An Investigation of Biofuel Production of Kapok Seed Oil Using Bimetal Supporting HZSM-5 Catalyst: Effect of Reaction Temperature by Catalytic Hydrocracking Process

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Abstract

The study aims to investigate the effect of reaction temperature variations on the catalytic hydrocracking process of kapok seed oil to produce biofuel using the CuMo/HZSM-5. The CuMo/HZSM-5 used has a CuMo loading of 5% on the mass of HZSM-5 as support. The catalyst is prepared through the impregnation method, Incipient Wetness Impregnation. Characterization of catalysts was analysed by X-ray Diffraction (XRD), BET, and EDX. Based on the results of EDX analysis denoted that Cu and Mo metals can be impregnated into the HZSM-5. This was indicated by detecting 1.45 wt% Cu metal and 7.46 wt% Mo metal. The catalytic hydrocracking process converted kapok seed oil into biofuel by adding hydrogen gas using a pressurized reactor equipped with a stirrer. This process occured at temperatures of 350-400^{o}C, 10-30 bar for 2 hours. Parameters used to investigate biofuel production, including the composition of hydrocarbons and yield of gasoline/kerosene/gas-oil range alkane. The gas chromatography-mass spectrometry (GC-MS) method analysed the liquid biofuel products. The best performance of CuMo/HZSM-5 was bio-kerosene (2.82%) and bio-gasoil (17.11%) for 2 hours at 400^{o}C. The selectivity of liquid hydrocarbon products was n-paraffin (50.12%) and cycloparaffin (32.57%).

Keywords: *Hydrocracking, Kapok Seed Oil, Bimetal Catalyst, CuMo/HZSM-5, Biofuel.*

Abstrak

Penelitian ini mempelajari pengaruh dari variasi temperatur reaksi pada proses catalytic hydrocracking minyak biji kapuk untuk memproduksi biofuel menggunakan katalis CuMo/HZSM-5. Katalis CuMo/HZSM-5 yang digunakan memiliki loading logam terhadap massa HZSM-5 Sebagian *support. Katalis tersebut disiapkan melalui metode impregnasi yaitu Incipient Wetness Impregnation. Karakterisasi katalis menggunakan metode X-ray Diffraction (XRD), BET, dan EDX. Berdasarkan hasil analisis EDX menunjukkan bahwa logam Cu dan Mo dapat terimpregnasi ke HZSM-5. Hal ini ditunjukkan dengan terdeteksinya logam Cu sebanyak 1.45 wt% dan logam Mo sebanyak 7.46 wt%. Proses catalytic hydrocracking digunakan untuk mengkonversi minyak biji kapuk menjadi biofuel dengan penambahan gas hidrogen menggunakan reaktor bertekanan yang dilengkapi dengan pengaduk. Proses tersebut terjadi pada temperature 350-400^oC, 10-30 bar selama 2 jam. Parameter yang digunakan untuk menginvestigasi produksi biofuel, termasuk komposisi hidrokarbon dan yield gasoline/kerosene/gas-oil range alkane. Produk cair biofuel dianalisis menggunakan metode gas chromathography-mass spectrometry (GC-MS). Performa katalis CuMo/HZSM-5 terbaik untuk memproduksi biofuel diperoleh bio-kerosene (2.82%) dan bio-gasoil (17.11%) selama 2 jam reaksi pada temperatur 400^oC. Selectivitas produk cair hidrokarbon untuk n-paraffin (50.12%) dan cycloparaffin (32.57%).*

Kata Kunci: *Hydrocracking, Minyak Biji Kapuk, Katalis Bimetal, CuMo/HZSM-5, Biofuel.*

