



Catalytic cracking of waste cooking oil for biofuel production using zirconium oxide catalyst



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ABSTRACT

In the present work, catalytic cracking of waste cooking oil (WCO) for the production of liquid fuels has been studied in a semi batch reactor under N₂ atmosphere. Catalytic cracking was performed at a temperature range of 400–500 °C using zirconium oxide (ZrO₂) as a catalyst. The influence of temperature, heating rate, residence time and catalyst loading on the product yield has been studied. The physicochemical properties such as calorific value, density, flash point and kinematic viscosity of organic liquid product (OLP) was analysed for its suitability for fuel application. The chemical compositions and functional groups present in OLP were identified by gas chromatography-mass spectrometry (GC-MS) and Fourier transformed infrared spectroscopy (FTIR). From the overall study, the optimal ZrO₂ load, reaction temperature, residence time and heating rate were found to be 4 wt%, 475 °C, 120 min and 10 °C/min, respectively at which 83 wt% of OLP was obtained. From the results of catalytic characterization, it was confirmed that the catalyst can be regenerated after use.

1. Introduction

The depletion of fossil fuels with a rapid growth of living standard inflates fuel prices, and its impact on the greenhouse gas attract the development of renewable and non-polluting fuels to promise energy security worldwide (Abnisa et al., 2014b; Mohanty et al., 2012; Zhang et al., 2014). Alternative fuels are obtained from different biomass sources such as cellulosic biomass, sugar and starchy crops (edible biomass), and oil-containing or oil-producing plants (triglyceride based biomass) (Huber and Corma, 2007; Shyamsundar et al., 2013). Among these, triglyceride based sources play a substantial role in biofuel generation (Doronin et al., 2013; Yu et al., 2013). These resources are clean and renewable due to their availability, biodegradability and CO₂ neutrality (Abnisa et al., 2014a; Taufiqurrahmi and Bhatia, 2011). Waste cooking oil (WCO) is one of the most prudent source of energy because of its restrictions for food use, cheap, availability and appropriateness for fuel production (Li et al., 2016). These sources are simply thrown out from cafeterias and could potentially play a significant role in biofuel production. The application of WCO for biofuel production addresses environmental problems concerning the treatment of WCO, but also generates a clean and renewable energy when used as a fuel source (Wiggers et al., 2009). Moreover, the type of biofuel production process used with this feedstock can play a major role in determining

the overall financial viability of biofuel (Zhang et al., 2003). Currently, the use of thermochemical conversion like pyrolysis, which can be done either thermal cracking (Iha et al., 2014; Kozliak et al., 2013; Kubátová et al., 2011; Luo et al., 2010; Mangas et al., 2012; Şensöz and Kaynar, 2006) or catalytic cracking (Chew and Bhatia, 2008; Dupain et al., 2007; Maher and Bressler, 2007; Ong and Bhatia, 2010; Xu et al., 2013) to convert WCO into liquid fuels have been developed (Doronin et al., 2013; Yu et al., 2013). The catalytic cracking process is one of the potential method and, is considered to be an inexpensive in terms of energy consumption to convert feedstocks to lighter fractions of gasoline boiling range (Zhang et al., 2005). The catalytic cracking of vegetable oils with selective heterogeneous catalyst is a potential candidate for renewable-process based industrialization to increase the yield and decrease the cost of liquid fuels (Huber and Corma, 2007; Mohanty et al., 2014). Moreover, these catalysts are easily regenerated, recycled and environmental friendly (Li et al., 2016). The product obtained from catalytic cracking of vegetable oils includes organic liquid product, gas, coke and water (Abnisa et al., 2014a; Mohanty et al., 2014).

The yield of OLP, gas, water and coke were influenced by various factors such as composition of feedstock (Bielansky et al., 2010), reaction temperature (Demirbas, 2009), residence/reaction time (Ong and Bhatia, 2010), nature and the type of catalyst used (Maher and Bressler, 2007; Yu et al., 2013).

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To the best of our knowledge, it is for the first time that the catalytic cracking of WCO with the help of pure zirconium oxide catalyst in batch system split type tubular reactor has been reported. In addition, the aim of this work was to investigate the effect of various process parameters such as temperature, catalyst loading, residence time and heating rates on the yield of OLP.

2. Materials and methods

2.1. Waste cooking oil

WCO was collected from student cafeteria, Indian Institute of Technology, Guwahati, Assam, India. The major fatty acid composition in WCO were myristic acid (C_{14:0}): 0.5%, palmitic acid (C_{16:0}): 20.4%, stearic acid (C_{18:0}): 4.8%, oleic acid (C_{18:1}): 52.9%, linoleic acid (C_{18:2}): 13.5%, linolenic acid (C_{18:3}): 0.8% and eicosanoic acid (C_{20:0}): 0.3% (Borugadda and Goud, 2012). Zirconium Oxide catalyst with a purity of 98% was supplied by Antares Chem. Pvt. Ltd., Mumbai, India. Deuterated chloroform (CDCl₃) was procured from Merck Pvt. Ltd., India.

