

Transesterification of vegetable oil to biodiesel fuel using alkaline catalyst

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ABSTRACT

Biodiesel is gaining more and more importance as an attractive fuel due to the depleting fossil fuel resources. Chemically biodiesel is monoalkyl esters of long chain fatty acids derived from renewable feed stock like vegetable oils and animal fats. It is produced by transesterification in which, oil or fat is reacted with a monohydric alcohol in presence of a catalyst to give the corresponding monoalkyl esters. This article reports experimental data on the production of fatty acid methyl esters from vegetable oils, soybean and cottonseed oils using sodium hydroxide as alkaline catalyst. The variables affecting the yield and characteristics of the biodiesel produced from these vegetable oils were studied. The variables investigated were reaction time (1–3 h), catalyst concentration (0.5–1.5 w/wt%), and oil-to-methanol molar ratio (1:3–1:9). From the obtained results, the best yield percentage was obtained using a methanol/oil molar ratio of 6:1, sodium hydroxide as catalyst (1%) and 60 ± 1 °C temperature for 1 h. The yield of the fatty acid methyl ester (FAME) was determined according to HPLC. The composition of the FAME was determined according to gas chromatography. The biodiesel samples were physicochemically characterized. From the results it was clear that the produced biodiesel fuel was within the recommended standards of biodiesel fuel.

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1. Introduction

Diesel fuel is largely utilized in the transport, agriculture, commercial, domestic and industrial sectors for the generation of power/mechanical energy. Thus the increased use of diesel fuel resulted in depletion of its fossil reserves [1,2]. The scientific community has been investigated new type of renewable energy sources, mainly due to the greenhouse effect brought about by the growing usage of fossil energies. Alternatives to petroleum-derived fuels [3], such as biodiesel, which is considered to be a possible substitute of conventional diesel, is biodegradable, non-toxic, renewable and has reduced emission of CO, SO₂, particulate matter, volatile organic compounds and unburned hydrocarbons as compared to conventional diesel [4,5].

Biodiesel seems to be a realistic fuel for future; it has become more attractive recently because of its environmental benefits. Biodiesel is an environmentally friendly fuel that can be used in any diesel engine without modification [6].

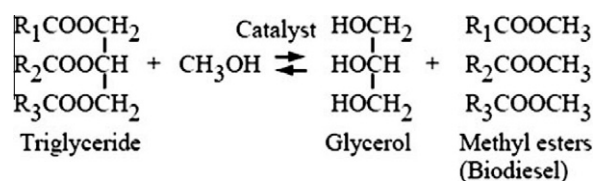
Biodiesel obtained from vegetable oil can be used as conventional diesel in diesel engines because its properties are very close to petroleum diesel. For example, biodiesel has the proper viscosity, high flash point; high cetane number and no engine modification are required when using biodiesel [7].

In the USA and Europe, the surplus edible oils like soybean oil, sunflower oil, and rapeseed oil are being used as feedstock for the production of biodiesel [8]. However, the viscosity of vegetable oil is several times higher than that of mineral diesel due to their large molecular mass and chemical structure. It ranges from 10 to 20 times greater than that of petroleum diesel fuel [9].

The high viscosity of vegetable oils (30–200 cSt) as compared to mineral Diesel oil (4 cSt) at 40 °C leads to unfavorable pumping and spray characteristics [10]. As a result, vegetable oils can cause poor fuel atomization, incomplete combustion, and carbon deposition on the injector and valve seats; all resulting in serious engine fouling [11].

Common methods employed to reduce the viscosity of vegetable oils include blending with diesel [11], micro-emulsions [12], thermal cracking [13], and transesterification [14].

Transesterification is a most common [15] and well established chemical reaction in which a primary alcohol reacts with the triglycerides of fatty acids (vegetable oil) in presence of a catalyst to form glycerol and esters [10,16–18].



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where R1, R2, R3 are long-chain hydrocarbons, sometimes called fatty acid chains.

The stoichiometry for the reaction is 3:1 M ratio of alcohol to oil, however, since the reaction is reversible, in practice, excess alcohol (6:1) is required to shift the equilibrium to the products side to raise the product yield and allow its phase separation from the glycerol to be formed [14,19]. Methanol and ethanol are used most frequently, especially methanol because of its low cost and its physical and chemical advantages (polar and shortest chain alcohol). The presence of a catalyst considerably accelerates the adjustment of the equilibrium.

The transesterification reaction can be catalyzed by both homogeneous and heterogeneous catalysts. Homogeneous catalysts include alkalis and acids. The most commonly used alkali catalysts are NaOH, KOH and the corresponding sodium and potassium alkoxides [14,20,21]. Sulfuric acid, sulfonic acid and hydrochloric acid are usually used as catalysts in the acid-catalyzed reaction.

The alkali-catalyzed transesterification of vegetable oils proceeds faster than the acid-catalyzed reaction [22]. The mechanism of the base-catalyzed transesterification of vegetable oils was discussed by Demirbas [23].

