JURNAL KIMIA VALENSI
p-ISSN: 2460-6065; e-ISSN: 2548-3013
Journal homepage: https://journal.uinjkt.ac.id/index.php/valensi
Research Article

Natural Zeolite as Mo and MoP Catalysts Support for Catalytic Deoxygenation of Jatropha Oil

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Article Info
Received: March 5, 2025
Revised: June 3, 2025
Accepted: June 6, 2025
Online: June 17, 2025

Citation:
Aziz, I., Farhan, M., Saridewi, N., Azizah, Y. N., Muawanah, A., Nurbayti, S., Dwiatmoko, A.A., & Adhani, L. (2025). Natural Zeolite as Mo and MoP Catalysts Support for Catalytic Deoxygenation of Jatropha Oil. *Jurnal Kimia Valensi*, 11(1), 116-125.

Doi:
10.15408/jkv.v11i1.45272

Abstract

Non-edible oil, such as Jatropha oil, is an interesting feedstock for the development of renewable diesel (green diesel). Catalytic deoxygenation using natural zeolite-supported Mo-based catalysts is a promising process for the conversion of Jatropha oil to green diesel. Mo and MoP catalysts supported on natural zeolite were synthesized by wet impregnation at a concentration of 5% (w/w). The catalysts were characterized by XRD, XRF, SAA and NH_3-TPD. The catalysts were successfully synthesized with the appearance of Mo and MoP peaks on the catalyst diffractogram. XRF results also showed that Mo and P were present in the catalyst. Metal impregnation decreased the surface area and pore volume of the catalyst, but increased the average pore diameter. The NH_3-TPD profile of the catalyst showed that the weak acid sites of both catalysts were larger than the strong acid sites. Based on the activity test of catalytic deoxygenation of Jatropha oil, the MoP/HZ catalyst produced a higher conversion (67%) and liquid product yield (79%) than Mo/HZ. This is associated with a larger pore diameter, so that the distribution of reactants on the catalyst surface is more optimal. However, the highest green diesel selectivity of 82% is produced by the Mo/HZ catalyst. The Mo/HZ catalyst is more oriented towards the HDO reaction, whereas the MoP/HZ catalyst is more oriented towards the DCO/DCO_2 reaction.

Keywords: Catalytic deoxygenation, green diesel, jatropha oil, natural zeolite

1. INTRODUCTION

The world's demand for energy is on the rise, but this state of affairs is not in balance with the increase in the number of energy sources. Fossil energy, which is the main source of energy, continues to decline. Oil reserves, especially in Indonesia, decreased from 3.31 (2016) to 2.41 million stock tank barrels MMSTB (2023) [1]. Another problem that arises is the increasing pollution caused by using fossil fuels [2,3,4]. This encourages the search for renewable and environmentally friendly alternative energy. Biodiesel has emerged as a first-generation biofuel that can replace conventional diesel [5]. The Indonesian government has developed renewable energy in the form of biodiesel (first-generation biofuel) as a substitute fuel for diesel. However, biodiesel has weaknesses such as low thermal stability, low calorific value, and low cetane number [6].

Green diesel has emerged as a second-generation biofuel that can overcome these shortcomings. Green diesel is a hydrocarbon compound derived from vegetable/animal oil with diesel-like characteristics. Green diesel contains no oxygen, therefore, its thermal stability, heating value, and cetane number are higher [7]. Green diesel is also environmentally friendly because it tends not to produce gas emissions in the form of SO_x and is able to reduce CO_x gas emissions [8].

Potential feedstocks for green diesel production include non-edible oils such as Jatropha oil. This oil is obtained from the seeds of the jatropha plant, and the plant is easy to cultivate [9]. The oil content in Jatropha seeds is quite high, reaching 40-60%. The composition of fatty acids in Jatropha oil consists of oleic acid (37.44-44.91%), linoleic acid (33.83-34.14%), stearic acid (7.33-15.59%), and palmitic acid (9.84-12.22%) [10,11]. The catalytic deoxygenation method using hydrogen can be used to convert Jatropha oil into green diesel. This method is able to produce a higher conversion and selectivity as well as a cetane number of the green diesel [12]. Some catalysts that have been used in the conversion of Jatropha oil to green diesel are $NiP/\gamma\text{-}Al_2O_3$ [13], $Ni_2P/Zr\text{-}SBA\text{-}15$ [14], Co/MWCNT, Ni/MWCNT, and Ni-Co/MWCNT [15], $Zr/\gamma\text{-}Al_2O_3$ [16], and $Ni\text{-}Mo_2C/MCM\text{-}41$ [17]. Catalysts typically use transition metals with good catalytic activity, enhanced by synthetic supports. Support synthesis requires a lot of chemicals (reagents) and the process is quite difficult. This affects the high cost of catalyst production. Therefore, in this study, natural zeolite was used as a support for Mo and MoP catalysts. In addition to its low price, zeolite has pores and contains Lewis and Bronsted acid sites on its surface, which can increase its catalytic activity. The surface area of zeolite can also be modified by the desilication and activation process. In previous studies, the surface area of zeolite can be increased from 43.570 to 155.402 m^2/g [18]. Natural zeolites have also been used as a NiMo catalyst support for the deoxygenation of waste cooking oil, palm oil, and palm fatty acid distillate [19-22].

