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CHEMICAL COMPOSITION OF BIO CRUDE OIL PRODUCED BY PYROLYSIS OF CRUDE PALM OIL USING THERMALLY ACTIVATED BENTONITE AS CATALYST

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ABSTRAK

Pada penelitian ini, metode kromatografi gas-spektrometri massa (GC-MS) diterapkan untuk menentukan komposisi kimia *bio crude oil* (BCO) yang dihasilkan melalui pirolisis minyak sawit mentah (CPO) menggunakan bentonit yang diaktifkan secara termal sebagai katalis. Untuk aktivasi, bentonit diberi perlakuan kalsinasi selama 8 jam pada suhu berbeda yaitu 600, 700, dan 800 °C. Sampel bentonit asli dan yang telah diaktifkan kemudian dikarakterisasi menggunakan XRD dan SEM untuk mengevaluasi pengaruh suhu kalsinasi terhadap struktur dan mikrostruktur sampel. Hasil yang diperoleh dari analisis GC-MS menunjukkan bahwa hidrokarbon merupakan komponen dominan dari sampel BCO, mengindikasikan bahwa bentonit memiliki potensi yang menjanjikan sebagai katalis untuk produksi biohidrokarbon dari biomassa melalui pirolisis. Hasil penelitian juga menunjukkan peningkatan signifikan kandungan hidrokarbon dalam BCO dari 58,98% dengan penggunaan bentonit yang tidak dikalsinasi menjadi 76,45% dengan penggunaan bentonit yang dikalsinasi pada suhu 800 °C sebagai katalis, yang juga berarti pengurangan oksigenat dari 41% menjadi 23,5%.

Kata kunci: GC-MS, *bio crude oil*, minyak sawit mentah, pirolisis, bentonit.

ABSTRACT

In this study, gas chromatography-mass spectrometry (GC-MS) method was applied to determine the chemical composition of bio crude oil (BCO) produced by pyrolysis of crude palm oil (CPO) using thermally activated bentonite as a catalyst. For activation, the bentonite was subjected to calcination treatment for 8 hours at different temperatures of 600, 700, and 800 ˚C. The original and activated bentonite samples were then characterized using XRD and SEM in order to evaluate the effect of calcination temperatures on structure and microstructure of the samples. The results obtained from GC-MS analysis indicate that hydrocarbons are the dominant components of the BCO samples, suggesting that bentonite has promising potential as a catalyst for production of biohydrocarbons from biomass by pyrolysis. The results also show a significant increase of hydrocarbon content of the BCO from 58.98% with the use of uncalcined bentonite to 76.45% with the use of bentonite calcined at 800 °C as catalyst, which also means a reduction of oxygenates from 41.0% to 23.5%.

Keywords: GC-MS, bio crude oil, crude palm oil, pyrolysis, bentonite.

INTRODUCTION

The ever-increasing need for energy sources, especially liquid fuels, is a global challenge facing the world today, as petroleum reserves which have traditionally been the mainstay, continue to deplete. This situation is a strong alarm that the development of renewable energy has become an unavoidable necessity. To address the challenges associated with fossil fuels, many countries, including Indonesia, consider biomass-based energy sources (bioenergies) as the most reliable renewable fuels and have therefore been extensively explored. The availability and renewability of different biomass around the globe make bioenergy a promising future energy source. In addition, different technology platforms for production of liquid bioenergies now exist, such as transesterification of vegetable oils or animal fats for production of biodiesel (Pandiangan *et al*., 2025; Vasić *et al*., 2020; Pandiangan *et al*., 2020) and pyrolysis for production of bio crude oil (BCO) from biomass feedstocks (Simanjuntak *et al*., 2021; Simanjuntak *et al*., 2024; Pandiangan *et al*., 2022; Morais *et al*., 2017; Pandiangan *et al*., 2024; Aulia *et al*., 2024).

