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Abstract

Charcoal of coconut shell is produced by pyrolysis at 300^{O}C and chemically activated using HCl. Both pyrolysis charcoal and chemical activation were measured and analyzed by Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM), and Energy Dispersive Spectroscopy (EDS). The functional groups found O-H, aliphatic C-H, aromatic C=C, C-O, and aromatic C-H. The distribution of pyrolysis charcoal pore length and chemical activation increased from 362 nm-1.02 μm to 1.10 μm-1.52 μm. The composition of carbon elements in the mass % of pyrolysis charcoal and chemical activation increased from 86.11% to 88.93%.

Keywords

Coconut Shell Charcoal, Pyrolysis, Activation, FTIR, SEM-EDS

RESEARCH PAPER

Study of Pore Length and Chemical Composition of Charcoal that Results From the Pyrolysis of Coconut Shell in Bolaang Mongondow, Sulawesi, Indonesia

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Abstract

Charcoal of coconut shell is produced by pyrolysis at 300 °C and chemically activated using HCl. Both pyrolysis charcoal and chemical activation were measured and analyzed by Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM), and Energy Dispersive Spectroscopy (EDS). The functional groups found O−H, aliphatic C−H, aromatic C=C, C−O, and aromatic C−H. The distribution of pyrolysis charcoal pore length and chemical activation increased from 362 nm-1.02 μm to 1.10 μm−1.52 μm. The composition of carbon elements in the mass % of pyrolysis charcoal and chemical activation increased from 86.11% to 88.93%.

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1. Introduction

The main products of coconut are coconut meat, coconut fruit, while the products are coconut water, coir, and shell. Coconut shells are considered waste. The amount of coconut shell waste is considered to affect the cleanliness of the environment. However, coconut shells are inexpensive and can be renewed. Activated carbon is one of the processed products from coconut shell charcoal. Activated carbon has a high economic value [1].

One of the products made from coconut shell has economic value is activated charcoal. Coconut shells are used as fire stoves (burned). Other alternatives are activated charcoal, liquid smoke, ornaments, and crafts [2]. Coconut shells go through a pyrolysis process into charcoal due to their excellent hardness, high bound carbon content, and low mineral ash content. The main product of pyrolyzed coconut shells is charcoal with volatile components, water, and ash. Bonded carbon, water, ash, nitrogen, and sulfur are the components contained in coconut shell charcoal [3].

Cellulose, hemicellulose, lignin with C, O, H, and N atoms are the main component of the coconut shell. The functional groups vary, namely carbonyl (R−CO−R'), ester (R−CO−O-R'), alkane (R-(CH2)n-R'), carboxyl (R−COOH), hydroxyl (R−OH), cyclic (RO-R'), and linear ether groups. The most common chemical reaction is combustion, which combines fuel with oxygen to form product compounds. This chemical transformation is potential energy at the molecular scale. In this case, it is related to atomic positions and molecular structures [4–7].

Charcoal is a porous material, where most of the pores are covered by tar, hydrocarbons, and other organic compounds. Charcoal can be made by direct or indirect heating in heaps or kilns. This

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decomposition process, apart from charcoal, can produce other products such as distillate and gas. Products which have commercial value are mainly charcoal [3].

There are two steps to make activated charcoal, namely pyrolysis (carbonization) and activation. Pyrolysis is an indoor combustion process without oxygen and other chemicals [8−10]. While the activation process aims to break the hydrocarbon bonds to increase pore length, the charcoal's chemical or physical properties can change [11,12].

Activated carbon contains 85%−95% carbon. Activated carbon can be produced by heating at high temperatures. The characteristic of activated carbon is porous, it is widely used as an adsorbent. For example, in the process of purification of taste, smell, purification materials in the food industry, it is even used in the water purification process.

Coconut shell charcoal has been studied as a carbon graphite material by heating treatment up to 1500 °C, using solvent mixing and powder mixing methods. XRD diffractogram pattern shows semi-crystalline carbon material [11].

In this study the coconut shells from Bolaang Mongondow district, with high air temperatures and located near the coast. Coconut trees near the coast affect the composition of the constituent elements of coconut shells. This study aimed to examine the changes in the character of coconut shell charcoal which was pyrolyzed at 300 °C and activated charcoal using an HCl activator. HCl activator has a high ability in the process of refining charcoal or carbon materials.

