

Synthesis of Hydroxy-Sodalite from Rice Husk Silica and Food-Grade Aluminum Foil as A Catalyst for Biomass Pyrolysis

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Abstract

In this research, food-grade aluminum foil (FGAF), rice husk silica (RHS), and sodium hydroxide were needed to synthesize hydroxy-sodalite (HS) using hydrothermal method. The raw materials were exposed to crystallization at 100 °C for 72, 96 and 120 h, and followed by calcination for 6 h at 550 °C. The products obtained were identified using X-ray diffraction (XRD) and scanning electron microscope (SEM) technique, and it applied as catalysts for pyrolysis of biomass. The bio-crude oils (BCO) produced were investigated adopting gas chromatography-mass spectrometry (GC-MS). The XRD results reveal that the products obtained with the crystallization time of 72 and 96 h are a mixture of zeolite A and HS and practically pure HS with a crystallization time of 120 h. The shapes of particles as displayed by SEM are in good agreement with the results of others. The main components of the BCO produced are hydrocarbons, with the biogasoline contents in the range of 81.67 - 96.24 % was resulted from the mixture of water hyacinth and palm oil, and 72.92 - 92.58 % from the mixture of cassava solid waste and palm oil. In this respect, it can be concluded HS is a prospective catalyst for the pyrolysis of biomass.

Keywords: Hydroxy sodalite, Hydrothermal, Pyrolysis, Water hyacinth, Cassava solid waste, Rice husk, BCO

Introduction

Hydroxy-sodalite (HS) is a type of synthetic zeolite with structural network characterized by varied ordered channel-like structures and different particle sizes. This unique network arrangement provides hydroxy-sodalite with characteristics suitable for diverse applications such as adsorbent for elimination of heavy metals from water [1-3], ion exchange agent [4], catalyst [5-7], and membrane [8,9]. Several preparation methods have been developed, such as in situ hydrothermal synthesis [2-4], vapor phase transport method [10], secondary growth method [11] or embedding microcrystals of zeolite into a matrix [12]. However, the most commonly used method is hydrothermal treatment and has been applied to a variety of raw materials, such as kaolin [3,10] fly ash [1,2], clay [4], and natural bentonite [14].

Another interesting finding regarding hydroxy-sodalite mentioned in the literatures is the existence of this zeolite as an additional phase in zeolite-A and zeolite-X. For this reason, it has been suggested that zeolite-A and zeolite-X can be transformed into pure hydroxy-sodalite by lengthening crystallization time or enlarge crystallization temperature [7,12]. This study was conducted with the main aim to obtain the required crystallization time for the transformation of zeolite-A into sodalite. For this purpose, food-grade aluminum foil (FGAF), rice husk silica (RHS), and sodium hydroxide (NaOH) were used to prepare reaction mixture following zeolite-A composition. The mixture was then subjected to crystallization treatment for different times of 72, 96 and 120 h at 100 °C.

The products were calcined at 550 °C for 6 h and characterized with X-ray diffraction (XRD) to identify the crystalline phase in the samples, which signifies whether the formation of sodalite was achieved. Characterization using scanning electron microscope (SEM) was also carried out to obtain information regarding the surface morphologies of the samples. The particular information provided by

SEM is the particle shape which is a unique feature of different phases and therefore can be used to support the presence of crystalline phase as seen by XRD.

