



Pyrolysis of Crude Palm Oil Using Modified Lampung Natural Zeolite as Catalyst and White Sand as Heat Exchanger

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Abstract. This work was carried out to study pyrolysis of crude palm oil (CPO) using modified Lampung natural zeolite (LNZ) as catalyst and white sand as heat exchanger. Modification was done to reduce the Si/Al ratio of LNZ from 6.4 to 3.0 by addition of food grade aluminium foil. The modified zeolite was then subjected to calcination temperature of 700 °C for six hours and then characterized using XRD and SEM and finally used as a catalyst in pyrolysis experiments. The results of XRD characterization revealed that the LNZ belongs to clinoptilolite type and modification led to formation of sodalite and faujasite structures. The change in the structure was also indicated by the results of SEM characterization, supporting the results of XRD technique. The liquid products obtained from pyrolysis experiments (bio crude oil, BCO) indicate that modification of the LNZ resulted in increased formation of hydrocarbon from 34.13% with the use of original LNZ to 76.09% with the use MLNZ plus heat exchanger, suggesting that combination of composition modification and the use of heat exchanger significantly enhanced the formation of hydrocarbon.

Keywords: Lampung Natural Zeolite, Catalyst, Pyrolysis, Bio Crude Oil, Crude Palm Oil.

1 Introduction

Continuous use of fossil fuels is a main concern since it has accelerated depletion of reserves of fossil energy sources which leads to a global energy crisis. Furthermore, combustion of fossil fuels releases greenhouse gases, CO₂ in particular, to the atmosphere and causes global climate change. To alleviate these two problems, associate with fossil fuels, development of carbon-neutral and renewable energy sources as alternatives to fossil fuels is crucially needed. In this regard, biomass derived energy sources (biofuels) are potential and futuristic solutions to ensure energy sustainability. In addition to availability and renewability of biomass around the globe, the potential of

biofuels is also supported by continuous development of biomass to energy conversion platforms, enabling the production of different forms of biofuel.

One method of converting biomass into energy that attracts massive interest worldwide is pyrolysis. This thermochemical method is a part of biomass to energy conversion platforms, in which the raw material is subjected to heating in closed reactor in the absence of oxygen, leading to cracking of large biomass molecules into smaller products, in the form of gas, liquid, and solid residue. Liquid, commonly known as bio crude oil (BCO), is of particular importance from renewable energy perspective, since one type of components composing the BCO is hydrocarbon which is considered as an ideal substitute for hydrocarbon fuels derived from fossil source (petroleum). Compared to the other existing methods, pyrolysis offers several advantages which make this method attractive. This method can be applied to treat all types of biomass, so it is not constrained by the availability of raw materials, and therefore offers the opportunities to apply it throughout the world. In addition, the process is significantly fast and can be run in a simple reactor. Moreover, this technique requires no additional reaction agent, except catalyst.

With the support of a number of favorable characteristics, the potential of pyrolysis has been intensively explored as reflected by its application for production of BCO from wide range of raw materials, including agricultural residues such as cassava solid waste [1] and sugarcane bagasse [2], vegetable oils such as palm oil [3], [4], rubber seed oil [5], [6], castor oil [7], and sludge palm oil [8], water hyacinth [9], water hyacinth and palm oil [10], mixed solid cassava residue and palm oil [11], grass [12], and forestry waste [13].

In principle, biomass pyrolysis can be carried out without or with the use of catalyst. However, in practice, catalytic pyrolysis is the most commonly applied since catalyst functions not only to lower the pyrolysis temperature, but also determine the chemical composition of the BCO produced. The most widely used catalysts are zeolites since they have been reported to possess ability for deoxygenation of oxygen-containing products formed during the pyrolysis process [14]-[16], leading to increased quantity of hydrocarbons in the BCO [17], [18].