The chemical activation process activates coconut shell charcoal by adding certain chemicals to the sample to reduce the water content that is still left on the surface of the coconut shell charcoal, the pores are more open [13,14]. Furthermore, coconut shells charcoal was characterized using Fourier Transform Infrared (FTIR), Scanning Electron Microscope (SEM), and Energy Dispersive Spectroscopy (EDS). FTIR is used to determine the functional groups contained in charcoal based on the absorption band pattern at wave number. SEM to determine the pore length and morphology of coconut shell charcoal. EDS to determine the composition of elements and compounds contained in coconut shell charcoal. The SEM-EDS instrument used in testing the chemical composition and morphology has the advantage of using digital data through measurements.

Coconut shell charcoal resulting from pyrolysis and activation with a high chemical composition of carbon elements is very potential as a raw material for SiC functional material that can be applied as a biosensor.

2. Material and methods

2.1. Tools and materials

The tools used are glassware commonly used in the laboratory, oven, furnace, porcelain dish, evaporation dish, analytical balance, spatula, magnetic stirrer, 100 mesh pass sieve, dropper pipette, desiccator, crucible, mortar and pestle, SEM-EDS JCM-6000 PLUS instrument. The materials used are coconut shell (from Bolaang Mongondow, Sulawesi, Indonesia), HCl, equates, filter paper, universal indicator, and aluminum foil.

2.2. Research procedure

The coconut shell samples were cleaned of the attached coconut husks and dried in the sun to remove the water content. Coconut shell samples were pyrolyzed in a pyrolysis reactor and heated to 300 °C for 4−6 h. The resulting charcoal is allowed to be cold, and then the charcoal is ground until smooth using a mortar and sieved using 100 mesh sieves.

A total of 20 g of coconut shell charcoal passed 100 mesh sieves was immersed in 100 mL of 3 M HCl solution for 24 h. The mixture was filtered using filter paper, then washed with distilled watermarked until the washing solution was neutral ± pH = 7. Then the residue was taken and dried in an oven at a temperature of 110−120 °C for 3 h. After that, it is stored in a desiccator until the charcoal is stable [8,13].

FTIR measurement using the Shimadzu FTIR Spectrophotometer IRprestige-21 type works in a scan range of 4000−340 cm^{-1}, resolution 4 cm^{-1}, and scans 2−3 s. FTIR analysis using IR solution software for spectrum measurement and peak detection. The JCM-6000 PLUS SEM tool works at a voltage of 15.00 kV, energy range 0−20 keV. The SEM tool is equipped with the JED-230 Analysis Station Plus for EDS analysis. The JED-2300 Analysis Station Plus is equipped with JEOL's DrySD™ (Dry Silicon Drift Detector), a high-speed analyzer, and analytical software specially designed for JEOL electron microscopes. Particle analysis software is used to determine the pore length of charcoal.

3. Results and discussion

3.1. Analysis of FTIR

FTIR analysis was used to determine the functional groups contained in charcoal based on the absorption band pattern at the wavenumber. The absorption band pattern of pyrolyzed coconut shell charcoal at 300 °C

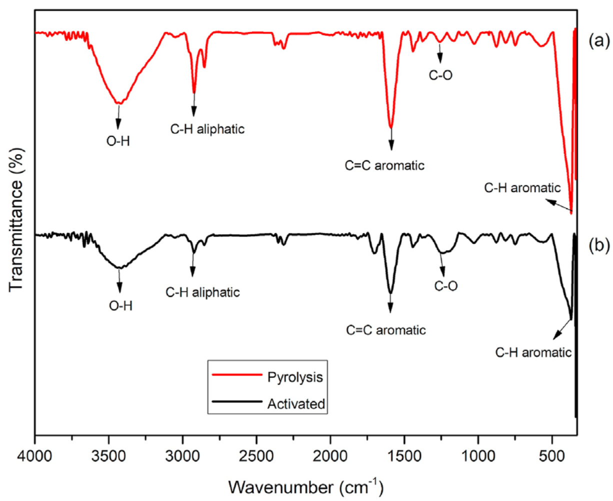

Fig. 1. FTIR spectrum of coconut shell charcoal (a) pyrolysis and (b) activated.