1. Introduction

The overall energy consumption has increased but has yet to be followed by the number of fossil fuels that continue to decline, so it requires alternative fuels that can meet energy needs in line with the population growth rate. Production of petroleum/fossil oil in Indonesia from 2005-2014 continued to experience a decline. in 2005 BP Global is capable of producing 1,090 thousand barrels per day (BPD), but in 2014 it was only able to produce 852 thousand BPD. That matter was exacerbated by unfulfilled domestic demand so Indonesia imported around 350,000 to 500,000 barrels of fuel. Oil consumption in Indonesia in 2005 as many as 1,299 BPD and continues to experience the increase until 2014 reached 1.641 thousand BPD (*BP Statistical Review of World Energy 2015 and SKK Migas 2015*). Alternative energy is energy where raw materials can be minimized so that they can be continuously produced. One alternative fuel is biofuel. It can be renewable, biodegradable, environmentally friendly, non-toxic, and economical. The potential of raw materials for biofuel production is from vegetable oil. First-generation biofuels are edible vegetable oils, but they are still debated because they belong in the food oil category. Therefore, vegetable oil or second-generation raw material is an alternative to biofuel production. One of the non-edible oil seeds for biofuel is kapok seed oil. The vegetable oils are extracted from seeds and mixtures of plants and mixtures of other organic compounds contained therein. It usually consists of 98% triglycerides and monoglycerides, diglycerides, and free fatty acids of long-chain carboxylic acids. Kapok seed oil (*Ceiba pentandra*) contains as much as 24-40wt% oil in the seeds and thus has the potential to be an alternative raw material for biofuel production (Atabani et.al., 2013).

Generally, biofuel production is based on vegetable oils via the transesterification or esterification process. The transesterification process uses an alkali catalyst to get a high yield, and the proper methanol/oil ratio is needed at moderate temperatures. The process needs to improve because of the need for large methanol. A more exciting technology has been developed that uses solid heterogeneous catalysts but still has disadvantages because it requires high temperatures to convert vegetable oils into biofuel. Catalytic hydrocracking technology is one of the alternative advanced technology for the transesterification process. Catalytic hydrocracking is one of the alternative technologies for producing biofuels (Bezergianni and Kalogianni, 2009). Catalytic hydrocracking is a technology that combines cracking and hydrogenation reactions using solid heterogeneous catalysts to produce saturated compounds. The catalyst used is acidic, i.e., in the form of amorphous (alumina-silicate), silicoaluminophosphates (SAPO), and crystal (zeolite) (Ancheyta et.al., 2009).

This work investigated biofuel production with hydrocracking of kapok seed oil using CuMo/HZSM-5 catalyst and the effect of reaction temperature parameters. In the previous study, Cu-HZSM5 is used for the hydrogenation of aromatic aldehydes in a fixed-bed reactor. Cu can as the main active species for the hydrogenation process in biofuel production (Mirzayanti et.al., 2019). This study focuses to used CuMo/HZSM-5 as a dual promotor to produce biofuel with gasoil-range alkanes. It has affected the distribution of product yield, selectivity, and conversion of liquid products such as bio-gasoline, bio-kerosene, and bio-Gasoil over CuMo/HZSM-5 catalyst. Preparation of CuMo/HZSM-5 catalyst used incipient wetness impregnation method. On wet impregnation, add the amount of active phase precursor solution greater than 1.5 times the volume of buffer pore. This method can produce active phase precursor deposition very much on the outside of the support after the drying process and also produces the distribution of the active phase on the outside of the support. Distribution this is beneficial to reduce the penetration of the reactants into the catalyst, so can increase the catalytic activity. While the dry impregnation method was carried out by adding less than 1.2 volume of active phase precursor solution buffer pore. The method commonly used in the manufacture of catalysts is wet impregnation. This is done because the process is easier the process but in wet impregnation, it requires heating and a relatively long time (Harber et al., 1995). Characterization of the catalyst was performed by X-ray diffraction (XRD), Brunauer-Emmett-Teller (BET), and scanning electron microscopy (SEM)-energy dispersive X-ray (EDX).

2. Method

2.1 Material

The kapok seed oil used was extracted from the kapok seed. The raw material for producing biofuels comes from kapok seed oil. The process used to convert kapok seed oil into biofuel is a hydrocracking process with a CuMO/HZSM5 catalyst. HZSM-5 is made by Ammonium ZSM-5 Zeolite (Zeolyst International, CBV 8014, a surface area of 425 $m^2.g^{-1}$) as in the previous study. Cu metal from Copper (II) Nitrate Trihydrate and Mo metal from Ammonium Molibdat Tetrahidrat were purchased from Merck with ≥98% purity, respectively.

