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Effect of Cu-MOF Catalyst Impregnated with K_2O to Enhance Pyrolytic Catalytic Cracking of Waste Cooking Oil for Biofuel Production

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ABSTRACT

Waste oil can be converted to fuel through complex processes due to its high impurity level and significant oxygen content. This research explores the use of pyrolysis together with metal-organic frameworks (MOFs) to enhance the conversion of waste oil. MOFs, porous crystalline materials formed by linking metal ions with organic linkers, provide moderate surface area and increased active sites for catalytic applications, especially in bioproduction. In this study, copper-based MOFs (Cu-MOF) were synthesized via a room temperature method using $CuSO_4$-$5H_2O$ and 2-methylimidazole, followed by wet impregnation with K_2O. The synthesized catalysts were characterized using FTIR, SEM, and BET and the biofuel production using GC-MS. The characterization results showed that K_2O impregnation enhanced the stability of MOF structure and significantly improved thermal stability as well as made it more efficient for the pyrolytic catalytic process for biofuel production. FTIR analysis confirmed the successful impregnation of K_2O, SEM analysis showed that the Cu-MOF particles did not have a clear crystal shape. From BET analysis, there is a decrease in surface area due to K_2O impregnation. Gas chromatography-mass spectrometry (GC-MS) confirmed that the pyrolytic reaction occurred by using Cu-MOF/K_2O catalyst can significantly increase the hydrocarbon compounds of the waste cooking oil to 39.25% as well as decrease the oxygen content contained in the oil to 56.65% according to the reaction in the pyrolytic catalysis cracking process. Importantly, the catalyst showed regeneration potential after use, proving its feasibility for repeated application in waste oil conversion processes.

Introduction

Metal-organic frameworks (MOFs) are versatile heterogeneous materials gaining attention due to their wide-ranging applications in energy storage, catalysis, and electromagnetism [1]. These porous crystalline structures are formed by linking metal ions with organic ligands, offering a significant surface area and active sites suitable for catalysis [2]. These properties make MOFs particularly promising for biodiesel production. Specifically, copper-based MOFs have shown great potential as heterogeneous catalysts for alcohol oxidation, which could serve as an effective pathway for achieving more stable biodiesel production. In biofuel research, numerous catalysts are employed in the transesterification process, including MOF-808, Mo-MOF, Zr-based MOFs, MOF-5, CuBTc-MOF, as well as copper- and calcium-based MOFs like BZnFMO [3]. Given the oxygenated hydrocarbons in biofuels and their growing significance in energy, economic, and environmental sectors, biofuel production has

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become increasingly critical. However, before biofuels can be effectively used, these oxygenated compounds need to be broken down into shorter hydrocarbon chains. While MOF-based catalysts have shown potential, their application in biodiesel synthesis is still underexplored. Therefore, this study seeks to investigate the use of MOF-based catalysts, particularly Cu-MOF, for biodiesel production from waste cooking oil (WCO). Previous research by Buchori et al. [4], examined the doping of CaO-ZnO catalysts with varying concentrations of K_2O (1%, 3%, and 5% by weight) and found that increased K_2O concentrations enhanced the basicity of the catalyst, leading to better oxygen removal and yielding hydrocarbons like alkanes and alkenes. Building on these insights, this study explores the doping of K_2O onto Cu-MOF catalysts to optimize the pyrolysis process for biofuel production.

The pyrolysis setup used in this study includes a catalytic bed reactor and an integrated condenser. The catalytic bed serves as the primary site where waste cooking oil is thermally decomposed into smaller fractions under oxygen-free conditions. The catalyst within the reactor is critical in accelerating the reaction and improving selectivity towards biogasoline. After pyrolysis, the resulting vapor is directed to the condenser, where it is cooled into a liquid phase. This integrated system enables efficient separation and collection of biofuel, particularly biogasoline. The reactor-condenser integration is key to achieving an efficient conversion process from waste oil to renewable fuel. A similar study by Ramli et al [5] focused on pyrolysis of plastics using a reactor integrated with a bubble cap distillation column, demonstrating reduced energy consumption through vapor-phase condensation on each tray.