BCO, which is a liquid product derived from biomass pyrolysis, is a complex mixture of various organic compounds, including hydrocarbons (biohydrocarbons) and other types of organic compounds, primarily oxygenates such as organic acids, aldehydes, and ketones (Simanjuntak *et al*., 2021; Jazie *et al*., 2023; Psarras *et al*., 2019; Pratiwi *et al*., 2025). The presence of biohydrocarbons makes BCO the most attractive from renewable energy perspective. Biohydrocarbons consist of various compounds with different carbon chain lengths, making them a promising replacement for hydrocarbon fuels derived from petroleum. With respect to this potential, extensive investigations have been carried out to produce BCO from different biomass feedstocks, such as mixture of palm oil and solid cassava residue (Simanjuntak *et al*., 2021), palm oil waste (Parthasarathy *et al*., 2024), pineapple crown leaves (Barbosa *et al*., 2019), water hyacinth (Pandiangan *et al*., 2022), mixture of sugar cane bagasse and sludge palm oil (Supryanto *et al*., 2018), mixture of cassava tubers and rubber seed oil (Aulia *et al*., 2024), and softwood and non-wood (Praserttaweeporn *et al*., 2022).

In practice, biomass pyrolysis is generally carried out in the presence of a catalyst, because apart from its main function of lowering the temperature, the catalyst also plays a role in determining the chemical composition of BCO. In relation to biohydrocarbons, the ideal catalyst is the one with deoxygenation activity, namely that can release oxygen as simple gaseous compounds. As a result, the optimum formation of biohydrocarbons is achieved.

In the search for an ideal catalyst, zeolites have attracted the most attention, because this group of compounds has been known to possess appreciable deoxygenation activity (Praserttaweeporn *et al*., 2022; Jazie *et al*., 2023; Garba *et al*., 2018). Broadly, zeolites are divided into natural zeolites and synthetic zeolites, both were acknowledged to have catalytic activity. However, the activity of natural zeolites is much less than that of synthetic ones. To improve their performance, the common methods applied are by subjecting the zeolites to activation treatment, using acid (Tišler *et al*., 2019), alkaline (Tišler *et al*., 2019; Monzón *et al*., 2019; Hrachovcová *et al*., 2020), or thermal (Tišler *et al*., 2019; Ibrahim *et al*., 2022).

Bentonite belongs to natural aluminosilicates that can be found in different parts of the world, including Indonesia. Several workers have reported applications of bentonite as catalyst for different purposes, such as production of biodiesel using bentonite clay pillared with Zr and Ti (Agustian *et al*., 2023), production of hydrogen by pyrolysis of corn cob powder using Na-bentonite (Irawan *et al*., 2025), hydrocracking of CPO using bentonite modified by zirconium nitrida and zirconium phosphide (Hasanudin *et al*., 2022). With respect to these previous studies, application of thermally activated bentonite as catalyst for pyrolysis treatment of biomass production of bio crude oil, particularly from crude palm oil, investigated in this work, is a new aspect of bentonite potential as promising catalyst.

In this study bentonite was subjected to thermal activation (calcination) treatment at different temperatures of 600, 700, and 800 $^{\circ}$C, and subsequently used as catalyst for pyrolysis of crude palm oil. The liquid products (BCO) obtained from different experiments were then analyzed using GC-MS method to identify the chemical components of the samples.

METHOD

Tools and Materials

The main equipments used in this research are laboratory scale pyrolysis reactor, furnace (Nabertherm, Germany), porcelain crucible, mortar and pestle, 300 mesh sieve, and supporting laboratory glassware. The instruments used in this study are X-ray fluorescence (XRF) Pan-Analytical Epsilon (Netherlands: elemental range: typically, Fluorine (F) to Americium (Am), X-ray tube: silver (Ag) anode, 10 W or higher, up to 50 kV, detector type: Silicon Drift Detector (SDD) with high resolution, and sample handling: benchtop, non-destructive), X-Ray Diffractometer (XRD) Pan-Analytical powder X-pert (Netherlands, X-ray source: Cu K radiation is standard (λ = 1.5418 Å), generator power: 3 kW (typically 45 kV and 40 mA), goniometer: vertical θ–θ (theta-theta) geometry with a radius of 240 mm, detector: X'Celerator

1D and angular range (2θ): -40° to +220°), Scanning Electron Microscope (SEM) model ZEISS EVO MA 10 (Country: Germany, imaging modes: high vacuum (HV) and variable pressure (VP), resolution: 3 nm at, magnification: 5x – 100,000x, specimen diameter: max 230 mm, and detectors: secondary electron (SE), variable pressure SE (VPSE), and 5-segment backscattered electron (BSD) detectors), and Gas Chromatography-Mass Spectroscopy (GC-MS) QP2010S SHIMADZU (Japan, high-performance metal quadrupole with pre-rods, mass range: up to m/z 1090, ion source: EI (electron impact ionization), 10 to 200 eV, detector: electron multiplier, vacuum system: turbo molecular pump (65 L/sec ffor He), and GC-MS interaction: direct connection with capillary column up to 350°C). Bentonite was purchased from a local supplier and crude palm oil (CPO) was obtained from a local market.