In this work, modified Lampung natural zeolite (MLNZ) was used as catalyst for pyrolysis of crude palm oil. Prior to modification, the LNZ investigated was analyzed using XRF method to obtain chemical composition of the sample and it was found that the sample has the Si/Al ratio of 6.4. Modification of the natural zeolite was carried out by changing the Si/Al ratio of the sample from 6.4 to 3.0 by addition of food grade aluminium foil. The MLNZ sample was subjected to calcination process at 700 °C for 6 hours, then characterized using XRD, SEM, and PSA, and finally tested as catalyst. Besides the catalyst, another factor that plays an important role in the biomass pyrolysis is distribution of thermal energy (heat) in the raw material during the process [19], [20]. Considering the important role of heat distribution, in this study, the effect of white sand as heat exchanger was also studied.

2 Materials and Equipments

Lampung natural zeolite (LNZ) investigated in this work was obtained from local supplier (CV. Minatama Utama). This zeolite was found to have the Si/Al ratio of 6.4 according to XRF analysis using XRF instrument (PANalytical Epsilon 3). Other materials used were crude palm oil obtained from local palm oil company (PT. Bhumi Segara), reagent grade NaOH (Merck), food grade aluminium foil (FGAF) from local food store, and crystalline white sands from local supplier (NAJ Fish Jogja). Laboratory instruments used for characterization include XRD instrument (PANalytical Xpert 3 Powder), SEM instrument (ZEISS EVO MA 10), PSA instrument (HORIBA SZ-100 for Windows, Z Type Ver2.50) and GCMS instrument (QP2010S SHIMADZU).

3 Methods

3.1 Preparation of Modified Lampung Natural Zeolite (MLNZ)

As previously stated, the LNZ used in this investigation has the Si/Al ratio of 6.4 and then modified to change the Si/Al ratio to 3.0. For this purpose, a typical sample was prepared by dissolving 4,68 grams FGAF in 250 mL NaOH 1 M, and then 50 g of LNZ was added into the solution. The mixture was aged for 24 hours in autoclave at room temperature and finally subjected to crystallization at temperature 100 °C for 72 hours. The sample was washed, filtered and dried for 8 hours at 100 °C then calcined for 8 hours at 700 °C. The sample was grounded into powder and sieved with 300 mesh sieve. The LNZ sample was also calcined under the same conditions as those applied to the MLNZ.

3.2 Characterization of LNZ and MLNZ

To study the effect of modification, both the LNZ and MLNZ samples were characterized using XRD, SEM, and PSA. Subsequently, the samples were then used as catalyst in pyrolysis experiments.

3.3 Pyrolysis Experiments

The pyrolysis experiment was conducted in a laboratory-scale pyrolysis reactor. Four experiments were conducted, using 100 mL oil in each experiment. In the first experiment, the oil was pyrolyzed without using catalyst and heat exchanger. In the second experiment, the oil was mixed with 5 grams of heat exchanger, in the third experiment the oil was mixed with 5 grams of activated MLNZ, and in the last experiment the oil was mixed with 5 grams heat exchanger and 5 grams MLNZ. The pyrolysis process was allowed to proceed for 90 minutes. The liquid product obtained was then transferred into a separating funnel and allowed to separate between the aqueous phase and the organic phase. This organic phase is known as bio crude oil (BCO).

3.4 Analysis of BCO Using GC-MS Technique

To identify the chemical constituents of the BCO produced and their relative percentages, the sample was analyzed using GC-MS. Identification of the components was carried with the aid of MS Library software NIST62.LIB and WILEY229.LIB programs.

4 Results and Discussion Methods

4.1 XRD Characterization

To compare the structure of OLNZ and MLNZ, the samples were characterized using XRD, and the diffractograms of the samples are presented in Fig 1.

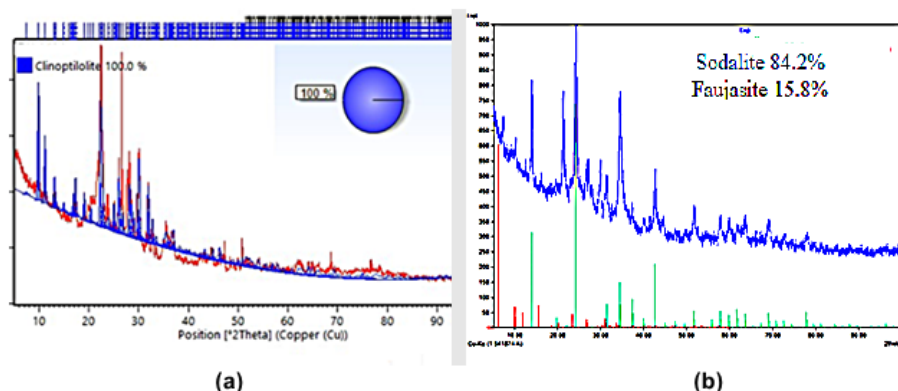


Fig. 1. XRD diffractograms of the samples investigated: (a) LNZ sample and (b) MLNZ sample.