2.2 Instrumentation

The pattern of XRD was recorded with a PANalytical X'Pert MPD diffractometer with Cu Kα as a source of X-ray operating at 40 kV and 30 mA. The sample was scanned in the range 2θ of 5-90° with a step size of 0.017, scan step time of 10.16 s, and PSD length of 2.12. Scanning electron microscopy (SEM) was used to determine the morphology of the catalyst crystals using Zeis s EVO MA 10 scanning electron microscopy. And the surface area, total pore volume and pore diameter were measured using an N_2 adsorption-desorption isotherm at liquid nitrogen temperature (77.35 K), using a NOVA instrument with a thermal delay of 600 sec.

Biofuel production from catalytic hydrocracking of kapok seed oil was investigated using a slurry pressure batch reactor with the stirrer. The type of reactor used is PARR 4563 with a maximum volume of 600 ml. Biofuel liquid products were determined using gas chromatography-mass spectrometry (GC Agilent 19091S-433). A capillary column (HP-5) with column length (30 m), diameter (250 μm), and 0.25 μm film thickness were used with helium as the carrier gas. The holding time temperature was carried out at 150°C for 2 min and the final temperature at 240°C for 13 min.

2.3 Catalyst Preparation

The HZSM-5 zeolite, 4.75 grams of Ammonium ZSM-5 Zeolite calcined at 550°C (5 hours) as previous study (Mirzayanti et.al., 2018). Then, the HZSM-5 zeolite was impregnated with an aqueous solution of Copper (II) Nitrate Trihydrate to generate a Cu/HZSM-5 catalyst by incipient wetness impregnation method. Based on the impregnation method used, namely incipient wetness impregnation, then based on the results of the BET analysis for HZSM-5, it was obtained that the pore volume of HZSM-5=0.2712 cm^3/g, the volume of precursor solution was obtained of ≈1.5 ml. The ratio of Cu:Mo used is 1:1 by loading 5% by total weight on the HZSM-5. Based on the wetness impregnation method, the volume of support was determined by adding water to 0.5 gr of Copper (II) Nitrate Trihydrate to a solution. The aqueous solution of Copper (II) Nitrate Trihydrate was slowly added to HZSM-5 with stirring at 30°C (3 hours) in 50 rpm. Then, for CuMo/HZSM-5 catalyst preparation, the solution of Ammonium Molibdat Tetrahidrat was impregnated to the Cu/HZSM-5 with stirring at 30°C (3 hours). The CuMo/HZSM-5 was dried in the oven at 120°C (12 hours) after each impregnation step. At last, the dried powder of CuMo/HZSM-5 was calcinated at 500°C (5 hours) in the air and reduced with hydrogen at 550°C (3 hours).

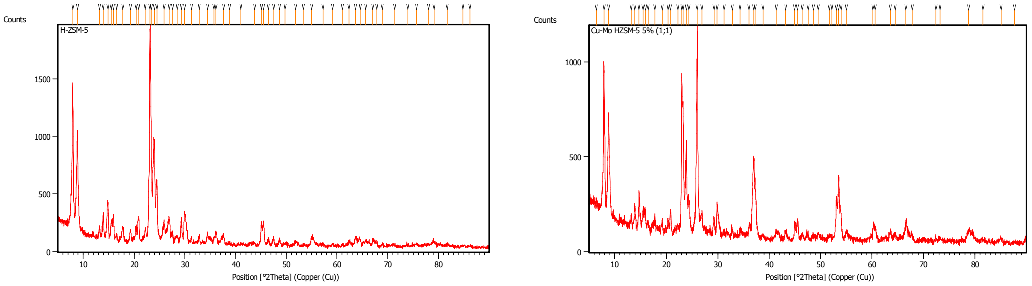
2.4 Catalytic Hydrocracking Reaction of Kapok Seed Oil

The catalytic hydrocracking of the biofuel kapok seed oil was carried out at different reaction temperature conditions in a batch reactor with a stirrer (PARR 4563, 600 ml volume). The hydrocracking reaction of kapok seed oil (200 ml) using 1 g of CuMo/HZSM-5 in reaction temperature of 350, 375 and 400°C and reaction pressure at the range 15 bar after flowing hydrogen for about an hour, were applied in the catalytic test. The hydrocarbon liquid products were collected for evaluation after 2 h of steady-state reaction. The reaction products were analysed by gas chromatograph-mass spectrometry (GC-MS). The hydrocarbon component of biofuel (i.e. paraffin, aromatic, cycloparaffin, and olefin) were defined as gasoline-like hydrocarbon (C_5-C_9), kerosene-like hydrocarbon (C_{10}-C_{13}) and gas-oil-like hydrocarbon (C_{14}-C_{22}), as reported previous study (Barron et al., 2011).