Procedure

The bentonite was first characterized using XRF to obtain the chemical composition of the sample, particularly the Si and Al contents as the main elements composing the sample. The original and activated bentonite samples were also characterized using XRD and SEM. Thermal activation of bentonite was conducted by subjecting the sample to calcination treatment at different temperatures of 600, 700, and 800 ˚C, for 8 hours, and then ground and sieved using a 300 mesh sieve to obtain a sample with relatively homogeneous particle size.

A laboratory-scale pyrolysis reactor with gas heater and equipped with water cooled was utilized to run pyrolysis experiments. An aliquot of 200 mL CPO was mixed with 5 grams of bentonite as catalyst, and the mixture was transferred into the pyrolysis reactor. The pyrolysis experiment was run for 90 minutes, at temperature around 300 °C and atmospheric pressure. The liquid product was collected and transferred into a separating funnel and left overnight to allow separation of the aqueous phase and organic phase (BCO). To identify chemical components of the BCO and their relative percentages, the sample was analyzed using GC-MS, which provides information regarding the compounds composing the sample, together with the similarity index of each component with standard compounds available in Mass Library Software, and relative percentage of each compound. To compare the composition features of the BCO samples obtained from different experiments, the components of the samples were then allocated into general groups of organic compounds together with relative percentage of each category. This grouping is applied to simplify the presentation of the relative composition of the samples, and to make it easier to compare samples obtained from different experiments.

RESULTS AND DISCUSSION

Chemical composition of bentonite

As previously stated, prior to thermal treatment, the bentonite was analyzed using XRF technique in order to obtain chemical composition of bentonite. This technique provides the composition data in the form of elements and oxides composing the sample. The results obtained are presented in Table 1.

Table 1. Chemical composition of bentonite used in this study

No.	Element	Relative percentage (%)	Oxide	Relative percentage (%)
1	Si	60.717	SiO_2	72.811
2	Al	7.004	Al_2O_3	8.517
3	Ca	10.588	CaO	6.020
4	K	9.827	K_2O	5.173
5	Fe	6.498	Fe_2O_3	3.059
6	P	2.884	P_2O_5	0.359
7	Ti	0.508	TiO_2	0.195
8	Ba	0.466	BaO	0.178

The presence of Si and Al as the main components forming the bentonite is a general characteristic of aluminosilicate minerals, including natural zeolites, and has been reported in various studies (Maj and Matus, 2023). The elements other than Si and Al found in the bentonite studied in this current investigation are also the elements commonly found in bentonite from different sources (Gilg *et al*., 2020).

XRD characterization

To obtain information regarding the structure, the original and activated bentonite samples were characterized using XRD technique, and the produced diffractograms are shown in Figure 1. For simplicity, the bentonite samples subjected to calcination treatment are specified as Bent-600, Bent-700, and Bent-800, respectively, with reference to the calcination temperature used for each of the samples. The XRD diffractograms compiled in Figure 1 clearly show that the samples consist of a mixture of amorphous and crystalline materials. This XRD feature of the samples is in agreement with the features of natural aluminosilicates reported by other workers (Holmboe *et al*., 2012). It is also observed (Figure 1) that no significant difference between the diffractograms of the original sample and those of the samples calcined at different temperatures, which means that structurally the bentonite remains stable within the applied calcination temperature range.