It is well known that zeolites are widely used for numerous applications; one of them is as a catalyst. One of the catalytic processes that continue to attract interest is the transformation of biomass into renewable liquid fuel (LF) through pyrolysis treatment [13,14]. Various catalysts including metal oxides, inorganic salts, zeolites, mesoporous silica, and carbon-based materials have been widely investigated in biomass pyrolysis treatment [18-20]. However, zeolites are acknowledged to promote deoxygenation reaction, resulted in higher hydrocarbon content of the liquid fuel [21,22]. Acknowledging this particular advantages, the zeolites synthesized in this study were evaluated as a catalyst for pyrolysis of 2 sets of biomass i.e. a mixture of cassava solid waste with palm oil and a mixture of water hyacinth plant with palm oil. Liquid fuels produced were then evaluated using gas chromatography-mass spectrometry (GC-MS), providing the information on the chemical content of the liquids, enabling the comparison of the performance of the zeolites synthesized to be made. The use of cassava solid waste is based on the availability of this agricultural waste in Indonesia, however, the utilization of this feedstock for renewable energy such as bioethanol production is still limited [23-27]. Water hyacinth is an aquatic plant that can be found in various water bodies. From environmental point of view, water hyacinth is considered a very problematic plant [19,20] because of the various problems it causes to the water ecosystem and the use of water for other purposes, such as transportation. From the biomass perspective, on the other hand, this plant is a very potent source for its fast-growing with high mass productivity, making it an interesting raw material for biomass utilization, such as production of renewable energy sources using pyrolysis [21-23]. In this study, a series of trials was operated with the purposes to investigate the effect of crystallization times on the formation of HS, to compare the compositions of the liquid fuels produced using the HS produced with different crystallization times, and to compare the compositions of the liquid fuels obtained from different raw materials pyrolyzed.

Materials and methods

Materials and instruments

Reagent grade NaOH pellet and HNO₃ were purchased from Aldrich. FGAF was bought from a sectional supplier, rice husk from a local farmer-owned rice mill, dry cassava solid waste was kindly provided by a cassava flour company in Bandar Lampung, and then ground into powder. Water hyacinth was taken from the field in the City of Bandar Lampung, washed with running water, chopped to small sizes, and dried under the sun. Dry sample was ground into powder, XRD instrument (PANalytical Empyrean type) and SEM (ZEISS EVO MA 10) were used for product characterization. An electrical furnace (Nabertherm, Lilienthal, Germany) was used for treatment of calcination. The pyrolysis test was executed adopting a laboratory-scale pyrolysis unit, and GC-MS analysis of liquid fuel was evaluated by GCMS-QP2010 SE SHIMADZU with column Agilent DB-624, length 30 m, internal diameter of 0.25 mm, carrier gas Helium, ionization energy 70 eV.

Extraction of rice husk silica (RHS)

RHS extraction was carried out by adopting the method of Pandiangan *et al.* [24]. A sample of 50 g of dried husk was put into 500 mL of 1.5 % NaOH solution. The mixture was boiled for 30 min, then cooled to room temperature and left for 24 h then filtered to obtain the filtrate (silica sol). For gel formation, 10 % HNO₃ solution was added to the silica sol until the pH of the silica sol was neutral (pH 6.8 - 7.0). The gel was left for 24 h and then washed repeatedly with distilled water to eliminate excess acid. The solid obtained was dried in an oven at 110 °C for 8 h. Furthermore, the solids were ground into powder and sieved through a 250 mesh sieve to obtain a relatively homogeneous sample of silica.

Preparation of hydroxy-sodalite

An attempt to synthesize HS was conducted using the raw material composition by zeolite-A formula Na₂O.Al₂O₃.2SiO₂.xH₂O. For this purpose, a typical synthesis was carried out using 40 g of NaOH pellet, 27 g of FGAF, and 60 g of RHS. The NaOH solution, as a solvent for silica and aluminum foil, was prepared by dissolving NaOH pellets in 350 mL of distilled water. An aliquot of 250 mL of the NaOH solution was used to dissolve 60 g of RHS and the rest 100 mL to dissolve the FGAF. The 2 solutions were mixed and homogenized using a laboratory mixture, and then placed in a polypropylene bottle for the crystallization process. Three samples were prepared and placed in the oven for crystallization, carried out at 100 °C for 72, 96 and 120 h. After the completion of crystallization, each of the samples was transferred into a filtering funnel and rinsed with distilled water to remove any excess of NaOH. The solid was oven-dried for 8 h at

110 °C, and then ground into powder and sieved with a 250 mesh sieve. The samples were calcined for 6 h at 550 °C, identified by XRD and SEM method, and finally tested as a catalyst in experiments of pyrolysis.

