

# Formation of hydrocarbon compounds during the hydrocracking of non-edible vegetable oils with cobalt-nickel supported on hierarchical HZSM-5 catalyst

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**Abstract.** The hierarchical Co-Ni/HZSM-5 catalyst with hierarchical pore structure was prepared by desilication and incipient wetness impregnation. Hydrocracking of non-edible vegetable oils at temperature of 400 °C, 20±5 bar for 2 h was performed in the presence of this type of catalyst under hydrogen initial pressure in pressured batch reactor. Non-edible vegetable oils, such as Reutealis trisperma (Blanco) airy shaw (sunan candlenut) and Hevea brasiliensis (rubber seed) were chosen to study the effect of the degree of saturation and lateral chain length on hydrocarbon compounds obtained through hydrocracking. Cerbera manghas oil was also tested for comparison because the composition of fatty acid was different with the other oils. The hydrocracking test indicated that liquid product produced has a similar hydrocarbon compounds with petroleum diesel. The most abundant hydrocarbon is pentadecane (n-C15) and heptadecane (n-C17). The high aromatic compounds were found in liquid product produced in hydrocracking of Sunan candlenut oil.

## 1. Introduction

The non-edible vegetable oils used as feedstock in biofuel production is considerable because of their chemical structure due to their energy capacity ability. The carbon chains of triglycerides are similar to the carbon chains of petroleum diesel. However, as reported by previous study [1], the vegetable oil has properties which are not beneficial if used directly as fuel. The high viscosity, high flash point and low heating value compared to the fossil diesel were related with the presence of oxygen atoms in the form of carboxyl or carbonyl groups. Several technology processes have been applied to reduce a high viscosity of vegetable oil such as transesterification in methanol. However, fatty acid methyl esters (FAMEs) produced through this technology have a poor energy density and oxidation stability compared to fossil diesel because of an excess of oxygen. In addition, according to Kim et al. [2] the impurities produced such as saturated free fatty acid, unreacted triglycerides, diglycerides and catalyst can cause problems on a machine seperti clogging fuel filters and swelling. Hydrocracking of non-edible vegetable oils with HZSM-5 catalyst is considerable to obtain free-oxygen biofuel containing paraffin-rich hydrocarbons as an alternative fuel. The content of olefins, iso-paraffins, cycloparaffins



and aromatics is found in a small amount during hydrocracking process through dehydrogenation, isomerization, cyclization and aromatization reactions.

The previous result stated that a certain level of poly-unsaturation of the fatty acids is required to produce relatively high aromatics content. When Pt/Al<sub>2</sub>O<sub>3</sub>/SAPO-11 catalyst is used, about 15 wt.% of aromatics are produced via hydrotreating of the poly-unsaturated soybean, sunflower and camelina oils while hydrotreating of palm or castor oils produces aromatics of 2 wt.%. In addition, the reaction condition and type of catalyst influence type and amount of hydrocarbon compounds [3]. The presence of hydrocarbon compound will determine biofuel properties so that it is important to understand and control these reactions during hydrocracking.

Da Rocha Filho et al. [4] stated that cyclization reaction was promoted at high temperature of hydrogenation favoured cracking and dehydrogenation and finally aromatics were formed. Sotelo-Boyas et al. [5] reported that Pt/zeolite catalyst used in hydrocracking of rapeseed oil produced more iso- than n-paraffin in the range of C5-C22. The presence of isoparaffin can improve cold flow properties. Chen et al. [6] produced C5-C18 of liquid paraffins from catalytic hydroprocessing of FAMES using Ni/HZSM-5 catalyst. According to Marlinda et al. [7], when hydrocracking of Cerbera manghas oil with Co-Ni/HZSM-5 was applied at temperature of 350 °C, a small alkylbenzenes were found such as n-pentylbenzene and hexylbenzene and cycloparaffins were detected in form of n-nonylcyclohexane and isobutylcyclopentane. A more high n-paraffins were produced at this reaction condition. Al-Muttaqii et al. [8] reported that when Co-Ni/HZSM-5 was used in hydrocracking of Sunan candlenut, a slightly n-paraffins were produced at temperature of 350 °C and aromatic hydrocarbons had been found in liquid product. Because the decarboxylation and/or decarbonylation reactions were endothermic reaction (Table 1), the reaction temperature above 350 °C was needed to produce more n-paraffins [6]. The presence of hierarchical pore structure on the Co-Ni/HZSM-5 catalyst increased n-paraffins (C11-C18) from 11.95 to 34.18 area% through hydrocracking of Sunan candlenut oil at 375 °C and produced a more high aromatics of 46.32 area% in liquid product, compared with HZSM-5 catalyst. It was different with a previous result [8], a various aromatic compounds were found and (1-methyldecyl) benzene was predominant in liquid product. A small cycloparaffin was also found [9]. Hierarchical pore structure was expected to solve problems about mass transport of triglyceride molecules. The hierarchical HZSM-5 has a mesoporous material properties (fast diffusion and accessibility of large molecules) and micropores (strong acidity and high hydrothermal stability). These hierarchical pore structure of zeolite allows large molecules that can not diffuse into the micropores will continue to experience a catalytic reaction in the mesoporous wall. According to Sandstede and Lehmann [10], the hydrophobic properties owned zeolite can crack vegetable oils containing triglycerides.

