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Ultrasound assisted transesterification of waste cooking oil using heterogeneous solid catalyst

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ABSTRACT

Transesterification based biodiesel production from waste cooking oil in the presence of heterogeneous solid catalyst has been investigated in the present work. The effect of different operating parameters such as type of catalyst, catalyst concentration, oil to methanol molar ratio and the reaction temperature on the progress of the reaction was studied. Some studies related to catalyst reusability have also been performed. The important physicochemical properties of the synthesized biodiesel have also been investigated. The results showed that tri-potassium phosphate exhibits high catalytic activity for the transesterification of waste cooking oil. Under the optimal conditions, viz. catalyst concentration of 3 wt% K_3PO_4, oil to methanol molar ratio of 1:6 and temperature of 50 °C, 92.0% of biodiesel yield was obtained in 90 min of reaction time. Higher yield was obtained in the presence of ultrasound as compared to conventional approach under otherwise similar conditions, which can be attributed to the cavitational effects. Kinetic studies have been carried out to determine the rate constant at different operating temperatures. It was observed that the kinetic rate constant increased with an increase in the temperature and the activation energy was found to be 64.241 kJ/mol.

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1. Introduction

Significant variation in the global prices of petroleum crude oil has an adverse impact on the national economy of crude oil dependant countries and developing countries such as India. The petroleum crude oil consumption in India was 148 million metric tonne per year in 2011–12 and demand for petroleum products was 141.79 million metric tonne. Diesel constitutes almost about 35.5% of all the petroleum product consumption in India. According to data from the Ministry of Petroleum and Natural gas, 8.2% of the total diesel consumed is for power generation where as agricultural activities use about 12.2% and transport guzzles a maximum of 65.3%. Apart from reducing the dependency of economy on the fossil fuels, depletion of petroleum sources and environmental concerns have also induced a recent interest in finding alternative sources for petroleum based diesel fuels [1].

Transesterified oil which is also commonly known as biodiesel can be a good alternative to petroleum based diesel. By chemical structure, biodiesel is mono-alkyl ester of long chain fatty acid that can be synthesized from a renewable lipid feedstock like used vegetable oil or animal fats. Generally biodiesel is considered as an oxygenated, renewable, biodegradable, environment friendly fuel with similar physical properties as that of petro-diesel with comparatively low emission profile [2]. Biodiesel can be produced using transesterification reaction between oil and alcohol in the presence of homogeneous or heterogeneous or enzymatic catalyst with glycerol as a by-product as shown in Scheme 1. Many alcohols can be used in transesterification reaction such as methanol, ethanol and isopropyl alcohol etc. but the most commonly used alcohol is methanol due its low cost and easy availability. Biodiesel production has a major problem in commercialisation in that the cost of biodiesel produced is about 1.5 times more as compared to that of petroleum based diesel. The production of biodiesel is economically not viable because of cost of raw materials, energy requirements for production and separation costs for the generally used homogeneous catalyst [3–4]. The present study reports an approach based on the use of waste cooking oil as a low cost feed stock and also the use of heterogeneous catalyst to minimise ease of separation. The studies related to the ultrasound induced intensification of biodiesel synthesis based on the use of heterogeneous catalyst and waste cooking oil are scarce and hence the present work is important.

Patil et al. [5] reported that about 4.1 kg of Waste Cooking Oil (WCO) is produced per person in a year. Considering the present

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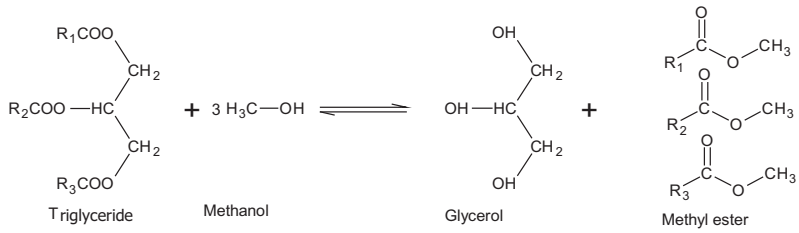

Scheme 1. Transesterification reaction of triglyceride with methanol.

scenario of world population as nearly 7.18 billion, around 29 million tons of WCO will be produced in a year. Disposal of WCO is a major task due to dumping problems and possible pollution of the land resources and water [6]. All these facts indicate that used cooking oil should be recycled and one of the options can be use as a potential source for biodiesel production. Heterogeneous catalyst has advantages like being non-corrosive, environmentally benign and present fewer disposal problems [7]. Also they are much easier to separate from liquid products and can be possibly reused [8]. Many researchers have reported the use of various heterogeneous catalysts like oxides, mixed oxides, phosphates, sulphates etc. [9]. The possible process intensification mechanism for synthesis of biodiesel using ultrasound is that ultrasound helps in the formation of fine emulsion of two immiscible liquids, generating large interfacial area and due to this fact transesterification reaction of waste cooking oil and methanol can reach equilibrium in a shorter time with a high yield of alkyl esters even at lower operating temperature. The enhancement in the mass transfer and interphase mixing between the heterogeneous reactant phases is also helpful in giving enhanced yields [10].

