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# Pyrolytic conversion of palm oil into liquid fuel using protonated zeolite-X prepared from rice husk silica and aluminum foil as catalyst

T D Febriyanti<sup>1</sup>, W Simanjuntak<sup>2,\*</sup>, K D Pandiangan<sup>2</sup>

<sup>1</sup> Postgraduate Student of Chemistry Department, University of Lampung, Jl. Sumantri Brodjonegoro no 1, Bandar Lampung 35145, Indonesia

<sup>2</sup> Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Lampung, Jl. Sumantri Brodjonegoro no 1, Bandar Lampung 35145, Indonesia

**email:** wasinton.simanjuntak@fmipa.unila.ac.id<sup>2,\*</sup>, tikadwifebriyanti16@gmail.com<sup>1</sup>,  
kamisah.delilawati@fmipa.unila.ac.id<sup>2</sup>

**Abstract.** Zeolite-X was successfully synthesized based on silica of rice husk and aluminum foil with the variation of sodium silicate aging time of 24, 48, 72, and 96 hours with a crystallization time of 96 hours as evidenced by XRD analysis with a  $2\theta$  angle peak diffractogram pattern similar to the IZA standard. Zeolite-X with sodium silicate aging time of 24 hours is the best zeolite-X with XRF analysis with components Na<sub>2</sub>O 0.436%, Al<sub>2</sub>O<sub>3</sub> 33.933%, and SiO<sub>2</sub> 65.631%, and the SEM surface morphology shape crystal was homogeneous cubes. Protonated zeolite-X or zeolite H-X has been successfully synthesized through the ion exchange process of zeolite-X with 2M NH<sub>4</sub>NO<sub>3</sub> solution which had been proven by XRF analysis to reduce Na<sub>2</sub>O from 0.436% to 0.179%. Zeolite-X and zeolite H-X have the same  $2\theta$  angle diffractogram pattern. In SEM analysis, the surface morphology of zeolite H-X had a formless surface morphology. Zeolite H-X was applied in pyrolysis experiments using palm oil to produce liquid fuel. Liquid fuel was analyzed by GC-MS with components 77% hydrocarbon, 16% acid, and 7% ketones.

**Keywords:** zeolite-X, zeolite H-X, pyrolysis, liquid fuel.

## 1. Introduction

The need for fuel was increasing along with human growth. All aspects of human needs including transportation, household and industrial needs require fuel. It had resulted in the depletion of petroleum reserves which can lead to fuel scarcity, so was is necessary to anticipate using alternative fuels, one of which is liquid fuel. Liquid fuel is a renewable alternative fuel produced from organic raw materials obtained from the cracking (pyrolysis) of biomass. Liquid fuel resulting from pyrolysis was a fuel that attracts attention because its production does not require specific raw materials, meaning that it can be applied to any biomass. Biomass that has been carried out includes a mixture of used cooking oil and plastic waste [1], karanja seeds [2], coconut oil and palm oil [3,4], palm oil bunches and polystyrene waste [5], a mixture of rubber seed oil and sugarcane bagasse [6], neem seeds [7], and methyl ester from



cooking oil [8]. In this paper using palm oil used as an alternative for liquid fuel due to abundant availability of palm oil in Indonesia and Indonesia is the largest palm oil producing country in the world.

The conversion of biomass to liquid fuel can use a catalyst or without a catalyst. However, the use of catalysts can improve fuel properties such as calorific value and reduce fuel viscosity and increase liquid fuel yield by reducing high pyrolysis conditions (350-450 °C, 5-15 MPa) which leads to the formation of gas and char [9-12]. This article uses a heterogeneous catalyst in the form of zeolites.

Zeolite was an important material used in various applications, one of which is applied as a catalyst used in the petrochemical and oil refining industries to separate and classify molecules according to their dimensions. In applications the adsorbent was used for the purification of air, soil and water, removing radioactive contaminants. Zeolite could be used in collecting waste heat and solar thermal energy, used for household purposes such as detergents. Increasing renewable energy and the environment also requires zeolites, such as converting biomass, fuel cells, capturing and converting CO<sub>2</sub>, and storing heat energy [13].