and activated by HCl is shown in Fig. 1. The FTIR spectrum of pyrolyzed and activated coconut shell charcoal has almost the same absorption band pattern. The comparison of the spectrum shows that absorption occurs but experiences a shift in wavenumber. The decrease in absorption intensity also occurred at wavenumbers of 3415.93 cm^{-1}, 2922.16 cm^{-1}, 2852.72 cm^{-1}, 1591.27 cm^{-1}, and 370.33 cm^{-1}. There is also a new absorption of activated charcoal, namely at wavenumbers 1283.30 cm^{-1} and 1199.72 cm^{-1}. The absorption loss and the decrease in the absorption intensity show the reduction of impurities in the charcoal. It indicates there will be the formation of aromatic compounds, which are the constituents of the hexagonal crystalline structure of charcoal.

The functional groups identified can be seen in Table 1. The absorption appears at a wavenumber of around 3400 cm^{-1} which indicates the presence of O−H groups. The absorption that occurs at wavenumbers 2900 to 2800 cm^{-1} indicates the presence of aliphatic C−H groups. Absorption at wave number 1500 to 1400 cm^{-1} indicates the presence of aromatic C=C, which is a hexagonal form of charcoal. The presence of C−O groups indicated the absorption at

Table 1. The absorption band of the FTIR spectrum of coconut shell charcoal produced by pyrolysis and activation.

Wavenumber (cm^{-1})		Functional groups
Pyrolysis	Activated	
3415.93	3415.93	O−H
2922.16	2922.16	C−H aliphatic
1589.34	1591.27	C=C aromatic
1259.52	1283.30	C−O
	1199.72	
370.33	370.33	C−H aromatic

wavenumber 1300 to 800 cm^{-1}. Furthermore, absorption at a wavenumber of 700 to 300 cm^{-1} indicates the presence of aromatic C−H groups from hydrocarbons. It shows the appearance of the C=C group is a characteristic of the structure of carbon charcoal [15–17].

3.2. Analysis of SEM

The pyrolysis or carbonization process is an important step to determine properties of final product obtained. Pyrolysis aims to increase carbon levels, where combustion without using oxygen (O_2). Ground the pieces of coconut shell charcoal produced by pyrolysis to increase the surface area. Further testing of the pore structure and functional groups was conducted. This procedure makes this research different from previous studies.

Fig. 2 shows an SEM image of coconut shell charcoal with ×1000, ×3000, and ×5000 magnifications. Figs. 2 (a to c) for pyrolyzed coconut shell charcoal. Fig. 2 (d to f) for activated coconut shell charcoal. The application of the chemical HCl to the coconut shell charcoal sample aims to dissolve the metals to increase C content. The distribution of the pores of coconut shell charcoal is not uniform [18]. It appears, the sample is in the form of granules caused by grinding. As a result of the grinding technique on the model resulted in the sample experiencing crack (Fig. 2f). This result agrees with the theory, which states coconut shell charcoal is a porous material. The non-uniformity of the pore structure indicates the tar filled in some of the pores [14]. In addition, the outer surface of the coconut shell charcoal looks rough associated with the rupture of the structure resulting from the release of more volatile elements due to heating treatment at high temperatures [19–21].

Fig. 3 shows the length of the pores in the SEM image of the pyrolyzed coconut shell charcoal sample and activated with a magnification of ×10000. It appears that pore length of coconut shell charcoal produced by pyrolysis and activation varies. The pore length of pyrolyzed and activated coconut shell charcoal can be seen in Table 2. The pore length of pyrolyzed coconut shell charcoal ranges from 362 nm to 1.02 μm. Meanwhile, the pore length of activated coconut shell charcoal is 1.10 μm−1.52 μm. The pore length of activated coconut shell charcoal is greater than pyrolysis. It was indicated by the presence of pores in the sample immersed in HCl. Metallic elements dissolve in HCl. Although the activated charcoal pore length is still not uniform, the pore lengths are not too considerable from one another.