The diffractograms in Fig 1 indicate the existence of amorphous and crystalline phase in both samples. To identify the type of crystalline phase composing the samples, the diffractograms were analyzed using software (Match Program), revealing that the LNZ sample belongs to clinoptilolite group, while in the MLNZ sample, two crystalline phases were identified, i.e. sodalite with relative percentage of 84.2% and faujasite with relative percentage of 15.8%. This change in the crystalline phase of the samples studied demonstrated that the crystalline phase in the LNZ (clinoptilolite) was transformed into sodalite and faujasite structures as a result of Si/Al reduction. This finding also implies that with the method applied in this study it is possible to transform natural zeolite into synthetic zeolite.

4.2 SEM Characterization

The samples were also characterized using SEM, and the micrographs obtained are shown in Fig 2.

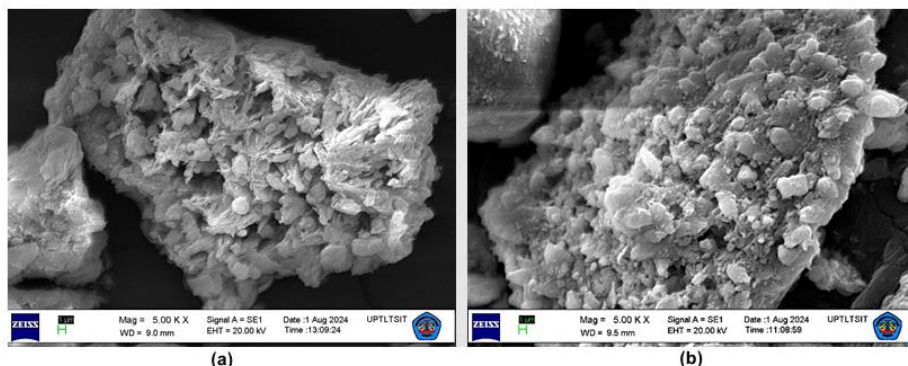


Fig. 2. SEM micrographs of the samples investigated: (a) LNZ sample and (b) MLNZ sample.

As displayed by the micrographs, the surface morphology of the samples is marked by the existence of amorphous and crystalline phase, which is in agreement with the information produced by the XRD characterization. In addition, the surface morphology of the MLNZ sample is characterized by the existence of crystalline particles with irregular shapes and of various sizes. Despite the existence of crystalline particles observed, it should be acknowledged the particles having the shapes of faujasite and sodalite particles were not evidently observed in the micro-graph, most likely due to amorphous particles which cover the crystalline particles on the surface of the sample.

4.3 GC-MS Analysis of the BCO Samples

To identify the chemical components composing the BCO samples obtained from the pyrolysis experiments were characterized using GC-MS method. The first experiment carried out was pyrolysis of the CPO alone (without catalyst and heat ex-changer). The GC chromatogram of the sample is presented Fig 3.

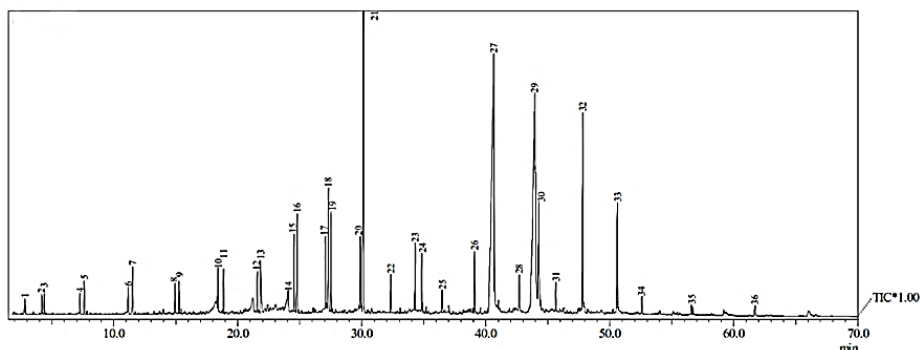


Fig. 3. GC-chromatogram of the BCO sample produced from pyrolysis of the CPO alone.

