



Influence of operating variables on the transesterification of waste cooking oil to biodiesel over sodium silicate catalyst: A statistical approach

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Abstract

This study examined the use of surface response methodology to investigate the influence of operating variables on the transesterification of waste cooking oil (WCO) to biodiesel over sodium silicate catalysts. The individual and interactive effects of three variables namely, reaction time, reaction temperature and amount of catalyst was evaluated using full 2^3 (+1) factorial design. The conversion of WCO to biodiesel was achieved through the transesterification reaction over the catalyst at a methanol-to-oil molar ratio of 6:1 in a batch reactor. Physicochemical properties of the sodium silicate catalyst were obtained using Fourier transform infrared spectroscopy (FT-IR) for surface chemistry, thermo-gravimetric analysis (TGA) for thermal stability, N_2 physisorption test for Brunauer–Emmett–Teller analysis and scanning electron microscopy (SEM) for morphology. The reaction temperature, reaction time and weight of the catalyst (expressed as a percentage of the amount of WCO) were varied to understand their effect on the yield of biodiesel via response surface methodology (RSM) approach. The BET analysis showed a surface area of $0.386 \text{ m}^2/\text{g}$ for the catalyst. Results from the transesterification reaction reveal that change in catalyst weight percentage had no considerable effect on the biodiesel yield and that there was no mutual interaction between the reaction time and catalyst weight percentage. The results also conveyed that the reaction temperature and reaction time were limiting conditions and a slight variation herein altered the biodiesel yield. The transesterification of WCO produced 57.92% maximum FAME yield at the optimum methanol to oil molar ratio of 6:1, catalyst weight of 2.5%, reaction time of 240 min and a reaction temperature of 64°C . The variance ratio, $VR < F_{value}$ obtained from the cross-validation experiments indicate perfect agreement of the model output with experimental results and also testifies to the validity and suitability of the model to predict the biodiesel yields.

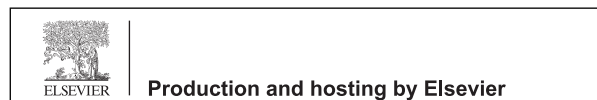
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Keywords: Biodiesel; Waste cooking oil; Transesterification; Heterogeneous catalysis

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1. Introduction

Combustion of fossil fuels has an immense detrimental effect on the environment. The release of pollutant gases such as NO_x, SO_x, and CO is inevitable during this process. In addition, the price instability of fossil fuels poses a serious threat to countries with limited resources. Taking into account the limited amount of energy resources, their increasing prices and environmental issues associated with the use of conventional fuels for energy, other means of producing energy sustainably have become the forefront of research. Amongst the studies for other means of providing sustainable energy is biodiesel, which is a branch of biofuels and has become an essential alternative for liquid fuels.

Previous studies have demonstrated the use of typical edible plant oils, such as soybean, rapeseed oil and palm oil for the production of biodiesel [1]. These raw materials are not entirely suitable, more especially in developing countries, due to the limited supply and high cost associated with their application, as well as competition with the food chain [2]. Therefore, low cost, non-edible oils such as jatropha oil, animal fat and waste cooking oil have been suggested and tested as alternatives [3–5]. However, the main disadvantage of these non-edible types of feedstock is the high content of free fatty acid (FFA) within the oils, which poses problems in the production process. Consequently, Biodiesel from high FFA content feedstock is conventionally produced by a two-stage process: esterification, followed by transesterification [6,7]. The esterification step serves to reduce the amount of FFAs present in the oil in order to allow for the transesterification reaction to commence [7,8]. The implication of this additional process unit is inevitably the additional costs associated with the biodiesel production.

In recent times, the use of heterogeneous catalysts has been proven to be very effective in converting high FFA feedstock directly to biodiesel, thereby by-passing the esterification stage [3,9,10]. The most commonly used heterogeneous catalysts for the production of biodiesel are ion-exchange resins, inorganic-oxide solid acids and supported noble-metal oxides. However, a dramatic decrease in the catalytic activity of these catalysts has been observed due to their absorption of water during biodiesel production [9]. Besides the sharp reduction in the catalytic activity, the catalysts can form a slurry with the products by absorbing water and carbon dioxide, thereby increases the viscosity of the product mixture, making product separation very difficult [11]. Guo et al. [11] reported that the use of sodium silicate as

solid catalyst suppresses the formation of soap because of the decreased water content (less than 4%). Guo et al. [3,12] demonstrated the excellent performance of calcined sodium silicate catalyst for the transesterification of soy bean oil to biodiesel. Sodium silicate has a high catalytic activity after calcination and is immiscible with triglycerides and alcohol [3]. During trans-esterification, hydrolysis reaction with sodium silicate and water resulted in the formation of NaOH and Si–O–H [3]. Furthermore, a yield of about 97% has been reported at catalyst amount of 7 wt.% and a methanol/oil ratio of 6:1 for the transesterification of soy bean oil to biodiesel using sodium silicate [12].

In spite of active research efforts in the development and use of heterogeneous catalysts for biodiesel production, only a few reports have been documented in literature related to the investigation of the influence of operating variables on the trans-esterification of waste cooking oil/soy bean oil to biodiesel over sodium silicate. Besides, most of these reports adopted a traditional approach whereby one variable is investigated at a time [12]. This approach overlooks the interactive effect of different variables on the results. Understanding these effects requires the use of an alternative approach. As a follow-up on our recent studies on the transesterification of WCO to biodiesel over calcined sodium silicate [13], in this article the use of response surface methodology (RSM) approach to investigate effect of operating variables is presented. The variables considered were reaction time, amount of catalyst and reaction temperature while the alcohol-to-oil ratio was fixed at 6:1 following report from [12].

2. Materials and methods

2.1. Determination of the free fatty acid (FFA) content of the oil

The waste cooking oil was obtained from a food vendor at the University of the Witwatersrand, and its FFA content in WCO was determined to confirm the need for a heterogeneous catalyst such as sodium silicate. It was also required to prove the tolerance of sodium silicate to a high content of free fatty acid in biodiesel production. The free fatty acid (FFA) content of the WCO was evaluated according to the procedure described elsewhere [13]. A 1.0 ml of the WCO diluted with 10 ml of 99% isopropyl alcohol was titrated against 0.025 M NaOH solution dropwisely using phenolphthalein solution (0.05 g of phenolphthalein to 50 ml of 95% pure ethanol and diluted to a 100 ml using distilled water)

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