3. Result and Discussion

3.1 Catalyst Characterization

The surface area and pore volume of CuMo/HZSM-5 were measured by the BET method. **Table 1** shows a decrease in the surface area and pore volume of the CuMo/HZSM-5. This is due to the impregnation of Cu and Mo metal into the HZSM-5 pore. This denoted that HZSM-5 crystallinity was not collapsed as the HZSM-5 framework atom was not replaced by metal. **Figure 1a** shows that the HZSM-5 framework is devoid of Cu and Mo metals, while **Figure 1b** shows that the HZSM-5 framework is unchanged and Cu and Mo metals are dispersed on the surface of the HZSM-5 crystals. The presence of Cu and Mo metals has been shown through the XRD pattern in Figure 1.



(a) (b)

Figure 1. XRD Pattern of (a) HZSM-5 and (b) Cu-Mo/HZSM-5_51

Figure 1 illustrates the XRD pattern of HZSM-5 and CuMo/HZSM-5. Figure 1a shows the XRD pattern of HZSM-5, which has specific and typical peaks for the MFI (Mobile Inverted Framework) at the highest intensity of $2\theta=7^{\circ}$-8° dan 23°-24°, as reported in a previous study (Hemalatha et.al., 2013; Mirzayanti, et.al., 2018). Figure 1b shows the XRD pattern of CuMo/HZSM-5 where Cu and Mo metals have successfully impregnated into the pore of HZSM-5. On fig. 1 shows the peak for HZSM-5 shown at a peak of $2\theta=7.93^{\circ}$; 8.82°; 23.04°; 23.28°, and 23.92°. In CuMo/HZSM-5 catalyst, Cu and Mo metals did not change the structure of the HZSM-5 catalyst. It can be seen from the results of the SEM-EDX analysis (**Figure 2**) that the structure of HZSM-5 which consists of Si and Al is still detected on the CuMo/HZSM-5 catalyst. Cu metal in CuMo/HZSM-5 catalyst was detected as Cu phase at peak $2\theta=45.00^{\circ}$ and 45.55°, while for CuO phase at peak $2\theta=38.80^{\circ}$ and 47.59°. And for Mo phase was detected at peak $2\theta=60{,}23^{\circ}$ while the MoO_2 phase is detected at peak $2\theta=26.02^{\circ}$; 36.97°; 37.29°; 53.08° and 53.54°.

Figure 2 illustrates the SEM image and the corresponding EDX patterns of the CuMo/HZSM-5 catalyst. Based on the SEM image and EDX pattern, it is shown that Cu and Mo crystals can be impregnated into the pore surface of the HZSM-5 catalyst. It is shown that

Cu and Mo metals have a loading of 1.45wt% for Cu and 7.46wt% for Mo. The HZSM-5 catalyst has a uniform crystal particle structure with a Si/Al ratio =25, as reported previous study (Mirzayanti et.al., 2017). Nevertheless, after the impregnation of Cu and Mo metal, it is shown that Cu and Mo metal covered the pore surface of HZSM-5 so the Si/Al ratio became 33.

Table 1. Properties of Catalyst

Catalyst	Surface Area, $m^2.g^{-1}$	Total Pore Volume, $cm^3.g^{-1}$	Average Pore Diameter, nm	Metal Content, wt.%	
				Cu	Mo
HZSM-5	375.121	0.2712	2.89	-	-
Cu-Mo/HZSM-5	281.140	0.1618	1.15	1.45	7.46