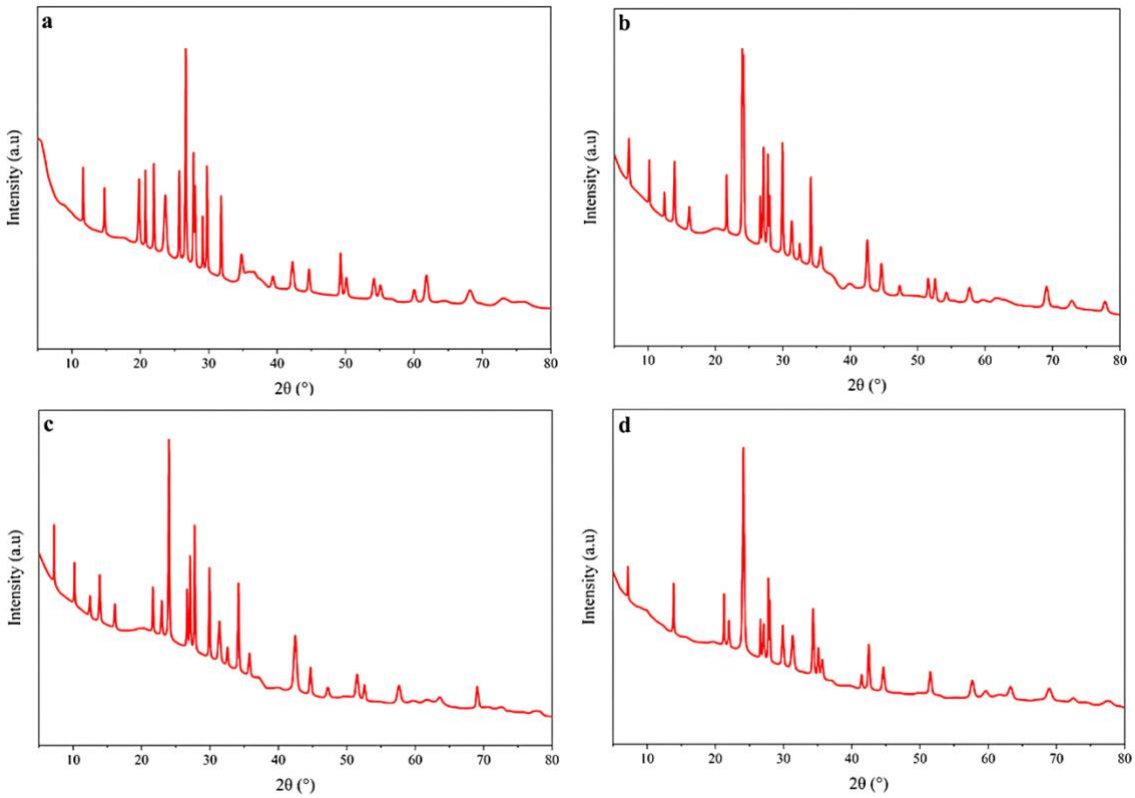

Figure 1. XRD diffractograms of the samples investigated in this study: (a) original bentonite, (b) Bent-600, (c) Bent-700, and (d) Bent-800

To clarify the effect of calcination temperatures on structure of the samples investigated, the crystallinity data of the samples are presented in Table 2.

Table 2. Crystallinity of the original and calcined bentonite investigated in this study.

Sample	Crystallinity (%)
Original bentonite	40.72
Bent-600	40.73
Bent-700	50.33
Bent-800	60.41

As displayed by the data presented in Table 2, gradual increases of the crystallinity following increased calcination temperature was observed. These results are in accordance with the characteristic of solid material, in which thermal treatment resulted in increased crystallinity of the sample. Despite the trend of crystallinity change following calcination temperature displayed by the samples investigated, it should be noted that no extreme change was observed, implying that the thermal stability of the samples was relatively retained.

SEM characterization

The four samples were also characterized using SEM, in order to gain information regarding surface morphology of the samples. Micrographs obtained are shown in Figure 2.

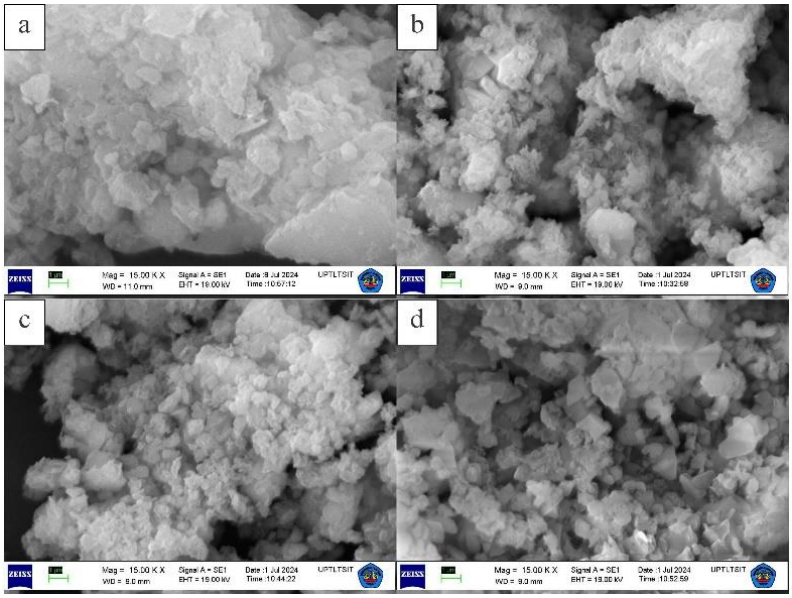

Figure 2. Micrographs of the samples investigated in this study: (a) original bentonite, (b) Bent-600, (c) Bent-700, and (d) Bent-800

