be performed prior to alkali transesterification. Trans-methylation occurs approximately 4000 times faster in the presence of an alkali catalyst than those catalysed by the same amount of acidic catalyst [3]

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A. Equipment Setup

For both the transesterification processes a hot plate magnetic stirrer device was used to provide continuous heating and stirring. An agitation speed of about 200 rpm was maintained for all reactions.

B. First Stage: Acid Catalysed Esterification

The first stage is considered as the pretreatment process. The entire oil was preheated first. Then acid esterification was carried out for the whole feed stock under a recommended condition of 0.50 w/w_{oil} methanol to oil ratio and1% w/w_{oil} concentrated H_2SO_4 at 60^oC for 60 minutes with constant mixing [5].

When the first stage of the acid esterification was complete, the reaction mixture was transferred to a separating funnel and allowed to settle for about 90 minutes [4]. The bottom layer was taken for the alkali transesterification. The top layer containing excess methanol, acid and other impurities was discarded.

C. Second Stage: Alkali Catalysed Transesterification

The bottom layer product from the first stage containing oil with FFA less than 3% was subjected to alkali transesterification using NaOH as the base catalyst. Here the catalyst has to be dissolved in methanol prior to the addition of oil to avoid moisture absorption by the catalyst. The sodium methoxide prepared was added to the preheated product of the acid esterification. The mixture was continuously stirred at constant speed keeping temperature constant at 65^oC. The temperature was not allowed to rise above this specified limit to avoid the methanol loss.

Afterwards the mixture was transferred to a separating funnel and kept undisturbed for a settling period of 60 minutes. On settling, the biodiesel forms the top layer and glycerine along with any impurities move to the bottom layer.

D. Post Treatment Process

The bottom layer was removed and biodiesel was collected for post treatment process. It was washed with distilled water at 60^oC to remove the presence of any excess methanol, soap and impurities like catalyst. The mixture was allowed to settle under gravity. The settled layer of mixture with impurities was drained out. Hot water wash was repeated two more times. The water content was removed by addition of Na_2SO_4 anhydrous. The Na_2SO_4 salt and any other remaining impurities present in biodiesel were removed by filtration through muslin cloth. Biodiesel was then heated to 110°C for 10 minutes to remove any moisture present in it. Finally, the finished biodiesel having a golden yellow colour was obtained.

V. EFFECT OF PARAMETERS ON ALKALI CATALYSED TRANSESTERIFICATION

In order to study the effect of various process variables such as methanol to oil ratio, reaction time and catalyst concentration on biodiesel yield, three consecutive sets of experiments were conducted in the second stage of transesterification process. The conversion efficiency resulting from different experiments were measured and its variation with respect to different parameters was studied to establish the optimum conditions. Conversion efficiency refers to the percentage of yield of biodiesel from the oil by transesterification. Reaction temperature of 65^oC was used

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for all experiments. In each set of experiments, the parameter to be analysed was varied while keeping the other parameters same for all the experiments of that set. The optimum value of the parameter (that resulted the maximum biodiesel yield) in the previous set of experiments was used for the succeeding sets of experiments.

A. Methanol to Oil ratio

The first parameter analysed was methanol to oil ratio. Biodiesel production process is incomplete when the methanol amount is less than the optimal value [2]. Experiments were conducted with four different methanol to oil ratio (0.30, 0.40, 0.50 and 0.60 w/w_{oil}), keeping other parameters constant (catalyst concentration of 1.5% w/w_{oil} NaOH and reaction time of 90 minutes). The plot of biodiesel yield to methanol to oil ratio is given in Figure 1. Maximum biodiesel yield was obtained at 0.4 w/w_{oil} methanol to oil ratio. Figure 1 makes clear that operation beyond the optimal value of methanol to oil ratio does not increase the ester yield.