One type, zeolite-X has a pore size around 8 Å with a cubic surface morphology, and a molar proportion between SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> between 2.2 to 3. The unit of zeolite-X forming structure consists of eight adjacent β-type spheres connected by six rings using oxygen bridges to connect to each other, forming a largest that can be accessed by a twelve-pore system with three-dimensional rings [14]. These distinctive crystal structure characteristics are advantageous for high exchange capacities, adsorption cationic and radionuclear [15].

In this article zeolite-X was synthesized using rice husk silica extracted by the alkaline method [6,16] as the Si source and aluminum foil as the Al source. The use of rice husk silica was profitable because rice husks were very available in nature and renewable. In addition, rice husk silica was cheaper than other types of silica that are commonly used. Use aluminum foil instead of aluminum salt as it was cheaper than aluminum salt. The resulting zeolite was calcined at temperature of 550 °C during six hours. The zeolite-X produced is known as zeolite Na-X. One of the efforts being developed was to convert zeolite Na-X into protonated zeolite-X or zeolite H-X.

Zeolite-X is used in many applications such as catalysts [17-19], gas separation in permanent gas streams [20], wastewater treatment [21], adsorption [22], biomedical [23], rapid softening of water hardness [24], and wastewater treatment [25]. The ability of zeolites as catalysts is related to the active availability centers in the channels between zeolites. These active centers are formed due to the presence of both Brønsted and Lewis-type acid functional groups [13]. Many modification strategies are used to improve the performance of the acid sites or active centers on zeolite-X, one of which is the ion-exchange process [25] In this method Na<sup>+</sup> is placed with H<sup>+</sup> ions.

In this study, zeolite H-X was synthesized from zeolite-X using rice husk silica and aluminum foil in two stages. The first stage was synthesizing zeolite-X then ion-exchange process using 2M NH<sub>4</sub>NO<sub>3</sub> solution [26] which was then recalculated at 550 °C for six hours to get the zeolite H-X. Zeolite H-X was applied to the pyrolysis method to produce liquid fuel.

## 2. Materials and methods

### 2.1. Materials

The materials used in this study included rice husks from rice mills Way Lima, Pesawaran, HNO<sub>3</sub> solution from Aldrich, NaOH pellets and NH<sub>4</sub>NO<sub>3</sub> from Merck, aquadest, universal indicators, filter paper, and aluminum foil.

## 2.2. Instruments

Zeolite-X and zeolite H-X were analyzed using PHILIPS-binary XRD to see the crystalline structure of the zeolite, XRF PANalytical Epsilon 3 to see the content contained in zeolites, and SEM FEI Inspeks S 50 to see the surface morphology of the zeolite. Zeolite H-X is applied to the pyrolysis method to produce liquid fuel which is then analyzed. GCMS-QP2010 SE SHIMADZU, comprehensive with the MS NIST17.LIB Library System is used for the analysis and chemical compounds of the resulting liquid fuel.

## 2.3. Synthesis of zeolite-X

Zeolite-X was synthesized using a hydrothermal method with a composition ratio of  $4\text{Na}_2\text{O} : \text{Al}_2\text{O}_3 : 4.8\text{SiO}_2 : 120\text{H}_2\text{O}$  prepared from a mixture of sodium silicate solution and aluminum foil. The synthesis of zeolite-X was initiated by weighing 11.11 grams of NaOH pellets, 1.875 grams of aluminum foil, and 10 grams of rice husk silica using previous research [6,16]. NaOH pellets were then dissolved with 120 mL of distilled water and added to silica rice husk slowly while stirring for 3 hours in a boiling state. After all the silica has dissolved, the sodium silicate solution was filtered using filter paper to separate it from the impurities that were still present in the solution. Furthermore, the sodium silicate solution was aging for 24, 48, 72, and 96 hours. Next, the sodium silicate solution is added with aluminum foil which has been cut into small pieces by spreading it evenly and then stirring for 3 hours until a homogeneous gel is formed. In the next stage, the gel was transferred to Teflon and put into an autoclave, then aging for 24 hours in a closed state to grow zeolite crystal nuclei. After that, the sample was transferred to an oven to undergo the crystallization stage at  $100\text{ }^\circ\text{C}$  for 96 hours. After the crystallization process was complete, the sample was slowly filtered with filter paper to separate the zeolite formed from the remaining filtrate in the autoclave and washed with distilled water until it reaches pH 8. The sample was then dried in an oven at  $80\text{ }^\circ\text{C}$  for 24 hours. In the final stage, the dried sample was calcined at  $550\text{ }^\circ\text{C}$  for 6 hours to activate the zeolite.