As can be observed in Figure 2, the micrographs indicate that the samples are porous materials and characterized by the existence of heterogeneous particles with different shapes and sizes. The presence of crystalline particles is also quite obvious, although the grain boundaries between particles are not clearly observable. Nevertheless, the visual appearance of the micrographs displays an indication of increased crystallinity of the samples following the increase in calcination temperature.

GC-MS analysis

The first pyrolysis experiment was conducted with the use of original bentonite as a catalyst. The GC chromatogram of the BCO sample produced is shown in Figure 3.

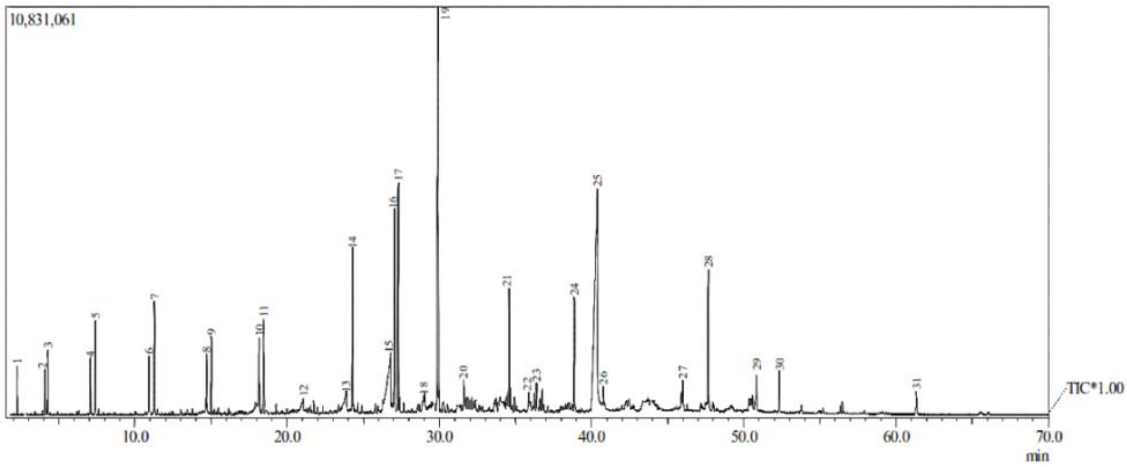

Figure 3. GC chromatogram of BCO obtained from pyrolysis using original bentonite as a catalyst

This chromatogram pattern shows that the BCO sample consists of a mixture of various organic compounds with different molecular weights and in varying relative amounts. The results obtained in this study are in accordance with the results reported by other researchers, who found that BCO produced from biomass pyrolysis, regardless of the type of biomass processed, consists of a mixture of various compounds in different amounts (Simanjuntak *et al*., 2021; Psarras *et al*., 2019; Pratiwi *et al*., 2025). The identified components of the sample are compiled in Table 3, together with some other related information.

Table 3. Chemical composition of BCO produced using the original bentonite as catalyst