Molybdenum (Mo) based catalysts show good catalytic activity in green diesel production. Molybdenum sulfide and oxide are the most common forms of Mo catalysts, and are widely used in hydrotreating and oxidation reactions [23-25]. The activity of the Mo catalyst can be increased by adding phosphorus (P) to form metal phosphide (MoP). The addition of P can prevent the deactivation of the metal into the oxide form [26], well-dispersed active phase [27], increase the acidity of the catalyst [28], and produce more primary alkane products through direct hydrodeoxygenation reactions [29]. Compared to others, MoP catalysts are more active and stable [30]. Alvarez-galvan et al. found that the order of the greatest catalytic activity of the metal phosphides is as follows $MoP/SiO_2 > CoP/SiO_2 > NiP/SiO_2 > WP/SiO_2$ [31].

In this study, Mo and MoP catalysts supported on natural zeolite were used for catalytic deoxygenation of Jatropha oil to green diesel. The catalyst was synthesized by wet impregnation method with Mo content of 5% and Mo/P molar ratio of 2:1. Before performing the catalytic activity test, the catalyst was characterized by surface area analyzer (SAA), X-ray diffraction (XRD), NH_3 temperature program desorption (NH_3-TPD), and X-ray fluorescence (XRF). The activity of the catalyst was tested at a temperature of 350 °C under the pressure of H_2 gas for 4 h with a catalyst concentration of 20%. The resulting product was analyzed for composition using gas chromatography-mass spectroscopy (GC-MS) to determine the yield, conversion and selectivity of green diesel.

2. RESEARCH METHODS

Materials and Tools

The materials used were jatropha oil from a supplier in Jakarta and natural zeolite from CV. Mina Tama Lampung. All reagents used were from Merck with pure quality (p.a) such as sodium hydroxide, ammonium acetate, ammonia, phosporic acid and molibdenum trioxide. The tools used were a set of stainslessteel batch reactor, Gas Chromatography Mass Spectroscopy (Shimadzu QP 2010), X-Ray Diffraction (Rigaku Miniflex 600), Surface Area Analyzer (Micromeritics TriStar II 3020), X-Ray Fluorescence (Bruker S2 PUMA), and Ammonia-Temperature Program Desorption (Micromeritics Chemisorb 2720).

Modification of Natural Zeolite

Natural zeolite was washed with distilled water and dried at 105 °C. Some 200 mL of 0.5 M NaOH was added to the zeolite (10 g) and heated at 75 °C for 2 h. The zeolite was neutralized with distilled water and dried at 100 °C for 12 h. The zeolite was activated with CH_3COONH_4 1 M at 90 °C for 5 h. After washing and drying, the zeolite was calcined at 450 °C for 3 h [32] and then labeled as HZ.

Synthesis of Mo/HZ Catalyst

The synthesis was carried out by wet impregnation method with 5% (w/w) Mo metal concentration. A total of 0.7485 g MoO_3 was dissolved in 25 mL of 25% NH_4OH, then HZ (10 g) was added. The mixture was stirred for 2 h at room temperature and dried at 100 °C for 12 h. The catalyst was then calcined at 500 °C for 3 h, followed by reduction with H_2 gas for 4 h without temperature change [33].

Synthesi of MoP/HZ Catalyst

For the MoP/HZ catalyst, after dissolving MoO_3 in 25% NH_4OH, 0.1752 mL of 85% H_3PO_4 was added dropwise. HZ (10 g) was then added and stirred for 2 h at room temperature. The dried mixture was reduced with H_2 gas for 3 h at 600 °C [34].