This research aims to explore the catalytic conversion of waste cooking oil into biofuel using Cu-MOF catalysts doped with potassium oxide (K_2O). The novelty of this study lies in the use of Cu-MOF in the catalytic cracking of WCO, a catalyst that has not been extensively studied for this purpose. By doping the Cu-MOF with K_2O, the catalyst's performance is expected to improve, particularly by enhancing its basicity and efficiency. Comprehensive characterization of the catalyst was performed to identify optimal conditions for the catalytic cracking process. Furthermore, a customized pyrolysis system integrated with a condenser was developed for efficient conversion of WCO into valuable biofuel products. This study hypothesizes that Cu-MOF doped with K_2O will increase catalytic activity, yielding higher hydrocarbon products and reducing oxygen content in the biofuel, thus enhancing its quality. The exploration of Cu-MOF doped with K_2O for pyrolytic catalytic cracking of WCO represents a novel and in-depth contribution to renewable biofuel research, filling a gap in current literature regarding its catalytic efficiency and mechanistic pathways.

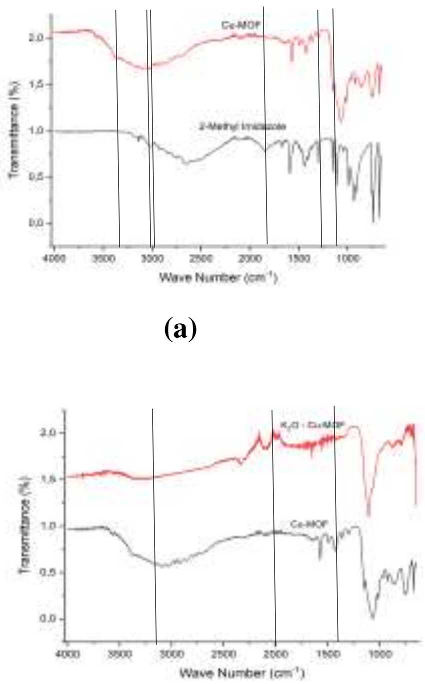
Methods

All the chemicals such as copper (II) sulphate anhydrous ($CuSO_4.5H_2O$), methanol (MeOH) (Sigma, 99%), 2-Methylimidazole (Sigma, 99%), pottasium acetate (CH_3COOK) (Sigma, 99%), and deionized (DI) water were purchased from Sumber Ilmiah Persada (Surabaya, Indonesia) and used without further purification. Waste cooking oil (WCO) as raw materials were collected from street vendors in Surabaya, Indonesia.

Cu-MOF was synthesized using a room temperature method with a metal-to-organic molar ratio of 1:2 ($CuSO_4\cdot5H_2O$ and 2-Methylimidazole). To create solution 'A,' $CuSO_4\cdot5H_2O$ was dissolved in distilled water, while solution 'B' was prepared by dissolving 2-Methylimidazole in methanol. Both solutions were stirred at 300 rpm at ambient temperature overnight, leading to the formation of a precipitate. This precipitate was then separated by centrifugation at 5000 rpm for 5 minutes and dried at 100°C overnight to produce Cu-MOF. Potassium loading on the Cu-MOF catalyst was carried out using the wet impregnation technique. A potassium solution was prepared by dissolving CH_3COOK in deionized water, based on an incipient wetness volume of 5 mL/g of Cu-MOF. The solution was added slowly to the catalyst under continuous stirring, followed by drying at 80°C overnight and calcination at 500°C for 5 hours to achieve K_2O-loaded Cu-MOF.

Results and Discussions

In order to understand the bonding interaction between Cu and 2-methylimidazole in all samples, FTIR (Fourier-transform Infrared Spectroscopy) analysis was performed. Figure 1 displays the FTIR spectra of the synthesized Cu-MOF and pure 2-methylimidazole at room temperature. The analysis was carried out at the Water Technology and Industry Consulting Laboratory (TAKI), Department of Chemical Engineering, Sepuluh Nopember Institute of Technology (ITS). Peaks observed between 650 and 1500 cm^{-1} correspond to the stretching and bending modes of the imidazole ring, characteristic of 2-methylimidazole [6]. The peak at 1578 cm^{-1} is assigned to the C=N stretching mode of 2-methylimidazole, while those at 2920 and 3131 cm^{-1} correspond to C-H bond stretching modes in the aliphatic hydrocarbon chain and the aromatic ring of 2-methylimidazolate [7]. Distinct peaks at 1116 cm⁻¹ and 1250 cm⁻¹ are attributed to C-O stretching vibrations, characteristic of the MOF structure [8]. Additionally, a band around 728 cm⁻¹ is indicative of Cu-O stretching vibrations, resulting from the coordination of oxygen atoms with Cu atoms, confirming the successful formation of Cu-MOF [9]. The pronounced peak near 3400 cm⁻¹ suggests O-H stretching vibrations, possibly from hydroxyl groups or adsorbed water [10].