According to the chromatogram in Fig 3, the sample is a mixture of various compounds with different intensities (peak areas) which also means different relative percentages of the components. With the aid of MS Library software, a total of 36 compounds in the sample were identified. The list of the compounds together with other relevant information, including relative percentage (%R) and similarity index (SI) are shown in Table 1.

Table 1. List of chemical components of the BCO obtained from pyrolysis of CPO.

Peak No.	Formula	Compound	SI	%R	Category
1	C ₆ H ₁₂	1-Pentene	85	0.28	Hydrocarbon
2	C ₇ H ₁₄	1-Heptene	97	0.38	Hydrocarbon
3	C ₇ H ₁₆	Heptane	97	0.38	Hydrocarbon
4	C ₈ H ₁₆	1-Oktene	98	0.45	Hydrocarbon
5	C ₈ H ₁₈	Octane	97	0.74	Hydrocarbon
6	C ₉ H ₁₈	1-Nonene	96	0.57	Hydrocarbon
7	C ₁₀ H ₂₀	1-Decene	97	1.64	Hydrocarbon
8	C ₁₀ H ₂₂	Decane	98	0.65	Hydrocarbon
9	C ₁₁ H ₂₂	1-Undecene	96	0.70	Hydrocarbon
10	C ₁₃ H ₂₈	Tridecane	97	0.81	Hydrocarbon
11	C ₉ H ₁₈ O ₂	Nonanoic Acid	97	1.00	Carboxylic Acid
12	C ₁₄ H ₂₈	1-Tetradecene	95	0.99	Hydrocarbon
13	C ₁₀ H ₂₀ O ₂	Decanoic Acid	98	1.13	Carboxylic Acid
14	C ₁₁ H ₂₂ O ₂	Undecanoic Acid	89	1.18	Carboxylic Acid
15	C ₁₅ H ₂₈	1-Pentadecene	84	2.35	Hydrocarbon
16	C ₁₅ H ₃₂	Pentadecane	96	2.10	Hydrocarbon
17	C ₁₆ H ₃₂	3-Hexadecene	97	2.49	Hydrocarbon
18	C ₁₆ H ₃₄	Hexadecane	97	2.82	Hydrocarbon
19	C ₁₇ H ₃₄	8-Heptadecene	96	2.16	Hydrocarbon
20	C ₁₈ H ₃₆	9-Octadecene	96	2.03	Hydrocarbon
21	C ₁₈ H ₃₆	3-Octadecene	96	7.82	Hydrocarbon
22	C ₁₉ H ₃₈	9-Octadecen-1-ol	91	0.85	Hydrocarbon

23	C ₁₉ H ₃₈	9-Nonadecene	96	2.00	Hydrocarbon
24	C ₁₉ H ₄₀	Nonadecane	91	1.39	Hydrocarbon
25	C ₂₀ H ₃₈	5-Ikosenyne	92	0.53	Hydrocarbon
26	C ₁₉ H ₃₈ O	2-Nonadecanoic	96	1.35	Ketone
27	C ₁₆ H ₃₂ O ₂	1-Hexadecanoic Acid	94	27.95	Carboxylic Acid
28	C ₂₀ H ₄₂	Eicosane	96	1.14	Hydrocarbon
29	C ₁₈ H ₃₂ O ₂	Linoleic Acid	94	18.98	Carboxylic Acid
30	C ₁₈ H ₃₄ O ₂	9-Octadecanoic Acid	93	4.19	Carboxylic Acid
		9,12-Octadecanoic			
31	C ₁₈ H ₃₄ O ₂	Acid	83	0.70	Carboxylic Acid
32	C ₁₉ H ₃₆ O ₃	Octadecanoic Acid	83	5.38	Carboxylic Acid
33	C ₂₃ H ₄₆	9-Trikosene	96	2.95	Alcohol
34	C ₁₉ H ₃₈ O	10-Nonadecanone	91	0.46	Ketone
35	C ₂₀ H ₄₂ O	1-Eicosanol	94	0.21	Alcohol
36	C ₂₃ H ₄₆ O	12-Tricosanone	83	0.39	Ketone