**Table 1.** Plausible reactions during hydrotreatment [6].

Liquid phase reactions			$\Delta G_{573}$ (kJ/mol)	$\Delta H_{573}$ (kJ/mol)
I.	Decarboxylation :	R-COOH → R-H+CO <sub>2</sub> (g)	-83.5	9.2
II.	Decarbonylation :	R-COOH → R'-H+CO(g)+H <sub>2</sub> O(l)	-17.0	179.1
		R-COOH+H <sub>2</sub> (g) → R-H+CO(g)+H <sub>2</sub> O(l)	-67.6	48.1
III.	Hydrodeoxygenation : (HDO)	R-COOH → R-CH <sub>3</sub> +2H <sub>2</sub> O(l) R=saturated alkyl group, R'=unsaturated alkyl group	-86.1	-115.0

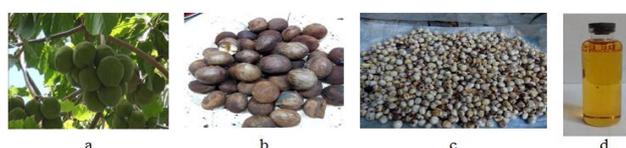
The hierarchical HZSM-5 catalyst was well prepared in order to enhance transport of reactant and product molecules from to zeolite pore. Desilication or alkaline treatment process was chosen to create intracrystalline in ZSM-5 framework. Although, the properties of the catalyst have been used in hydrocracking of vegetable oil to biofuel which had hydrocarbon compounds similar to petroleum diesel, the effects of different degrees of saturation and chain lengths on hydrocarbon compounds distribution obtained through hydrocracking of non-edible vegetable oil are still unclear for the hierarchical HZSM-5 catalyst. In this work, the reutealis trisperma (Blanco) airy shaw (sunan candlenut) and hevea brasiliensis (rubber seed) were chosen to investigate the effects. Cerbera manghas oil was also tested for comparison because the composition of fatty acid was different with

the oils used in this work. In addition, according to the result in previous work [8], [9], the effect of the high temperature (at 400 °C) in forming the hydrocarbon compounds, such as paraffin-rich hydrocarbon, cycloparaffins, aromatics, and olefins in liquid product were also studied.

## 2. Material and Methods

### 2.1. Material

Sunan candlenut oil was extracted from seeds, as shown in Figure 1. The fruits was collected from Bogor, West Java, Indonesia and afterwards the seeds was pressed with screw press mechine to obtain oil. The oil content obtained was about 50 wt.% of dried seeds, as reported by Syafaruddin and Wahyudi [11]. While, rubber seeds was collected from Jember, East Java, Indonesia and pressed with screw press mechine to obtain rubeer seed oil. Non-edible vegetable oils were analyzed using gas chromatography-mass spectrometry (GC-MS) to determine composition of fatty acids.