Experiment of catalytic activity

To examine the catalytic activity of zeolites, the 3 zeolites were used as a catalyst for pyrolysis of a mixture of water hyacinth with palm oil, and a mixture of cassava solid waste with palm oil. Experiments were conducted with a raw material consists of 100 g of solid biomass (cassava solid waste or water hyacinth), 300 mL of palm oil and 10 g of catalyst were mixed and then removed to a pyrolysis reactor. The pyrolysis experiment was run at temperature of 350 °C and left to continue for 1 h at peak temperature. The product of liquid was collected in a separating funnel and left 24 h, to achieve the separation between organic phase and water phase or bio-crude oil (BCO). The water phase was drained out of the funnel and the BCO was refined using Whatman® filter paper 42. The BCO was investigated by the GC-MS method and the components were determined with the assist of the Wiley 7 database and the software MS Library System NIST62.

Results and discussion

Structure of hydroxy-sodalite

The XRD diffractograms the samples produced at different crystallization times and the crystallite phases identified using the software Match! software (version 3.4.2. Build 96) are presented in **Figure 1**.

As indicated by the XRD patterns shown in **Figure 1**, the existence of the samples as crystalline materials is obvious, although the existence of an amorphous phase should also be acknowledged. These features imply that silica and aluminum have reacted under the experimental conditions applied to produce crystalline materials, but some of the silica and alumina remained unreacted. As can be seen in the results of XRD characterization (**Figure 1**), the products synthesized at 72 and 96 h are characterized by existence of HS as a prominent phase and zeolite A as a minor one. For this reason, it has been suggested that zeolite-A and zeolite-X can be transformed into pure HS by lengthening crystallization time or increase crystallization temperature [7,12]. Naskar *et al.* reported that a small amount of zeolite A particles were observed in HS produced at 90 °C with crystallization time of 6 h, and pure HS particles were obtained when the crystallization times were extended to 10 and 15 h [33]. The results of this current investigation indicate that the formation of HS has taken place with 72 h crystallization time **Figure (1a)** and the HS remains exist for 96 h crystallization time **Figure (1b)**. As can be observed in **Figure (1c)**, when the crystallization time was extended to 120 h, the diffraction peaks associated with zeolite A disappeared, displaying the existence of HS as a pure crystalline phase. In this regard, it can be concluded that high purity HS was produced with 120 h crystallization time.

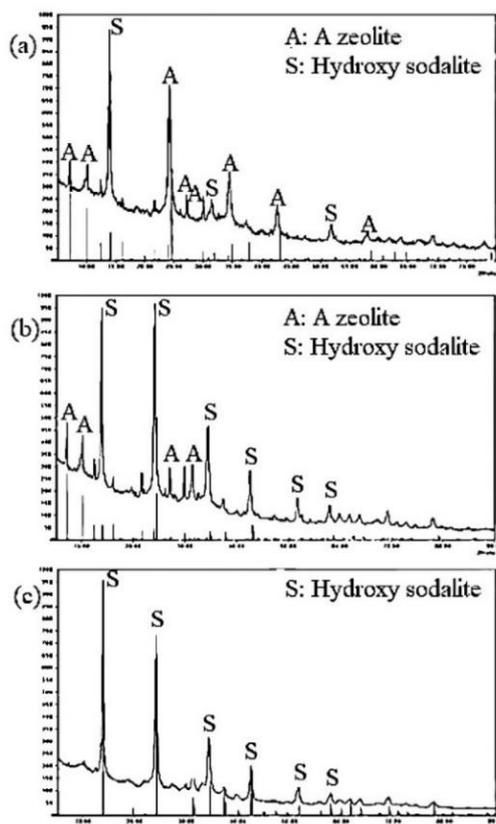


Figure 1 XRD patterns of different crystallization time of (a) 72 h, (b) 96 h, and (c) 120 h.

