Optimization of Biodiesel Production and its Characterization from Soybeans

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ABSTRACT
In recent years, the fossil fuel resources are depleting rapidly with consequent environment degradation. Further, India is importing more than 80% of its fuel requirement and spending a huge amount of foreign currency on fuel. In view of this, it becomes highly imperative to search alternative fuel options based on renewable energy. Biodiesel, the monoalkyl esters of long chain fatty acids derived from a renewable lipid feedstock’s such as vegetable oil or animal fat is providing a substitute to diesel. In the present study the parameters affecting solvent extraction and transesterification of oil was investigated. Also the characterization of the produced biodiesel was done. The highest oil extraction was obtained under the conditions of 0.425 mm particle size, 8:1 solvent to feed ratio, 6 h extraction time and extraction temperature of 60°C. The results showed that biodiesel produced from soybeans was the maximum at 1:1 methanol to oil ratio, 2 h reaction time, 0.5% NaOH catalyst, 250 rpm and methanol as alcohol type. The different properties obtained for the biodiesel produced from soybeans conformed to the biodiesel standard values.

Keywords: Biodiesel, Transesterification, Soybeans, Solvent extraction, Purification, Characterization.

INTRODUCTION
In view of the fast depletion of fossil fuel, the search for alternative fuels has become inevitable, looking at huge demand of diesel for transportation sector, captive power generation and agricultural sector [1]. The major part of all energy consumed worldwide comes from petroleum, charcoal and natural gas. These sources are limited and would be exhausted in the near future. Historically, many biomass and agricultural derived materials have been suggested as alternative energy sources and the use of biodiesel as fuel presents promising potential [2][3]. Biodiesel has attracted much attention all over the world because of its availability, renewability, non-toxicity, better gas emissions and its biodegradability [4]. Chemically Biodiesel is defined as the mono-alkyl esters of fatty acids derived from vegetable oil or animal fat. It is compatible with conventional diesel fuel and already used as a commercial fuel in Europe.

The raw material being exploited commercially by the developed countries constitutes the edible fatty oils derived from rapeseed, soybean, palm, sunflower, coconut, linseed etc. Soybean oil derived biodiesel possess enhanced biodegradation, increased flashpoint, reduced toxicity, lower emissions and increased lubricity. Various oils like algal oil, sunflower and palm oil have been used in different countries as raw materials for biodiesel production owing to its availability [5]. The different methods that are used to extract oil from seeds are hydraulic presses, screw presses, extrusion methods and solvent extraction method. The method by which a particular oil seeds are extracted depends on the type of seed, the seed characteristics and the oil content of the seed. The biodiesel is prepared by transesterification process combining vegetable oils with alcohol in the presence of the catalyst to form fatty acid alkyl esters and glycerol.

Transesterification has been described as a chemical reaction between triglycerides and alcohol in the presence of catalyst to produce mono-esters that are termed as biodiesel. The three basic methods of ester production from oil/fat are the base-catalyzed transesterification, the acid catalyzed transesterification and enzymatic catalysis. Alkali catalyzed transesterification process is the common process for production, because it can achieve high purity and high yield of biodiesel in short time [6]. Transesterification reactions have been studied for many vegetable oils such as soybean [7], rapeseed [8], sunflower [9], safflower [10], canola [6], palm [11] and fish oil [12].The determination of biodiesel fuel quality is an issue of great importance to the successful commercialization of this fuel [13]. The objectives of the present study were to investigate the factors affecting the solvent extraction of oil from soybeans and conversion of it to biodiesel. In addition, characterization of the biodiesel produced was also done.
MATERIALS AND METHODS:

Preparation of raw material
Soybean was brought from local market, Hubli, Karnataka state, India. Seeds were cleaned for foreign materials and were dried in an oven at 103°C for one hour to remove any moisture content present in it. The dried seeds were ground with Mortar and Pestle. After grinding, the ground seeds were separated, according to their size, using sieve shaker. Then the seeds were subjected for oil extraction using soxhlet extractor.

Solvent extraction of oil
The extraction process was studied for particle size of 0.425 mm, 0.850 mm and 1.275 mm, solvent to feed ratio of 2:1, 4:1, 6:1 and 8:1 and extraction time of 2 h, 4 h, 6 h and 8 h. These parameters were varied one at a time to study the effect of these on oil extraction process. 150 ml of n-hexane was poured into round bottom flask. 50 grams of ground meal was placed in the thimble and was inserted in the centre of the extractor. The soxhlet was heated at 60°C. When the solvent was boiling, the vapor rises through the vertical tube into the condenser at the top. The liquid condensate drips into the filter paper thimble in the centre, which contains the solid sample to be extracted. The extract seeps through the pores of the thimble and fills the siphon tube, where it flows back down into the round bottom flask. This was allowed to continue for 2 h. It was then removed from the tube, dried in the oven, cooled in the desiccators and weighed again to determine the amount of oil extracted. At the end of the extraction the solvent was separated from the oil using rotary vacuum evaporator [14]. The Percentage of oil extracted was determined by dividing the amount of oil obtained by the amount of the seeds multiply by 100.