**Figure 1.** Sunan candlenut (a) fruit, (b) seed before skin removed, (c) seed after skin removed, (d) oil.

### 2.2. Catalyst preparation and characterization

The Ammonium-ZSM-5 zeolite was purchased from Zeolyst International (CBV 8014, a surface area of 400 m<sup>2</sup>/g, Na<sub>2</sub>O content less than 0.05 wt.%) and calcined at 550 °C for 5 h to HZSM-5 zeolite. Another chemicals were Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O purchased from Merck with ≥98 % purity. The hierarchical Co-Ni/HZSM-5 catalyst used in this work had been prepared with procedure as reported by Marlinda et al. [9]. The first step, the hierarchical HZSM-5 catalyst was prepared in stainless steel vessel through desilication on HZSM-5 zeolite within 0.2 M NaOH solution and kept under stirring at 65 °C for 2 h. The second step, the sample was modified by designed concentration of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O aqueous solution with incipient wetness impregnation, then calcined at 400 °C for 2 h with air and reduced at 450 °C for 3 h to obtain metallic phase. Finally the hierarchical Co-Ni/HZSM-5 catalyst was obtained. Furthermore, the detailed characterizations of the hierarchical Co-Ni/HZSM-5 catalyst have been described in previous report [9]. The creating mesoporosity in this catalyst was also effected by cobalt-nickel supported on hierarchical HZSM-5.

### 2.3. Hydrocracking process

As reported by previous study [12], the batch reactor equipped with a mechanical stirrer was used to make biofuel from non-edible vegetable oil by hydrocracking process. The hydrocracking process was conducted under hydrogen initial pressure in pressured batch reactor equipped with a mechanical stirrer with procedure as reported by Marlinda et al. [12]. The non-edible vegetable oil of 200 ml was placed into the reactor, which was containing the catalysts of 1 g. Then, a reactor was purged with nitrogen to remove air dissolved in oil for 30 min, as reported in previous study [13]. The catalyst was activated under flowing H<sub>2</sub> for 1 h. In previous study, Martinez-Grimaldo et al. [14] activated hydrocracking catalyst at 350 °C for 1 h. The reaction temperature was carried out at a temperature of 400 °C for 2 h. In practically, the reaction pressure changed between 10 bar and 15 bar.

Liquid products were analyzed by gas chromatography-mass spectrometry with standard GC : Agilent HP 6890 models 19091S-433, HP-5MS capillary column 30 m x 250 μm x 0.25 μm. This analyze was held at nominal initial pressure 13.3 psi and the temperature for the oven was at 150 °C for 2 min, then it increased to 240 °C at a rate of 10 °C/min for 11 min. The Wiley275 and NIST02 mass spectral library of data were used to determine hydrocarbon compounds. The hydrocarbon components with a probility match equal to or higher than 80% were considered. N-paraffin, aromatic, cyloparaffin, and

olefin contained in liquid product were grouped as gasoline-like hydrocarbon (C5-C9), kerosene-like hydrocarbon (C10-C13), and gasoil-like hydrocarbon (C14-C22), as reported by Barron et al. [15].

### 3. Result and Discussion

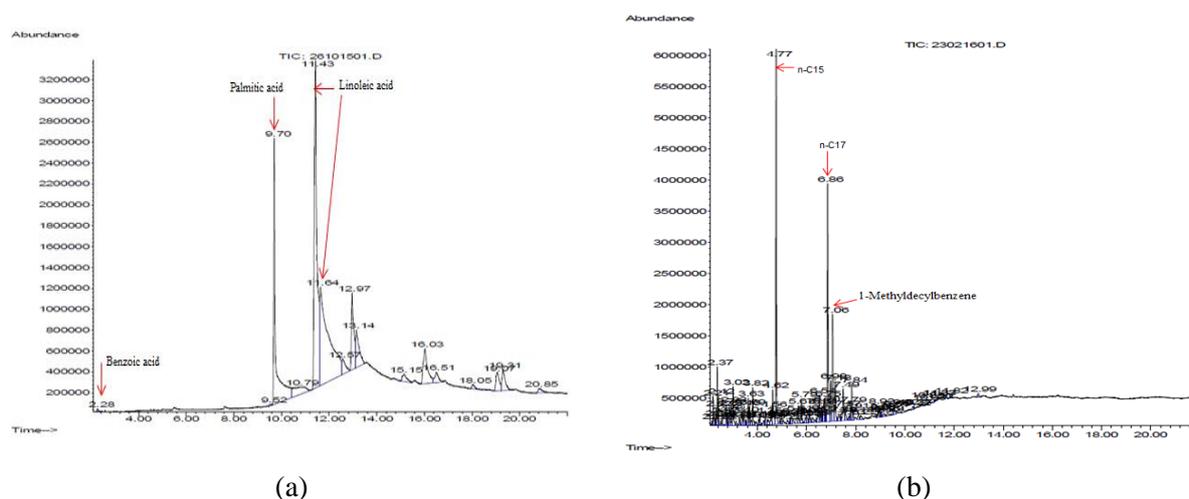
#### 3.1. Characterization of non-edible vegetable oils