Coexistence of HS with different zeolite phases has been reported by others carrying out synthesis works using various raw materials. Shabani *et al.* [7] studied the synthesis of HS with hydrothermal method from coal fly ash, mentioned presence of HS together with mullite and quartz. Using coal fly ash for the preparation of HS has also been attempted by Golbad *et al.* [2] and published the formation of HS together with hematite and quartz. The use of the same raw material was also described previously by Yu *et al.* [34], and the product obtained was a mixture of sodalite, nepheline, mullite, and quartz. The synthesis of HS from natural bentonite was carried out by Liu *et al.* [14] and noted the coexistence of HS and 4A zeolite. The use of pure chemicals i.e. sodium aluminate and sodium silicate as raw materials has also reported the coexistence of HS and Linde type A (LTA) zeolite [35]. The used of rice husk silica as raw material for preparation of HS was reported by Naskar *et al.*, in which it was obtained that coral-like HS was produced under hydrothermal condition at 90 °C for 15 h [33].

Hydroxy-sodalite microstructure

The effect of crystallization times on the zeolite microstructure was studied by characterizing the sample by SEM technique, micrographs of the samples are presented in **Figure 2**. The micrographs in **Figure 2**, clearly demonstrate morphology of the surface of the samples, described by the existence of clusters having different sizes and shapes.

The coexistence of HS and zeolite-A as suggested by XRD results is supported by the morphology of surface of the samples as demonstrated by micrographs in **Figure 2**. Crystallization times have an effect on the formation of the HS phase is quite evidently reflected by the images. As shown in the sample produced at 72 h of crystallization time (**Figure 2a**), rod structures spread over the surface and separated from each other. Quite different profiles are observed for the samples prepared with crystallization time of 96 h (**Figure 2b**) and 120 h (**Figure 2c**), in which the images are characterized by the existence of spherical agglomerates of the HS phase. Increased purity of the HS with crystallization times is also reflected by the SEM images as indicated by the XRD results. Concerning the morphology of surface of the samples as viewed by SEM, it can be noted that the shape of HS particles as rod-like structure and formation of spherical agglomerates observed in the present study are comparable with the surface morphology of HS reported by others [2,7,11].

