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Detailed investigation of optimized alkali catalyzed transesterification of Jatropha oil for biodiesel production

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Abstract

The non-edible oils are believed to be one of the major feedstock for the production of biodiesel in future. In the present study, we investigated the production of Jatropha oil methyl esters (JOMEs) via alkali-catalyzed transesterification route. The biophysical characteristics of Jatropha oil were found within the optimal range in accordance with ASTM standards as a substitute diesel fuel. The chemical composition and production yield of as-synthesized biodiesel were confirmed by various analytical techniques such as FT-IR, ^{1}H NMR, ^{13}C NMR and gas chromatography coupled with mass spectrometry. A high percentage conversion, ~96.09%, of fatty acids into esters was achieved under optimized transesterification conditions with 6 : 1 oil to methanol ratio and 0.9 wt% NaOH for 50 min at ~60 °C. Moreover, twelve fatty acids methyl esters (FAME) were quantified in the GC/MS analysis and it was interesting to note that the mass fragmentation pattern of saturated, monounsaturated and diunsaturated FAME was comparable with the literature reported values.

Key words

Jatropha oil methyl esters; transesterification; biodiesel fuel; gas chromatography; mass spectrometry

1. Introduction

Due to the rapid depletion of fossil fuels resources and global warming effects, the entire world is tending to shift its energy dependence from the fossil fuel economy to a more cleaner renewable energy based economy [1−3]. As per International Energy Outlook 2013 (IEO2013) report, the global energy consumption has increased by 56 percent i.e. ~2.5%/y [4]. This figure indicates an expected demand of energy that will rise from 524 quadrillion Btu in 2010 to 630 quadrillion Btu in 2020 and to 820 quadrillion Btu in 2040. Hence, at present, one of the major challenges in front of scientists and technologists is to find alternate resources of energy, which potentially exhibit the capacity to meet the growing demand of energy with minimum environmental impact. In this context, a significant level of efforts in past few years is to explore the renewable energy resources with the aim to smoothly shift the energy dependence from fossil fuel based economy to more cleaner renewable energy based economy [5,6].

Over the last decade, the biomass resources such as vegetable oils and tree borne oil seeds have received immense attention owing to their profitability of producing high quality biodiesel fuel [7−9]. The high viscosity of vegetable oil (30−200 cSt) as compared with mineral diesel oil leads to unfavorable pumping and spray characteristics [10]. In order to address the energy crisis to some extent, biofuels, particularly the biodiesel must be used as an alternate to the conventional petroleum supply. The 95% of biodiesel production feedstock comes from the edible oils and their properties are very promising in terms of diesel fuel substituent [11]. The most commonly used edible feedstock for the production of biodiesel are soybean [12,13], rapeseed [14], canola [15], safflower [16,17], barley [18], cotton seed [19], almond [20], brassica carinata [21], sesame and rice bran [22].

However, the diminution of edible oils and their rapidly increasing cost led the researchers to explore non-edible oils as biodiesel feedstock. Recently, Jatropha curcas [23−25], jojoba oil [26], mahua [27], tallow and poultry fat, microalgae and waste cooking oil [10] have been successfully utilized as non-edible feedstock for the production of biodiesel. The

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selection of vegetable oils as biodiesel feedstock mainly depends on the region and climate. The biodiesel such as fatty acid alkyl ester is intrinsically non-toxic, renewable, and biodegradable, which upon use results in reduced emission of GHG, particulate matter, volatile organic compounds and unburned hydrocarbons as compared with the mineral diesel [14,28].

The transesterification of triglycerides with alcohols, which in practice is usually catalyzed by an alkali catalyst, is the most commonly used process in biodiesel production technology. Due to low-energy and cost-effective transesterification route, the homogenous alkaline media based catalysis appears to be a quite general approach and extremely useful in biodiesel industry [29−32]. However, the reaction conditions for this alkali catalyzed transesterification of triglycerides with alcohols need to be controlled in order to obtain high yields of fatty acid methyl esters (the biodiesel). It is commonly observed that the homogenous alkaline catalysis is usually affected by the presence of water, as water has strong tendency to react with esters to produce acids. The acids thus formed can rapidly react with alkaline species in the media to form soaps, which acts as an impurity. Hence, for homogenous alkaline catalysis, the total free fatty acids content must not exceed 0.5% by weight; otherwise the yield of fatty acid methyl esters would reduce significantly [32].

