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ORIGINAL ARTICLE

Synthesis, spectroscopic and chromatographic studies of sunflower oil biodiesel using optimized base catalyzed methanolysis

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Transesterification;
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Abstract Methyl esters from vegetable oils have attracted a great deal of interest as substitute for petrodiesel to reduce dependence on imported petroleum and provide an alternate and sustainable source for fuel with more benign environmental properties. In the present study biodiesel was prepared from sunflower seed oil by transesterification by alkali-catalyzed methanolysis. The fuel properties of sunflower oil biodiesel were determined and discussed in the light of ASTM D6751 standards for biodiesel. The sunflower oil biodiesel was chemically characterized with analytical techniques like FT-IR, and NMR (^{1}H and ^{13}C). The chemical composition of sunflower oil biodiesel was determined by GC–MS. Various fatty acid methyl esters (FAMEs) were identified by retention time data and verified by mass fragmentation patterns. The percentage conversion of triglycerides to the corresponding methyl esters determined by ^{1}H NMR was 87.33% which was quite in good agreement with the practically observed yield of 85.1%.
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1. Introduction

In the last few decades energy needs have increased enormously and most of the energy needs are supplied through petrochemical sources (Vicente et al., 2007). In addition to rapid consumption, the rising costs, environmental problems and unavailability of the fossil fuel have led to look for the alternate sources of energy (Bajpai and Tyagi, 2006). The need of the hour is to have an alternative fuel which is technically feasible, economically competitive, environmentally acceptable and readily available (Demirbas, 2009). The most common fuel

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that is being focused upon and having brighter aspects is biodiesel. Biodiesel refers to a diesel-equivalent, processed fuel derived from biological sources.

Biodiesel is produced as a result of transesterification between a fat or oil and an alcohol (usually methanol) resulting in ester and a by-product, glycerol (Dorado et al., 2000; Angelo et al., 2005). Transesterification is a three step reaction with formation of mono and diglycerides as intermediate products (Vicente et al., 2007). Biodiesel is biodegradable, non-toxic, non aromatic and does not contain sulfur. Being plant based it does not emit CO on burning and is environmentally safe (Haq et al., 2006). Aromatic, ash, sediments, water, glyceride and methanol content if exist are less than 2% in biodiesel, in total showing that it has no waste products to be disposed off.

The commercial preparation of biodiesel from the oils of rapeseed (Komers et al., 2001), soybean (Candeia et al., 2009), rocket seed (Tariq et al., 2011), palm (Benjumea et al., 2008), sesame (Tariq et al., 2013), jojoba seeds (Mazloom et al., 2014), etc., has been reported in the literature. The present study deals with the synthesis of biodiesel from sunflower oil by base catalyzed transesterification with methanol to check the potential of this oil source for the commercial production of biodiesel. The importance of using base catalyzed transesterification is its economic process, less time for reaction, low temperature and high conversion rate. However, base catalyzed transesterification has limitations of hygroscopic nature of base catalyst, saponification, more washing and emulsion formation. The need of the present study is to find out an alternative renewable energy source in order to decrease dependence on crude oil and to check the potential of this oil source for the commercial production of biodiesel. Sunflower seeds contain about 42–50% high quality edible oil (Haq et al., 2006; Baksh et al., 1999). Sunflower has a high place in international agricultural market due to its varied uses. The biodiesel formed was characterized by different physico-chemical properties and its composition was determined by employing various instrumental techniques like FT-IR, NMR and GCMS.

2. Materials and method

2.1. Materials

Sunflower seeds were purchased from a local market. The seeds were washed with distilled water to remove the dirt and were oven dried at 60 °C till constant weight. The oil was extracted by using electric oil expeller (KEK P0015-10127), Germany. Methanol (99.9% purity), sodium hydroxide (NaOH) and anhydrous sodium sulfate (Na_2SO_4) were of analytical grade and were obtained from Merck (Germany) and used as such without any purification.

2.2. Analytical methods

The fuel properties of synthesized sunflower oil biodiesel were determined by ASTM methods. Sunflower oil biodiesel was characterized by FT-IR, using a Bio-Rad Excalibur Model FTS3000MX in the range 4000–400 cm^{-1}. The resolution was 1 cm^{-1} and 15 scans. NMR analyses were performed using Avan CE 300 MHz spectrometer equipped with 5 mm BBO probes at 7.05 T. Deuterated chloroform ($CDCl_3$) and tetramethylsilane (TMS) were used as solvent and internal standard, respectively (Ahmad et al., 2010). The ^{13}C (75 MHz) spectra were recorded with a pulse duration of 30°, a recycle delay of 1.89 s and 160 scans. The fatty acid methyl ester (FAME) contents of sunflower oil biodiesel were determined by gas chromatography, model GC-6890 N coupled with mass spectrometer, model MS-5973 MSD (mass selective detector). Separation was performed on a capillary column DB-5MS (30 m × 0.32 mm, 0.25 μm of film thickness). The mass spectrometer coupled with GC was set to scan in the range of m/z 50–550 with electron impact (EI) mode of ionization.

2.3. Experimental procedure

2.3.1. Transesterification

The transesterification was carried out using 2000 mL round bottom flask equipped with reflux condenser, magnetic stirrer, thermometer and sampling outlet. One liter vegetable crude oil was filtered and preheated up to 120 °C to remove moisture on a heating mantle. The transesterification of sunflower oil was carried out with a 6:1 M ratio of methanol/oil and 0.7% NaOH (as catalyst) w/w of the oil. Sunflower oil (500 mL) was transesterified with methanol (100 mL) in the presence of NaOH (3.25 g) as catalyst. The temperature of the reaction was maintained at 60 °C and the contents were stirred with a constant speed of 600 rpm for 1.5 h. The resultant mixture was cooled to room temperature for the separation of two phases. The upper lighter phase of biodiesel and lower denser phase of glycerin (by-product) were separated by simple decantation. Crude biodiesel contains the excess methanol, unused catalyst, soap formed, some entrained methyl esters and partially reacted glycerides. The remaining catalyst was removed by successive rinsing with distilled water followed by the addition of 1–2 drops of acetic acid to neutralize the residual catalyst and conversion of soap to free fatty acids (FFA). The residual water contents were eliminated by treatment with anhydrous Na_2SO_4, followed by filtration. A transparent yellow liquid was obtained as the final product.

The yield of biodiesel formed was calculated by using the expression

$$\%\text{Yield} = \frac{\text{Grams of methyl ester produced}}{\text{Grams of oil taken}} \times 100 \qquad \text{(i)}$$

3. Results

Biodiesel was synthesized from sunflower oil by using NaOH as catalyst for transesterification with methanol. The molar ratio of alcohol to oil (6:1) and temperature of 60 °C, used were selected on the basis of our earlier reports (Tariq et al., 2011; Mazloom et al., 2014) as well as other reported optimal conditions for the transesterification of vegetable oils (Meher et al., 2006). The general reaction of the base catalyzed transesterification can be represented as:

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