



## Conversion of Sunan Candlenut Oil to Aromatic Hydrocarbons with Hydrocracking Process Over Nano-HZSM-5 Catalyst

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### Abstract

In this paper, the catalysts (Nano-HZSM-5 and Fe-La/nano HZSM-5) were prepared with incipient wetness impregnation and applied for hydrocracking of Sunan candlenut oil. The hydrocracking process was conducted in a batch reactor with a pressure of 20-30 bar H<sub>2</sub> gas for 2 h under various temperatures. The results demonstrated that hydrocracking of Sunan candlenut oil using nano HZSM-5 and Fe-La/NHZ catalysts could be converted into aromatic hydrocarbons, and the reaction temperature affected hydrocarbon production. The aromatic compounds, such as propylbenzene, 1-ethyl-3-methylbenzene, heptylbenzene, 2-ethyl-naphthalene, etc., reached 35.51% over the Fe-La/NHZ<sub>2</sub> catalyst. In all cases, the zeolite-based catalysts are the most suitable to produce aromatic hydrocarbons. Metal impregnated (Fe and La) on nano HZSM-5 catalyst could improve the aromatics compounds due to increased metal and acid sites.

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**Keywords:** Hydrocracking; nano HZSM-5; Fe-La/NHZ catalysts; Sunan candlenut oil; aromatics compounds

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### 1. Introduction

The production of fuel from fossil raw materials produces very high carbon emissions. These carbon emissions can increase the earth's surface temperature and cause increased global warming. This can be a disaster for the survival of life on earth, such as extreme weather, natural disasters, changes in food chain production, the spread of disease, damage to marine ecosystems,

the melting of polar ice, reduced clean water, and so on. A solution is needed to the problem of lack of energy availability and high carbon emissions from energy production. The solution can be developed by implementing a sustainable energy transition as proposed in Indonesia's G20 presidency. This energy transition aims to replace fossil raw materials for energy production with clean, sustainable natural materials. This can support Indonesia's target of using 23% of new and renewable energy (EBT) in the national mix 2025. This target combines Indonesia's

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commitment to reduce emissions by 29% by 2030 and achieve net zero emissions (NZE) by 2060.

One energy transition solution can be implemented and developed by producing is biofuel. Biofuel is a liquid fuel produced from biomass that can substitute fossil fuels. Biofuel can be made from various biomasses that contain oil, one of which is oil from the sunan candlenut plant. Sunan candlenut oil is a biomass that has the potential to be developed as a raw material for biofuel. Sunan candlenut oil content reaches 45-55% of the seeds and has an acid number of 2.4-6.3 mg KOH/g. Sunan candlenut oil consists of myristic acid, palmitic acid, stearic acid, oleic acid, linoleic acid, linolenic acid, and traces of other acids. The highest acid content is palmitic acid (16.93%), oleic (38.63%), and linoleic acid (35.06%) [1].

Biofuel can be obtained through the cracking method of sunan candlenut oil. The cracking way is a process of breaking long-chain hydrocarbon compounds into short hydrocarbon compounds. Cracking produces compounds such as methane, ethane, propane, butane, gasoline, kerosene, and diesel. One cracking process that has advantages is hydrocracking because it can provide high conversion, the addition of hydrogen gas, the yield towards the middle distillate is also high, and the quality of the alkane produced has a high cetane number [2,3].

In the hydrocracking process, operating conditions and the type of catalyst greatly influence the quality of the product produced. Various types of catalysts are used in the cracking process because, by using a catalyst, a reaction can run in a short time and with an excellent conversion value. The catalysts widely applied in cracking reactions are heterogeneous catalysts with the advantages of easy separation and reusability. Currently, zeolite-based catalysts are effectively used for hydrocracking processes. HZSM-5 is catalyst and capable for the conversion of vegetable oil into biofuel. This can happen because it is supported by the active site, structural selectivity, ion exchange properties, and specific pore structure of H-ZSM-5 itself [4].