In the present work, potassium and sodium phosphate have been used as the heterogeneous catalysts for transesterification of waste cooking oil. Since phosphates of potassium and sodium are insoluble in methanol, such a process possesses the advantage of easy recovery of the used catalyst from the reaction medium. The influence of various reaction parameters, such as type of catalyst and its loading, oil to methanol molar ratio (OMMR), reaction temperature and catalyst reusability on the yield of fatty acid methyl ester (FAME) has been investigated. Kinetic constant as well as activation energy for the transesterification reaction have been also determined at optimum operating conditions. Also, the properties of the synthesized biodiesel have been evaluated in order to compare with ASTM standards.

2. Materials and methods

2.1. Materials

Waste cooking oil was obtained from a local restaurant in Mumbai. The procured WCO was subjected to physical treatment for removing the suspended solid contaminants by filtration and repeated water washing for the separation of water soluble salt impurities present in the WCO. After the water washing, dehydration was carried out by heating the sample at 80 °C under a vacuum of 25 mmHg for 10 min to remove the traces of water. Table 1 gives the properties of treated WCO being used as feed stock for biodiesel production. As the free fatty acid content in the WCO obtained was observed to be very less (less than 2% i.e. acid value of 3.75), WCO was used directly for the transesterification reaction without any deacidification treatment. Methanol (99%) (AR grade), different catalysts viz. K_3PO_4, Na_3PO_4, Na_2HPO_4, NaH_2PO_4, KH_2PO_4 and molecular sieves were procured from M/s S. D. Fine Chem. Ltd., Mumbai. All the catalysts used in the present work were in powder form. Acetonitrile and acetone (HPLC grade) used as solvent for HPLC analysis were procured from M/s Hi Media, Mumbai whereas methyl oleate and methyl linoleate standards were procured from Sigma–Aldrich.

Table 1
Physical and chemical properties of waste cooking oil.

Property	Value
Physical	
Saponification value (mg KOH/g of oil)	207.7
Density (kg/m^3)	928
Acid value (mg KOH/g oil)	3.75
Viscosity (mm^2/s)	96.55
Water content (%)	0.7
Fatty acid composition	
Linoleic acid (%)	73.4
Oleic acid (%)	18.3
Palmitic acid (%)	6.7
Stearic acid (%)	1.6

2.2. Experimental methodology

The transesterification experiments were performed in a 200-mL three-neck batch reactor with a diameter of 55 mm. The three openings in the reactor were used for accommodating the condenser (to condense the evaporating methanol), introduction of ultrasonic horn having diameter of 12 mm or stirrer (based on the approach of ultrasound assisted or the conventional approach) and inserting the temperature sensor to monitor the temperature during the reaction. The schematic of the experimental setup has been shown in Fig. 1. The reactor was initially charged with used vegetable oil and heterogeneous catalyst and heated to the desired reaction temperature. Once the desired temperature was achieved, known quantity of methanol was added to the reactor. The biodiesel yield was determined by sampling 3 ml of the mixture at different pre-designated time. The withdrawn samples were centrifuged, and the upper phase was withdrawn and the samples were washed with water in order to remove the excess methanol from the mixture. Finally, molecular sieves (3 Å) were added to each sample to adsorb the trace amount of moisture. Samples were then finally analysed using HPLC as per the details described later. The different operating parameters used in the present work to optimise the yield of biodiesel from waste cooking oil, include type of heterogeneous catalysts (K_3PO_4, Na_3PO_4, Na_2HPO_4, NaH_2PO_4 and KH_2PO_4), catalyst concentrations (1, 2, 3 and 4% by weight of the charged oil), oil to methanol molar ratios (1:4, 1:6 and 1:8) and operating temperatures (30, 40, 50 and 60 °C) at fixed 50% amplitude (supplied power of 375 W) of ultrasonic horn of frequency 22 kHz. Calorimetric study has been carried out with known amount of methanol where the time required to increase the temperature by 15 °C was measured. The energy transfer efficiency at different percentage amplitude from 30 to 90% has been calculated based on the input energy (percentage amplitude × rated power) and actual energy dissipation ($m \times Cp \times \Delta T$) into the methanol solution. From Fig. 2, it can be established that maximum energy

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