## 2.4 Synthesis of zeolite H-X

Zeolite H-X was made using the ion exchange method, which was exchanging sodium metal with hydrogen metal in zeolite-X. Modification of zeolite-X to zeolite H-X by using the adsorption ion-exchange process [26] used zeolite-X which had been calcined by dissolving zeolite-X into  $2\text{M NH}_4\text{NO}_3$  solution for 6 hours at  $80\text{ }^\circ\text{C}$  with a zeolite/solution ratio of 1/10 mL to obtain zeolite  $\text{NH}_4^+\text{-X}$ , then zeolite  $\text{NH}_4^+\text{-X}$  results was washed using distilled water, filtered, and dried at  $80\text{ }^\circ\text{C}$ . The zeolite  $\text{NH}_4^+\text{-X}$  formed was calcined at  $550\text{ }^\circ\text{C}$  for 6 hours to obtain zeolite H-X.

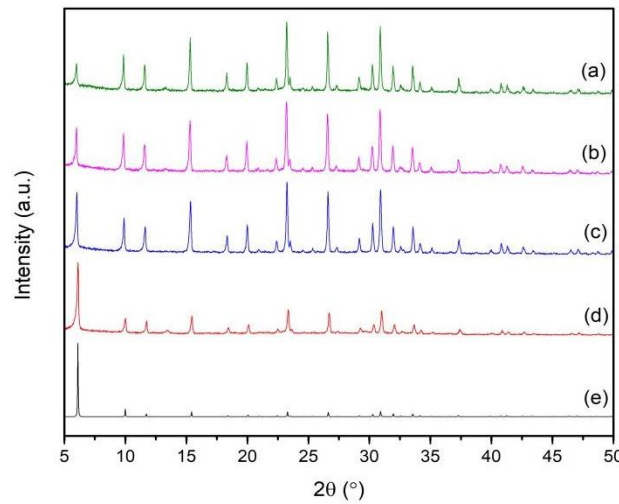
## 2.5 Pyrolysis experiment

Pyrolysis experiment to produce liquid fuel, the first step was to mix 250 mL of palm oil with 12.5 grams zeolite H-X catalyst and then mix well. Then the mixture was put into the pyrolysis reactor which had been assembled on the pyrolysis instrument, then heated to a temperature of  $450\text{ }^\circ\text{C}$ . The steam formed was flowed from the reactor and then cooled in the condenser to produce distillate and the liquid produced was collected. Pyrolysis was carried out for 3 hours. The liquid was transferred into separatory funnel for separation of organic phase (liquid fuel) and water phase. The liquid fuel obtained was then characterization by GCMS, with the support of MS List17.LIB Library System for tentative identification of the chemical compounds of the liquid fuel.

# 3. Results and discussion

## 3.1 Characterization of zeolite-X and zeolite H-X

XRD analysis was carried out to determine the compounds or crystals formed in a crystalline material so that the structure, orientation, and size of the crystals are known. The resulting XRD pattern was matched pair with the XRD pattern of IZA (International Zeolite Association). Zeolite-X had been synthesized using a different sodium silicate aging method has the same diffractogram pattern as the diffractogram pattern of IZA. This proves of zeolite-X had been successfully synthesized in Figure 1.