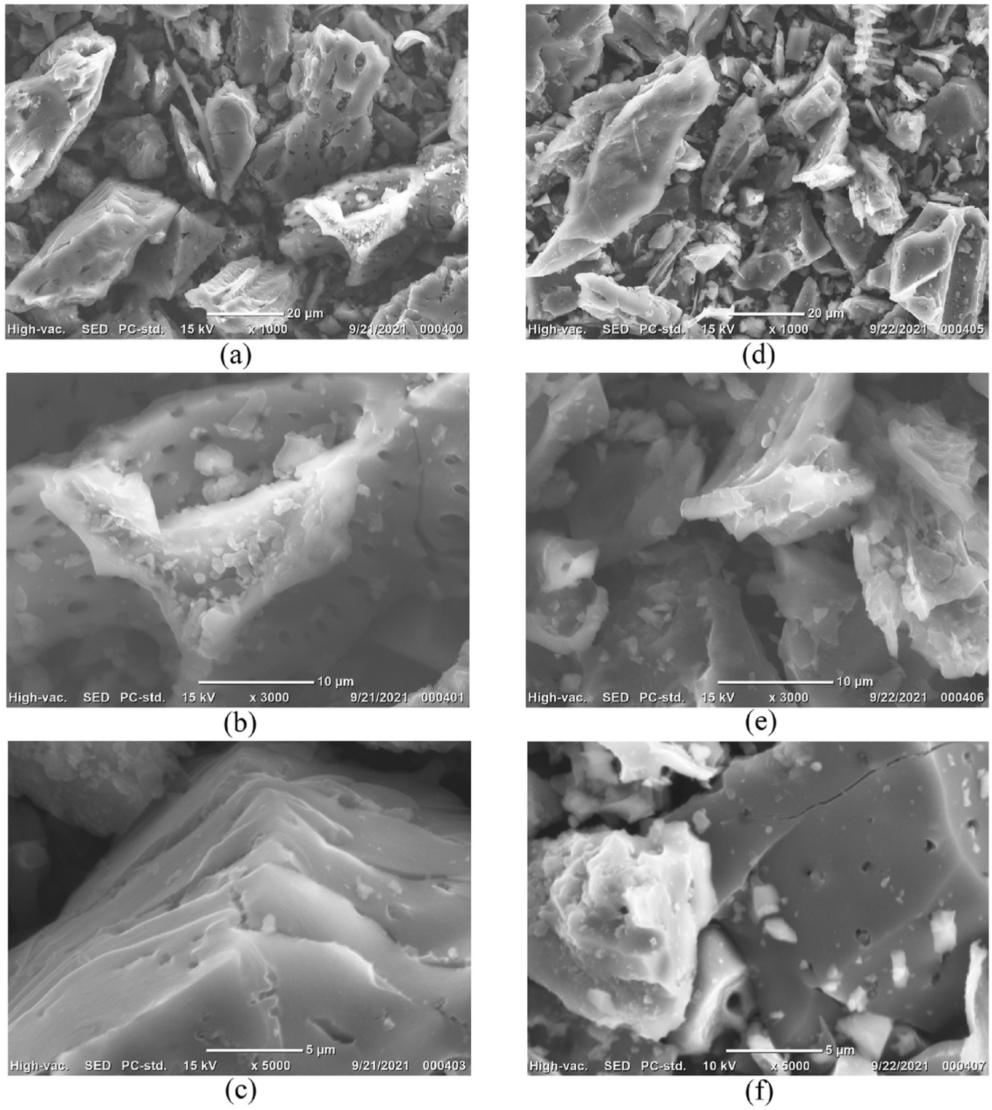

Fig. 2. SEM image of coconut shell charcoal with magnifications of ×1000, ×3000, and ×5000 (a–c) pyrolysis (d–f) activated.

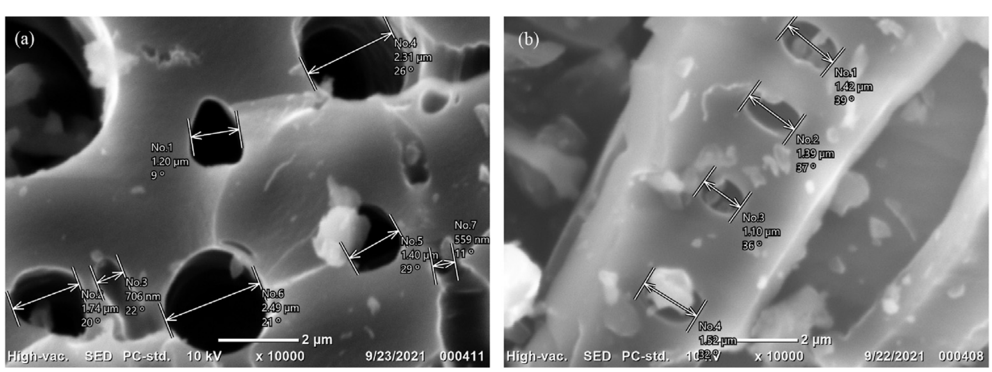

Fig. 3. SEM image of coconut shell charcoal pyrolysis with ×10000 magnification (a) pyrolysis (b) activated.