According to previous work [9], [17], a high content of poly-unsaturated fatty acids found in both the sunan candlenut and rubber seed oils were around 50 % and 95.2 %, respectively. Linoleic acid was the main component. In addition, palmitic acid as saturated fatty acids was found in around 3.67 - 20.3 area%. While, cerbera manghas oil contained mono-unsaturated fatty acid [7].

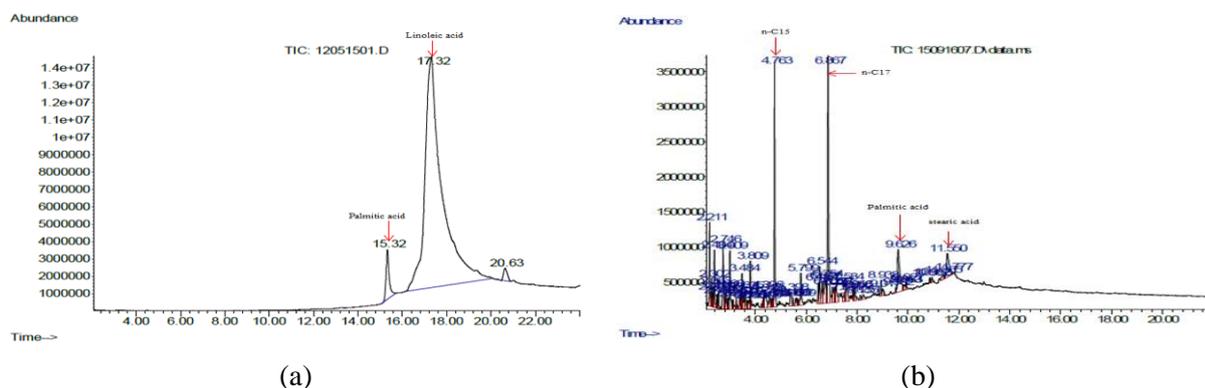
#### 3.2. Analysis of Biofuel

GC-MS chromatogram of Sunan candlenut oil and liquid product produced at temperature of 400 °C are shown in Figure 2. The compounds with retention time of 10-20 min in the Sunan candlenut oil chromatogram disappear after hydrocracking reaction [9]. As shown in Figure 2 (b), the chromatograms of liquid product showed that peaks at retention time of 0-12 min appeared and indicated the increasing a large amount of hydrocarbon compounds. It implied that triglycerides were converted into short chain molecules by several reaction sequence [16], including interconnected reactions of triglyceride molecules and intermediates such as hydrogenolysis forming fatty acids and propane, deoxygenation of carboxylic groups in fatty acids, cyclization, dehydrogenation of cyclic ring to aromatic ring, hydrocracking of alkyl chains, and isomerization of alkyl chains [3].

The similar phenomenon can be also observed in GC-MS chromatogram of rubber seed oil and liquid product produced at temperature of 400 °C, as shown in Figure 3. A small peaks intensity of fatty acid such as palmitic acid and stearic acid were detected in GC-MS chromatogram of rubber seed oil liquid product so that it can be implied that trilinoleic was successfully hydrogenated and cracked into saturated fatty acids at 400 °C with hierarchical Co-Ni/HZSM-5 catalyst. It was interesting to be studied later, a peaks intensity of aromatic (e.g. 1-methyldecylbenzene) were detected in GC-MS chromatogram of sunan candlenut oil liquid product. In addition, pentadecane and heptadecane, the most abundant n-paraffin were detected at retention time of 4.73-4.77 and 6.67-6.86 respectively, as presented in Figure 2 and Figure 3.