Characterization of the Catalyst

X-ray diffraction (XRD) measurements for crystal phase identification were performed at a voltage of 40 kV, a 2θ angle in the range of 5-80°, and a scan rate of 2°/min. XRD patterns were compared with JCPDS (The Joint Committee on Powder Diffraction Standards) for material identification. Crystal size was calculated using the Debye-Scherrer equation. The surface properties of the catalyst were determined using a surface area analyzer (Micromeritics TriStar II 3020) after degassing at 320 °C for 5 h. The pore size distribution and pore volume of the catalyst were calculated using the Barret Joyner Halenda (BJH) model, and the specific surface area was obtained from the Brunauer, Emmett, and Teller (BET) equation. The elemental composition was analyzed by X-ray fluorescence (XRF). Sample (0.5 g) was placed into the holder and compacted using a presser. The pressed sample was irradiated with X-rays at a voltage of 40 kV and a current of 45 mA. The ammonia temperature desorption (NH_3-TPD) program was used to determine the acidity of the catalyst.

Test of Physical and Chemical Properties of Jatropha Oil

The compound composition of jatropha oil was analyzed using a GCMS instrument. The sample was first esterified and then injected up to 0.1 mL into the Agilent HP-5 column at 50 °C. The temperature was increased at a rate of 5 °C/min to 260 °C. Moisture content, free fatty acids, iodine number and saponification were analyzed using the SNI method 01-3555-1998.

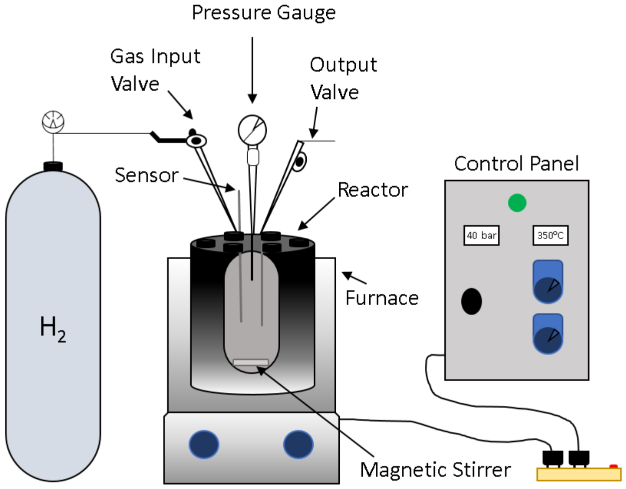

Figure 1. Schematic of experimental devices [35]

Catalytic Deoxygenation of Jatropha Oil

Jatropha Oil (10 g) and the catalysts (2 g) were introduced into the reactor (**Figure 1**). H_2 gas was injected at a pressure of 40 bar. The reactor heater was ignited to a temperature of 350 °C. The stirrer was turned on at 300 rpm and the reaction was allowed to proceed for 4 h. The resulting product was filtered to separate the catalyst and the filtrate was analyzed for chemical composition using GC-MS. The yield of liquid/gas products and the selectivity of the hydrocarbon fractions (gasoline, kerosene and green diesel) produced were determined based on equations (1) and (2) [22].

$$\text{Liquid/gas fraction} = \frac{\text{Weight of liquid/gas product}}{\text{Weight of feed (jatropha oil)}} \text{ x } 100\% \quad (1)$$

$$\text{Selectivity} = \frac{\text{Area of desired hydrocarbon fraction}}{\text{Total of area products}} x100\% \quad (2)$$

3. RESULTS AND DISCUSSION

Physicochemical Characteristics of the Catalyst

The diffraction pattern of Mo/HZ catalyst shows the presence of clinoptilolite zeolite phase with the appearance of peaks at 2θ: 9.92°, 11.18°, 13.06°, 17.3°, 26.04°, and 30.18° (JCPDS No. 25-1349) (**Figure 2**). This phase is also present in the MoP/HZ catalyst (2θ: 10.1 °, 11.32 °, 13.2 °, 17,56 °, 22.62 °, 26.39 °, and 30.36 °). Thus, metal impregnation has no significant effect on the support phase. Crystal peaks of Mo on Mo/HZ catalyst appeared at 2θ: 36.96°, 49.82°, and 70.06° (JCPDS No. 01-1208). Peaks of MoP detected at 2θ: 28.14° and 31.68° (JCPDS No. 24-0771). Usman et al. [36] and Nie et al.[37] got MoP peak on 2θ: 32.1°, 43.1°, and 57.0°. The crystal size of the Mo produced, as determined by the Debye-Scherrer equation, was 35.512 nm and the MoP was 28.822 nm (**Table 1**). The addition of P can reduce the crystal size of the catalyst.