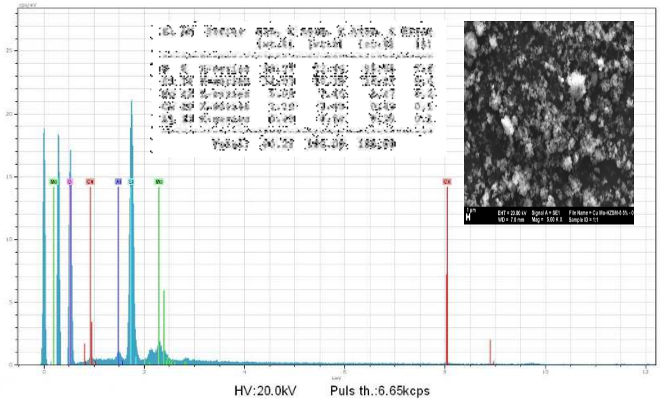


Figure 2. SEM Image and The Corresponding EDX Patterns of CuMo/HZSM-5 Catalyst

3.2 Catalytic Hydrocracking Performance

This study did not compare the performance of catalysts using support and without support. Catalytic hydrocracking of kapok seed oil produces liquid organic products (OLP) using a CuMo/HZSM-5 catalyst. The process of catalytic hydrocracking kapok seed oil was carried out at temperatures of 350 - 400ºC. The conversion of kapok seed oil was achieved at 21.22% at 350ºC and 45.72% at 400ºC. OLP products are classified into n-paraffins, cycloparaffins, aromatic compounds, olefins, oxygenated compounds, carboxylic acids, and other hydrocarbons. The composition of OLPs is presented in Table 2. **Table 2** shows that hydrocarbon products increase with increasing reaction temperature. However, there was a decrease in carboxylic acid from 78.78% at 350ºC to 54.28% at 400ºC. It shows that triglycerides first turn into free fatty acids through elimination reactions (Romero et.al., 2015). The presence of carboxylic acid is a compound produced to produce liquid hydrocarbons during decarboxylation reactions and/or decarbonylating for n-paraffins. The formation of cycloparaffins and aromatics proves cyclization reactions in catalytic hydrocracking. Olefins are formed due to dehydrogenation reactions and/or n-paraffin hydrogenolysis (Kim et.al., 2013). In hydrocracking reactions, the formation of alcohols and esters can also occur during triglyceride hydroprocessing so these reactions occur depending on the type of catalyst and hydroprocessing reaction conditions. The typical reaction that occurs during the catalytic hydrocracking process of vegetable oils is the cracking reaction of triglycerides into carboxylic acids (fatty acids) and propane (Sotelo-Boyas et al., 2012).

The biofuel product that is formed leads to bio-gasoline, bio-kerosene and bio-gasoil, while gasoline is not formed in temperature variations of 350-400ºC. Bio-Gasoline consists of compounds (C_5-C_9, cycloalkanes, aromatics), bio-kerosene consists of compounds (C_{10}-C_{14}), and bio-gasoil consists of compounds (C_{15}-C_{23}) (Sotelo-Boyas et al., 2012). **Figure 3** shows the product distribution of biofuel. In Figure 3, bio-gasoil has been formed (10.40% area), bio-kerosene (1.38% area), and decreased bio-gasoline (0.87% area). The temperature of 375ºC shows only bio-kerosene products (1.45%) and bio-gasoil (11.72%) are formed. The increase in the number of biofuel products occurred at 400ºC where the products produced were bio-kerosene (2.82%) and bio-gasoil (17.11%). Cu metal supported by an HZSM-5 catalyst has excellent performance for hydrogenation reactions (Kong and Chen et.al., 2014).

Table 2. Composition (%area) of Organic Liquid Products Under Various Reaction Temperatures

Component	Reaction Temperature		
	350ºC	375ºC	400ºC
n-paraffin	1.53	9.84	16.11
isoparaffin	0.00	0.00	0.00
Cycloparaffin/naphtene	2.48	0.60	10.47
Aromatics	0.76	0.86	2.75
Olefin/Alkena	9.79	5.00	2.81
Oxygenated compound (ester, aldehyde, ketone, alcohol)	5.93	1.87	1.15
Carboxylic acid	78.78	74.19	54.28
1,2-Benzisothiazole, 3-(hexahydro-1H-azepin-1-yl)-, 1,1-dioxide	-	5.52	7.30
14-.BETA.-H-PREGNA $$ 14-.BETA.-PREGNA $$ 14B-PREGNANE	-	-	5.12
1-Bromo-11-iodoundecane	-	4.03	-
Other organic liquid products	0.73	-	-