Transesterification of oil
Transesterification is the displacement of alcohol from an ester by another alcohol in a process similar to hydrolysis except that an alcohol is used instead of water. The effect of three main parameters, which are different alcohol types like Methanol, Ethanol and Butanol, Alcohol to oil ratio of 1:1, 1:2, 1:3 and 1:4, and reaction time of 2 h, 4 h, 6 h and 8 h were studied for transesterification of oil. An appropriate volume of alcohol was measured and poured into a 500 ml conical flask. The catalyst in pellet form was weighed and mixed with alcohol. The mixture was then shaken for about 1 h until all the catalyst dissolved. 50 ml of oil was measured using a measuring cylinder and poured into a conical flask containing the mixture of alcohol and catalyst. The reactions were carried out by using 0.5% NaOH (wt/wt), 1:1 alcohol to oil ratio for 2 hrs at room temperature of 28°C and stirring speed of 250 rpm. After the reaction, the mixture was transferred to a separating funnel and allowed to stand overnight. The lower layer consisting of glycerol, methanol and catalyst was drained out [4].

Purification of biodiesel
The upper layer contains some impurities like excess alcohol, excess catalyst and glycerin. Since all the impurities are polar groups, water is a suitable solvent for dissolving them. The volume of distilled water added was approximately 30% of the biodiesel volume. The flask was shaken gently for one minute and placed on the table to allow separation of biodiesel and water layers. After separation, the biodiesel was transferred to a clean conical flask. The washing process was repeated for several times until the washed water became clear [4]. Biodiesel yield was calculated by dividing the amount of biodiesel produced by amount of oil used multiply by 100.

Biodiesel Characteristics

Determination of Flash Point
Flash point is that minimum temperature of fluid at which combustible fluid under consideration releases sufficient vapors such that on mixing with air a combustible mixture is formed that can sustain a momentary combustion on being ignited from external source. The biodiesel sample was placed in the Pensky Martin’s cup such that it touches the required mark of the cup. The cup was covered with a lid and Bunsen burner was used to supply heat to the oil sample at the rate of about 3°C per minute. During heating, the biodiesel sample was stirred continuously. As the sample approaches its flashing, the injector burner was lighted and injected into the sample container after every 12 seconds intervals until a distinct flash was observed within the container. The temperature at which the flash occurred was then recorded [14].

Determination of Carbon residue
Carbon residue is measured by Ram’s bottom apparatus. Initially weighed crucible (W1) was taken and four grams of biodiesel sample was taken in it and reweighed (W2). Crucible was placed in position and assembled the ram’s bottom apparatus for closed cup test. Crucible was heated for about 10 minutes, till the biodiesel sample is completely burned, leaving behind carbon residue. The crucible was cooled for about 10 minutes and weighed (W3). Percentage of carbon residue is given by the expression: \[\left(\frac{W_3- W_1}{W_2- W_1}\right) \times 100\] [14].

Determination of Specific Gravity
A clean and dry bottle of 25 ml capacity was weighed (Wo) and then filled with the biodiesel sample, stopper inserted and reweighed to give (W₁). The bottle was washed, dried, filled with water and weighed to give (W₂). Specific gravity is determined by the expression: \((W₁ - Wo) / (W₂ - Wo) = \text{Mass of the substance/Mass of an equal volume of water}\) [14].

**Determination of Viscosity**

A clean, dried viscometer with a flow time above 200 seconds for the fluid to be tested was selected. The sample was filtered through sintered glass to eliminate dust and solid material in the Biodiesel sample. The viscometer was charged with a sample by inverting tubes thinner arm into the sample and suction force drawn up to the upper timing mark of the viscometer, after which instrument was turned to its normal vertical position. The viscometer was placed into a holder and inserted to a constant temperature bath set at 29°C and allowed approximately 10 minutes for the sample to come to the bath temperature at 29°C. The suction force is then applied to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time by timing the flow of the sample as it flow freely from upper timing mark to the lower timing mark was recorded [14].

**Results and Discussion**

**Influence of particle size on oil extraction**

Figure 1 shows the effect of particle size on oil extracted. The extraction was carried out using particle size of 0.425 mm, 0.85 mm and 1.275 mm. The results show that increasing the meal size decreased the percentage of oil recovered. Less oil is extracted from the larger particles compared to the smaller size particles. The reason is that larger particles with smaller contact surface areas are more resistant to solvent entrance and oil diffusion. Therefore, less amount of oil will be transferred from inside the larger particles to the surrounding solution in comparison with the smaller ones [15].