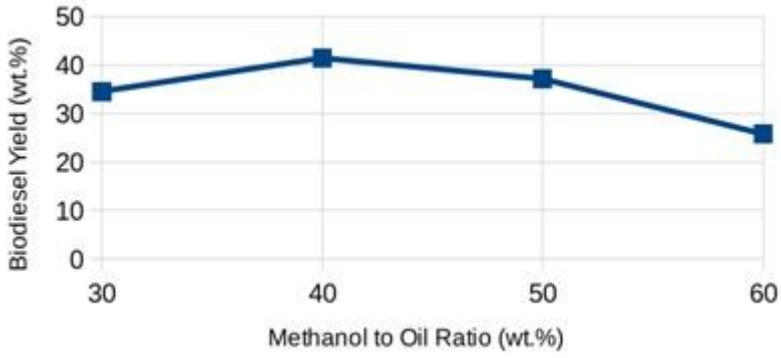

Figure 1: Effect of methanol to oil ratio on biodiesel yield with catalyst concentration of 1.5% w/w_{oil} NaOH, reaction time of 90 minutes and reaction temperature of 65°C

B. Reaction Time

Sufficient reaction time should be allowed to ensure complete conversion of triglycerides into esters. However, excess reaction time did not promote the conversion but favours the reverse reaction of transesterification which resulted in a reduction in the ester yield [2]. The minimum reaction time required for maximum conversion efficiency was investigated by varying reaction time from 30 to 90 minutes. The methanol to oil ratio of 0.4 w/w_{oil}, found out as optimum earlier, and 1.5% w/w_{oil} NaOH were used in all cases. Figure 2 shows the effect of reaction time on biodiesel yield. 45 minutes was found to be sufficient for completing the alkali transesterification as the biodiesel yield started declining thereafter.

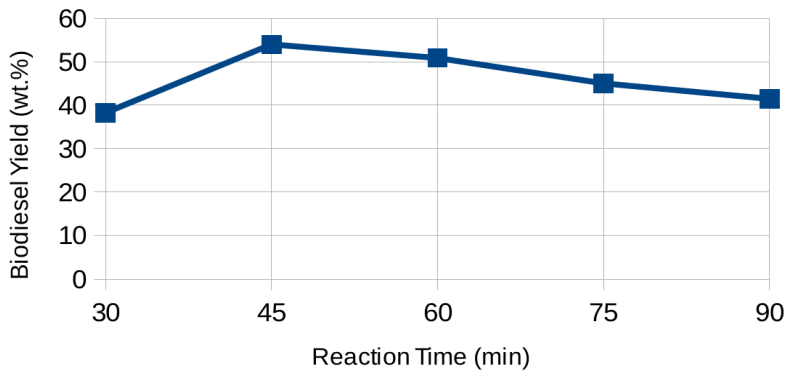

Figure 2: Effect of reaction time on biodiesel yield with methanol to oil ratio of 0.4 w/w_{oil}, catalyst concentration of 1.5% w/w_{oil} NaOH and reaction temperature of 65°C

C. Catalyst Concentration

Addition of excess catalyst reduces the conversion efficiency due to the saponification reaction that leads to the gel formation which increases viscosity [2]. For analysing the effect of catalyst concentration on biodiesel yield, alkali

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transesterification of ayurvedic waste oil was carried out with NaOH concentration of 0.5–1.5% w/w_{oil} in steps of 0.25%. The methanol to oil ratio of 0.4 w/w_{oil} and reaction time of 45 minutes were used. The yield of biodiesel versus catalyst concentration is shown in Figure 3. Maximum biodiesel yield occurred at a catalyst concentration of 0.75% w/w_{oil} and further increase in catalyst concentration lowered the biodiesel yield.

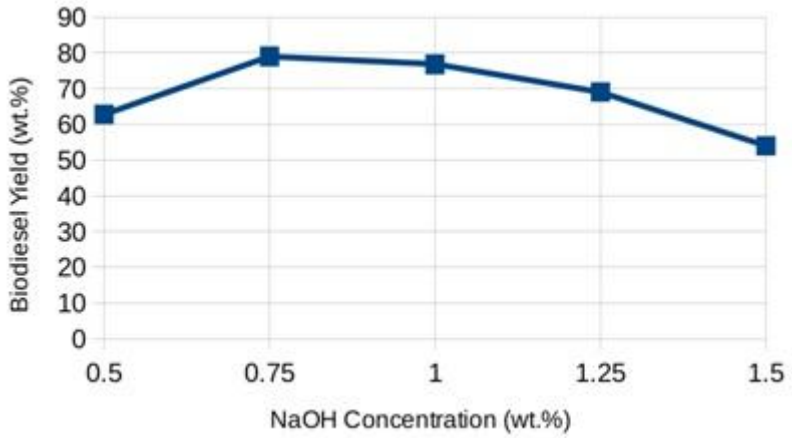

Figure 3: Effect of catalyst concentration on biodiesel yield with methanol to oil ratio of 0.4 w/w_{oil}, reaction time of 45 minutes and reaction temperature of 65°C