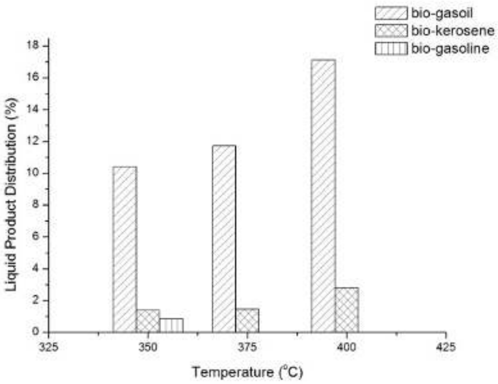


Figure 3. Effect of Reaction Temperature (ºC) on Biogas Oil, Bio-kerosene and Bio-gasoline Yield (as %Area of Liquid Product Distribution)

The effect of temperature was successfully investigated on the CuMo/HZSM-5 catalyst. The selectivity of n-paraffin and cycloparaffin is presented in figure 4. In **Figure 4**, the reaction temperature from 350ºC to 375ºC showed an increase in selectivity to n-paraffin from 10.48 % to 58.23 % and selectivity from cycloparaffin shows an increase from 17.03 % to 13.66 %. But, at 400ºC there was a decrease in the selectivity of n-paraffin which was 50.12 %, and cycloparaffin was 32.57 %. This shows that at 400ºC, the reaction turns water gas into water. This water is formed during the catalytic process Hydrocracking will result in the deactivation of the catalyst.

$$\text{Selectivity} = \frac{\text{mole of B formed}}{\text{moles of A consumed x stoichiometric factor}} \qquad (1)$$

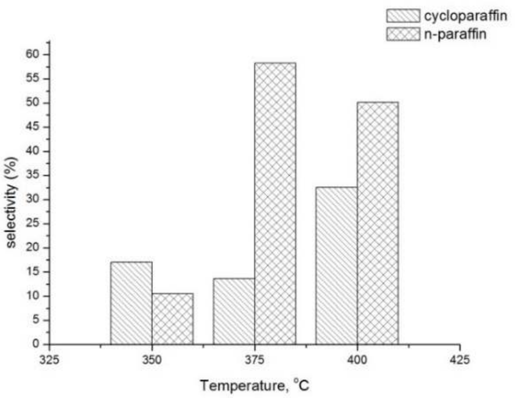


Figure 4. Effect of Reaction Temperature (ºC) on The Selectivity of n-Paraffin and Cycloparaffin

Figure 5 shows the effect of temperature variation on the conversion of kapok seed oil using a CuMo/HZSM-5 catalyst. In Figure 5, the higher the reaction temperature for catalytic hydrocracking processes, the conversion of kapok seed oil to biofuel is also increasing. It can be seen that at 350ºC the conversion is 21.22%, 375ºC is 30.22%, and 400ºC is 45.72%. This shows at 400ºC that have succeeded in breaking the double chains on the long chain of fatty acids.

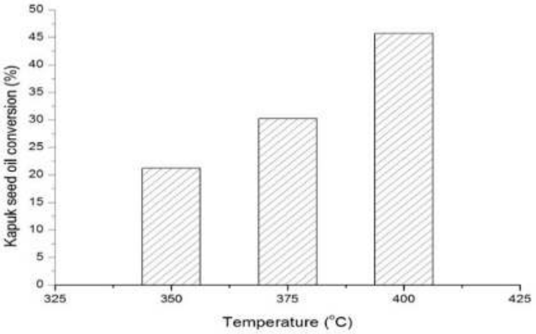


Figure 5. Effect of Reaction Temperature (ºC) Variation Over the CuMo/HZSM-5 Catalyst on the Conversion of Kapok Seed Oil

4. Conclusion

The production of hydrocarbons in the biogas oil and bio-kerosene was performed in the catalytic hydrocracking of kapok seed oil at 400ºC and a pressure of 10-30 bar for 2 h using CuMo/HZSM-5 catalyst. The analysis of conversion and selectivity results were obtained at different temperatures in the experiments. Liquid products that are formed predominantly lead to biogas oil range diesel. In this investigation, the CuMo/HZSM-5 catalyst performed best and produced bio-kerosene (2.82%) and bio-gasoil (17.1%) in a 2 h reaction at 400ºC. In addition, the selectivity of n-paraffin was 50.12% and cycloparaffin was 32.57%.

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