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Quality of Microporous Activated Charcoal from Coconut Shell Waste in Industrial Scale

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Due to its diverse applications in the adsorption process, the activated charcoal encourages the manufacturing technology to produce high-quality products. In present study, an industrial-scale activated charcoal was prepared in activated charcoal manufacturing companies using coconut shells waste as raw material. The process of making activated charcoal begins with preparation, carbonization and activation of charcoal into activated charcoal using the addition of steam as an activator. The resulting activated charcoal products meet the quality requirements of the Indonesian National Standard (SNI). The best-activated charcoal product from PT Nui Hia Prima produces iodine adsorption reaching 876.2 mg/g with the surface area of 504.306 m^2/g and a total pore volume of 0.298 cm^3/g. After some treatment with steam addition, the best product resulting in iodine and methylene blue adsorption are 973.9 mg/g and 121.25 mg/g, respectively. The development of the activated charcoal products expected to adjust with a technology that is more capable of producing better quality activated charcoal.

Keywords: Activated charcoal, Industry, Coconut shell.

INTRODUCTION

Activated charcoal can be used as an adsorbent for contaminants of organic compounds, heavy metals [1], synthetic dyes [2], polluted gases, plant media and fertilizers [3]. Even as technology advances, activated charcoal can be used as a catalyst [4-9], supercapacitor material [10], food packaging development, smart textiles, and more advanced nanomaterial products. Activated charcoal itself can be produced from various raw materials, especially lignocellulose from the forestry, agriculture, plantation and even biomass waste sectors [11-13].

A large number of activated charcoal uses have resulted in a high market demand. Therefore, it cannot even be handled domestically so that the activated charcoal in Indonesia is mostly supplied by foreign products (imports). The import value of activated charcoal on the last record was US $ 16 million, with an increasing trend of around 5% per year. This unfortunate condition because Indonesia has enormous potential as an activated charcoal producer with a demographic bonus so that the raw materials can be obtained diversely and abundantly.

One of the raw materials which is abundant, easy, cheap and renewable is coconut. Coconut is a tropical commodity which is generally used as food additives as drinks, oil and flour. Thus, in addition to obtaining these products, coconut processing produces lot of waste in the form of coconut shells. Indonesia is the largest coconut producer with a production output of around 18 million tons. Coconut plants in the Maluku Islands, to be precise in the Sula district are quite a lot and not fully utilized. Coconut shell is also known to have high carbon content with low ash content, so it meets good criteria for processing into activated charcoal. Coconut shells can be used as raw materials for activated charcoal with a high iodine adsorption capacity ranging from 448-1599 mg/g [14]. Several studies have also examined activated charcoal from coconut shells for various applications including colour removal [15], nickel waste removal [16], electrodes [17], adsorbents enrofloxacin [18], phenol adsorbent [19], gold leaching [20], methylene blue adsorbent [21], *p*-cresol adsorbent [22,23], catalysts [24] and water quality improvement [25]. From this explanation, it is very promising to produce activated charcoal on a large scale and provide economic added value to coconut shell waste.

To meet the high demand for activated charcoal, we should develop technology for making activated charcoal so that it can be used appropriately. Therefore, the production of coconut shell-activated charcoal on an industrial scale is conducted so that it can be studied more deeply.

PT Nui Hia Prima is one of the start-up company located in Sula District, North Maluku, established in 2019 as a local pioneer of industrial manufacture with coconut as a raw material in order to utilize a natural resources around this company. The business process based on good intention to develop local potential for economy improvement. PT Nui Hia Prima collaborated with Forestry Research and Development Center to develop charcoal production and process including technology manufacture. This research aims to study the quality of activated charcoal from industry and hopefully, in the future, it can become an economic source of the community in the Sula area of Indonesia.

EXPERIMENTAL

The instrument used in this study were carbonization reactor, activation reactor, porcelain dish, oven, furnace, shaker, four decimal analytical balance, 100 mesh sieve, a set of glassware and universal pH. Ultimate analyzer (LECO), surface area analyzer BET (JWGB Meso 112, China), UV-Vis spectrophotometer (Shimadzu Japan) and scanning electron microscope (SEM) (ZEISS) were used for analysis. The materials used are coconut shell from Sula area, 0.1 N iodine solution, 1% starch solution, and 0.1 N $Na_2S_2O_3$. The experiments were performed in the Forest Product Research and Development Center. The product quality was assessed using the Indonesian National standards.

Preparation and carbonization of coconut shells waste: The research procedure has several stages, namely preparation of coconut shell raw materials, dehydration and carbonization to produce charcoal products and activation of charcoal products with various conditions. Charcoal production was carried out by collecting coconut shell waste in the PT Nui Hia Prima factory area, Sula District, then cleaning it of contaminants. The coconut shells were then carbonized by a pyrolysis process using oxygen limitation. The pyrolysis temperature was adjusted to 400 ºC for 4 h. Then, the charcoal is allowed to reach room temperature and the coconut shell charcoal product is obtained

Charcoal activation: Charcoal activation is carried out physically (steam) with heating time variations, steam time variations and holding time variations (Table-1).

Coconut shell charcoal, as much as 300 kg was heated with time variation (Table-1). The addition of steam flowed then the storage was carried out by holding it for 4 h. Furthermore, the activated charcoal products were characterized according to the SNI 06-3730-1995 method. The analysis includes water content, volatile matter content, ash content, fixed carbon content, iodine adsorption and methylene blue adsorption conducted to determine the quality of activated charcoal. The surface area and porosity analysis conducted using the surface area analyzer (JWGB Meso 112, China). The ultimate analysis was carried out using Ultimate Analyzer (LECO) to determine the content of elements C, H, N, O and S from each sample of activated charcoal. For the morphological features, the analysis conducted with a scanning electron microscope (SEM) (ZEISS).