Several studies have discussed about the use of the HZSM-5 catalyst to produce biofuel. Lobo *et al.* [5] reported that adding Fe to HZSM-5 zeolite via dehydrogenation reaction can help the formation of alkane free radicals, which produce light olefins. Zhao *et al.* [6] also used the Fe/HZSM-5 catalyst by impregnation method for the isobutane catalytic cracking reaction. Fe metal was chosen because it is efficient and has a low cost. Besides that, Fe metal can also enter the zeolite framework, produce the Fe-OH-Si (FeZSM-5) structure, and control the acidity of the zeolite [7,8]. In some previous studies, Li *et al.* [9] reported using LaNiMo/ZSM-5 catalysts to

convert jatropha oil with a hydrocracking process. The results showed that adding La into NiMo/ZSM-5 improves the aromatic yield and reduces coke formation. Fan *et al.* [10] used La to convert jatropha oil using NiMoLa/Al<sub>2</sub>O<sub>3</sub>, NiMoLa/HY and NiMoLa/nHA catalysts. The results showed that the product contains renewable C<sub>15</sub>-C<sub>18</sub> alkanes, and La contributes to reducing coke in the cracking of Jatropha oil.

Based on this, it is desirable to impregnate Fe and La metals into nano HZSM-5. In addition, Fe and La metals impregnation was also used to increase the active sites of the H-ZSM-5 catalyst. Metals can modify the properties of the H-ZSM-5 catalyst, such as catalytic activity and selectivity, because transition metals, can add acid sites to H-ZSM-5 [11]. In this study, we have developed a nano HZSM-5 catalyst with impregnated Fe and La metals, denoted Fe-La/Nano HZSM-5 catalysts, which is promising as a heterogeneous catalyst for hydrocracking reaction to produce biofuel from Sunan candlenut oil. The effect of reaction temperature on the chemical composition of biofuel, consisting of olefin, aromatics, cycloparaffin, isoparaffin, and n-paraffin, was also studied.

## 2. Materials and Methods

### 2.1 Materials

The materials used in this research was Sunan candlenut oil from Bandung, West Java. Sunan candlenut oil was extracted from seeds and pressed with press machine. Then, it was analyzed using gas chromatography-mass spectrometry (GC-MS). The composition of fatty acid in sunan candlenut oil contains palmitic acid (23.89%), stearic acid (8.48%), oleic acid (12.45%), linoleic acid (16.80%), and linolenic acid (8.14%). The main abundant component of sunan candlenut oil in this research has a high content of poly-unsaturated fatty acids. The metal transitions were ferric nitrate nonahydrate (Fe(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O) and Lanthanum nitrate hexahydrate (La(NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O) purchased from Merck (with purity 98%).

### 2.2 Catalyst Preparation

Fe-La/Nano HZSM-5 catalysts were synthesized using impregnation method. Fe-La was impregnated on nano HZSM-5 by incipient wetness impregnation with variations ratio 1:1 and 1:2, and 5% loading of metals in catalyst. First, the solution of Fe(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O and La(NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O were put into nano HZSM-5, stirred for 1 h and dried overnight. Then, the samples were dried in oven at 110 °C to remove the water. After that, the samples were calcined at 500 °C for 3 h. The obtained catalysts were named Fe-La/NHZ\_1 and Fe-La/NHZ\_2.

### 2.3 Catalyst Characterization

X-Ray Diffraction (XRD) with diffractometer (PANalytical EMPYREAN) was used to determine the crystalline structure of the catalysts using the Cu-K $\alpha$  radiation ( $\lambda = 0.15406$  nm) operated at voltage 40 kV, and 30 mA. The analysis scanning in the range  $2\theta$  of 5-100°. The textural properties of the catalyst were characterized by nitrogen physisorption (Micromeritics TriStar II 3020). Before the measurement, the catalysts were degassed at 300 °C for 3 h under vacuum conditions with nitrogen gas. The instrument operated at -196 °C to measure surface area, pore volume, and average pore diameter. Scanning electron microscopy-Energy Dispersive X-ray (SEM-EDX, SEM Jeol JSM-IT200) was used to determine the morphology of catalysts as well as metal contents. X-ray dot mapping was used to investigate the metal dispersion of the catalyst.