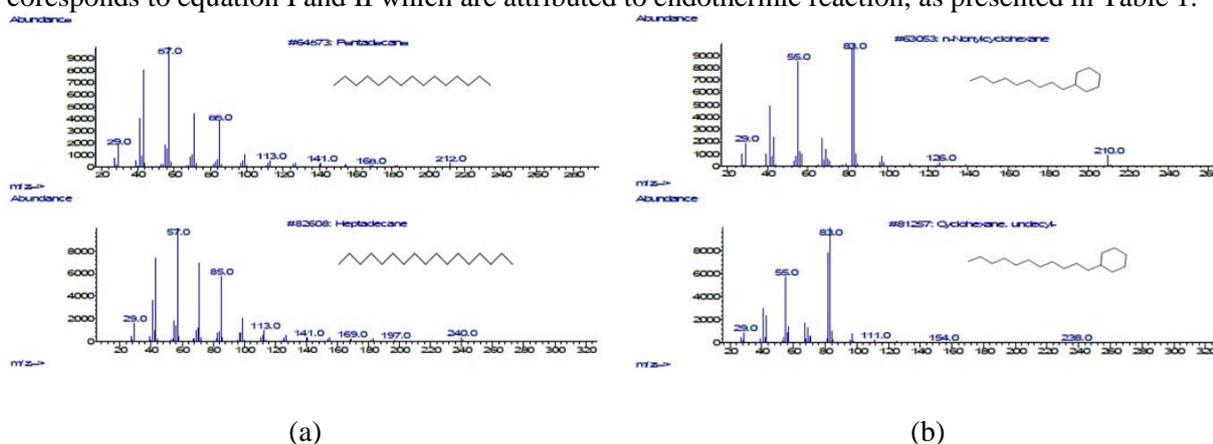
**Figure 2.** GC-MS chromatogram of (a) Sunan candlenut Oil [9], (b) liquid products produced at 400 °C using hierarchical Co-Ni/HZSM-5 catalyst.



**Figure 3.** GC-MS chromatogram of (a) Rubber seed oil [17], (b) liquid products produced at 400 °C using hierarchical Co-Ni/HZSM-5 catalyst.

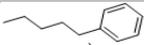
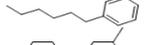
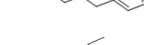
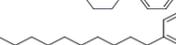
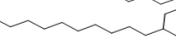
### 3.3. Effect of non-edible vegetable oil composition on formation of paraffins, cycloparaffins and alkylbenzenes

The presence of n-paraffins was identified on mass spectra in Figure 4 (a). It was found that pentadecane (molecular weights of 212) and heptadecane (molecular weights of 240) were produced from hydrocracking of all non-edible vegetable oils with the hierarchical Co-Ni/HZSM-5 catalyst. While hydrocracking of cerbera manghas oil with the Co-Ni supported on HZSM-5 catalyst also indicated the presence of n-C15 and n-C-17 in liquid product, as listed in Table 2. The abundant carboxylic acids in liquid product from hydrocracking of cerbera manghas oil showed that the HZSM-5 catalyst without hierarchical pore structure less increased the conversion level of carboxylic acid to hydrocarbon compounds. In the previous report [7], [8], a high n-paraffin content in liquid product was reached for hydrocracking of cerbera manghas oil. According to Da Rocha Filho et al. [4], fatty acid composition of oil was significant factor on the formation of n-paraffin. It can be also observed in Table 2, large amount of alkylbenzenes were only produced via hydrocracking of sunan candlenut oil. While, long chain saturated fatty acids and olefins were not found. Small amount of olefins (less than 4 area%) and aromatic were found in cerbera manghas oil liquid product. It can be said that degree of saturation of vegetable oil and reaction temperature also effected the hydrocarbon product distribution. As reported by previous study [7], [12], olefin content decreased with the increasing reaction temperature. While, the normal paraffins were produced via decarboxylation and/or decarbonylation with elimination of oxygen atoms as CO<sub>2</sub>, CO and H<sub>2</sub>O. This phenomenon corresponds to equation I and II which are attributed to endothermic reaction, as presented in Table 1.

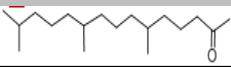


**Figure 4.** Mass spectra of (a) n-paraffins, (b) cycloparaffins in liquid product produced from hydrocracking of non-edible vegetable.

**Table 2.** Hydrocarbon composition of liquid product (area% of liquid product) at temperature of 400 °C, 20±5 bar for 2 h.