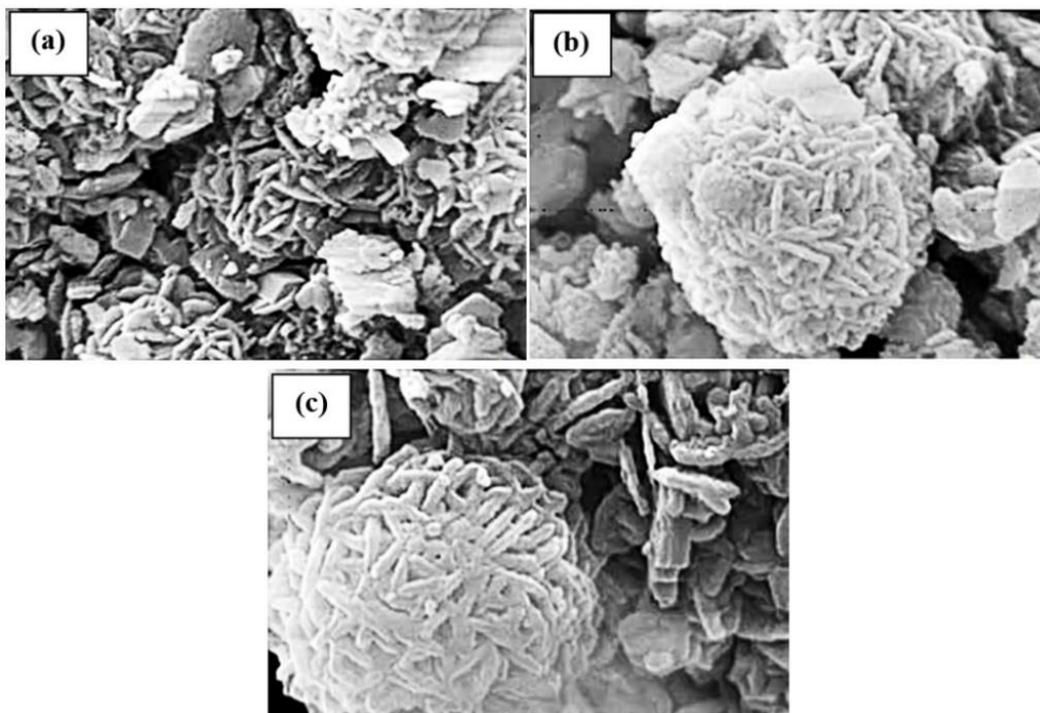


Figure 2 SEM images of the products obtained using crystallization time of (a) 72 h, (b) 96 h, and (c) 120 h.

GC-MS Analysis of pyrolysis products

The first set of pyrolysis experiments was carried for a mixture of water hyacinth with palm oil as raw material and the 3 HS samples as a catalyst. The GC chromatograms of the BCO produced by using the different HS were presented in **Figure 3**.

Figure 3 shows 3 chromatograms that display same general trend, in which most of the compounds have been eluted in less than 30 min suggesting that the components are of relatively low molecular weights. It is also noticed that most of the components have practically the same retention times, indicating that they are the same compounds, but with quite significant differences in relative intensities, suggesting that to some extent the crystallization times applied for preparation of the HS have the effect on the formation of the components during the pyrolysis. With the assist of database of MS Library, the components of the sample were temporarily classified and cataloged in **Table 1**. The relative percentage of each component, determined by dividing the peak range of the component with total peak range of all determined components multiplied by 100, is also included in **Table 1**. The components of the 3 BCO samples are very similar with only view different components.

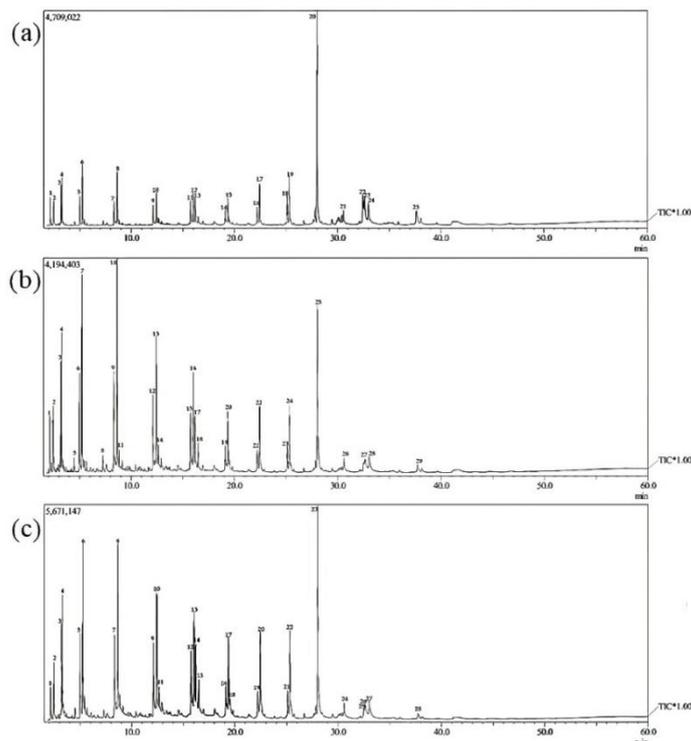


Figure 3 GC chromatograms of the BCO produced by pyrolysis of a mixture of water hyacinth and palm oil using a catalyst prepared with crystallization time of (a) 72 h, (b) 96 h, and (c) 120 h.