### 2.4 Hydrocracking of Sunan Candlenut Oil

The hydrocracking process of sunan candlenut oil was carried out in a batch reactor as shown in Figure 1. First, the catalyst was put in the reactor with mass 0.5 g and 15 ml of sunan candlenut oil. Then, hydrogen gas flowed 4 times to 20-40 bar. The reactor heater was switched on and stirred. The temperature variations used were 250, 300, and 350 °C with a reaction time of 2 h. After the reaction, the biofuel samples were

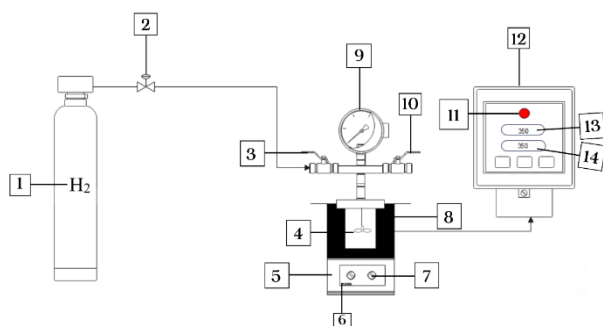


Figure 1. The experimental reactor for hydrocracking of sunan candlenut oil, consist of: 1) Gas tube H<sub>2</sub>, 2) tube valve gas H<sub>2</sub>, 3) Pressure inlet valve gas, 4) Stirrer, 5) Hotplate, 6) Power stirrer, 7) Stirrer speed control, 8) Reactor, 9) Reactor pressure indicator, 10) Valve gas outlet, 11) Power light, 12) Reactor heater control panel, 13) Display temperature, and 14) Display set point temperature.

collected and centrifuged to separate the catalyst and oil. The biofuel was centrifuged for 30 min at 8000 rpm. The liquid products of biofuel were obtained. Finally, liquid products of biofuel were analyzed using gas chromatography-mass spectrometry (GC-MS) and Fourier transform infrared (FT-IR). The liquid product was determined using GC-MS with Model Agilent Technologies to identify hydrocarbon in the liquid product. The column type of GC-MS was 19091S-433 and 93.92873 HP-5MS UI column (5% Phenyl Methyl Siloxane, 30 m  $\times$  250  $\mu$ m  $\times$  0.25  $\mu$ m). A biofuel product was further analyzed using FT-IR (Bruker Tensor II, frequency range 500-4000 cm<sup>-1</sup>) to determine the process of removing oxygen atoms and forming C-H groups.

## 3. Results and discussion

### 3.1 Catalyst Characterization

Figure 2 displays the diffractograms of nano HZSM-5, Fe-La/NHZ-1, and Fe-La/NHZ-2. The nano HZSM-5 has characteristic diffraction peaks at  $2\theta$  of 7.9124°, 8.801°, 9.067°, 9.75°, 21.719°, 22.142°, 23.0569°, 23.9115°, 24.3905°, and 24.728° in the  $2\theta$  region between 7-9° and 22-24°. Metal oxide of Fe<sub>3</sub>O<sub>4</sub> was detected at  $2\theta$  of 35.6° with JCPDS No. 19-629, and La<sub>2</sub>O<sub>3</sub> was detected at  $2\theta$  of 30.1°, and 48.2° based on JCPDS No. 35-0609 [12,13]. Furthermore, the diffractogram of Fe-La/NHZ-1 and Fe-La/NHZ-2 catalyst shows the

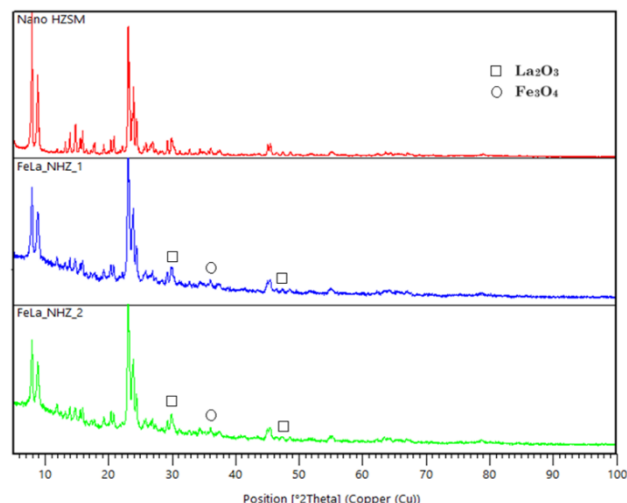


Figure 2. XRD diffractograms of nano HZSM-5, Fe-La/NHZ-1 and Fe-La/NHZ-2.