Compound/Formula	Structure	Abundance (area%)		
		I <sup>a</sup>	II <sup>b</sup>	III <sup>c</sup>
<b>n-paraffin</b>				
Dodecane/C <sub>12</sub> H <sub>26</sub>		1.53	1.05	2.03
Tridecane/C <sub>13</sub> H <sub>28</sub>		2.06	2.02	2.96
Tetradecane/C <sub>14</sub> H <sub>30</sub>		1.60	1.75	1.86
Pentadecane/C <sub>15</sub> H <sub>32</sub>		11.49	17.48	11.04
Hexadecane/C <sub>16</sub> H <sub>34</sub>		1.43	1.58	2.25
Heptadecane/C <sub>17</sub> H <sub>34</sub>		7.24	13.42	14.67
Octadecane/C <sub>18</sub> H <sub>38</sub>		0.50	0.52	0.65
Nonadecane/C <sub>19</sub> H <sub>40</sub>		1.12	0.65	0.82
Eicosane/ C <sub>20</sub> H <sub>42</sub>		0.6	0	0.55
Heneicosane/C <sub>21</sub> H <sub>44</sub>		0.91	0	0
Tricosane/C <sub>23</sub> H <sub>48</sub>		0.69	0	0
Pentacosan /C <sub>25</sub> H <sub>52</sub>		0	0	0.31
<b>Aromatic</b>				
Pentylbenzene/C <sub>11</sub> H <sub>16</sub>		0.38	0.54	0
(3-Methylbut-2-en-2-yl)benzene/C <sub>11</sub> H <sub>14</sub>		0	0.26	0
Cyclopentacycloheptene/Azulene/C <sub>10</sub> H <sub>8</sub>		0	0.57	0
Hexylbenzene/C <sub>12</sub> H <sub>18</sub>		0	0.82	0
1,3-Dimethylbutylbenzene/C <sub>12</sub> H <sub>18</sub>		0	0.86	0
1-Methylnaphthalene/C <sub>11</sub> H <sub>10</sub>		0	0.39	0
2-Methylnaphthalene/C <sub>11</sub> H <sub>10</sub>		0.47	0.66	1.42
1-Heptylbenzene/C <sub>13</sub> H <sub>20</sub>		0	1.39	0
1-Methyl-2-n-hexylbenzene/ C <sub>13</sub> H <sub>20</sub>		0	0.89	0
1-Ethyl-naphthalene/ C <sub>12</sub> H <sub>12</sub>		0	0.31	0
2-Ethyl-naphthalene		0	0.55	0
1,3-Dimethylnaphthalene/ C <sub>12</sub> H <sub>12</sub>		0	0	0.35
Octylbenzene/ C <sub>14</sub> H <sub>22</sub>		0	1.05	0
n-Nonylbenzene/ C <sub>15</sub> H <sub>24</sub>		0	0.89	0
1-Methyldecylbenzene/C <sub>17</sub> H <sub>28</sub>		0	9.58	0
n-Undecylbenzene/C <sub>17</sub> H <sub>28</sub>		0	0.88	0
<b>Cycloparaffin</b>				
(1,3-Dimethylbutyl)cyclohexane		0	1.07	0
Nonylcyclohexane/ C <sub>15</sub> H <sub>30</sub>		1.46	0.71	1.46
n-Undecylcyclohexane/ C <sub>17</sub> H <sub>34</sub>		1.80	1.02	2.05
1-(Cyclohexylmethyl)-2-ethylcyclohexane /C <sub>15</sub> H <sub>28</sub>		0	0	1.42
Cyclopentadecane		0.69	0	0
Cyclopentane, nonyl-		0.65	0	0
Cyclotetracosane		0.31	0	0
<b>Olefin</b>				
1-Heptadecene/C <sub>17</sub> H <sub>34</sub>		0.51	0	0.68
1-Nonadecene		0	0	0.47
1-Docosene		0	0	0.11
4-Dodecene		0.51	0	0
2-Tridecene		1.05	0	0
3-Heptadecene		1.41	0	0
<b>Carboxylic acid</b>				
Octanoic Acid/ C <sub>8</sub> H <sub>16</sub> O <sub>2</sub>		1.79	1.22	3.38
Nonanoic acid/ C <sub>9</sub> H <sub>18</sub> O <sub>2</sub>		3.03	1.05	3.64
n-Decanoic acid/C <sub>10</sub> H <sub>20</sub> O <sub>2</sub>		3.29	1.19	1.94
Undecanoic acid/C <sub>11</sub> H <sub>22</sub> O <sub>2</sub>		1.33	0	1.78
n-Hexadecanoic acid		19.03	0	6.8
Octadecanoic acid		11.21	0	4.6
Oleic Acid		4.71		

Continued . .