(a)

(b)

Figure 1 FTIR spectra of 2-Methylimidazole(a), Cu-MOF(a), and Cu-MOF/K_2O(b)

Following the successful formation of Cu-MOF, the catalyst was impregnated with K_2O to enhance its catalytic efficiency are presented in Figure 1(b). A shift in the vibration peaks associated with the imidazole ring is observed in the spectral region between 1200 cm⁻¹ and 1600 cm⁻¹, with a prominent peak at 1400 cm⁻¹, indicating successful K_2O impregnation.

Brunauer, Emmett, and Teller (BET) analysis was used to assess the specific surface area, pore volume, and size of the materials. The physical properties of Cu-MOF and Cu-MOF/ K_2O obtained through N_2 physisorption analysis, as shown in Table 1. Cu-MOF's surface area decreases to 371.514 m²g⁻¹ after potassium oxide impregnation. The reduction in surface area is likely due to the blockage of small pores by potassium oxide particles [11] and accompanied by a reduction in pore volume after impregnation with potassium oxide particles. The pore size increases from 3.7636 nm to 3.9665 nm after impregnation with 1% potassium oxide, indicating that Cu-MOF undergoes pore restructuring, resulting in an increase in pore diameter [12].

Table 1. The nitrogen adsorption–desorption method was used to examine catalysts

Catalyst sampel	**Spesific surface area (SA_{BET}) (m^2g^{-1})**	**Pore volume (V_{pore}) (cm^3g^{-1})**	**Pore diameter (D_{Pore}) (nm)**
Cu-MOF	435.123	0.4094	3.7636
Cu-MOF/K_2O	371.514	0.368	3.9665

Previous studies on potassium oxide impregnation on dolomite indicated that increasing the concentration of potassium oxide from 15% to 20% led to a decrease in the basic site capacity of the catalyst. Potassium oxide impregnation leads to a decrease in surface area and indicates a saturation limit, the results still demonstrate that potassium oxide impregnation can enhance catalytic activity due to the introduction of new active sites [13]. The experimental results show that using imidazole as an organic solvent in the synthesis of Cu-MOF yields a specific surface area of 435 m²g⁻¹, significantly higher than the study [14], which used dimethylformamide (DMF) and reported a surface area of 118 m²g⁻¹.

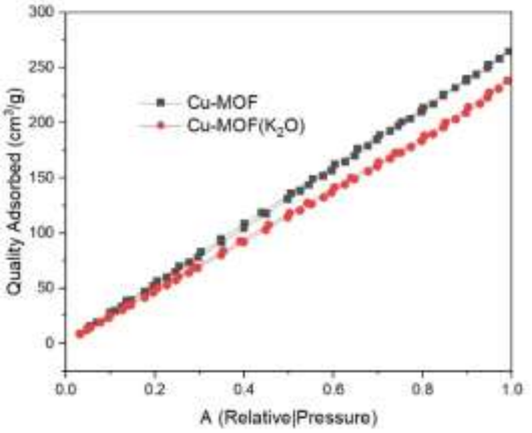


Figure 2 Nitrogen sorption isotherm

For further clarity, Figure 2 illustrates the adsorption and desorption isotherm peaks of nitrogen from the synthesized catalysts. Both Cu-MOF and Cu-MOF/

K_2O exhibit hysteresis, indicating that both catalysts possess mesoporous materials.