The surface properties of the catalysts, including BET surface area, average pore diameter and pore volume, are shown in **Table 1**. The Mo/HZ catalyst produced a higher surface area (40.4649 m^2/g) and pore volume (0.0620 cm^3/g) than the MoP/HZ catalyst (surface area 30.7816 m^2/g and pore volume (0.0492 cm^3/g). The addition of P can reduce the surface area of the catalyst by partially covering the catalyst pores. Compared to the support (HZ), the surface area of the catalyst is smaller. The surface area of HZ is 155.402 m^2/g [35]. Metal impregnation on the surface of the support may cover the pores of the catalyst, thus reducing the surface area [38]. NiP/SiO_2 catalyst also produces lower surface area and pore volume than SiO_2 [39].

In contrast, the pore diameter of the catalyst increases relative to its support (HZ). The presence of metal causes the micropores to clog, increasing the average pore diameter. The large pore diameter is beneficial for reactant diffusion while increasing deoxygenation capability [40]. Mo/HZ and MoP/HZ catalysts showed an average pore diameter of 12.9900-14.7338 nm, indicating the presence of mesopores. This is supported by the nitrogen adsorption-desorption isotherms of both catalysts (**Figure 3a**).

The resulting isotherms are Type IV and Type IV hysteresis loop. These are attributed to the mesoporous structure of the catalyst. Pore distribution of the catalyst (**Figure 3b**) The pore diameter shows that the catalyst has a pore size of <2 nm (micropore) and >2 nm (mesopore), which indicates that the catalyst support is a hierarchical type zeolite because it has two different pore sizes [41]. This result is supported by Aziz et al. who also obtained NiAg/HZ catalyst pore distributions of 1-23 nm [42].

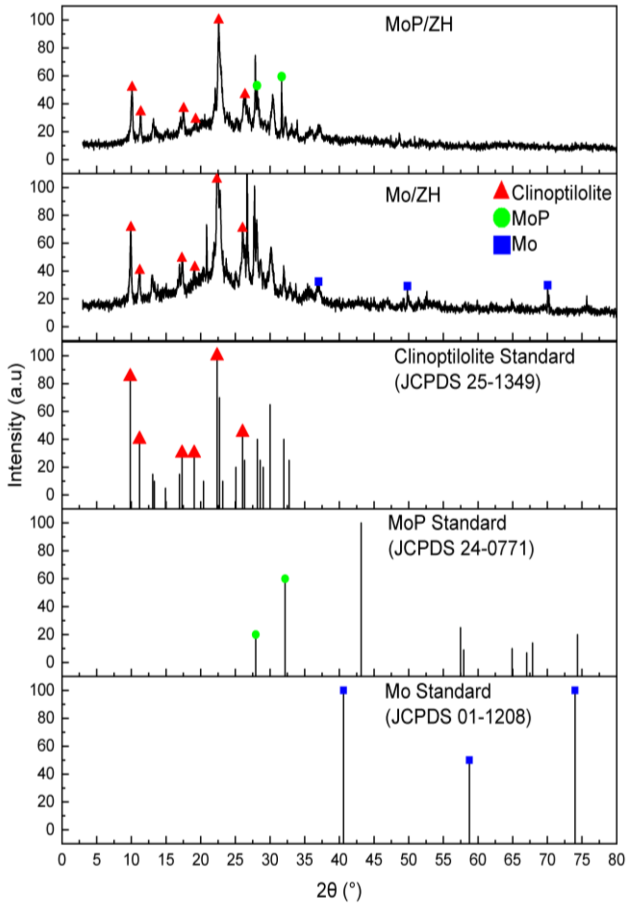

Figure 2. XRD patterns of catalysts

The composition of the element contained in the catalyst is given in **Table 1**. The impregnation of Mo and P elements on the catalyst decreased the Si/Al ratio compared to HZ, but the Si/Al ratio of both catalysts (Mo/HZ and Mop/HZ) was almost the same. The P impregnation was successfully carried out with the increase of P concentration on the MoP/HZ catalyst. Therefore, XRF analysis verified the effectiveness of wet impregnation in loading accurate amounts of metal precursors onto the catalyst support[7]. Mo/HZ catalyst has higher acidity (1.4832 mmol/g) than MoP/HZ (1.2368 mmol/g). The increased surface area of Mo/HZ relative to MoP/HZ results in a larger surface area at the active site. However, the acidity of Mo/HZ is lower compared to HZ. HZ has a large surface area (155.5700), meaning it has strong acidity. This acidity comes from Lewis and Bronsted sites on the zeolite surface after desilication and activation. Based on profile of NH_3-TPD (**Figure 4**), Both catalysts have peaks in the temperature range of 100-250 °C and 400-600 °C, indicating weak and strong acid sites, respectively [43]. Weak acid sites (100-250 °C) are Lewis acid sites, and strong acid sites (> 400 °C) are Bronsted acids [44]. As with the support (HZ), both catalysts have weak acid sites with larger peaks than strong acid sites [35].