Table 1. Textural properties of nano HZSM-5 and FeLa/NHZ catalysts.

Catalyst	Surface Area (SA, m <sup>2</sup> /g)	Pore Volume (cm <sup>3</sup> /g)	Size Pore (nm)	Metal Content (wt%)	
				Fe	La
Nano HZSM-5	307.8874	0.2407	3.1275	-	-
Fe-La/NHZ-1	294.0965	0.2241	3.1252	2.09	2.02
Fe-La/NHZ-2	286.7340	0.2273	3.0913	1.78	3.35

similar diffraction peak characteristic with nano HZSM-5 support. This result shows that the addition of metal (Fe and La) to the support nano HZSM-5 did not change the structure and affect the degree of crystallinity of the nano HZSM-5 [14].

Table 1 shows the textural properties of nano HZSM-5 and Fe-La/NHZ catalysts. Nano HZSM-5 high surface area 307.8874 m<sup>2</sup>/g than the FeLa/NHZ catalysts. The surface area of Fe-La/NHZ-1 and Fe-La/NHZ-2 catalyst was lower than parent nano HZSM-5 after the impregnation of metal transition (Fe and La), 294.0965 and

286.7340, respectively. This is in accordance with previous research [15,16]. Based on Table 1, it can be concluded that the particles of metal were successfully filled on the surface and incorporated into nano HZSM-5 pore.

The morphology of the catalyst was obtained from SEM analysis while the composition of metal obtained from EDX. Figure 3 shows images of Images of (a) Nano HZSM-5, (b) Fe-La/NHZ\_1 and (c) Fe-La/NHZ\_2 with 5000x magnification. The results of the morphologies catalysts showed the agglomeration and uniformity of the crystal. The metal contents as shown in Table 1, the contain of