Peak No.	Retention time (minutes)	SI	Compound name	Molecular formula	Relative percentage (%)	Category
1	2.26	96	Acetone	C_3H_6O	0.72	Ketone
2	4.09	96	1-Heptene	C_7H_{14}	0.91	Hydrocarbon
3	4.27	96	Heptane	C_7H_{16}	1.34	Hydrocarbon
4	7.07	96	1-Octene	C_8H_{16}	1.41	Hydrocarbon
5	7.39	96	Octane	C_8H_{18}	2.25	Hydrocarbon
6	10.93	96	1-Nonene	C_9H_{18}	1.65	Hydrocarbon
7	11.28	96	Nonane	C_9H_{20}	2.72	Hydrocarbon
8	14.69	96	1-Decene	$C_{10}H_{20}$	1.66	Hydrocarbon
9	15.02	96	Decane	$C_{10}H_{22}$	1.80	Hydrocarbon
10	18.16	96	1-Undecene	$C_{11}H_{22}$	2.37	Hydrocarbon
11	18.45	96	Dodecane	$C_{12}H_{26}$	2.10	Hydrocarbon
12	21.04	94	Octanoic acid	$C_8H_{16}O_2$	0.77	Acid
13	23.89	96	Nonanoic acid	$C_9H_{18}O_2$	1.69	Acid
14	24.31	96	1-Tetradecene	$C_{14}H_{28}$	4.16	Hydrocarbon
15	26.79	97	Decanoic acid	$C_{10}H_{20}O_2$	7.09	Acid
16	27.07	97	3-Hexadecene	$C_{16}H_{32}$	5.28	Hydrocarbon
17	27.29	97	Heptadecane	$C_{17}H_{36}$	6.06	Hydrocarbon
18	28.96	90	Undecanoic acid	$C_{11}H_{22}O_2$	0.85	Acid
19	29.91	96	Octadecane	$C_{18}H_{38}$	15.24	Hydrocarbon
20	31.76	94	9-Nonadecene	$C_{19}H_{38}$	0.74	Hydrocarbon
21	34.58	96	Nonadecane	$C_{19}H_{40}$	3.08	Hydrocarbon
22	35.88	95	Tetradecanoic acid	$C_{14}H_{28}O_2$	0.63	Acid
23	36.37	92	10-Heneicosene	$C_{21}H_{42}$	0.79	Hydrocarbon
24	38.86	94	2-Heptadecanone	$C_{17}H_{34}O$	2.87	Ketone
25	40.36	94	Hexadecanoic acid	$C_{16}H_{32}O_2$	22.69	Acid
26	40.77	94	3-Octadecanone	$C_{18}H_{36}O$	0.91	Ketone
27	45.97	94	9-Tricosene	$C_{23}H_{46}$	1.10	Hydrocarbon
28	47.67	96	9-Hexacosene	$C_{26}H_{52}$	4.30	Hydrocarbon
29	50.83	87	1-Heptacosanol	$C_{27}H_{56}O$	0.96	Alcohol
30	52.33	91	10-Nonadecanone	$C_{19}H_{38}O$	1.02	Ketone
31	61.33	84	Vinyl ester	$C_{20}H_{38}O_2$	0.82	Ester

As shown in Table 3, the components of the sample are dominated by the hydrocarbon group, which contributes 58.98%, suggesting that the bentonite used has not only able to

support cracking process but also exhibits deoxygenation activity, which promotes hydrocarbon formation during pyrolysis process. Apart from the production of hydrocarbon as main components, the presence of oxygen containing compounds (oxygenates) should also be acknowledged. The presence of different chemical groups composing BCO is acknowledged as general characteristic of BCO regardless of the raw materials treated, as has been reported by other workers (Simanjuntak *et al*., 2021; Jazie *et al*., 2023; Pratiwi *et al*., 2025).