Table 1. Physicochemical properties of catalysts

Sample	**Mo/HZ**	**MoP/HZ**	**HZ** [35]
Surface area (m^2/g)	40.4649	30.7816	155.5700
Avarage pore diamater (nm)	12.9900	14.7338	3.824
Pore volume (cm^3/g)	0.0620	0.0492	0.190
Acidity (mmol/g)	1.4832	1.2368	2.431
Crystal size (nm)	35.512	28.822	-
Composition (%):			
Mo	5.5	6.0	0
P	0.2	0.8	0
Si	31.6	30.9	35.7
Al	8.8	8.7	8.7
Si/Al ratio	3.59	3.51	4.10

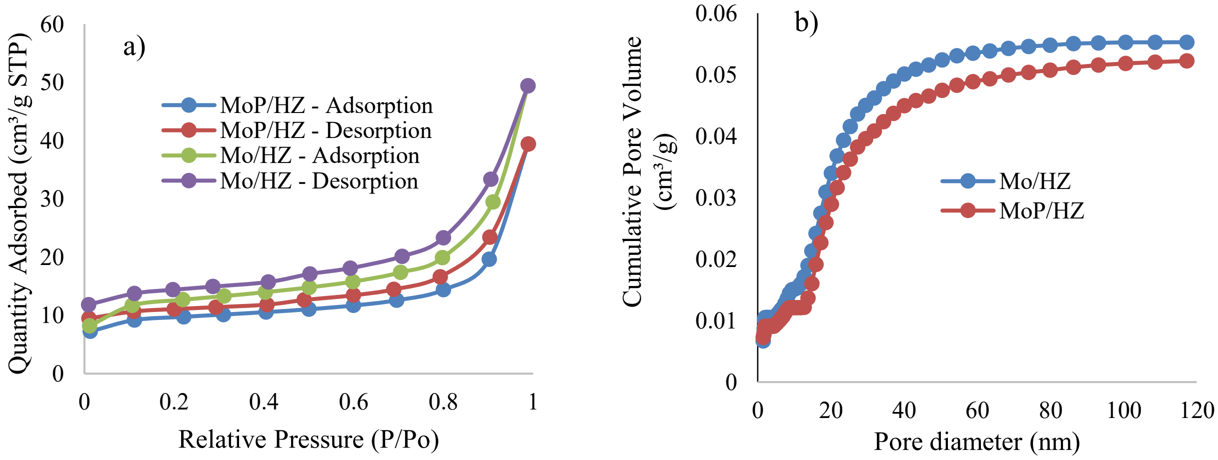

Figure 3. a) Nitrogen adsorption-desorption isotherms, b) Pore diameter distribution of catalysts

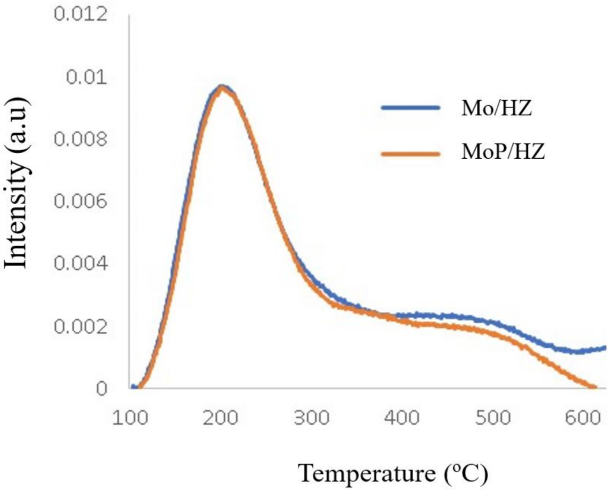

Figure 4. TPD-NH_3 profile of catalysts

Physicochemical Properties of Jatropha Oil

The fatty acid composition of Jatropha oil was analyzed by GC-MS instrument. Based on **Table 2**, the largest fatty acid composition is oleic acid 51.28%, followed by linoleic acid 35.61%, palmitic acid 8.08%, pentadecanoic acid 4.69%, and stearic acid 0.25%. Oleic acid is the largest component of Jatropha oil. This has also been found by other researchers [15,14]. The water content and density of the Jatropha oil produced were 0.05 wt% and 0.966 g/cm^3, higher than Asikin-Mijan et al. (0.01 wt% and 0.920 g/cm^3) [15], with lower free fatty acid content. This difference is due to the different raw material sources used.