As shown in Table 1, each of the compounds identified was allocated in a more general organic category. This categorization of the components was carried out to simplify the composition of the sample, by summation the relatives of the components in each category. By using this method, it was found that the sample is composed of 4 classes of organic compounds, with hydrocarbon contributes 34.13%, acid 60.51%, ketone 2.20%, and alcohol 3.16%. This composition feature also indicates that the sample is dominated by oxygenated compounds (65.87%), with acid as a dominant component. As a consequence, this particular BCO is not suitable as practical fuel.

The BCO samples obtained from the other three experiments were analyzed in a similar way, and the GC-chromatograms of the samples are compiled in Fig 4.

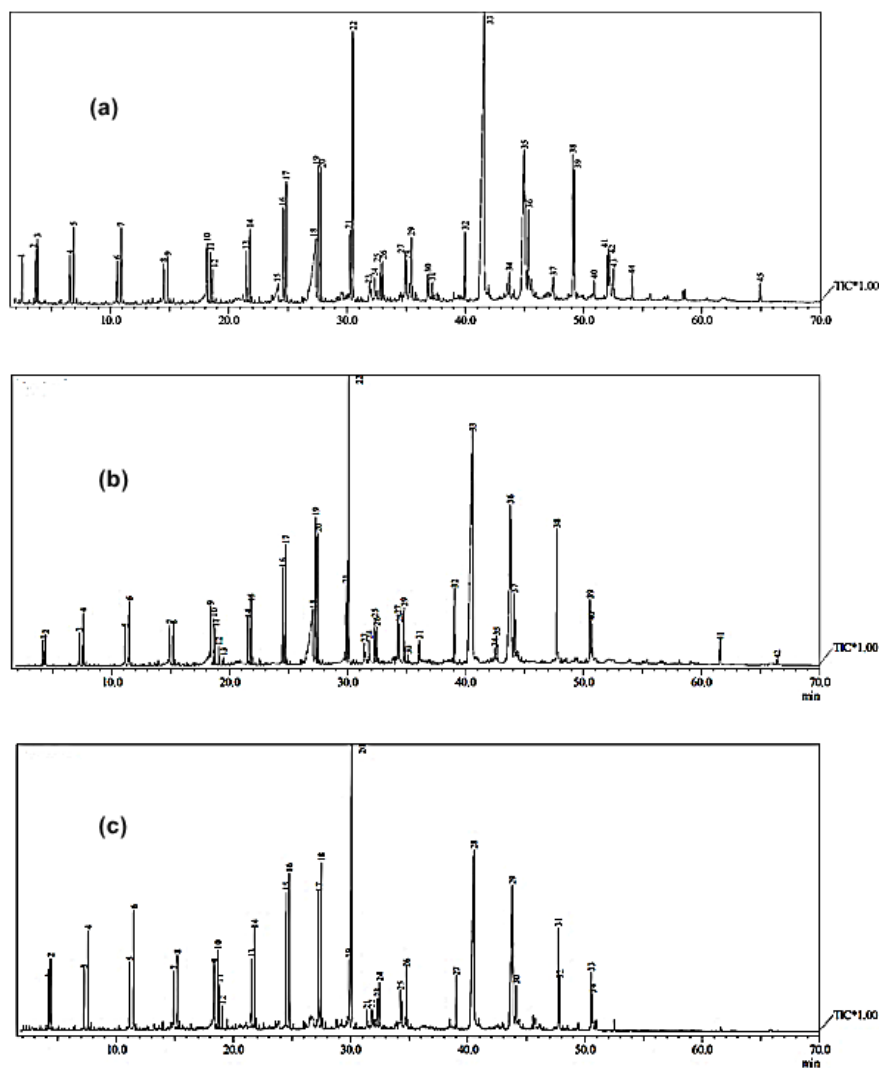


Fig. 4. GC chromatograms of the BCO samples produced from pyrolysis experiment with the use of heat exchanger (a), with the use of MLNZ (b), and with the use of MLNZ + heat exchanger (c).