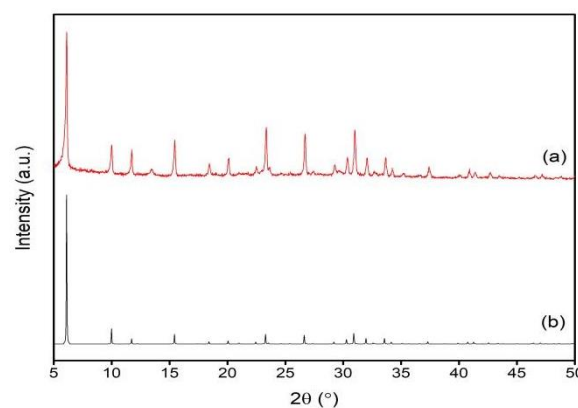


**Figure 1.** XRD zeolite-X diffractogram (a) aging 96 hours, (b) aging 72 hours, (c) aging 48 hours, (d) aging 24 hours, and (e) IZA.

**Table 1:** Comparison of 2θ and the intensity of the XRD diffractogram for zeolite-X IZA standart and zeolite-X.

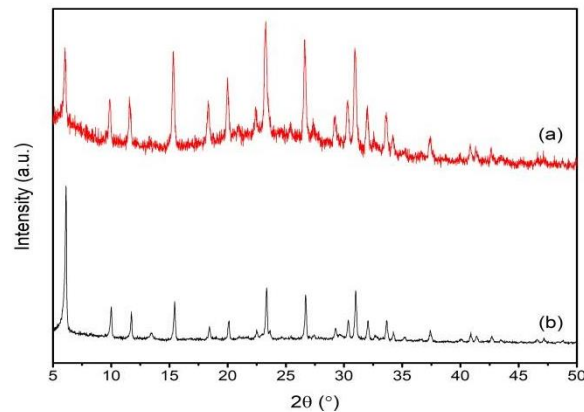
Zeolit-X IZA		Zeolit-X (24 h)		Zeolit-X (48 h)		Zeolit-X (72 h)		Zeolit-X (96 h)	
2θ	%	2θ	%	2θ	%	2θ	%	2θ	%
6,11	100	6,12	100	6,03	80,83	5,99	58,25	5,99	34,42
9,98	10,15	10,01	20,16	9,98	46,44	9,86	48,73	9,85	50,59
15,41	6,09	15,45	25,78	15,34	71,30	15,32	69,81	15,31	76,79
23,28	6,28	23,35	45,74	23,24	100	23,20	100	23,21	100
30,91	5,84	30,37	14,27	30,90	88,99	30,87	90,00	30,88	89,22

From Table 1, it is presented that the four zeolite-X synthesized matching with the 2θ angle of the IZA standart. Zeolite-X synthesized by sodium silicate aging method did not affect the 2θ angle peak shift but did affect its intensity. Zeolite-X with the best 24 hours sodium silicate aging which best comform the peak of the 2θ angle and intensity in Figure 2. Zeolite-X was analyzed XRF containing 0.436% Na<sub>2</sub>O, 33.933% Al<sub>2</sub>O<sub>3</sub>, and 65.631% SiO<sub>2</sub>. This proves that zeolite is the main component of aluminum and silicates.



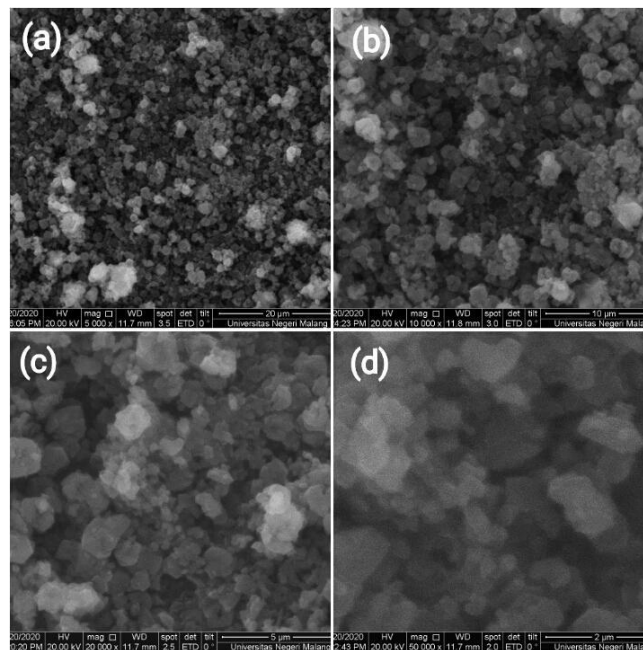
**Figure 2.** XRD zeolite-X diffractogram (a) aging 24 hours (b) IZA.