Table 2. Physicochemical properties of the jatropha oil

Oil properties	This study	Asikin-Mijan et al. [15]	Tan et al. [14]
Water content (wt%)	0.05	0.01	-
FFA content (wt%)	10.781	15.4	-
Density (g/cm^3)	0.966	0.920	-
Fatty acid composition (%):			
Oleic acid (C18:1)	51.28	39.77	40.7
Linoleic acid (C18:2)	35.61	31.52	37.6
Palmitic acid (C16:0)	8.08	20.16	13.3
Pentadecanoic acid (C15:0)	4.69	-	-
Stearic acid (C18:0)	0.25	7.22	6.7

Catalytic Activity

The catalyst activity was tested on the catalytic deoxygenation of Jatropha oil at a temperature of 350 ºC, reaction time of 4 hours, catalyst concentration of 20% (w/w), stirring speed of 400 rpm, and H_2 gas pressure of 40 bar. Catalytic deoxygenation of Jatropha oil produces liquid and gaseous products. The conversion of the Jatropha oil produced ranges from 52-67% (**Figure 5**). The support (HZ) produces a lower conversion compared to Mo/HZ and MoP/HZ catalysts, as well as a lower yield of liquid products. This is due to the smaller pore diameter of HZ compared to Mo/HZ and MoP/HZ, which hinders the diffusion of reactants into the catalyst pores.

On the other hand, HZ produced a higher yield of gas products. This can be attributed to the high acidity of HZ (especially the strong acid site), which triggers side reactions in the form of cracking, producing light fractions in the form of gas. Mo and P impregnation in HZ can increase the conversion and yield of liquid products. The MoP/HZ catalyst produced the highest Jatropha oil conversion of 67% and liquid product yield of 79%. The larger pore diameter of the MoP/HZ catalyst increases the diffusion of the reactants, thereby increasing the catalyst activity. The composition of the resulting liquid product was determined by GC-MS. Based on **Figure 6,** the highest selectivity of green diesel (C_{15-18}) was produced by the Mo/HZ catalyst (82%), followed by MoP/HZ (55%) and HZ (66%). Deoxygenation of palm fatty acid distillate using Mo/AC and Cr_2O_3/AC catalysts resulted in higher C_{15-17} selectivity compared to AC [38,40]. This shows that the addition of metal can increase the activity of the catalyst deoxygenation.

The highest selectivity of kerosene (C_{14-15}) is produced by the HZ catalyst (22%), while the highest selectivity of gasoline (C_{5-12}) is produced by the MoP/HZ catalyst (38%). The addition of P to the catalyst can increase the selectivity of gasoline. In previous studies, the deoxygenation of Jatropha oil using the Ni_2P/Zr-MCM-41 catalyst resulted in a green diesel selectivity of 50.37% [45]. The use of the Ni_xP_y catalyst in the hydrodeoxygenation of palm oil resulted in a green diesel yield of 63% [46]. The results obtained were lower than the Mo/HZ catalyst but close to the MoP/HZ catalyst.

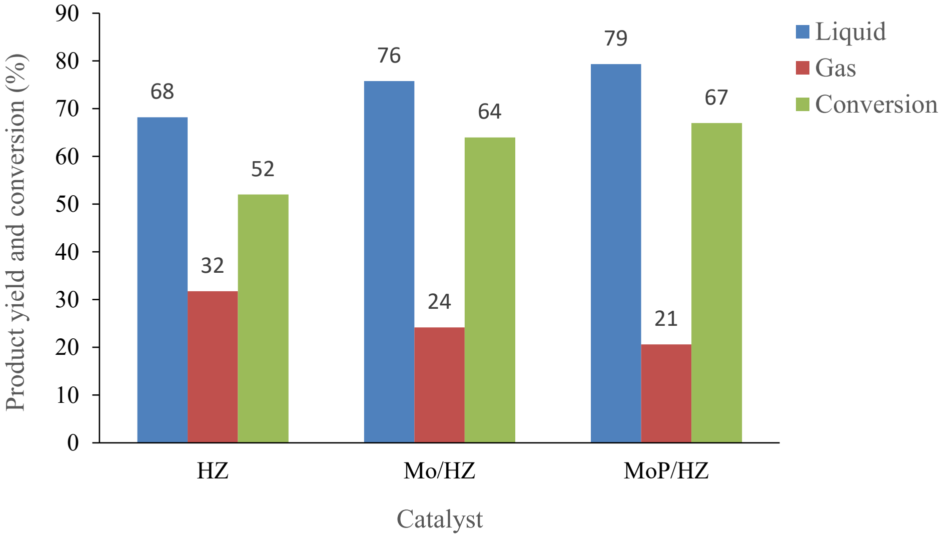

Figure 5. Product yield from catalytic deoxygenation of jatropha oil. Reaction conditions: T = 350 ◦C, t = 4 h, P_{H2} = 40 bar and catalyst concentration = 20 wt%.