![Figure 1: Effect of particle size on oil extraction.](image)

**Influence of Solvent to feed ratio on oil extraction**

Four different solvent to feed ratios (v/w) were used to study the effect on oil extracted. From figure 2, it is found that there is increase in the total amount of oil extracted with the increase in solvent to feed ratio from 2:1 to 8:1. Increasing solvent to solid ratio will increase the yield since the concentration gradient between the solid and the liquid phase becomes greater which favors good mass transfer [15].

![Figure 2: Effect of Solvent to feed ratio on oil extraction.](image)
**Influence of reaction time on oil extraction**

Figure 3 shows the percentage of oil extracted from soybean seeds at different reaction times. The results were obtained for a particle size of 0.425 mm, reaction temperature of 60°C and solvent to feed ratio of 8:1. There is increase in the oil extraction with the increase in the reaction time. At 6 hrs, oil extraction is found to be maximum. There is no increase in oil extraction at 8 hrs. The oil recovery is fast at the beginning of the extraction process, and slows down gradually. This is because when the seed is exposed to the fresh solvent, the free oil on the surface of meal is solubilized and oil gets extracted quickly and also the oil concentration is low in the solvent at the beginning of the extraction process [15].

![Figure 3: Effect of reaction time on oil extraction.](Image)

**Influence of different alcohol types on biodiesel yield**

Three different alcohols: methanol, ethanol and butanol were used in the study. Figure 4 shows the yield of Biodiesel from soybean oil by using different types of alcohols. The transesterification was carried out by using 0.5% NaOH, 1:1 alcohol to oil molar ratio for 2 hours at room temperature. The result shows that methanol is the best alcohol, which gave a biodiesel yield of 69.8%, followed by ethanol and butanol. The methanol is simpler in terms of chemical structure than butanol, so it is more likely for transesterification to occur. Also the base catalyzed formation of ethyl ester is difficult compared to the formation of methyl esters. In the case of methanolysis, formation of emulsions quickly and easily breaks down to form a lower glycerol rich layer and upper methyl ester rich layer. In ethanolysis, these emulsions are more stable and severely complicate the separation and purification of esters [4].

![Figure 4: Effect of different alcohol types on biodiesel yield.](Image)

**Influence of different alcohol to oil ratio on biodiesel yield**

From the figure 5, it can be seen that 1:1 methanol to oil ratio gave the higher ester yield compared with other ratios. The result shows that increasing the alcohol to oil ratio decreased the biodiesel yield. The high ratio of alcohol to oil interferes with the separation of glycerin because there is an increase in solubility. When glycerin remains in solution it helps to drive the equilibrium to back left, thus lowering the ester yields [16]. Maximum and minimum biodiesel yield of 69.8% and 58.2% was observed for methanol to oil ratio of 1:1 and 1:4 respectively.
Influence of reaction time on biodiesel yield
Figure 6 shows the effect of reaction time on ester yield. Results show that reaction time of 2 h gave the better ester yield than 8 h. It is observed that there is decrease in Biodiesel yield with the increase in reaction time. The longer the reaction time, the more the hydrolysis of ester would occur. It might produce many free fatty acids at the end, and these FFAs would participate in soap formation, thus reducing the ester yield. Excess reaction time did not promote the conversion, but favored the reverse reaction of transesterification which resulted in a reduction in the ester yield [4].

Analysis of different properties of Biodiesel produced
The results obtained for the analysis of viscosity, specific gravity, carbon residue and flash point of Biodiesel produced are given in table 1. The experimental values were compared with those of biodiesel standard values and were found to be satisfactory.

<table>
<thead>
<tr>
<th>Property</th>
<th>Biodiesel Standard values</th>
<th>Experimental Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flash Point (°C)</td>
<td>130</td>
<td>106</td>
</tr>
<tr>
<td>Carbon Residue (% mass)</td>
<td>0.050</td>
<td>0.025</td>
</tr>
<tr>
<td>Viscosity (mm²/s)</td>
<td>1.9-6.0</td>
<td>4.7919</td>
</tr>
<tr>
<td>Specific gravity (g/cc)</td>
<td>0.860-0.900</td>
<td>0.868</td>
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</tbody>
</table>

Conclusion
In the present study, Biodiesel from soybean has been successfully produced under varying operating conditions in the laboratory. The optimal values of the parameters affecting solvent extraction of soybean oil were found to be 0.425 mm particle size, 8:1 solvent to feed ratio and extraction time of 6 hrs. It was also observed that the Biodiesel yield was the maximum for methanol alcohol type, 1:1 alcohol to oil ratio and 2 hrs of reaction time. Further produced Biodiesel was characterized for viscosity, specific gravity, carbon residue and flash point which were found to be in close agreement with the Biodiesel standards.
References