Since, the reaction conditions can strongly affect the yield of biodiesel produced; this study aims to determine the optimum conditions of reactions to produce biodiesel from Jatropha oil via a single step process of alkali catalyzed transesterification reaction. In addition, we also investigated the effects of different process parameters on the progress of overall reaction and the final biodiesel product.

2. Experimental

2.1. Reagents and materials

Jatropha curcas oil was purchased from the local market and was used as received. Other reagents like sodium hydroxide (99.9%), methanol (99.8%; density: 0.791 g/mL) and anhydrous sodium sulphate (99.9%) were purchased from Sigma Aldrich. Deuterated chloroform used for NMR was of spectroscopic purity and argon gas was of 99.9% pure. All of the chemicals were used without further purification.

2.2. Methyl esters characterization

The fatty acids methyl esters (FAMEs) obtained after the completion of transesterification reaction were tested for their physical and fuel properties (density, specific gravity, kinematic viscosity, cloud point, pour point, flash point, acid value), according to the ASTM standard test methods of analysis. FAME composition was determined by gas chromatography using GC-6890 N directly uploaded to MS 5973 MSD. The MS was set within the range of *m/z* 02−600 and ionization potential. The JOB was characterized by FTIR using Excalibur Model FTS3000MX in the range of 4000–400 cm^{-1}. The scan rate was 15 scans and resolution was 8 cm^{-1}. ^{1}H and ^{13}C NMR was performed using Avan Ce 300 MHz spectrometer equipped with 5 mm BBO probes at 7.05 T. Deuterated chloroform $CDCl_3$ was used as solvent. ^{1}H (300 MHz) spectra were recorded with scan rate of 8 scans; recycle delay of 1.0 s, with pulse duration of 30°. ^{13}C NMR (75 MHz) spectra were recorded with pulse duration of 30°; recycle delay of 2 s and scan rate of 160 scans.

2.3. Procedure for JCO transesterification

The transesterification process was carried out in a 250 mL three necked round bottom flask equipped with a reflux condenser, thermometer and magnetic stirrer. At the start, the crude Jatropha oil was filtered and dehydrated in a rotary evaporator under vacuum for 30 min at 130 °C. This dehydrated oil was later warmed by placing the flask at 60 °C on a water bath. In a separate flask, a known amount of sodium hydroxide was dissolved in methanol with continuous stirring to form sodium methoxide. After that, the transesterification reaction was carried out by mixing 6 : 1 molar ratio of methanol/oil and sodium hydroxide (0.9 wt% of oil) at 60±1 °C. The sodium methoxide formed earlier was then added to the pre-heated Jatropha oil and the mixture was continuously stirred at 450 rpms. The reaction was continuously stirred for 70 min using magnetic stirrer. At the completion of pre-established time, the mixture was transferred to the separating funnel. The bottom layer of glycerin was removed, whereas the upper layer of biodiesel was washed with warm distilled water (50 °C) in order to remove impurities including unreacted methanol, unreacted oil and sodium hydroxide catalyst. The washing was continued further until the lower layer had a pH equivalent to pH of distilled water, showing that biodiesel is free of impurities. The final product of biodiesel (methyl esters) was dried with anhydrous sodium sulphate to remove any trace water. The practical yield was calculated by using the Equation (1):

$$\text{Yield}\ (\%) = \frac{\text{Gram of biodiesele produced}}{\text{Gram of oil used}} \times 100\% \quad (1)$$

Figure 1 shows the trans-esterification mechanism of triglycerides with alcohols catalyzed by alkali catalyst for the biodiesel production technology.

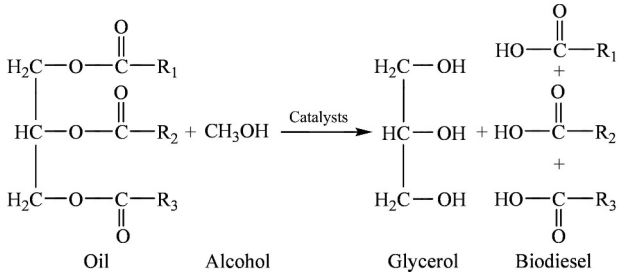

Figure 1. The transesterification of triglycerides with alcohols catalyzed by alkali catalyst for the biodiesel production technology