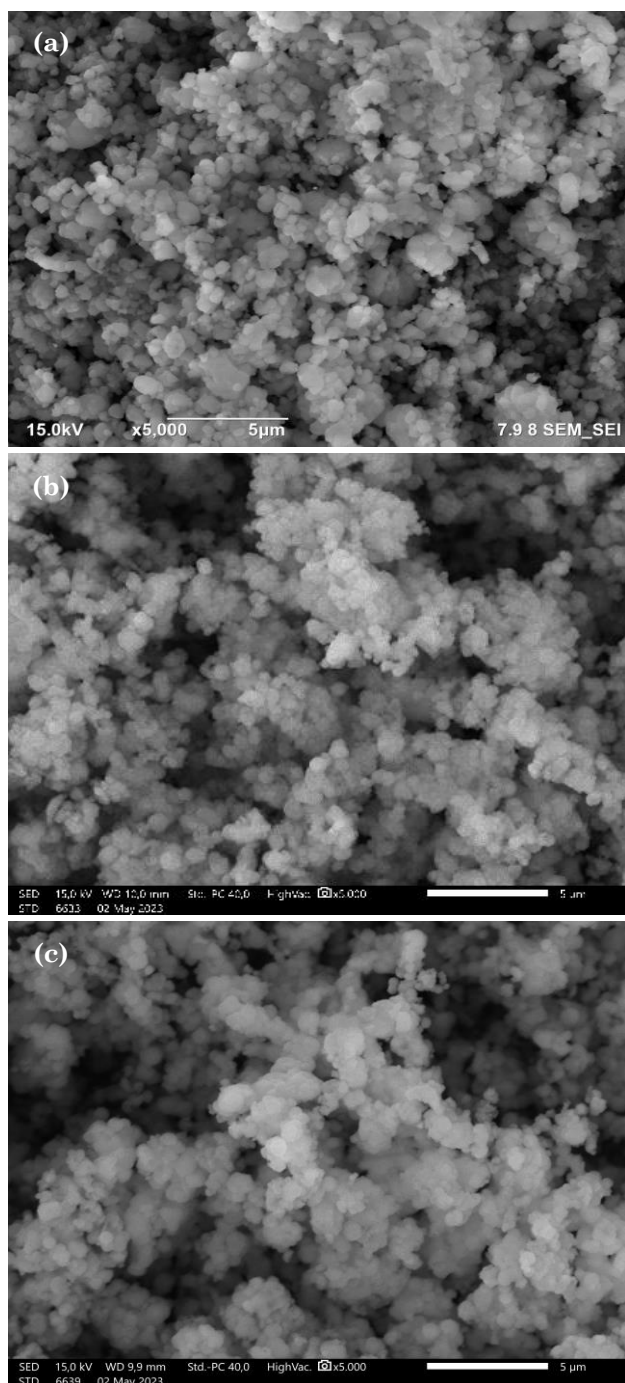


Figure 3. SEM Images of (a) Nano HZSM-5, (b) Fe-La/NHZ\_1 and (c) Fe-La/NHZ\_2.

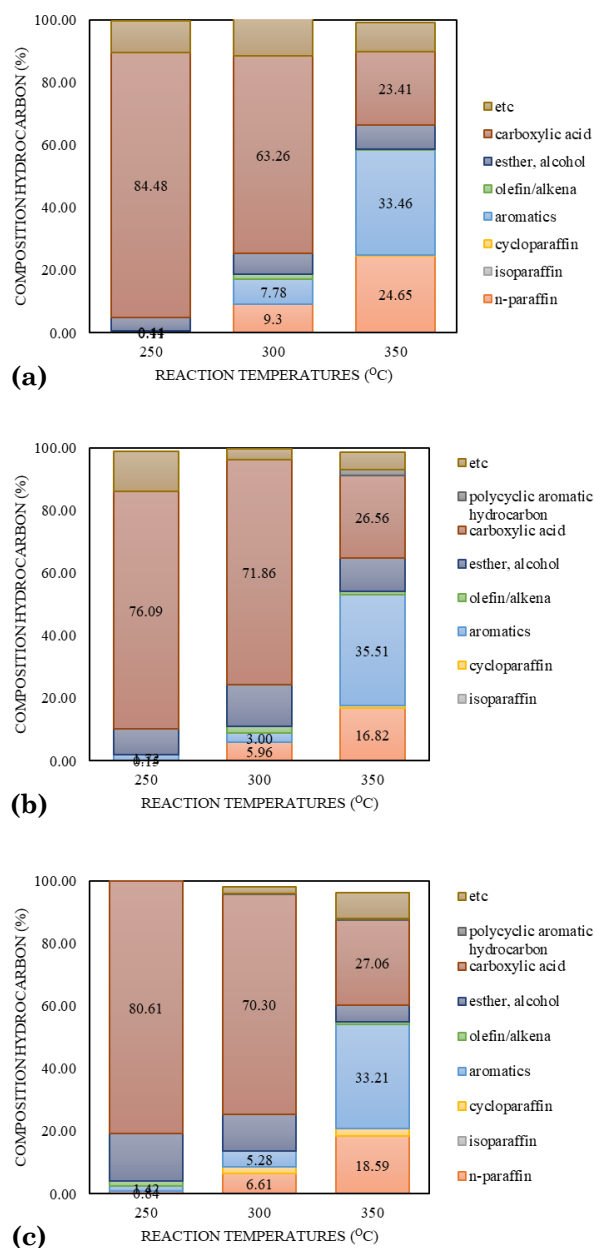


Figure 4. The effect of reaction temperature on chemical composition of biofuel from hydrocracking process using a) nano HZSM-5, (b) Fe-La/NHZ\_1 and (c) Fe-La/NHZ\_2

metal in the nano HZSM-5 support bit lower than calculated metal contents. It caused non uniform distribution of the metal on the support [17].