As can be observed, the general patterns of the chromatograms are practically similar, implying that the samples are composed of mostly the same compounds. Despite this similarity in the patterns, it can be noted that the difference in the intensities of the peaks are quite obvious, suggesting the difference in the relative compositions of the

samples. For more detail comparison, the GC-MS data of the samples were treated in similar way applied for the first sample, and the results are compiled in Table 2.

Table 2. List of chemical components of the BCO samples obtained from different pyrolysis experiments and their relative percentage (%R).

Chemical Component		%R of Components with Reaction Mixture		
Compound	Formula	Oil + HE	Oil + MLNZ	Oil + HE + MLNZ
1-Hexene	C ₆ H ₁₂	0.49	nd	1.28
1-Heptene	C ₇ H ₁₄	0.80	0.52	1.18
Heptane	C ₇ H ₁₆	0.91	0.65	nd
3-propyl-1,4-Pentadiene	C ₈ H ₁₄	nd	0.22	nd
1-Octene	C ₈ H ₁₆	0.85	0.81	1.43
Octane	C ₈ H ₁₈	1.31	1.16	1.55
1-Nonene	C ₉ H ₁₈	0.81	0.96	2.32
Nonane	C ₉ H ₂₀	1.42	1.54	nd
1-Decene	C ₁₀ H ₂₀	0.80	1.11	1.63
Decane	C ₁₀ H ₂₂	0.81	0.87	3.10
1-Undecene	C ₁₁ H ₂₂	1.17	1.80	
4-Undecene	C ₁₁ H ₂₂	nd	0.77	1.76
1-pentyl-2-propyl-cyclopropane	C ₁₁ H ₂₂	nd	0.42	1.86
1-heptyl-2-methyl-cyclopropane	C ₁₁ H ₂₂	nd	nd	2.27
Undecane	C ₁₁ H ₂₄	0.91	0.99	nd
2-Undecene	C ₁₁ H ₂₂	0.63	nd	nd
1-Dodecene	C ₁₂ H ₂₄	0.99	1.39	1.75
Dodecane	C ₁₂ H ₂₆	1.31	0.18	1.14
Nonanoic acid	C ₉ H ₁₈ O ₂	0.47	nd	1.80
1-Tridecene	C ₁₃ H ₂₆	1.99	2.37	nd
Tridecane	C ₁₃ H ₂₈	2.31	3.35	0.55
Decanoic Acid	C ₁₀ H ₂₀ O ₂	nd	7.53	nd
Undecanoic acid	C ₁₁ H ₂₂ O ₂	7.20	nd	nd
1-Tetradecene	C ₁₄ H ₂₈	2.89	2.57	2.33
Tetradecane	C ₁₄ H ₃₀	2.63	2.93	4.09
1-Pentadecene	C ₁₅ H ₂₈	1.79	0.78	nd
Pentadecane	C ₁₅ H ₃₂	6.34	nd	3.65
Tridecanoic acid	C ₁₃ H ₂₆ O ₂	0.47	nd	nd
1-Pentadecyne	C ₁₅ H ₂₈	0.62	nd	4.44
1-Pentadecene	C ₁₅ H ₂₈	0.84	nd	nd
Tetradecane	C ₁₄ H ₃₀	0.82	nd	nd
Pentadecane	C ₁₅ H ₃₂	1.37	nd	nd
9-Nonadecene	C ₁₉ H ₃₈	1.40	nd	nd
1-Hexadecene	C ₁₆ H ₃₂	1.08	nd	nd
3-Hexadecene	C ₁₆ H ₃₂	nd	1.85	2.08
Dexylcycloheane	C ₁₆ H ₃₂	nd	1.16	nd
Octyl cyclohexane	C ₁₄ H ₂₈	nd	nd	3.46