In previous studies, pyrolysis of water hyacinth as a single substrate has been conducted by other workers and showed varied chemical compositions of the BCO produced. In the study by Biswas *et al.* [36], it was expressed the composition of BCO encompasses a series of compounds belong to aliphatic hydrocarbons, phenols, ketones, and nitrogen-containing compounds. In another study, Gulab *et al.* [32] was reported that pyrolysis of water hyacinth using Cu and Al catalyst produce BCO with aliphatic hydrocarbons and aromatic hydrocarbons. Santos *et al.* [31] reported BCO produced by pyrolysis of water hyacinth with the composition of o-benzenediol, glycerol, p-benzenediol, hexadecanoic acid, arabinic acid, and levoglucosan and composed of phenols, furans, and nitrides were interpreted by Huang *et al.* [37].

Table 1 Chemical composition of BCO produced by pyrolysis of a mixture of water hyacinth and palm oil by different catalysts.

Compound name	Chemical formula	Catalyst		
		HS-72	HS-96	HS-120
Butane	C ₅ H ₁₂	-	1.74	0.92
Acetone	C ₃ H ₆ O	1.63	-	-
Hexane	C ₆ H ₁₄	2.29	3.33	2.34
Heptene	C ₇ H ₁₄	2.46	3.36	2.67
Heptane	C ₇ H ₁₆	2.83	3.91	4.12
Octene isomer	C ₈ H ₁₆	2.65	3.91	3.61
Octane	C ₈ H ₁₈	5.35	7.90	6.70
Octene isomer	C ₈ H ₁₆	-	1.15	-
Ethylbenzene	C ₈ H ₁₀	-	1.02	-
Nonene isomer	C ₉ H ₁₈	2.22	4.29	3.20
Nonane	C ₉ H ₂₀	5.37	9.60	7.60

Compound name	Chemical formula	Catalyst		
		HS-72	HS-96	HS-120
Nonene isomer	C ₉ H ₁₈	-	1.04	-
Decene isomer	C ₁₀ H ₂₀	2.24	4.38	3.99
Decane	C ₁₀ H ₂₂	3.51	6.75	5.97
Decene isomer	C ₁₀ H ₂₀	-	1.81	1.95
Undecene isomer	C ₁₁ H ₂₂	2.60	3.39	3.89
Undecane	C ₁₁ H ₂₄	3.22	5.08	5.37
Undecene isomer	C ₁₁ H ₂₂	2.88	3.39	4.77
Undecene isomer	C ₁₁ H ₂₂	-	1.61	2.77
Undecene isomer	C ₁₁ H ₂₂	2.78	3.53	4.08
Dodecene isomer	C ₁₂ H ₂₄	-	-	1.43
Dodecene isomer	C ₁₂ H ₂₄	2.21	1.47	1.71
Dodecene isomer	C ₁₂ H ₂₄	4.24	4.14	5.45
Dodecene isomer	C ₁₂ H ₂₄	3.48	1.61	1.82
Dodecene isomer	C ₁₂ H ₂₄	5.72	3.93	5.35
Dodecane	C ₁₂ H ₂₆	24.25	11.73	12.98
Tridecane	C ₁₃ H ₂₈	1.65	0.76	1.24
Tetradecene isomer	C ₁₄ H ₂₈	3.79	-	0.59
Tetradecene isomer	C ₁₄ H ₂₈	4.86	1.55	1.66
Hexadecane	C ₁₆ H ₃₄	2.72	1.31	1.76
2-Heptadecanone	C ₁₇ H ₃₄ O	3.68	0.90	0.49

Remark:

HS-72 indicates to sample prepared with 72 h of crystallization time.

HS-96 indicates to sample prepared with 96 h of crystallization time.

HS-120 indicates to sample prepared with 120 h of crystallization time.