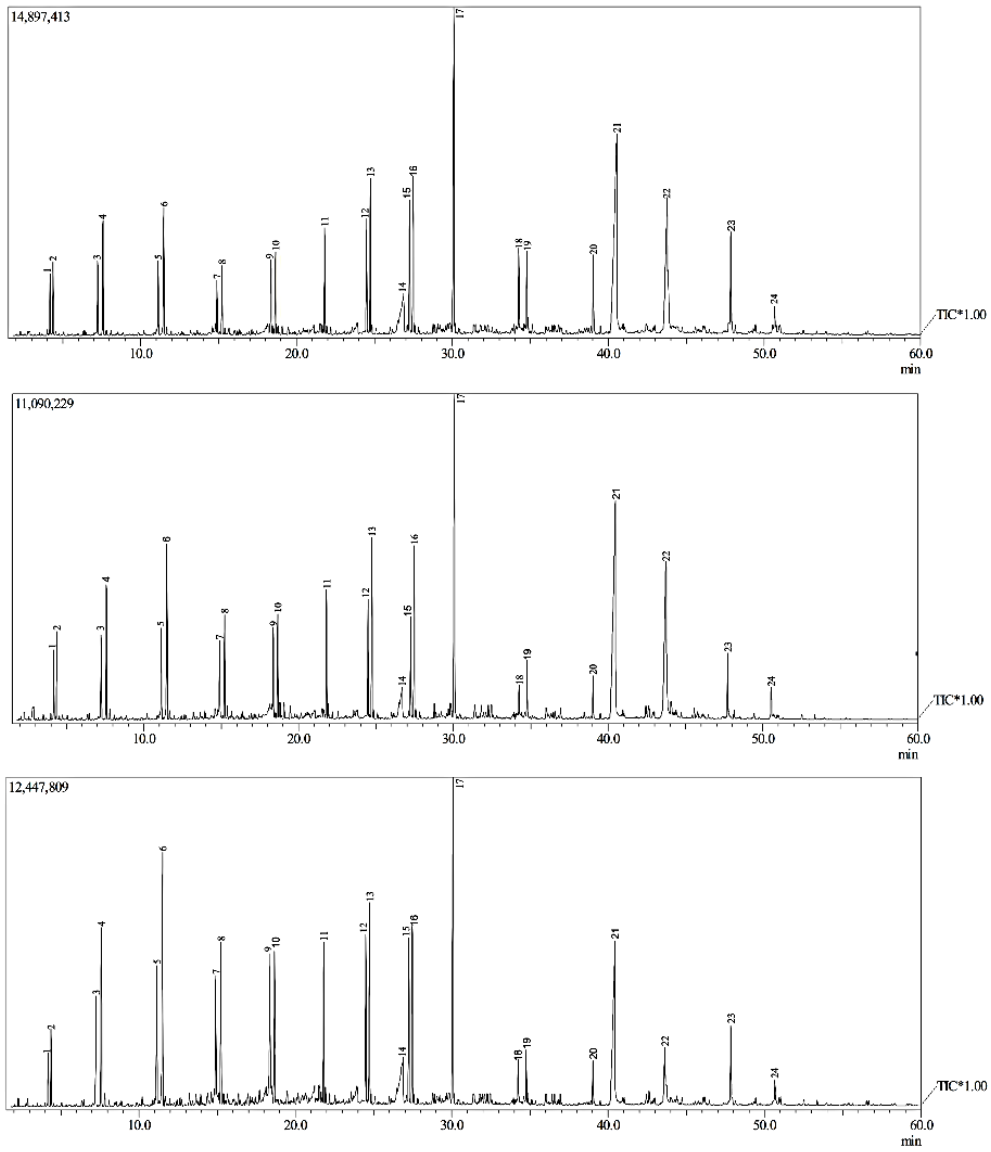

Figure 4. GC chromatograms of BCO samples produced using calcined bentonites catalysts: (a) Bent-600, (b) Bent-700, and (c) Bent-800

To evaluate the effect of calcination temperatures on catalytic performance of bentonite, the BCO samples produced from pyrolysis experiments using bentonite subjected to different calcination temperatures were analyzed using GC-MS. The GC chromatograms of the samples

were compiled in Figure 4, and the identified compounds composing the sample is listed in Table 4.

Table 4. List of compounds composing BCO samples produced using different catalysts

No.	Compound name	Molecular formula	Relative percentage (%) using different catalysts			Category
			Bent-600	Bent-700	Bent-800	
1	1-Heptene	C_7H_{14}	1.30	1.46	1.03	Hydrocarbon
2	Heptane	C_7H_{16}	1.66	1.86	1.52	Hydrocarbon
3	1-Octene	C_8H_{16}	1.94	2.12	2.57	Hydrocarbon
4	Octane	C_8H_{18}	2.99	3.41	4.25	Hydrocarbon
5	1-Nonene	C_9H_{18}	2.00	2.57	3.84	Hydrocarbon
6	Nonane	C_9H_{20}	3.70	5.04	7.31	Hydrocarbon
7	1-Decene	$C_{10}H_{20}$	1.48	2.46	4.60	Hydrocarbon
8	Decane	$C_{10}H_{22}$	1.72	2.67	3.97	Hydrocarbon
9	1-Undecene	$C_{11}H_{22}$	2.15	2.84	5.91	Hydrocarbon
10	Dodecane	$C_{12}H_{26}$	2.04	2.62	3.65	Hydrocarbon
11	Tridecane	$C_{13}H_{28}$	2.64	3.17	3.86	Hydrocarbon
12	1-Tetradecene	$C_{14}H_{28}$	3.13	3.03	4.10	Hydrocarbon
13	Pentadecane	$C_{15}H_{32}$	4.16	4.60	5.36	Hydrocarbon
14	Decanoic acid	$C_{10}H_{20}O_2$	4.47	3.12	5.19	Acid
15	3-Hexadecene	$C_{16}H_{32}$	3.67	2.62	4.23	Hydrocarbon
16	Heptadecane	$C_{17}H_{36}$	4.05	4.71	4.47	Hydrocarbon
17	Octadecane	$C_{18}H_{38}$	12.35	10.33	10.61	Hydrocarbon
18	9-Eicosene	$C_{20}H_{40}$	2.72	0.76	1.21	Hydrocarbon
19	Eicosane	$C_{20}H_{42}$	2.16	1.63	1.22	Hydrocarbon
20	2-Nonadecanone	$C_{19}H_{38}O$	2.09	1.08	1.14	Ketone
21	Hexadecanoic acid	$C_{16}H_{32}O_2$	23.27	20.52	12.46	Acid
22	Oleic acid	$C_{18}H_{34}O_2$	10.33	14.65	3.76	Acid
23	Tricosene	$C_{23}H_{46}$	3.14	1.78	2.74	Hydrocarbon
24	Oleyl alcohol	$C_{18}H_{36}O$	0.84	0.95	1.00	Alcohol