Zeolite-X with the 24-hours aging with the ion-exchange process using 2M  $\text{NH}_4\text{NO}_3$  solution to form zeolite H-X which was then analyzed using XRF and XRD. In XRF analysis, zeolite-X had been successfully converted into zeolite H-X as evidenced by a decrease in the  $\text{Na}_2\text{O}$  component from 0.436% to 0.179%. In the XRD analysis, there wasn't change in the XRD diffractogram pattern which showed that the ion-exchange process didn't change the crystal structure of the zeolite which was presented in Figure 3.

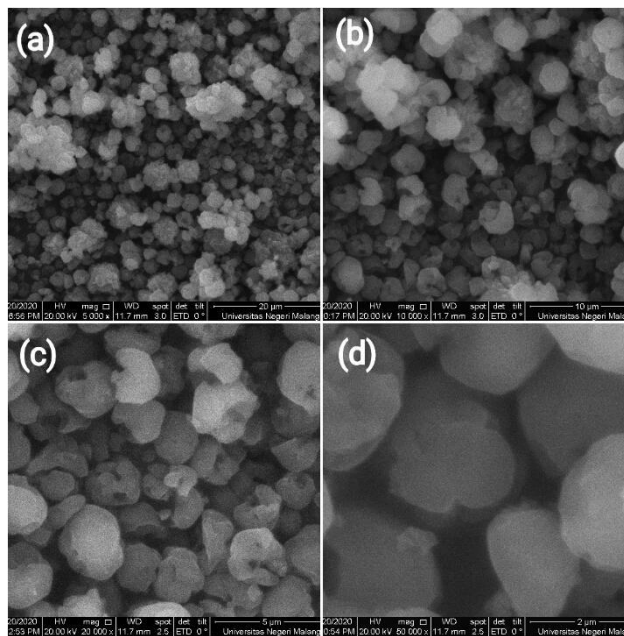


**Figure 3.** XRD diffractogram (a) zeolite H-X, (b) zeolite X.

The results of SEM morphology (Figure 4) depicts zeolite-X synthesized by 24 hours aging method homogeneous cuboid shape and size according to the literature [27]. However, the morphology of SEM zeolite H-X (Figure 4) was synthesized from zeolite-X using the ion-exchange process with 2M  $\text{NH}_4\text{NO}_3$  solution with a different appearance from zeolite-X (Figure 5). In zeolite H-X, the structure changes to be irregular but with homogeneous shape and size.