### 3.2 Effect of Temperature on the Product Composition

The effect of reaction temperature on chemical composition of biofuel from hydrocracking process using a) nano HZSM-5, (b) Fe-La/NHZ\_1 and (c) Fe-La/NHZ\_2 with variation of temperatures 250, 300, and 350 °C are shown in Figure 4. The chemical components were detected on the biofuel consisted of carboxylic acids, ester, alcohol, olefin, aromatics, cycloparaffin, isoparaffin and n-paraffin. Carboxylic acids, esters, and alcohols are undesirable compounds for producing energy, while hydrocarbon fractions such as olefins, aromatics, cycloparaffins, isoparaffins, and n-paraffins are desirable compounds. After the hydrocracking process was enhanced, the amount of carboxylic acid, esters, and alcohol compounds decreased while the number of aromatic compounds and n-paraffin tended to increase.

As shown in Figure 4 (a–c), at a temperature of 300 °C, the component of carboxylic acids was around 76–84%. The presence of C=O in the product shows the hydrocracking process needs to be completed. It indicates that the nano-HZSM-5 and Fe-La/NHZ catalysts are not beneficial for removing carbonyl groups in raw materials at relatively low reaction temperatures. The reaction required high temperatures to cleavage double bond C=O into C–C. The increasing temperature in the hydrocracking process is needed for the catalytic activity and contributes to breaking the long molecular chain of triglyceride compounds into shorter molecular chains, following research by Zhao *et al.* [18] and Yang *et al.* [19].

Figure 4(a) shows when used nano HZSM-5 catalyst, after raised the temperature to 350 °C, the carboxylic acids were converted into n-paraffin (24.65%), cycloparaffin (0.32%), aromatics (33.46%), and olefin (0.37%). The main compounds of n-paraffin, *i.e.* pentadecane and heptadecane; aromatic compounds, *i.e.* heptyl-benzene, 1-methylheptyl-benzene, 1-methylnonyl-benzene, *etc.* The temperature reaction was raised, the component of carboxylic acids was reduced, and the content of hydrocarbon increased. This shows that the HZSM-5 catalyst can form aromatic compounds due to its high acidity and good thermal stability. Furthermore, based on BET characterization, the nano-HZSM-5 catalyst has a large surface area (307.8874 m<sup>2</sup>/g) so that molecules from vegetable oil can diffuse and enter the pores, resulting in high aromatic compounds. These results indicated that nano HZSM-5 has a good catalyst to conversion of

triglycerides into formation of aromatic compounds [20–25].

Figure 4(b) shows the chemical composition of biofuel over Fe-La/NHZ-1 catalyst at temperature of 350 °C. The component of biofuel such as n-paraffin (18.59%) *i.e.* decane, undecane, dodecane, tridecane, tetradecane, pentadecane, hexadecane, and heptadecane, *etc.*; cycloparaffin (2.32%) *i.e.* decyl- cyclopentane, undecyl- cyclohexane; aromatics (33.21%) *i.e.* propyl-benzene, 1-methyl-3-propyl-benzene, 1-methyl-4-butyl-benzene, 1,3-dimethylbutyl-benzene, heptyl-benzene, *etc.*; and olefin (0.72%) *i.e.* 1-3-methylphenyl-4-methyl-3-pentene. Pentadecane (C15) and heptadecane (C17) were the main product in the biofuel through decarbonylation and decarboxylation reaction with odd carbon number of n-paraffins. In addition, the EDAX results revealed that Fe and La in the nano HZSM-5 catalyst were well dispersed and had enhanced effects to improve the reaction efficiency to produce aliphatic and aromatic compounds. This result is consistent with previous studies [9,26].

Figure 4(c) shows the chemical composition of biofuel over Fe-La/NHZ-2 catalyst. The component of biofuel, such as n-paraffin (16.82%); cycloparaffin (0.79%); aromatics (35.51%), *i.e.* propyl-benzene, 1-methyl-propyl-benzene, 1-methyl-4-butyl-benzene, 1,3-dimethylbutyl-benzene, heptyl-benzene, 1-methylheptyl-benzene, *etc.*; and olefin (1.07%), *i.e.* tetradecane. The data shows that the aromatic compounds increased by 2% compared to using the Fe-La/NHZ-1 catalyst. Li *et al.* [27] reported with Fe metal loading on ZSM-5 increased Mono Aromatic Hydrocarbon (MAH) and decreased coke content significantly. La loading on the nano HZSM-5 had positive effects improve the aromatics due to the increase of metal active sites and acidity sites. This suggested that nano HZSM-5 loaded Fe-La facilitated for the promotion of oligomerization and aromatization reactions, with bifunctional character of acidic and metal sites [9].