Pyrolysis experiments with the second raw materials (a mixture of cassava solid waste and palm oil) produced the GC chromatograms presented in **Figure 4**. By comparing the chromatograms in **Figure 4** to those displayed in **Figure 3**, it shows that the chromatograms present very identical general features, implying that the BCO samples obtained from different raw materials investigated share the same components. The components of the samples presented in **Figure 4** were identified and their relative percentages were calculated with the same method applied to the data obtained from the first raw material. The results are tabulated in **Table 2**.

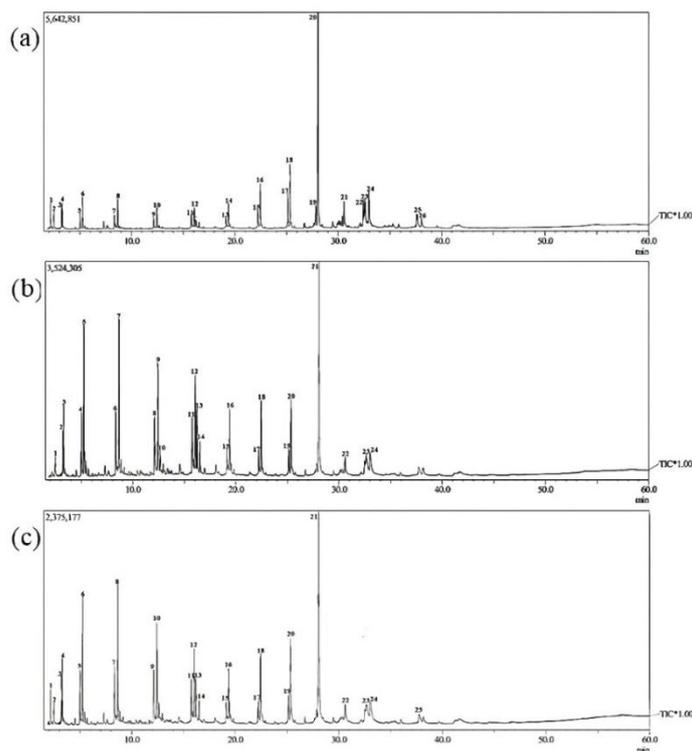


Figure 4 GC chromatograms of BCO produced by pyrolysis of cassava solid waste using a catalyst prepared with crystallization time of (a) 72 h, (b) 96 h, and (c) 120 h.

Table 2 shows the compositional features of the samples are very similar to those of the results presented in **Table 1**, both characterized by the presence of hydrocarbons as the main constituents by only 2 oxygen compounds identified. The types of components are also very similar.

Table 2 Chemical composition of BCO produced by pyrolysis of a mixture of cassava solid waste and palm oil by different catalysts.

Compound name	Chemical formula	Catalyst		
		HS-72	HS-96	HS-120
Acetone	C ₃ H ₆ O	1.92	-	1.20
Hexane	C ₆ H ₁₄	1.93	1.15	1.39
Heptene	C ₇ H ₁₄	1.28	1.77	1.64
Heptane	C ₇ H ₁₆	1.61	2.52	2.48
Octene isomer	C ₈ H ₁₆	1.53	3.12	2.88
Octane	C ₈ H ₁₈	3.23	7.31	6.92
Nonene isomer	C ₉ H ₁₈	1.45	3.37	3.15
Nonane	C ₉ H ₂₀	3.27	8.30	7.69
Decene isomer	C ₁₀ H ₂₀	1.52	4.22	4.00
Decane	C ₁₀ H ₂₂	2.51	6.79	6.27
Decene isomer	C ₁₀ H ₂₀	1.76	1.86	-
Decene isomer	C ₁₀ H ₂₀	-	3.98	3.17
Undecane	C ₁₁ H ₂₄	2.42	5.89	4.68
Undecene isomer	C ₁₁ H ₂₂	1.34	4.27	3.09
Undecene isomer	C ₁₁ H ₂₂	2.90	1.86	1.31

