



Ultrasound-assisted biodiesel production from waste cooking oil using hydrotalcite prepared by combustion method as catalyst



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ABSTRACT

Ultrasound-assisted biodiesel production from waste cooking oil catalyzed by hydrotalcite (HT) catalyst prepared using combustion method was studied. Two important parameters in the HT synthesis i.e., calcination temperature (550–850 °C) and fuel type (saccharose, glucose and fructose) were particularly investigated. The dependence of HT's characteristics on the synthesis parameters and correlations with their catalytic performance under ultrasound condition were successfully elucidated. The HT catalyst prepared using saccharose and calcined at 650 °C was the best catalyst to be used in the transesterification reaction. It showed high biodiesel yield (about 76.45%) in just 60 min in the presence of low ultrasound amplitude (~11 kHz). The enhancement effect of ultrasound was successfully demonstrated. The reaction only needed short reaction time (about 1 h) to give a biodiesel yield of up to 76.45% compared to conventional stirring method that needed about 5 h to achieve the same yield.

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1. Introduction

Sustainable energy supply in the future is of great concern due to the depletion of petroleum fuel resources. The quest for replacement fuel is currently underway. The use of vegetable oil as biodiesel feedstock provides interesting alternative as sustainable carbon cycle and environmentally friendly objectives are guaranteed. Meanwhile, the enhancement of biodiesel production process can be achieved through the use of sophisticated equipment such as membrane reactor, reactive distillation column, high frequency magnetic impulse cavitation reactor, centrifugal contact separator and ultrasound-assisted system [1]. However, mature understanding and the full potential of these systems are yet to be achieved. Development of advanced technologies that can stimulate an economical biodiesel production is highly needed. Therefore, it has motivated this present study on the use of an efficient and novel catalytic reaction system for biodiesel production.

Feedstock is an important aspect that can affect the economic viability of biodiesel production process. Atabani et al. [2] have reviewed various feedstock consisting of both edible and non-edible oils suitable for biodiesel production. However, edible feedstock might face the fuel vs food dilemma as well as the deforestation issue [3]. Meanwhile, non-edible feedstock usu-

ally encounter limitations with regards to impurities that can affect the production process [4]. However, such problems motivate researchers to come out with new inventions and solutions. Research on waste cooking oil as the non edible feedstock has enticed many researchers to come up with new ideas [5–7] to ensure the future relevance of biodiesel industry. In line with that, the present study evaluates the prospects of waste cooking oil in a positive way for commercial use in biodiesel industry. However, the use of waste cooking oil as feedstock is rather challenging as it basically contains high concentrations of 2 major impurities i.e., free fatty acid and moisture. However, with the available technology, the problem can be solved with several pre-treatment techniques and the use of specific types of catalyst [8,9]. Based on such concerns, this work has been designed to demonstrate an innovative and suitable catalytic reaction system that could possibly address the challenges of using waste cooking palm oil as the feedstock.

Biodiesel production process has been successfully intensified with recently developed processing technologies including pyrolysis, dilution, micro-emulsion, transesterification and esterification [2]. In recent years, most of the studies have been focusing on the role of heterogeneous catalysts in transesterification or esterification reaction. The conventional production processes of biodiesel i.e., through the use of homogeneous acid and base catalysts such as sulfuric acid and potassium hydroxide have been successfully performed [10–13]. The main drawback experienced when dealing with homogeneous catalyst with regards to the product separation has enticed the study on the application of heterogeneous catalysts

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such as CaO, hydrotalcite, dolomite, silica, SiO₂ etc. [14] which are sufficiently active and the need for further product purification can be avoided.

Conventional biodiesel production processes based on heterogeneous catalysis still show some drawbacks with regards to long processing time and poor rate of reaction. Many factors should also be considered when dealing with the immiscible reactants i.e., oil and alcohol. The reactants usually experience mass transfer limitation and emulsification problem so that longer time is needed for the reaction to complete. These problems can be effectively solved by conducting the biodiesel production process in the presence of ultrasound and solid catalysts. Badday et al. [15,16] have performed various studies on the enhancement of biodiesel processing using an ultrasound system. The reaction period was efficiently shortened to 60 min and the function of ultrasound had been technically elucidated. Poor mass transfer and emulsification limitation are no longer the issues toward achieving high transesterification rate to achieve high yield of biodiesel. Super vigorous effect due to high mechanical agitation provided by ultrasound can facilitate the contact between oil and alcohol. Besides, the boundary limitation between both reactants can be eliminated to force the reaction to occur mostly inside the catalyst pores. Therefore, this pivotal role of ultrasound could efficiently assist the catalytic reaction system and it is thoroughly addressed in this study.