### 3.3 Effect of Reaction Temperature on the Gasoline / Kerosene / Gasoil Yields

The effect of reaction temperature on the gasoline / kerosene / gasoil yields from the hydrocracking process using a) nano HZSM-5, (b) Fe-La/NHZ\_1, and (c) Fe-La/NHZ\_2 with a variation of temperatures 250, 300, and 350 °C are shown in Figure 5. The fraction of product was divided into three fractions, such as gasoline / kerosene / gasoil. Hydrocarbons with a chain length of C<sub>5</sub>-C<sub>9</sub> are known as the gasoline fraction, the chain length of C<sub>10</sub>-C<sub>13</sub> is known as the kerosene fraction, and the chain length of C<sub>14</sub>-C<sub>22</sub> is known as the diesel fraction. Figure 5 shows that increasing the temperature of the

hydrocracking reaction causes triglycerides with long carbon chains to break down into shorter molecules, forming gasoline, kerosene and gasoil fraction components.

Figure 5(a) shows at a temperature of 250 °C, the yield of gasoline fraction (C<sub>5</sub>-C<sub>9</sub>) was not detected over nano HZSM-5 catalyst. The yield of kerosene and gasoil was increased when the temperature was raised. At a temperature of 300 °C, the yield of kerosene was 2.71% and changed to 30.81% at temperature of 350 °C. This is also the same as the yield from gasoil. At a temperature of 300 °C was 13.59% changed to 25.9%. Figure 5(b) shows while using Fe-La/NHZ\_1, at a temperature of 250 °C, a small yield of kerosene and gasoil was detected, 1.41% and 2.65%, respectively. When the temperature raised from 300 to 350 °C, small yield of gasoline was detected, 0.77%. Yield of kerosene was 2.26% and changed to 24.99%, and yield of gasoil was 12.11% changed to 26.26%.

Figure 5(c) shows while using Fe-La/NHZ\_2, at a temperature of 250 °C, small yield of kerosene and gasoil was detected, 0.8% and 0.84%, respectively. When the temperature raised from 300 to 350 °C, small yield of gasoline was detected, 1.63%. Yield of kerosene was 4.2% and changed to

27.62%, and yield of gasoil was 6.12% changed to 21.97%. According to Qi and Fan [28], the gasoline fraction (C<sub>5</sub>-C<sub>9</sub>) produced very little because it cracks in the gas phase, or the cracking process is more stable towards shorter carbon chains when using high temperatures. As reported by Mo and Savage [29], the temperature significantly affected on the yields of hydrocarbon biofuel. In addition, metals, such as Fe and La, could change the acidity strength of HZSM-5 as support and show higher catalytic C-C bond cleavage activity [30,31].

#### 4. Conclusions

In this study, the influence of the reaction temperature on the hydrocracking of Sunan candlenut oil into biofuel has been studied in a batch reactor using nano HZSM-5 and Fe-La/NHZ catalysts. The obtained data confirmed that the raised temperature affected the conversion of triglycerides into hydrocarbons over zeolite-based catalysts. In all cases, zeolite-based catalysts are the most suitable to produce aromatic hydrocarbons. The use of Fe-La/NHZ\_2 catalyst, the highest aromatic component, reached 35.51%. The Fe and La loading into nano HZSM-5 could enhance the acid and metal sites to convert longer carbon chain molecules into shorter ones, such as n-paraffin (aliphatic) and aromatic compounds.

