



# Preparation, characterization, and modelling activity of potassium fluoride modified hydrotalcite for microwave assisted biodiesel conversion



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## ABSTRACT

In this paper, potassium fluoride (KF) modification of hydrotalcite (HT) and its activity as a solid base catalyst for microwave (MW) assisted biodiesel conversion was investigated. The catalyst was prepared simply and rapidly by the solid reaction of KF and HT, and characterization was performed using x-ray diffraction, surface profile analysis using gas sorption analyses, SEM, FTIR, and the determination solid basicity. The effect of KF content on the physicochemical character, yield, and turn over frequency (TOF) of the reaction is presented. A statistical optimization of the effects of the reaction parameters on yield using logit transformation regression showed that the reaction time and the KF content of the catalyst strongly affected the reaction's yield. The data shows the potency of the modified HT as a catalyst for biodiesel conversion. From the TOF data, it is suggested that the MW-irradiation method gives a better yield compared to the reflux method. It was also found that the catalysts are reusable.

## 1. Introduction

Biodiesel is still the most high potential energy resource substitute in developing countries. The main features of its potency are the renewable resources of plant oil and its simple production reaction procedure. Traditionally, biodiesel has been obtained from the transesterification of triglycerides using methanol over a basic catalyst of NaOH or KOH. However, further work is required for green and environmental efficiency reasons. Proposed schemes for creating a greener process is the use of a heterogeneous catalyst for the reaction so that the catalyst can be recycled and reused for more economical cost, and the use of other efficient energy sources for biodiesel production (Martins et al., 2013; Mittelbach, 2009). The use of NaOH as a homogeneous catalyst in the transesterification process has some advantageous, such as rapid reaction due to the miscibility of the reactant; however, NaOH being a non-recyclable catalyst is a disadvantage. Another problem faced by soluble, basic catalysts is the saponification reaction; plant oils contain high levels of free fatty acid. For these reasons, some solid base catalysts were simulated and applied (Rustamaji et al., 2011; Santos et al., 2009). Another captured technique for fast biodiesel production is the utilization of the microwave- (MW) irradiation method. The reaction can be conducted in a few minutes instead of hours for a targeted yield.

As catalyst alternatives, basic oxides of Ca, Mg, and Ba have been

reported as active in biodiesel conversion as well as some zeolites, clays, and silica-alumina, and its modified forms (Atadashi et al., 2013; Helwani et al., 2009). Within the clay scheme of materials, hydrotalcite (HT) is a well-known base material. HT, or layered double hydroxide (LDH), is an anionic and basic clay with the general formula  $(M^{2+}_{1-x}M^{3+}_x)(OH)_2]^{b+}[A_n^{b-}]_m H_2O$ , where  $M^{2+}$  is a divalent or monovalent cation and  $A_n$  is the interlayer anion. The basic properties of HT as a reusable catalyst in the transesterification of biodiesel production have been reported to be active and efficient. However, many attempts have been made to enhance the basicity and effectivity of HT as a catalyst; an increase in the basicity of the catalyst has been attributed to HT's increased intralayer electron density and pore volume by calcination and the surface modification of HT (Climent et al., 2004). Factors that determine the solid basicity, for the modification concept, include the Mg/Al mole ratio, the calcination temperature, and metal framework modification (Coriolano et al., 2017; Teng et al., 2010; Xie et al., 2006). Previous investigations have reported the effect of HT modification using potassium salts such as  $K_2CO_3$ , KI, and KF for the enhancement of biodiesel conversion (Guzmán-Vargas et al., 2015; Katekong et al., 2017; Teng et al., 2010). It was observed that impregnation with potassium generates basic surface sites and directly plays a role in increasing the catalytic activity in the transesterification mechanism. As a background for improving the effectivity and efficient production of biodiesel, this work aimed to study the effect of modified HT and the

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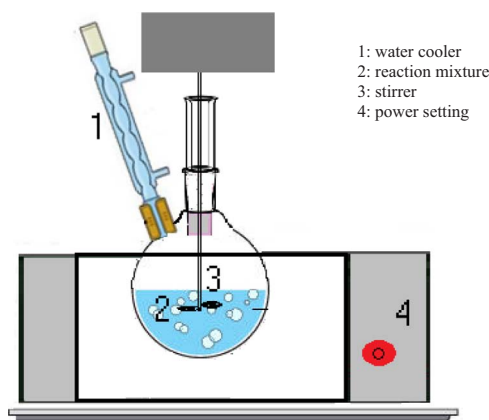


Fig. 1. Schematic representation of microwave reactor.

use of MW for biodiesel production. Although a similar studies to this have been reported, the effect of the KF content on the physicochemical character and catalytic activity in the modified HT has not been reported. Furthermore, it is also important to study catalyst reusability related to the catalyst's character.

## 2. Material and methods

HT (Sorbacite®) was obtained from Süd-Chemie Ltd., Munchen, Germany. Jatropha oil was obtained from PT. Cipta Kimia, Indonesia, and directly used without further purification. Chemicals used comprised methanol, KF,  $\text{NH}_4\text{OH}$ , citric acid, and n-hexane (Merck, Berlin, Germany).