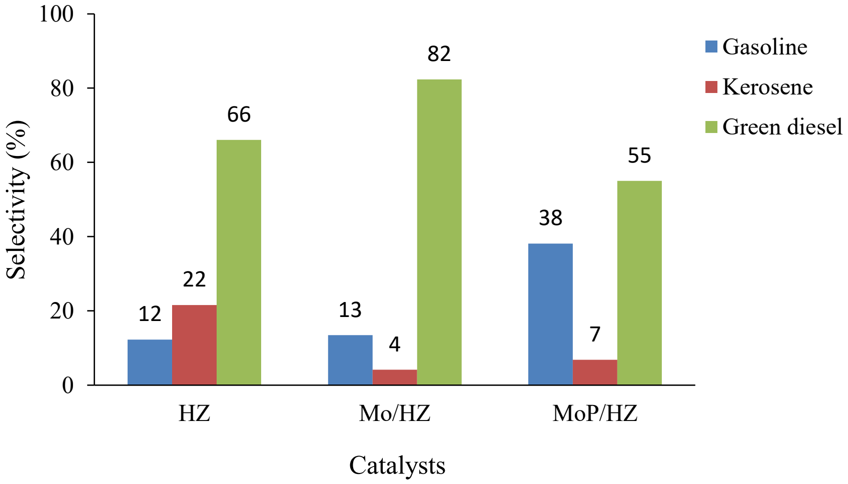

Figure 6. Biofuel fraction from catalytic deoxygenation of jatropha oil. Reaction conditions: T = 350 ◦C, t = 4 h, P_{H2} = 40 bar and catalyst concentration = 20 wt%.

Carbon distribution from biofuel fraction ranges from C_6 to C_{18} (**Figure 7**). In general, C_{17} and C_{18} compounds produce the highest selectivity. This is because oleic acid (C18:1) and linoleic acid (C18:2) are the major components of Jatropha oil. The presence of the C_{17} and C_{18} compounds can be explained by the proposed mechanism of the reaction (**Figure 8**).

Triglycerides (TGs) in Jatropha oil undergo hydrogenation reactions to convert unsaturated fatty acids (oleic and linoleic acids) to saturated fatty acids (stearic acid) (Stage 1) [46]. In addition, hydrogenolysis reactions occur with the conversion of TGs to stearic acid and propane gas (stage 2). Stearic acid undergoes three reactions in the presence of hydrogen. First, the hydrodeoxygenation (HDO) reaction, in which stearic acid is converted to octadecane ($C_{18}H_{38}$) and water (stage 3). Second, the decarboxylation reaction (DCO_2) produces heptadecane ($C_{17}H_{36}$) and CO_2 gas (stage 4). Third, the decarbonylation reaction (DCO) produces heptadecane ($C_{17}H_{36}$), CO gas, and water (stage 5). Octadecane and heptadecane compounds were detected from the GC-MS results. The appearance of other compounds (C<15) is caused by the occurrence of side reactions in the form of cracking of HDO, DCO and DCO_2 products (stage 6). The Mo/HZ catalyst produces the highest C_{18} selectivity compared to MoP/HZ and HZ. This shows that the Mo/HZ catalyst is more oriented towards the HDO reaction, while the MoP/HZ catalyst is more oriented towards the DCO/DCO_2 reaction.