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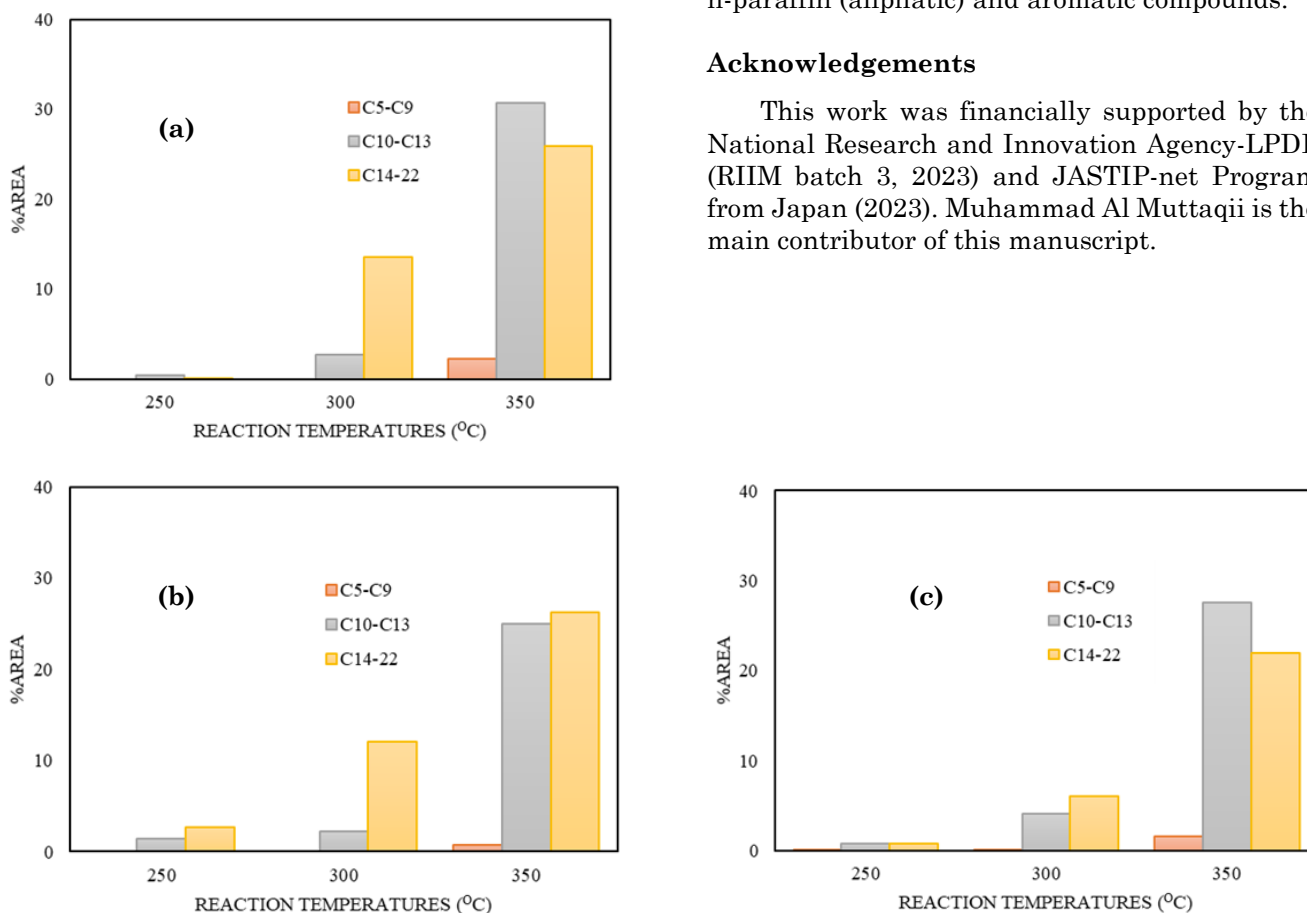


Figure 5. The effect of reaction temperature on chemical composition of biofuel from hydrocracking process using a) nano HZSM-5, (b) Fe-La/NHZ\_1 and (c) Fe-La/NHZ\_2.

**CRedit Author Statement**

Author Contributions: *M.A. Muttaqii*: Conceptualization, Methodology, Investigation, Resources, Data Curation, Writing, Review and Editing, Supervision; *N. Rinaldi, M.P. Marbun and S. Sudibyo*: Draft Preparation, Methodology, Investigation; *A. Aunillah, D. Pranowo*: Investigation, Resources, Writing; *H. Hasanudin*: Data Curation, Writing Draft Preparation; *T.B. Bardant*: Validation, Writing, Review and Editing. All authors have read and agreed to the published version of the manuscript.

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