For an alkali catalyzed transesterification, the triglycerides should have lower free fatty acid (FFA) content, and the alcohol must be anhydrous to render soap formation. Soap formation lowers the yield of esters and renders the separation of esters and glycerol [14,24,25]. Up to about 5% FFA, the reaction can be catalyzed using an alkali catalyst [26].

The extent of transesterification and side reactions depends upon the type of feedstock, catalyst formulation, catalyst concentration, alcohol-to-oil ratio, reaction temperature and reaction time [27,28].

Cottonseed oil was converted into biodiesel by alkali-catalyzed transesterification reaction at different factors. It was found that catalyst concentration 0.75%, temperature 65 °C, methanol to oil molar ratio 6:1 and agitation intensity 600 rpm provided optimum condition producing excellent yield (96.9%) of cottonseed methyl ester [29].

This current work was focused on the production of biodiesel from soybean and cottonseed oils using sodium hydroxide as alkaline catalyst.

2. Materials and methods

2.1. Materials

In this study two commercially vegetable oils (soybean and cottonseed) were used in the methyl ester production. Some physical properties of the vegetable oils are shown in Table 1. Pure sodium hydroxide as alkaline catalyst and methyl alcohol (Merck) of 99.5% purity (density: 0.791–0.792 kg/l) were used in the transesterification process.

2.2. Experimental procedures

The transesterification was carried out in 1 l reaction flask equipped with reflux condenser, magnetic stirrer and thermometer. The transesterification process was studied at three catalyst

loadings (0.5%, 1.0% and 1.5% NaOH wt/wt), one reaction temperature (60 ± 1 °C) and three alcohol-to-oil molar ratios (3:1, 6:1 and 9:1) at a reaction time (1–3 h). The catalyst was dissolved into methanol by stirring. Five hundred grams of the oil was introduced into the reaction flask. After the appropriate temperature was reached, NaOH previously dissolved in methanol was added and the mixture was continuously stirred at 400 rpm by means of a magnetic stirrer. After the preestablished time, the mixture was carefully transferred to a separating funnel and allowed to stand there overnight. The lower layer (glycerol, methanol and most of the catalysts) was drained out. The upper layer (methyl esters, some methanol and traces of the catalyst) was then cleaned thoroughly by washing with warm (50 °C) de-ionized water in order to remove the impurities like uncreated methanol, uncreated oil and catalyst. The process of washing was repeated until the lower layer had a pH similar to the pH of distilled water, indicating that the biodiesel is free of catalyst. The methyl ester was heated to 110 °C then dried with anhydrous Na_2SO_4 to get rid of any water.

2.3. Ester characterization

The composition of the transesterification products was determined by high performance liquid chromatography (HPLC) using a Waters GPC-Styragel 0.5 column, ($7.8 \times 300 \text{ mm}^2$) with tetrahydrofuran as the mobile phase combined with Detector Waters 2410 refractive index. The column was operated at 40 °C.

The fatty acid composition was determined by gas chromatography using Agilent 6890 plus, equipped with HP50 + capillary column ($0.53 \text{ mm} \times 30 \text{ m}$, $0.5 \mu\text{m}$ film) and flame ionization detector. Pure nitrogen was used as a carrier gas. The oven temperature was varied from 60 to 260 °C at a fixed rate of 4 °C/min. The temperature of the injector and detector was 300 °C.

The esters obtained by transesterification were then tested for estimating their fuel properties according to the IP and ASTM standard test methods of analysis [30,31].

3. Results and discussion

Alkaline catalyst transesterification of soybean and cottonseed oils was carried out taking consideration to achieve maximum yield of biodiesel methyl ester, so factors affecting the yield of the methyl ester were studied.

3.1. Factors affecting the yield of the methyl ester

3.1.1. Effect of molar ratio

One of the most important parameters affecting the yield of ester is the molar ratio of alcohol to vegetable oil. The stoichiometry of the transesterification reaction requires 3:1 M ratio to yield 3 mol of ester and 1 mol of glycerol, but most researchers found that excess alcohol was required to drive the reaction close to completion. Higher molar ratios result in greater ester production in a shorter time [1].

In the present research, methanol was used. The effect of methanol in the range of 3:1 to 12:1 (molar ratio) at 60 ± 1 °C was investigated, keeping other process parameters fixed (Fig. 1).

The reaction was incomplete for a molar ratio less than 6:1 and no separation was observed during settling. The ester yield percentage increases with increasing methanol: oil molar ratio from 3 to 6 giving a methyl ester yield of a percentage of 90% and 98.5% for soybean and cottonseed, respectively. It is shown that beyond the molar ratio of 6:1, further methanol addition had no effect on ester formation; rather it complicated ester and raised process cost. When the ratio increased to 12:1 high methanol amounts interfere with the separation of glycerin because of an

Table 1
Some physical properties of soybean and cottonseed vegetable oils.

Physical properties	Soybean oil	Cottonseed
Kinematic viscosity, cSt at 40 °C	32.50	35.42
Density, g/cm ³	0.9330	0.9241
Pour point, °C	4	15
Total acid number, mg KOH/g	0.415	0.602

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