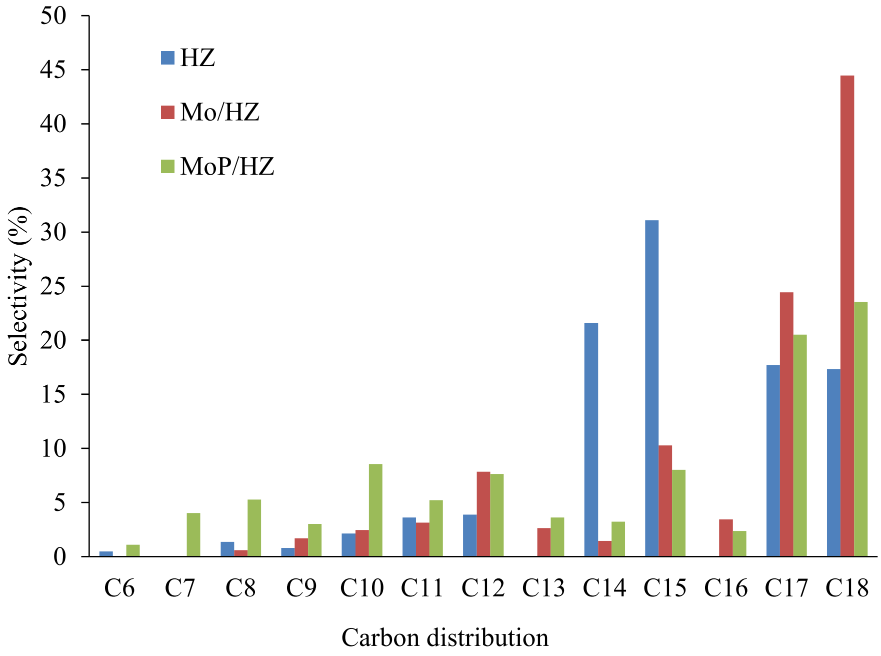

Figure 7. Effect of catalysts on hydrocarbon selectivity reaction conditions: T = 350 ◦C, t = 4 h, P_{H2} = 40 bar and catalyst concentration = 20 wt%.

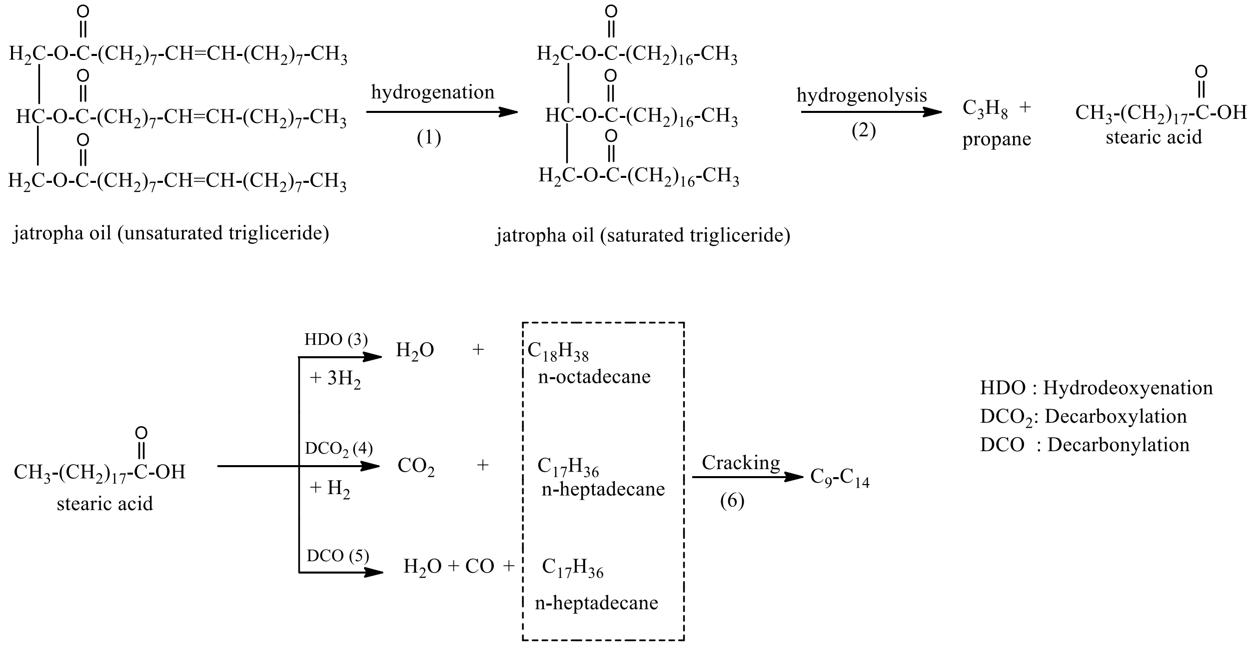

Figure 8. Proposed catalytic deoxygenation reaction pathway of jatropha oil to hydrocarbon over MoP/HZ catalysts

4. CONCLUSIONS

Mo and MoP catalysts supported on natural zeolite have been successfully synthesized by the wet impregnation method. This is evidenced by the appearance of Mo and MoP peaks on the catalyst diffractogram. The resulting metal concentration is close to the theoretical concentration. The surface area and pore volume of the catalyst decreased after impregnation, but the average pore diameter increased. Catalytic deoxygenation of Jatropha oil using MoP/HZ catalyst resulted in conversion and liquid product yield of 67% and 78% respectively, higher than those of Mo/HZ and HZ catalysts. However, the highest green diesel selectivity was produced by the Mo/HZ catalyst (82%). The Mo/HZ catalyst is more oriented towards the HDO reaction, while MoP/HZ is more oriented towards the DCO/DCO_2 reaction.

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