Remarks: Bent-600 refers to bentonite calcined at 600 ˚C, Bent-700 refers to bentonite calcined at 700 ˚C, and Bent-800 refers to bentonite calcined at 800 ˚C.

To clarify the comparison, the relative composition of samples based on the categories of the components is summarized in Table 5. Relative composition of the BCO produced with the use of original bentonite is included in the table, for comparison. As can be seen in Table 5, all samples are composed of compounds in four categories, with varied relative percentages, and the hydrocarbon category emerges as the dominant component. As displayed by the data, the use of Bent-800 as catalyst resulted in a significant increase of hydrocarbon category composing the BCO from 58.98% with the use of the original bentonite to 76.45% with the use of bentonite subjected to calcination at 800 ºC (Bent-800), accompanied by a significant decrease in the oxygenates from 41.0% to 23.5%. These results demonstrate that calcination at 800 ºC led to significant improvement of deoxygenation activity of the bentonite, although it

should be acknowledged that complete deoxygenation has not been achieved, suggesting the need for further investigation.

Table 5. Category composition (%) of BCO produced using different catalysts

Category	Relative composition (%) of BCO produced using different catalysts			
	Original bentonite	**Bent-600**	**Bent-700**	**Bent-800**
Hydrocarbon	58.98	59.00	59.68	76.45
Acid	33.73	38.06	38.29	21.41
Ketone	5.52	2.09	1.08	1.14
Alcohol	0.96	0.84	0.95	1.00
Ester	0.82	-	-	-

There are three deoxygenation mechanisms commonly encountered during the pyrolysis of biomass, namely decarbonylation, decarboxylation, and hydrodeoxygenation. Decarbonylation is a reaction in which organic compounds containing carbonyl groups, such as aldehyde and ketone, decompose into hydrocarbons and carbon monoxide. In decarboxylation reaction, carboxylic acid decomposes into hydrocarbon and carbon dioxide, whereas in hydrodeoxygenation alcohol reacts with hydrogen to produce hydrocarbon and water (Praserttaweeporn *et al*., 2022; Jazie *et al*., 2023).

In addition to chemical composition, the feasibility of a fuel is also determined by several physicochemical properties, such as caloric value, cetane number, and viscosity. However, in this current study, physicochemical properties of the BCO produced were not determined. The main reason is the presence of non-hydrocarbon components (acid, ketone, ester, and alcohol) which are unwanted substances in the light of fuel, therefore determination of physicochemical properties are considered not necessary at this stage.

CONCLUSION

The results obtained in this study demonstrated that GC-MS is a reliable method for analyzing the chemical composition of bio crude oil (BCO) produced by pyrolysis of crude palm oil. The use of this analytical method allows comparison between BCO samples obtained using bentonite samples subjected to calcination at different temperatures as catalyst can be made. With the aid of GC-MS results, it can be concluded that bentonite has promising potential as catalyst for biomass pyrolysis, which is a rapidly developing technology for the production of biomass-based renewable energy. The results also demonstrate that significant improvement of deoxygenation activity of the bentonite was achieved by calcination treatment, with the highest activity exhibited by the bentonite subjected to calcination at 800 ºC. Using

this particular activated bentonite resulted in increased hydrocarbon content from 58.98% (in the BCO sample produced using the original bentonite) to 76.45%, which means a reduction of oxygenates from around 41.0% to around 23.5%. Despite the promising results obtained, it should be acknowledged that complete deoxygenation has not been achieved, suggesting the need for further investigation in order to develop bentonite to become high performance and low-cost catalyst.

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