Hydrotalcite (HT) is commercially recognized as an applicable catalyst in many reactions. HT generally possesses layered structure and its properties are often differentiated based on the composition with respect to the type of cation and the intercalated anion [17]. HT is predominantly basic in which the cations (usually Mg) in the layered structure belong to alkaline earth metals and the intercalated anions are basic (usually CO₃²⁻). Besides, HT catalyst is relatively robust where it shows no leaching problem during transesterification reaction [18]. These desired properties of HT justify its suitability to be used as high mechanical strength catalyst in an ultrasound-assisted system. In previously reported works, HT has shown good performances in catalyzing transesterification reaction for biodiesel production in which the highest FAME conversions and yields were recorded to be up to 77% [18,19] and 95.2% [20,21], respectively. HT might provide adequate hydroxyl and carbonate ions to create highly basic condition to initiate the transesterification reaction. The reaction starts when the carbonyl carbon of triglyceride is attacked by alkoxide ion from alcohol producing intermediates with the aid of hydroxyl ions from HT [22]. Then, further reaction between these intermediates would form methyl ester (biodiesel). The presence of sufficient ions provided by HT would speed up the reaction rate. Thus, by considering all the chemical and physical properties as well as the mechanistic advantages of HT, this catalyst has been chosen to be used in this study.

There are 8 general methods identified to be possibly used in preparing HT including co-precipitation, urea hydrolysis, hydrothermal treatment, combustion method, sol-gel, microwave irradiation, steam activation and solvothermal methods [23]. From all methods reviewed, the method that needs close attention is combustion method as significantly shorter preparation time is needed. In this study, by considering all advantages and disadvantages of each HT preparation method, combustion method has been selected. There are 2 major determinants that should be focused on in the combustion method which are the calcination temperature and fuel type. HT is activated by thermal decomposition and this stage is critical and needs to be emphasized. Suitable calcination conditions should be provided in order to achieve complete decomposition of interlayer anions and to avoid detrimental effects of periclase-like phases (MgO segregated phases) on the HT structure [24]. Heating of HT to a certain high temperature usually causes dehydration, decomposition of anions and segregation of oxides.

Fuel is needed in the synthesis of HT to initiate and influence the reaction. The types of fuel used in previously reported studies include sugars, glycine and urea [24–26]. The combustion process generally takes less than 5 min to complete and the reaction is actually based on the principle of explosive decomposition of fuel mixtures [25,26]. Fuel is important in producing CO₂ and H₂O compounds that are highly required for the formation of layered structure by means of various forms of probe molecules such as monodentate, bidentate and bridge [25,27]. Thus, a good preparation method would ideally result in proper layered structure of HT catalyst that is desirable in the transesterification reaction.

In this study, the production of biodiesel from waste cooking oil has been conducted in the presence of hydrotalcite as the catalyst coupled with the application of ultrasound to further enhance the transesterification reaction. Particular focus has been given to the capability of HT synthesized using combustion method. Two major important parameters i.e., calcination temperature and fuel type have been investigated. Effects of those parameters on HTs' characteristics and their catalytic performance (biodiesel yield) under ultrasound system have been elucidated. Comparison between ultrasound and conventional stirring methods has been technically performed in order to demonstrate on the benefits of an ultrasound-assisted system in producing biodiesel.

2. Experimental

2.1. Materials and reagents

Magnesium nitrate hexahydrate (Merck), aluminum nitrate nonahydrate (Ridel de Hein), sodium carbonate (Fischer Scientific), D-glucose (R&M Chemical), saccharose (Merck), fructose (Merck) were used to synthesize the hydrotalcite. Waste palm oil (from the campus's cafeteria) and methanol (Merck) were used as the reagents for the transesterification reaction. Meanwhile, *n*-hexane (Merck), methyl heptadecanoate (Fluka), fatty acid methyl esters (FAME) standards (Sigma and Fluka) were used for fatty acid methyl ester (FAME) analysis.

2.2. Catalyst preparation

Hydrotalcite was synthesized using combustion method in which the preparation of MgAlO oxide mixture was adapted from Anuar et al. [24] and Davilla et al. [25]. The Mg/Al ratio was maintained at 3. Pre-calculated amounts of magnesium nitrate and aluminum nitrate were first suspended in 80 ml of deionized water that was placed in two different beakers for better suspension and then heated to 80 °C. Then, it was thoroughly mixed in a beaker and kept at 80 °C. 20% wt of Na₂CO₃ and 10 wt% of fuel (sugars) from the total weight of sample were then added. Next, the mixture was stirred vigorously and maintained at 80 °C until all water had evaporated. The resulting solid was then calcined in a furnace for 5 min. Calcination of MgAlO was then carried out at 4 different temperatures i.e., 550 °C, 650 °C, 750 °C and 850 °C. The fuels used in the catalyst preparation were saccharose, glucose and fructose. The amount of fuel used was kept constant at 10 wt% of the total mixture. The effects on the catalytic reaction were studied. Then, the resulting MgAlO catalysts were ground into powder form and put in contact with 60 ml of 0.05 M anion solution for 5 min at room temperature. The solid products were then filtered and washed 2 times with deionized water and dried in an oven at 100 °C.

2.3. Catalyst characterization

The analysis of surface area was carried out using a Micromeritics ASAP 2020 V3.02 H surface analyzer based on nitrogen adsorption/desorption. X-ray diffraction (XRD) was used to study

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