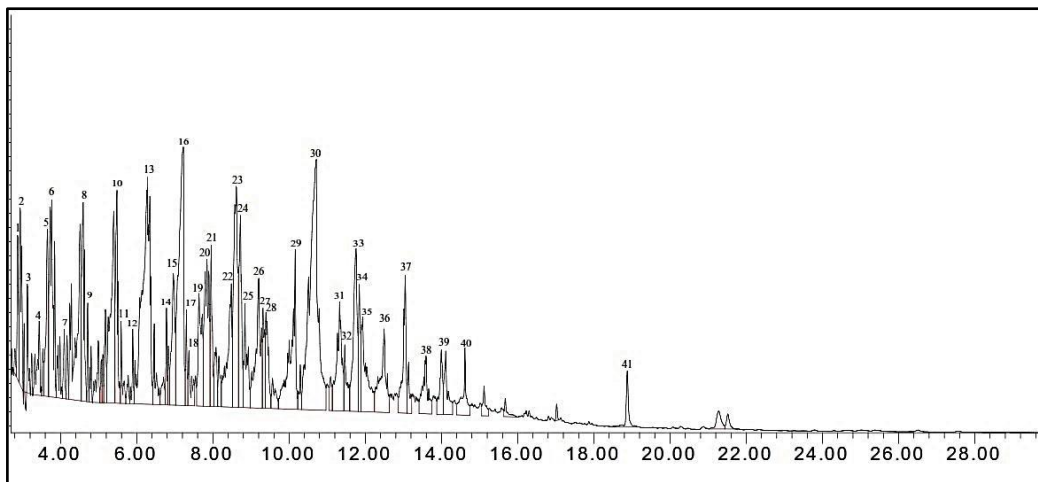
**Figure 4.** The results of the SEM morphology of zeolite-X aging 24 hours with a magnification of (a) 5000x, (b) 10000x, (c) 20000x, and (d) 50000x.



**Figure 5.** The results of the SEM morphology of zeolite H-X aging 24 hours with a magnification of (a) 5000x, (b) 10000x, (c) 20000x, and (d) 50000x.

**3.2 Characterization of liquid fuel**

The GC chromatogram resulting from liquid fuel with palm oil using zeolite H-X is shown in Figure 6.



**Figure 6.** GC-Chromatogram of palm oil fuel with zeolite H-X.

The pyrolysis product of the biomass sample in the form of liquid fuel is reported in the literature [2-7], the liquid fuel GC-chromatogram presented in Figure 6 shows that the liquid fuel consists of many compounds, and it need help with MS NIST17.LIB Library System for identified compounds, as many as 41 temporary identified compounds were described in Table 2.

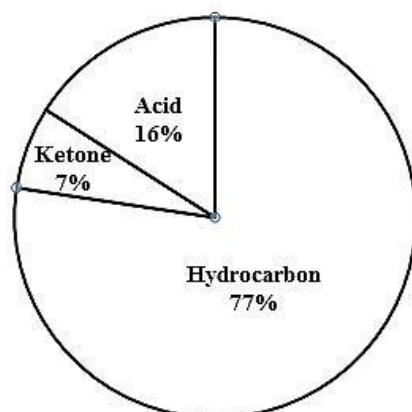
**Table 2:** Compound of palm oil pyrolysis liquid fuel with zeolite H-X catalyst.