Tetradecanoic acid	C ₁₄ H ₂₈ O ₂	0.90	0.21	nd
8-Heptadecene	C ₁₇ H ₃₄	nd	1.42	8.89
1-Heptadecene	C ₁₇ H ₃₄	nd	0.39	nd
Heptadecane	C ₁₇ H ₃₆	nd	7.40	0,90
3-Octadecene	C ₁₈ H ₃₆	nd	1.04	0,90
Octadecane	C ₁₈ H ₃₈	nd	1.06	1.18
9-Octadecene	C ₁₈ H ₃₆	nd	1.15	nd
9-Nonadecene	C ₁₉ H ₃₈	0.35	nd	0.89
2-Nonadecanone	C ₁₉ H ₃₈ O	1.66	1.94	nd
Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	25.86	25.19	1.32
2-propenil nonanoic	C ₁₂ H ₂₂ O ₂	nd	0.60	nd
4-Tetradecanol	C ₁₄ H ₃₀ O	0.94	nd	nd
9-Hexadecenoic acid	C ₁₆ H ₃₀ O ₂	9.43	14.30	18.34
Octadecanoic acid	C ₁₉ H ₃₆ O ₃	2.93	2.27	nd
Oleyl Alcohol	C ₁₈ H ₃₆ O	nd	1.76	1.69
3-Octadecanone	C ₁₈ H ₃₆ O	nd	0.98	nd
12-Tricosanone	C ₂₃ H ₄₆ O	nd	0.57	nd
1-Hexacosene	C ₂₆ H ₅₂	0.73	nd	nd
9-Tricosene	C ₂₃ H ₄₆	3.42	0.39	2.70
1-Docosene	C ₂₂ H ₄₄	3.33	nd	nd
1-Heptacosanol	C ₂₇ H ₅₆ O	0.60	nd	nd
9-Eicosyne	C ₂₃ H ₄₆ O	1.28	nd	nd
Loxanol	C ₂₀ H ₃₈ O ₂	1.23	nd	nd
9-Octadecen-1-ol	C ₁₈ H ₃₆ O	nd	nd	1.47
Nonadecane	C ₁₉ H ₄₀	nd	0.98	1.20
1-Nonadecene	C ₁₉ H ₃₈	0.44	1.51	13.64
10-Nonadecanone	C ₁₉ H ₃₈ O	0.63	nd	1.65
Eicosane	C ₂₀ H ₄₂	nd	nd	1.56
Vinyl stearate	C ₂₀ H ₃₈ O ₂	0.84	0.91	nd

Based on the GC-MS data obtained from different experiments, the relative compositions of the samples are compiled in Table 3.

Table 3. Relative compositions of BCO samples produced from different experiments investigated in this work.

Sample	Component (%)				
	Hydrocarbon	Carboxylic Acid	Ketone	Alcohol	Ester
Palm Oil	34.13	60.51	2.20	3.16	0
Palm Oil + HE	46.84	47.26	2.29	2.77	0.84
Palm Oil + MLNZ	43.74	50.41	3.49	1.76	0.60
Palm Oil + MLNZ + HE	76.09	22.03	0	1.88	0

As can be seen in the data presented in Table 3, different reaction mixtures (samples) led to significant differences in the chemical compositions of the samples, most evidently in terms of two predominant categories, i.e. hydrocarbon, carboxylic acid, and ketone. As can be seen, very significant increase in hydrocarbon content, from only 34.13% in the sample produced from CPO alone to 76.09% with the use of MLNZ as

catalyst plus HE. This increased hydrocarbon content was accompanied by decrease carboxylic acid from 60.51% to 22.03%. These composition profiles suggest that the use of MLNZ in combination with HE promoted deoxygenation reactions during the pyrolysis, leading to increased hydrocarbon content, suggesting that the method developed in this study has the potential for production of biohydro-carbons as a promising renewable energy.

5 Conclusions

The experimental data obtained in this work suggest the opportunity to transform natural zeolites into synthetic zeolites by adjusting the Si/Al ratio to satisfy the Si/Al ratio range of synthetic zeolites (1 to 5). The pyrolysis experiments conducted revealed that the best BCO, in term of hydrocarbon content, was produced with the use of MLNZ as catalyst in combination with white sand as heat exchanger. In over-all, the method developed in this study has the potential for production of biohydrocarbons as a promising renewable energy source derived from biomass by pyrolysis technique.

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