The results show these pores are macropores [22–24], with more than 50 nm pore length. The non-uniformity of pore length is influenced by chemical factors [25], in this case, HCl. In addition, it is influenced by chemical composition, which still contains other chemically bound elements such as

Table 2. Pore length of pyrolysis and activated coconut shell charcoal.

Pyrolysis		Activated	
Length	Unit	Length	Unit
1.02	μm	1.42	μm
525	nm	1.39	μm
555	nm	1.10	μm
362	nm	1.52	μm
513	nm		
692	nm		
714	nm		
432	nm		
466	nm		
521	nm		

oxygen and hydrogen [26,27]. These elements resulted from raw materials left behind due to incomplete carbonization processes. Therefore, the pyrolysis process requires higher heating for the cellulose, hemicellulose, and lignin evaporate [28].

3.3. Analysis of EDS

Furthermore, the pyrolyzed and activated coconut shell charcoal was subjected to EDS testing to show the elemental composition based on energy levels. The EDS spectrum and the results of the EDS analysis of pyrolysis and activated coconut shell charcoal samples can be seen in Fig. 4 and Table 3. It appears that the amount of kinetic energy of the photo-electrons of each element is different (Table 3). The graph of the photo-electron kinetic energy of coconut shell charcoal produced by pyrolysis can be seen in Fig. 5a. Meanwhile, the chart of the kinetic energy of activated photo-electrons of