Catalyst preparation was conducted by the solid reaction method. A mixture of KF and HT was ground for 20 min followed by heating to  $200^\circ\text{C}$  for 2 h. KF content varied from 5, 10, 20, and 30 wt% and the results obtained were designated KF5/HT, KF10/HT, KF20/HT, and KF30/HT, indicating the KF percentage, respectively. Characterization of the prepared catalysts was performed using x-ray diffraction (XRD), gas sorption analyses, pyridine FTIR, and solid basicity measured by the titration method. XRD (X6000; Philips) with Ni-filtered  $\text{Cu-K}\alpha$  radiation was utilized with a step size of  $0.4^\circ$ . For surface profile analysis, a NOVA 1200e instrument was used, and FTIR analyses were conducted using a Perkin Elmer ATR instrument with  $4\text{ cm}^{-1}$  resolution over a scanning range of  $400\text{--}4000\text{ cm}^{-1}$ .

Solid basicity was determined by the potentiometric titration method. The sample was mixed with citric acid under stirring for 24 h, and unreacted citric acid was measured by titration with  $\text{NH}_4\text{OH}$ . Solid basicity was defined as the total citric acid adsorbed by the surface of the catalyst (mg/g).

The biodiesel conversion reactions were performed using the reflux method and the MW-irradiation method. For the MW-irradiation technique, a modified, commercial MW oven was utilized as shown in the schematic presented in Fig. 1. The machine worked at a frequency of 2.45 GHz and the power output was from 0 to 900 W. The reaction parameters, the catalyst, the methanol to oil molar ratio, and the reaction time, and the reaction method uses were studied. From each experiment, the reaction result was separated using a separating funnel and the biodiesel fractions were analyzed with a gas chromatography mass spectrometer (GCMS) (Shimadzu) equipped with a capillary column (TG-5MS). The condition of GCMS analysis is as follow: column oven temperature of  $80.0^\circ\text{C}$ , injection temperature of  $300.00^\circ\text{C}$ , column flow of  $1.24\text{ mL/min}$ , and the split ratio of 59.1.

Testing of the catalysts' activities was conducted by identifying the influencing parameters such as the methanol to oil molar ratio, catalyst loading, irradiation time, and the identification of the applied recycled catalyst.

Eqs. (1) and (2) were used to calculate the yield (%) and the turn

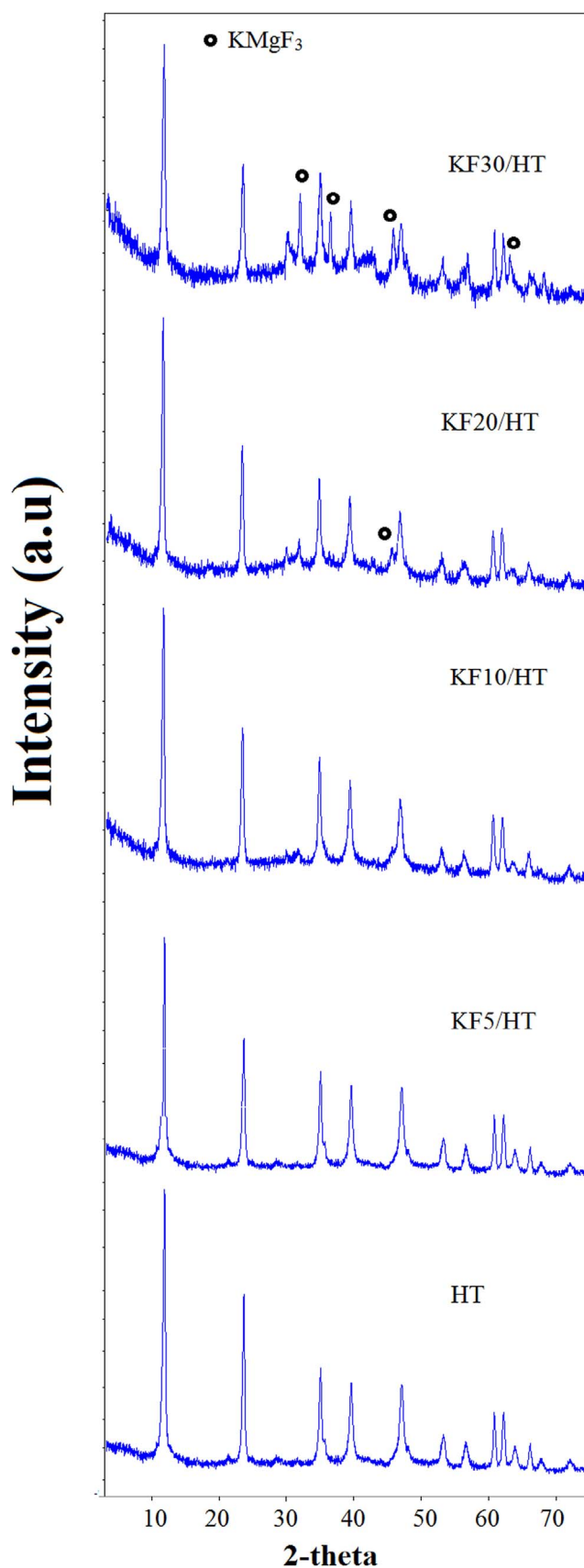


Fig. 2. XRD pattern of prepared catalysts.

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