Compound name	Chemical formula	Catalyst		
		HS-72	HS-96	HS-120
Undecene isomer	C ₁₁ H ₂₂	-	1.69	1.45
Undecene isomer	C ₁₁ H ₂₂	-	4.31	3.85
Undecene isomer	C ₁₁ H ₂₂	-	1.56	1.81
Dodecene isomer	C ₁₂ H ₂₄	2.04	5.02	5.16
Dodecene isomer	C ₁₂ H ₂₄	4.83	1.92	2.30
Dodecene isomer	C ₁₂ H ₂₄	4.28	5.08	6.37
Dodecene isomer	C ₁₂ H ₂₄	7.21	-	-
Dodecene isomer	C ₁₂ H ₂₄	2.91	-	-
Dodecane	C ₁₂ H ₂₆	24.89	16.59	20.40
Tridecene isomer	C ₁₃ H ₂₆	1.76	-	-
Tridecane	C ₁₃ H ₂₈	3.36	1.05	1.36
Tetradecene isomer	C ₁₄ H ₂₈	4.38	-	-
Tetradecane	C ₁₄ H ₃₀	6.00	-	-
Hexadecene isomer	C ₁₆ H ₃₂	-	3.22	3.35
Hexadecane	C ₁₆ H ₃₄	6.26	3.15	4.09
2-Heptadecanone	C ₁₇ H ₃₄ O	3.40	-	-

Remark:

HS-72 indicates to sample prepared with 72 h of crystallization time.

HS-96 indicates to sample prepared with 96 h of crystallization time.

HS-120 indicates to sample prepared with 120 h of crystallization time.

Several workers have conducted pyrolysis of cassava solid waste, however, the main purpose of the studies was for the production of biochar [29-32]. For the production of BCO, pyrolysis of cassava solid waste was described by several workers with various compositions. The composition encompasses acids, aldehydes, ketones, phenols, and hydrocarbons was noted by Pattiya *et al.* [42], composed of acids, alcohols, aldehyde, ketone, ester, ethers, and phenol was informed by Zhang *et al.* [43], and composed of methyl cis-9octadecenoate, octadecanoic acid and hexadecanoic acid methyl ester as the prominent constituents were detailed by Nonchana and Pianthong [44].

The results of pyrolysis experiments indicate that hydrocarbons are the main constituents of the BCO samples obtained from the 2 sets of raw materials. For the samples produced from pyrolysis of a mixture of water hyacinth and palm oil, hydrocarbons were found to contribute 94.69 - 99.51 % to the composition. Another interesting compositional profile of the BCO samples observed is that in terms of the carbon chains, most of the hydrocarbons are in the gasoline range (C₅-C₁₂), account for 81.67 - 96.24 % of the composition. For the samples produced from the mixture of cassava solid waste and palm oil, the products obtained indicate content of hydrocarbons in the range of 94.68 - 100 %, with gasoline contents, account for 72.92 - 92.58 %. The formation of only 2 oxygenated compounds in the BCO samples indicates that the zeolites synthesized have high activity to promote deoxygenation reactions during the pyrolysis process, which is acknowledged as the main advantage offered by zeolites in general.

Conclusions

The experimental results of this research exhibited that hydroxy-sodalite (HS) could be produced from food-grade aluminum foil and rice husk silica as raw materials. Results from XRD technique indicate that a mixture of HS and zeolite-A was produced by application of crystallization time of 72 and 96 h, and practically pure HS was produced with extended crystallization time to 120 h. The pyrolysis experiments suggest that HS either coexists with zeolite-A or in a pure state display attractive activity as a catalyst for biomass pyrolysis as a catalyst for pyrolysis of biomass capable of producing biogasoline type as a liquid hydrocarbon product. The BCO with biogasoline contents in the range of 81.67 - 96.24 % has resulted from the mixture of water hyacinth and palm oil, and in the range of 72.92 - 92.58 % was produced from the mixture of cassava solid waste and palm oil.

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