Peak No.	Ret. Time (Minute)	Percentage (%)	Compound Name	Formula
1.	2.881	1.04	1-Decene	C <sub>10</sub> H <sub>20</sub>
2.	2.942	2.00	Decane	C <sub>10</sub> H <sub>22</sub>
3.	3.133	0.80	Cycloheptanone	C <sub>7</sub> H <sub>12</sub> O
4.	3.444	1.10	n-Butylbenzene	C <sub>10</sub> H <sub>14</sub>
5.	3.656	1.95	1-Undecene	C <sub>11</sub> H <sub>22</sub>
6.	3.770	4.06	2-Undecene	C <sub>11</sub> H <sub>22</sub>
7.	4.097	0.51	2-Methyldecalin	C <sub>11</sub> H <sub>20</sub>
8.	4.514	4.32	1-Dodecene	C <sub>12</sub> H <sub>24</sub>
9.	4.588	2.68	Dodecane	C <sub>12</sub> H <sub>26</sub>
10.	5.396	4.02	1-tridecene	C <sub>13</sub> H <sub>26</sub>
11.	5.474	2.46	Tridecane	C <sub>13</sub> H <sub>28</sub>
12.	5.594	0.62	3-Tridecene	C <sub>13</sub> H <sub>26</sub>
13.	6.269	9.12	1-Tetradecene	C <sub>14</sub> H <sub>28</sub>
14.	6.460	0.86	3-Tetradecene	C <sub>14</sub> H <sub>28</sub>
15.	6.782	0.59	Nonyl-cyclopentane	C <sub>14</sub> H <sub>28</sub>
16.	7.213	7.14	Pentadecane	C <sub>15</sub> H <sub>32</sub>
17.	7.305	0.63	5-Octadecene	C <sub>18</sub> H <sub>36</sub>
18.	7.370	0.82	1-methyl-4-(1-methylethenyl)-cyclohexane	C <sub>10</sub> H <sub>18</sub>
19.	7.639	2.25	Heptylcyclohexane	C <sub>13</sub> H <sub>26</sub>
20.	7.845	3.45	7-hexadecene	C <sub>16</sub> H <sub>32</sub>
21.	7.953	1.57	Hexadecane	C <sub>16</sub> H <sub>34</sub>
22.	8.079	0.81	Cyclohexadecane	C <sub>16</sub> H <sub>32</sub>
23.	8.614	4.64	8-Heptadecene	C <sub>17</sub> H <sub>34</sub>
24.	8.720	2.70	Heptadecane	C <sub>17</sub> H <sub>36</sub>
25.	8.843	1.60	3-Heptadecene	C <sub>17</sub> H <sub>34</sub>
26.	9.318	1.08	9-Octadecene	C <sub>18</sub> H <sub>36</sub>
27.	9.393	1.51	3-Octadecene	C <sub>18</sub> H <sub>36</sub>
28.	9.566	0.57	1-Octadecene	C <sub>18</sub> H <sub>36</sub>
29.	10.161	4.04	2-Heptadecanone	C <sub>17</sub> H <sub>34</sub> O
30.	10.700	11.31	Palmitic Acid	C <sub>16</sub> H <sub>32</sub> O <sub>2</sub>
31.	11.332	2.85	1-Heneicosene	C <sub>21</sub> H <sub>42</sub>
32.	11.751	3.36	Oleic acid	C <sub>18</sub> H <sub>34</sub> O <sub>2</sub>
33.	11.845	1.27	Petroselinic acid	C <sub>18</sub> H <sub>34</sub> O <sub>2</sub>
34.	11.926	2.64	1-Docosene	C <sub>22</sub> H <sub>44</sub>
35.	12.495	2.44	1-Nonadecene	C <sub>19</sub> H <sub>38</sub>
36.	13.050	2.19	Cyclotetracosane	C <sub>24</sub> H <sub>48</sub>
37.	13.134	0.55	Tetracosane	C <sub>24</sub> H <sub>50</sub>
38.	13.590	1.49	12-Pentacosene	C <sub>25</sub> H <sub>50</sub>
39.	14.112	1.12	1-Hexacosene	C <sub>26</sub> H <sub>52</sub>
40.	14.617	1.15	10-Nonadecanone	C <sub>19</sub> H <sub>38</sub> O
41.	18.880	0.69	16-Hentriacontanone	C <sub>31</sub> H <sub>62</sub> O

In the palm oil pyrolysis experiment using zeolite H-X catalyst, 41 compositions were identified with the help of the MS NIST17.LIB library system which were classified into three categories of more general chemical compounds, namely hydrocarbons, acids, and ketones as shown in Table 2. chemistry was useful for further simplifying the data and comparing the chemical components in the produced liquid fuels with the commonly used liquid fuels. Figure 7 shows a diagram of the relative compounds



of the liquid fuel which explains that the main component of the liquid fuel was hydrocarbon at 77% which is the main component of the ideal fuel.



**Figure 7.** Relative compounds of palm oil pyrolysis liquid using zeolite H-X.

#### 4. Conclusion

Zeolite-X was synthesized with sodium silicate which was aged for 24 hours to produce zeolite with the best conditions as evidenced by the XRD diffractogram pattern results that was similar with IZA standard. Zeolite-X was carried out by an ion-exchange process to produce zeolite H-X. The ion exchange process on the synthesized zeolite did not change the resulting XRD diffractogram pattern but did change the zeolite surface morphology observed from the SEM analysis results. Zeolite H-X was applied in pyrolysis experiments to produce liquid fuel. Liquid fuel has a component of 77% hydrocarbons, 16% acids, and 7% ketones.

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