The surface morphologies of the Cu-MOF catalyst were examined using Scanning Electron Microscopy (SEM) at 50,000x magnification. Figure 3a, representing Cu-MOF/K_2O, exhibits a more homogeneous and less intricate surface structure. The particles appear discrete and well-defined, suggesting minimal agglomeration. This uniformity serves as a baseline morphology for comparative analysis of K_2O impregnation effects. Figures 3b, c, and d demonstrate of K_2O impregnation effects, its enhanced surface irregularities and porosity indicate that K_2O dopping significantly alters the material's morphology. These morphological changes potentially increase the surface area and number of active sites, which could enhance catalytic activity - a beneficial attribute for applications such as gasification or catalysis.

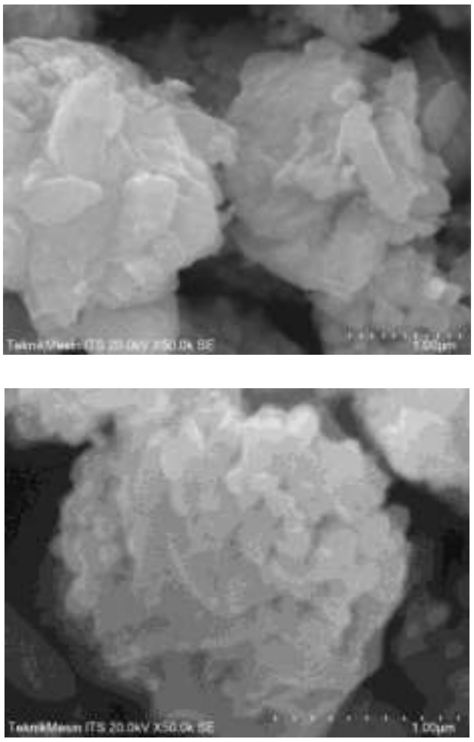


Figure 3 SEM images of (a) Cu-MOF, (b) Cu-MOF/K_2O

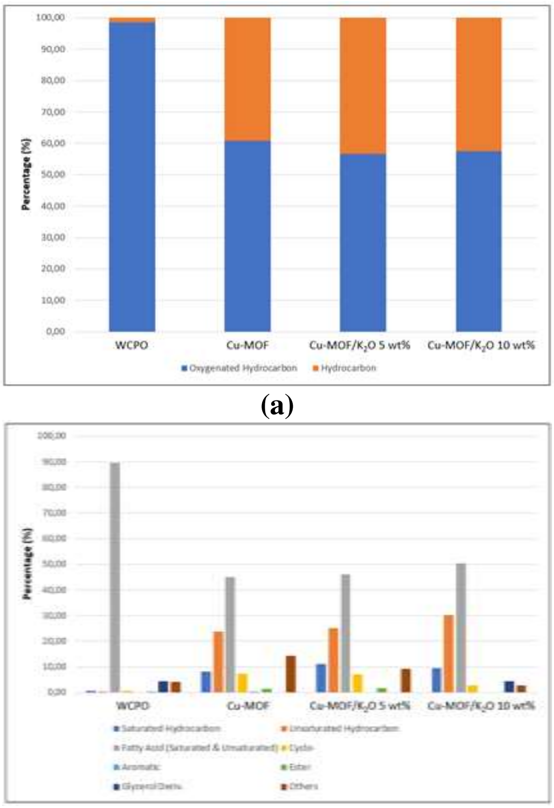
Waste cooking oil from palm oil (WPCO) can be converted into petroleum-like products such as gasoline, kerosene, and diesel through pyrolytic catalytic cracking. WPCO is rich in fatty acids, primarily palmitic acid (C_{16}) and oleic acid (C_{18}), which are present in a triglyceride structure derived from palm oil. Due to its high kinematic viscosity and elevated acid value, WCO is often considered low-quality oil. These properties can negatively impact engine performance, leading to problems such as increased smoke emissions, carbon deposition, and incomplete combustion. Moreover, the acidity of WCO poses a risk of corrosion to both engines and storage systems [15]. The pyrolytic cracking of WCO begins at approximately 300°C and is completed around 600°C, with significant hydrocarbon weight loss typically occurring between 300°C and 500°C [16]. A common operating temperature range is 350°C to 450°C, which optimizes the production of lighter hydrocarbons, including those beneficial for biodiesel synthesis. According to [17], the biofuel production process from off-grade crude palm oil operates in two stages: an initial reaction at 300°C followed by a second stage at 400°C, at which the cracking of hydrocarbons occurs. Based on these findings, a reaction temperature of 400°C was selected for this study.