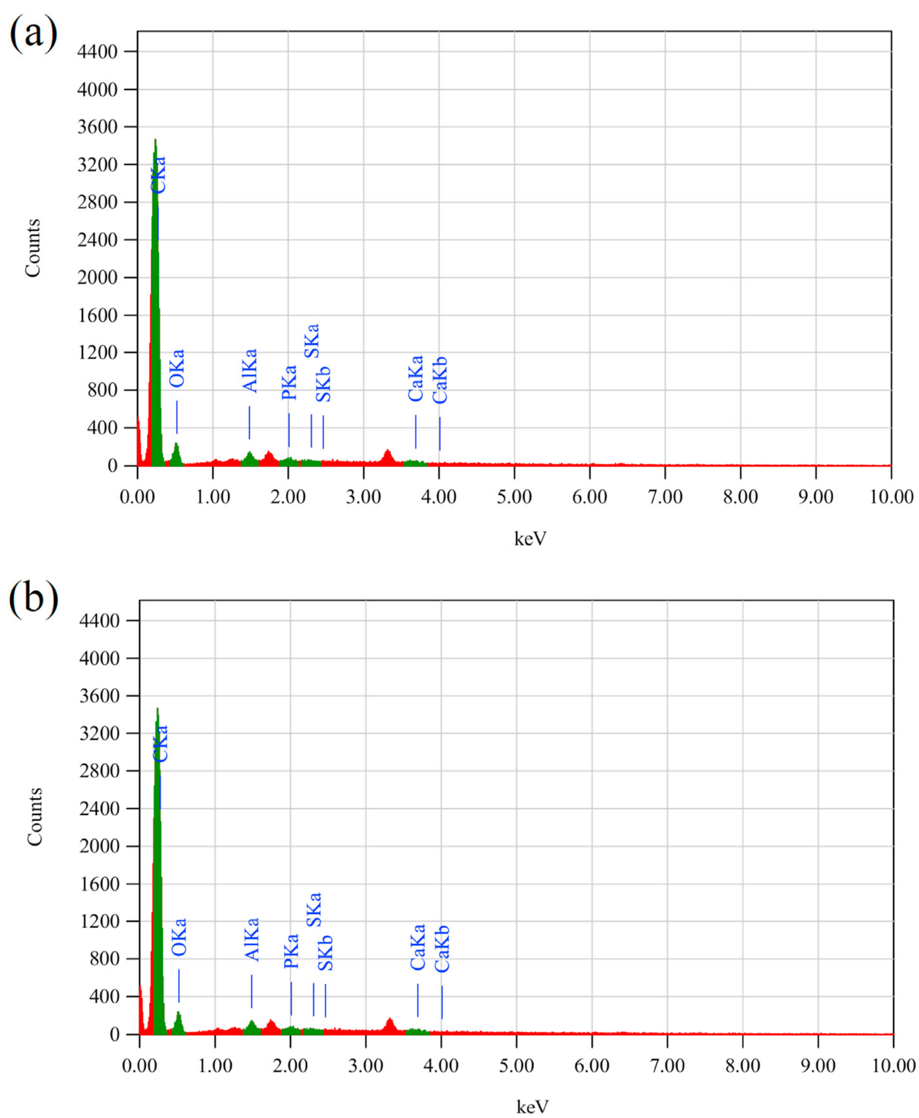

Fig. 4. EDS spectrum of coconut shell charcoal (a) pyrolysis and (b) activated.

Table 3. Results of EDS analysis of pyrolysis and activated coconut shell charcoal.

No	Pyrolysis					Activated			
	Element	Photo-electron KE (keV)	Mass (%)	Atom (%)	Mole (%)	Photo-electron KE (keV)	Mass (%)	Atom (%)	Mole (%)
1	C	0.277	86.11	91.29	98.25	0.277	88.93	92.60	99.11
2	O	0.525	7.46	5.94	ND	0.525	7.64	5.97	ND
3	Al	1.486	3.63	1.72	0.92	1.486	1.69	0.78	0.42
4	P	2.013	1.43	0.59	0.32	2.013	1.00	0.40	0.22
5	S	2.307	0.44	0.18	0.19	2.307	0.11	0.04	0.05
6	Ca	3.690	0.93	0.3	0.32	3.690	0.64	0.20	0.21
	Total	10.298	100	100	100	10.298	100	100	100

coconut shell charcoal can be seen in Fig. 6a. The amount of photo-electron kinetic energy of coconut shell charcoal resulting from pyrolysis and activation is the same. Element C has the minor photo-electron kinetic energy of 0.277 keV, while element Ca has the most considerable photo-electron kinetic energy of 3.690 keV.

There is no difference in the photo-electron kinetic energy of each element in the pyrolysis and activated coconut shell charcoal samples due to the characteristics possessed by each element. It means each element of the EDS test results has a different atomic number, atomic mass, and density. In the JCM-6000 PLUS instrument, the electrons generated by the electron gun are accelerated and illuminate the sample. Electrons have kinetic energy is proportional to accelerate voltage. If the incoming electron has sufficient acceleration and enough power to strike the electron from the atom's inner shell, it will create an electron–hole. It causes the bit to become unstable. As a result, an electron from the outer shell will fill the gap, producing energy emission of X-rays. The X-ray characteristics of each element are different, and their intensity will be proportional to the element's concentration. At the same time, the EDS background spectrum shows a