Oxygenated compound				
6,10,14-Trimethyl-2-pentadecanone/ C <sub>18</sub> H <sub>36</sub> O		0	0.36	0

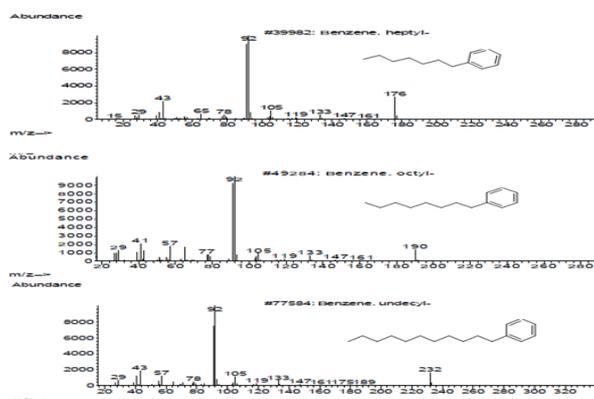
<sup>a</sup>Liquid product produced from hydrocracking of cerbera manghas oil over Co-Ni/HZSM-5 catalyst,

<sup>b</sup>Liquid product produced from hydrocracking of sunan candlenut oil,

<sup>c</sup>Liquid product produced from hydrocracking of rubber seed oil.

The cyclization and C-C bond cleavages were enhanced by the presence of a large amount of unsaturated component in vegetable oil, as reported Da Rocha Filho et al. [4]. Rabaev et al. [3] also stated that the formation of alkylbenzenes were determined by poly-unsaturation fatty acids in vegetable oil. As presented in Figure 4 (b), cycloparaffins with molecular weights of 210 and 238 found in liquid product for all vegetable oils showed that sites migration of unsaturation have occurred. Only in liquid product produced from hydrocracking of sunan candlenut oil, some of alkylbenzenes with molecular weights of 176, 190, and 232 were found, as shown in Figure 5. Small amount of naphthalene compounds found in liquid product for rubber seed oil were 2-methylnaphthalene and 1,3-dimethylnaphthalene.

It is interesting to study that the presence of alkylcycloparaffin was proportional to the presence of corresponding n-paraffin, such as if a large amount of n-C<sub>17</sub>H<sub>36</sub> with molecular weight of 240 was present, a large amount of the corresponding n-undecylcyclohexane with molecular weight of 238 could be observed. This phenomenon can be explained with multistep sequence for the hydrocracking of the vegetable oils over a NiMo/Al<sub>2</sub>O<sub>3</sub> catalyst [4]. In practically, triglyceride hydrogenation is uncomplete reaction at certain reaction temperature so that unsaturated fatty acids will produce and the decarboxylation and/or reduction on them can produce unsaturated hydrocarbons due to proceed cyclization into alkylcycloparaffins. In addition, alkylcycloparaffin was obtained via cyclization of alkenes produced by the thermally and/or catalytically dehydrogenation on n-paraffins as the main product hydrocarbons.



**Figure 5.** Mass spectra of alkylbenzenes in liquid product produced from hydrocracking of sunan candlenut oil.

#### 4. Conclusion

According to the result, the hierarchical Co-Ni/HZSM-5 catalyst with hierarchical pore structure can contribute to control various reactions occurred in hydrocracking process at 400 °C, 20±5 bar for 2 h, including hydrogenation, cracking, cyclization, and aromatization. Large amount of alkylbenzenes were produced via hydrocracking of sunan candlenut oil. It can be said that degree of saturation of vegetable oil and reaction temperature effected the hydrocarbon product distribution. The most abundant hydrocarbon is pentadecane (n-C<sub>15</sub>) and heptadecane (n-C<sub>17</sub>). The future work including biofuel purification into gas oil and development catalyst modified was expected to be applied in low reaction temperature with remain keep the quality of biofuel.

## 5. References

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