D. Optimum Parameter Values

The optimum conditions were established based on the observations with varying conditions. Maximum biodiesel yield of 79% was obtained with a production condition of methanol to oil ratio of 0.40 w/w_{oil}, catalyst concentration of 0.75% w/w_{oil} NaOH, reaction time of 45 minutes, reaction temperature of 65°C and agitation rate of 200 rpm.

VI. QUALITY ASSESSMENT OF PRODUCED BIODIESEL

Biodiesel properties such as density, kinematic viscosity, acid value, iodine value, saponification number, cetane number and flash point were estimated as per IUPAC procedures. Determination of density, kinematic viscosity and flash point were carried out using specific gravity bottle, Ostwald viscometer and Pensky-Martens closed cup flash tester respectively. The acid value, saponification value and iodine value were determined by titrimetry [6]. The cetane number was determined from the following simple empirical correlation by using the estimated iodine value (IV) and saponification number (SN) [7].

$$CN = 46.3+(5458/SN)-(0.225IV) \quad (1)$$

The estimated properties were then compared with standards specified for biodiesel and petroleum diesel. The observations of quality assessment of biodiesel produced from ayurvedic waste oil are summarized in Table 2.

Table 2: Quality assessment of produced biodiesel

Property	**Biodiesel**	**ASTM D6751 Standards for Biodiesel**	**ASTM D975 Standards for Diesel**
Density (kg/m^3)	874.18	860-900	850
Kinematic viscosity (mm^2/s)	5.94	1.9-6.0	1.9-4.1
Acid value (mg KOH/g)	1.57	<0.5	-
Saponification no. (mg KOH/g)	224.44	-	-
Iodine value (g I_2/100g)	25.69	-	-
Cetane number	64.83	48-65	40-55
Flash point (K)	423	373-443	333-353

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It was found that all the fuel properties of biodiesel other than the acid value were within the prescribed limits and even better compared to those of petroleum-derived diesel. The two stage transesterification process has reduced the initial kinematic viscosity of 72.16 mm^2/s to a lower value of 5.94 mm^2/s. The low viscosity will make the biodiesel easier to pump and atomize, thus ensures the better performance of engine [8]. The produced biodiesel offers high safety against fire hazard and handling benefits over diesel fuel because of its high flash point of 423K, which is the minimum temperature at which the fuel will ignite on application of an ignition source [8]. Cetane number (CN) is an important parameter in evaluating the quality of biodiesel fuel, especially ignition quality. CN of produced biodiesel was identified to be very near to the upper limit of the reference range. It measures the readiness of the fuel to auto-ignite when injected into the engine. A fuel with high cetane number has good ignition quality, where the ignition delay period between the start of fuel injection and the onset of auto ignition is short and provides smoother engine operation than diesel fuel. The high CN of biodiesel may be influenced by their characteristics of the feed stock [8]. This shows that the ayurvedic waste oil is a good source for biodiesel.

VII. CONCLUSION

The properties of biodiesel produced from ayurvedic waste oil conform to the ASTM standards, except for the acid value. Certain fuel characteristics are even better than petroleum diesel. The biodiesel yield obtained by conventional heating procedure is also considerably good. Hence, the ayurvedic waste oil can be used as an excellent source of biodiesel. This also gives a remedy for oil waste management problems. Optimisation of production parameters of acid esterification process can bring down the acid value and thereby can improve the biodiesel yield. The work is placed as an initial investigation for a software assisted optimisation of acid and alkali transesterifications for biodiesel production from ayurvedic waste oil.

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