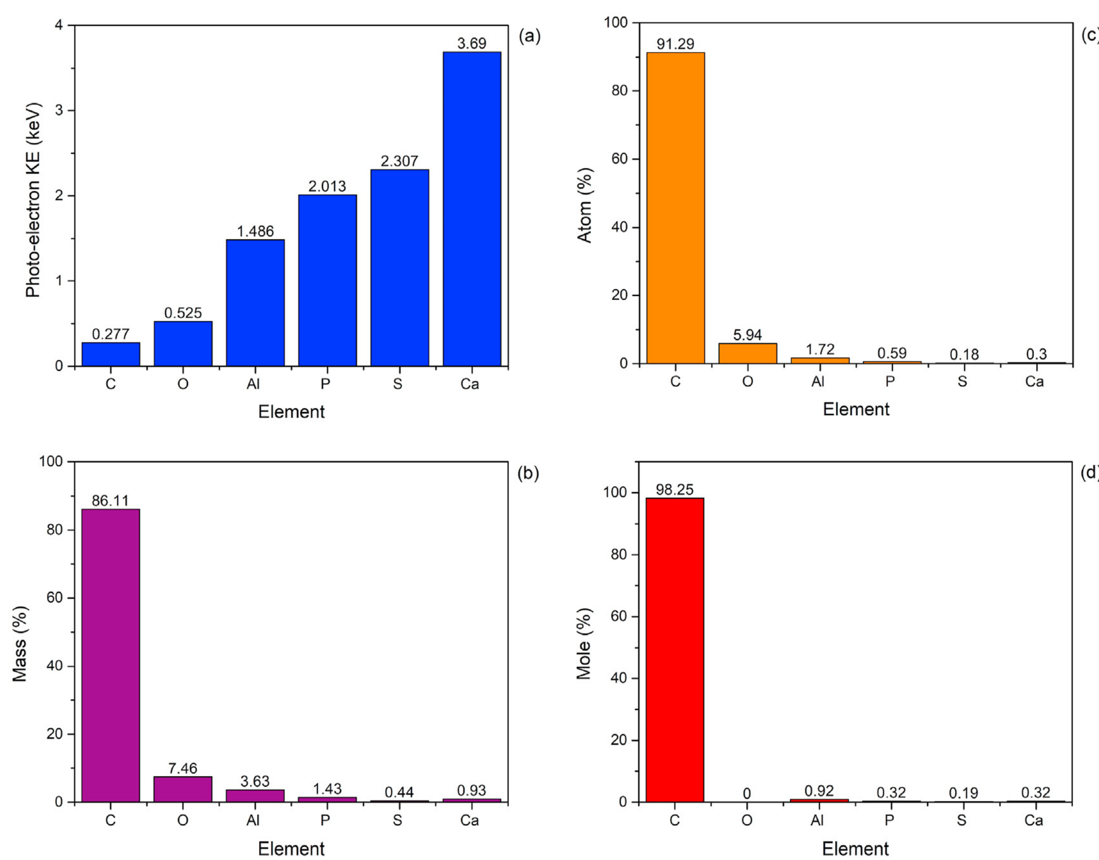

Fig. 5. Graph of (a) KE of photoelectrons, (b) % mass, (c) % atoms, and (d) % moles of pyrolysis coconut shell charcoal.