The catalytic pyrolytic cracking process occurs in two stages. In the first stage, the waste cooking oil undergoes thermal decomposition of triglyceride and fatty acid molecules, resulting in the formation of oxygenated products. In the second stage, these oxygenated products react through cracking over the catalyst to form hydrocarbon compounds [18]. Three concurrent reaction mechanisms occur in the gas and liquid phases namely hydrodeoxygenation (HDO), decarbonylation (DCO), and decarboxylation (DCO_2) [19]. H_2 can be produced from the breakdown of tar into gaseous components and through the water-gas shift reaction [20]

The presence of K_2O in the catalyst introduces more basic sites, which help reduce the oxygen content in the pyrolytic oil. This is evident from the data, where the oxygenated hydrocarbon compounds decreased from an initial 98.47% in waste cooking oil to 60.75% for pyrolytic oil produced with Cu-MOF, and Cu-MOF/K_2O. Hence, it can be concluded that Cu-MOF/K_2O catalysts are more effective and efficient in reducing oxygen content during the catalytic pyrolytic cracking process compared to Cu-MOF alone.

The GC-MS chromatogram (Figure 4b) shows that the primary components of the pyrolytic oil are hydrocarbons, including alkanes, cycloalkanes, and aromatic compounds, with significantly reduced concentrations of oxygenated compounds. In contrast, the waste cooking oil is primarily composed of 89.56% carboxylic acids, 0.37% esters, and 4.30% glycerol derivatives, which contribute to its elevated acidity. The use of the Cu-MOF/1-K_2O catalyst led to a marked decrease in oxygenated compounds (Figure 4a) from 94.23% to 46.31%, along with an increase in hydrocarbon content to 43.35%, as compared to 39.25% when using the Cu-MOF catalyst alone.

These results align with previous studies, such as those by [21], which similarly identified aliphatic and aromatic hydrocarbons as the dominant components in pyrolytic oil. The superior performance of the Cu-MOF/K_2O catalyst is likely due to its enhanced basicity, which promotes more efficient catalytic cracking and deoxygenation, making it significantly more effective for pyrolytic catalysis than Cu-MOF alone.



(a)

(b)

Figure 4 GCMS Results (a) classification based on oxygenated compounds (b) components of OLP

Conclusions

This study investigates the conversion of used cooking oil into biofuel using a Cu-MOF/K_2O catalyst. The physical and chemical properties of the catalyst were characterized through various analytical techniques, demonstrating that K_2O was successfully doped onto Cu-MOF, as confirmed by FTIR analysis, SEM analysis revealed no significant crystalline structures in the catalyst, while the incorporation of K_2O increased the surface area and neutralized acid sites. The used of K_2O enhanced catalytic efficiency, contributing to improved biofuel production through pyrolytic catalytic cracking (PCC).The use of the best catalyst for biofuel production in this study was Cu-MOF/1-K_2O as evidenced by the results of GC-MS analysis which showed a reduction of fatty acids from used cooking oil from 89.56% to 45.92% and caused hydrocarbons to increase to 43.35% as well as a decrease in oxygen content from 98.47% in used cooking oil to 56.65%. This study shows that Cu-MOF/K_2O catalyst is highly effective and efficient for biofuel production via pyrolytic catalysis, although further research is needed to optimize the catalyst composition for maximum performance.

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Author Contributions

Conceptualization, Tri Widjaja and Joni Prasetyo; methodology, Ali Altway and Hendro Yuwono; software, X.X.; validation, Deliana Dahnum, and Dinda Amelia Nurhanifa; formal analysis, Nadiyah Salsabil Zahidah; investigation, Shofia Khoirunnisa' ; resources, Shofia Khoirunnisa'; data curation, Dinda Amelia Nurhanifa; writing—original draft preparation, Tri Widjaja; writing—review and editing, Ali Altway; visualization, Dinda Amelia Nurhanifa; supervision, Nadiyah Salsabil Zahidah; project administration, Shofia Khoirunnisa'; funding acquisition, Tri Widjaja. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest and the funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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