2.2. Catalyst characterization

X-ray powder diffraction (XRD) patterns of fresh, after one cycle (before calcination) and calcinated ZrO₂ catalyst were obtained on *DB Advance* instrument using monochromatic Cu Ka radiation ($k = 0.15418$ nm). It was operated at 40 kV and 40 mA. The experimental conditions corresponded to a step width of 0.05° and a scan speed of 1°/sec. Diffraction were monitored over a scan range of 2θ from 10° to 80°. Thermogravimetric analysis (TGA) technique was used to estimate the thermal stability of a catalyst. The TGA of fresh, after one cycle and calcinated ZrO₂ catalyst were measured as a function of initial decomposition temperature at a temperature range of 30–900 °C with 10 °C/min heating rate using thermogravimetric analyser (*Model no: TGA 851e/LF/1100*) with the carrier gas flow rate of 60 mL/min (99.99% purity of N₂). Approximately 10 mg of ZrO₂ catalyst was used for analysis. The surface area of ZrO₂ catalyst was calculated using a BET method (*Model Autosorb-IQ MP*). The surface area, total pore volume and average pore diameter (APD) were determined from the amount of N₂ adsorbed at P/P₀ = 0.1–1. The morphology of fresh, after one cycle and calcinated ZrO₂ were analysed using Field Emission Scanning Electron Microscope (FESEM) (Zeiss, *Model: Sigma*). A small powders of ZrO₂ were placed on double-sided carbon tape and attached on a suitable support. The samples were then inserted into the micro-scope chamber and analysed.

2.3. Catalytic cracking of waste cooking oil

Catalytic cracking experiments were carried out at various temperature, range between 400 and 500 °C in a split type tubular semi batch reactor using 1, 2, 4, 6 and 8 wt% ZrO₂ catalyst (Fig. 1). For each experiment 30 g of sample was placed in a reactor. The reactor was heated externally by an electric furnace to achieve final temperature of 400, 425, 450, 475, and 500 °C, at heating rates of 10 and 20 °C/min and kept at isothermal condition from 60 min to 300 min. The vapour are allowed to pass through a condenser fitted on neck of the flask and collected as OLP. The residue left in the reactor was heated in a furnace at 500 °C for 2 h for catalyst regeneration. The yield of OLP was calculated using Eq. (1). (Abnisa et al., 2013)

$$\text{Yield of OLP(wt\%)} = \left(\frac{\text{Mass of OLP collected}}{\text{Mass of feed}} \right) \times 100\% \quad (1)$$

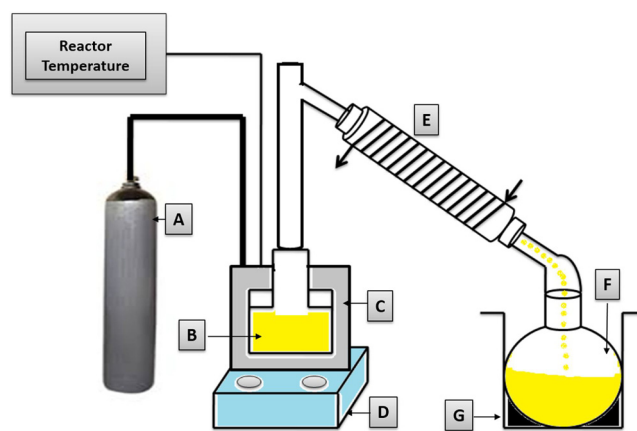


Fig. 1. Experimental setup of catalytic cracking of WCO.

2.4. Feedstock and organic liquid product characterization

Density, kinematic viscosity, flash point and the calorific value of WCO and OLP have been studied. A specific gravity bottle of 25 mL was used for the experiment to measure the sample volume. Density was calculated by taking weight to volume ratio at 25 °C temperature (Tat and Van Gerpen, 2000). Kinematic viscosity was determined using U-tube viscometer and the results were calculated by multiplying the constant of U-tube viscometer with measured average efflux time (time at which a known volume of liquid flowing under gravity to pass through a calibrated glass capillary viscometer tube) (Alptekin and Canakci, 2008). Flash point of OLP was determined by pouring 100 mL of OLP into the flash cup and the lighted test flame was passed along the circumference of the cup. When a flash appeared at any point on the surface of the sample, the temperature was recorded and that temperature was considered as flash point according to ASTM D 92. The calorific value of the samples were determined using bomb calorimeter in which about 0.5–1 g of each sample was taken and kept in a crucible. When the temperature became constant, the bomb was fired and successive readings of temperature rise was recorded to calculate the calorific value according to ASTM D 2015-85.

Fourier Transform Infrared Spectroscopy (FTIR) (*Shimadzu Model: IR Affinity1*) was used for the identification of functional groups present in WCO and OLP with the aid of attenuated total reflectance (ATR). In FT-IR, the samples were analysed in the mid IR region of 400–4000 cm⁻¹ with 16 scan speed. FTIR spectrum of the samples were used for further analysis. The FTIR analysis of the catalyst was done in the same procedure by using potassium bromide (KBr) plate.

Nuclear magnetic resonance spectrometer analyses (NMR) of WCO and OLP were performed on *Bruker Ascend™ 600 MHz*, to know the chemical shifts. The calibration of signals were done by *Mestere Nova* software and chemical shifts were reported in ppm using solvent proton signal as a standard in which chemical shift of CDCl₃ at 7.2 ppm was taken as a reference.

Further, gas chromatography-mass spectroscopy (GC-MS) (*model no: 450-GC, 240-MS; make: M/s Varian*) was used for identification of organic fractions present in OLP sample. Capillary column (VF-5 ms) of dimension 50 m × 0.2 mm (0.33 μm film thickness) was used. Nitrogen was used as a carrier gas under the following temperature conditions. The oven temperature range from 70 °C (2 min) to 300 °C (7 min) at a heating rate of 10 °C/min with injector temperature of 250 °C and holding time of 3 min. The ion source temperature was set at 300 °C; MS detector was operated in the mass scan range of 50–550 atomic mass unit (amu) and the spectra were taken at 70 electron volt (eV). The

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