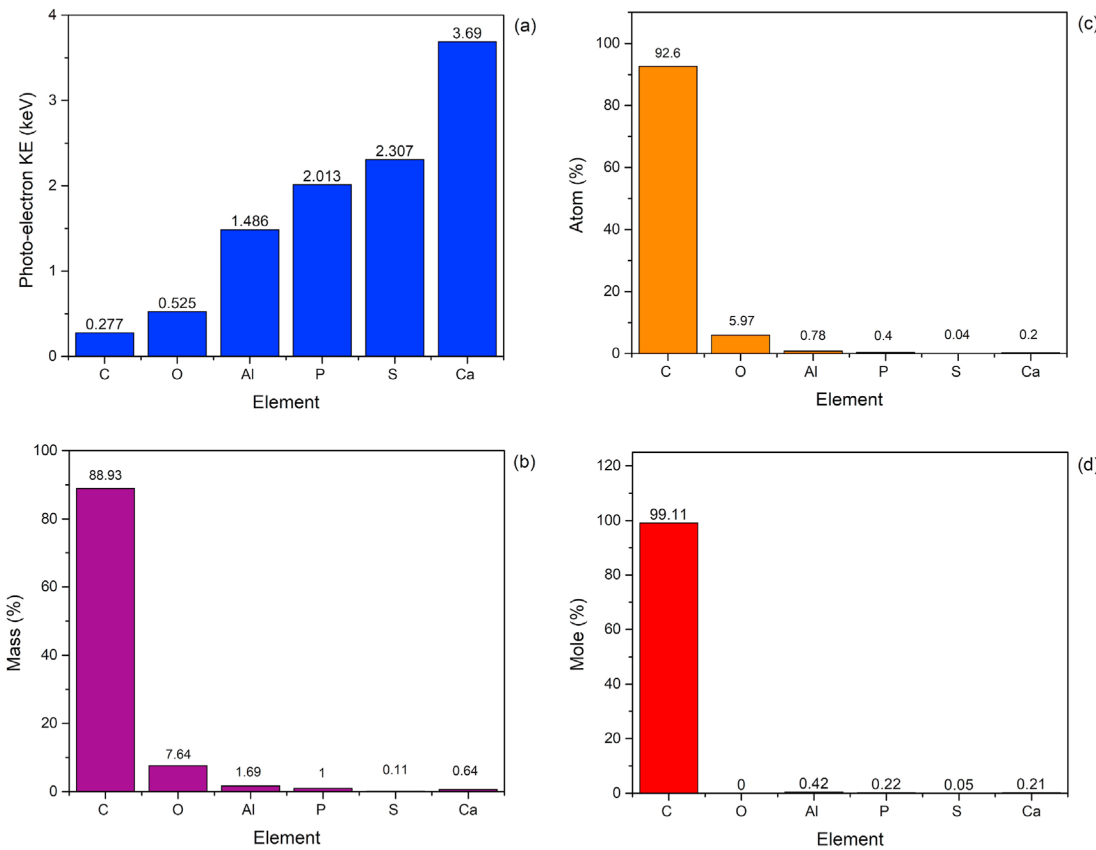

Fig. 6. Graph of (a) KE of photo-electrons, (b) % mass, (c) % atoms, and (d) % moles of activated coconut shell charcoal.

continuum of X-rays emitted when a strong electric field slows down the primary electrons near the atomic nucleus. Continuous X-rays have kinetic energy is lost during deceleration.

Other information obtained from the results of the EDS analysis is mass %, atomic %, and mole % of each element contained in the pyrolysis and activated coconut shell charcoal samples (Table 3). The graph of % mass, % atom, and % mole of pyrolysis coconut shell charcoal can be seen in Fig. 5 (b−d). In contrast, the graph of % mass, % atom, and % mole of activated coconut shell charcoal can be seen in Fig. 6 (b−d). The results show that element C has the most significant value for each of these items. % mass, % atom, and % mole of element C charcoal pyrolysis results are 86.11%, 91.29%, and 98.25%, respectively. Meanwhile, % mass, % atom, and % mole of element C charcoal were activated respectively 88.93%, 92.60%, and 99.10%. Inactivated coconut shell charcoal increased in % mass, % atom, and % mole for element C and decreased in other elements contained in the sample. The presence of O, Al, P, S, and Ca indicates the carbonization process is not perfect. It is in agreement with the SEM results. An increase in carbon content is associated with increasing the number of pores [29,30].

The pore length of pyrolyzed coconut shell charcoal and activated charcoal showed an increase in the pore length of coconut shell charcoal. This increase was due to the loss of impurity elements, namely O, Al, P, S, and Ca. The breaking of the bonds of the minor elements causes the arrangement of the carbon atoms in the activated coconut shell charcoal to occur. The chemical composition of coconut shell charcoal as a result of pyrolysis and activated charcoal results in an increase in the composition of carbon (C) and a decrease in the composition of minor elements. This also corresponds to a decrease in the intensity of the impurity and an increase in the intensity of the carbon functional group. These results indicate activated coconut shell charcoal is getting purer by using an HCl activator. Pore length and chemical composition indicate the purity of activated coconut shell charcoal. Activated coconut shell charcoal is controlled by the purity and regularity of the structure of the carbon material for pyrolysis temperature and activation method can determine the specificity of the physicochemical character of the activated charcoal produced.

4. Conclusions

The results of the FTIR analysis showed the appearance of the O−H groups, aliphatic C−H groups, aromatic C═C, which is a hexagonal form of charcoal, C−O groups, aromatic C−H groups from hydrocarbons. The formation of the two functional groups shows the characteristics of the structure of carbon charcoal. The SEM analysis results showed an increase in the pyrolysis pore length of coconut shell charcoal. It activated coconut shell charcoal in the range of 362 nm to 1.02 μm and 1.10 μm−1.52 μm, respectively. The surface structure shows the particle distribution is not homogeneous. The percentage of carbon elements from the EDS analysis showed an increase in the elemental composition of carbon (C) produced by pyrolysis and activated charcoal, namely by 86.11% and 88.93% by mass. The activation process with 3 M HCl can enlarge the pores of the charcoal and increase the amount of carbon and reduce the composition of minor elements contained in coconut shell charcoal. The pore length of charcoal from SEM analysis showed an increase in pyrolysis charcoal. The increase in the carbon composition of activated charcoal shows the charcoal is getting purer where there is a regular arrangement of carbon atoms. The increase in pore length, loss of functional groups, and increase in carbon composition showed an increase in the pattern of structural regularity in the charcoal. Activated charcoal as a carbon material has the potential to be applied as a functional Si−C material for biosensors.

Acknowledgments

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