The industrial product (AK1 and AK7) then treated with steam addition with three various contact time (60, 90 and 120 min) at 800 ºC. The final product analyzed based on Indonesian National Standard (SNI) in order to know the product quality improvement.

RESULTS AND DISCUSSION

Activated charcoal was produced from coconut shells by following a two-step process. This process involves pyrolysis for converting biomass into charcoal and chemical/physical activation. Physical activation requires three reagents: steam, air and CO_2. Physical activation is highly more cost efficient and effective for generating activated porous charcoal than chemical activation. Compared with chemical activation, this method can prevent corrosive properties.

Proximate analysis of activated charcoal according to SNI-06-3730-1995: In this study, activated charcoal acquired from coconut shell was characterized to investigate its quality of the charcoal products manufactured. Some tested parameters included ash content, moisture content, volatile matter, iodine adsorption and fixed carbon were performed on the basis of the SNI 06-3730-1995 method (Table-2). The results were compared with related references and SNI.

The moisture content of seven activated charcoal samples was 4.88-8.21% (Fig. 1). The lowest and highest water content were obtained for AK2 and AK3, respectively; these results satisfy the standard requirements. For SNI moisture content, reference is < 15%. The ash content of activated charcoal was 1.26-2.19%. The lowest and highest ash contents were obtained for AK1 and AK5, respectively. These results met the SNI quality standards of activated charcoal (< 10%). For the samples,

TABLE-1
EXPERIMENTAL DATA FOR ACTIVATED CHARCOAL FROM INDUSTRIAL-SCALE PRODUCTION AT PT NUI HIA PRIMA

Samples	Heating time (h)	Steam time (h)	Holding time (h)	Remarks
AK1	4	1	4	–
AK2	7	2	4	AK2 is continuation of AK1
AK3	10	3	4	AK3 is continuation of AK2
AK4	14	4	4	AK4 is continuation of AK3
AK5	17	6	4	AK5 is continuation of AK4
AK6	21	8	4	AK6 is continuation of AK5
AK7	25	10	4	AK7 is continuation of A61

AK is activated charcoal

TABLE-2
QUALITY STANDARD OF ACTIVATED CARBON ACCORDING TO SNI 06-3730-1995

Parameters	Quality requirements
Moisture (%)	< 15
Ash content (%)	< 10
Volatile matter (%)	< 25
Fixed carbon (%)	> 65
Iodine adsorption (mg/g)	> 750
Methylene blue adsorption (mg/g)	> 120

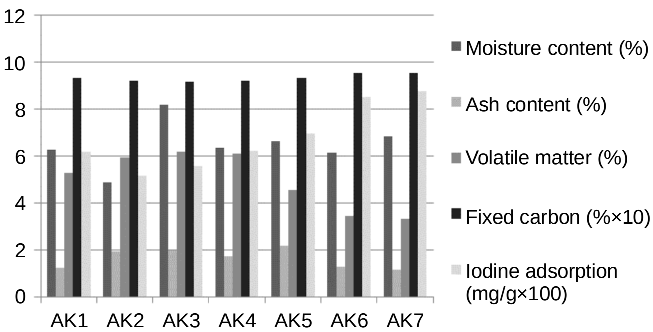

Fig. 1. Proximate analysis of activated charcoal from industrial scale production

the volatile matter content was 3.35-6.18%. The highest and lowest volatile matter contents were obtained from AK3 and AK7, respectively. The fixed carbon value was 91.85-95%. Its highest and lowest values were obtained from AK7 and AK3, respectively.

Activation and carbonization can render organic molecules unstable; thus, bonds between these molecules can be damaged. These processes convert volatile matter into liquid and gas products. Hence, the fixed carbon value increases [2,26]. The values of fixed carbon (> 65%) and volatile matter (< 25%) satisfy the SNI requirements. For the seven samples, the iodine adsorption value was 519-876 mg/g. Only AK7 and AK6 samples met the requirements of iodine adsorption set by SNI (> 750 mg/g). In coconut shell charcoal, activation process tend to opening pores *via* volatile matter removal to increase the adsorption capacity [27].

The moisture contents of the seven products are higher than those of previous studies (Fig. 1) [28]. Activated charcoal produced from coconut shell, with a surface area of 185 m^2/g and an iodine adsorption of 300-500 mg/g, does not meet SNI standards for ash and moisture contents. Compared with the activated charcoal products made from coconut shell available in the market (*e.g.* products produced by PT Inti Alam Kimia), our products require quality improvement, especially for iodine adsorption. The iodine adsorption of the products of PT Inti Alam Kimia is 1000-1200 mg/g. Thus, these products can be utilized for various applications, such as gold processing, water treatment, water/gas treatment, food and beverage industries. Moreover, AK1-AK7 (except AK3) sample meet the requirements of American Water Work Association (AWWA). According to the AWWA standard, the moisture of activated charcoal should not be >8%, and the iodine adsorption capacity should be 500 mg/g for water applications. Thus, the activated charcoal products of PT Nui Hia Prima were possibly used in the water treatment. To elevate the global competitiveness of the company products, further treatments are required to meet community/market demands and use activated charcoal products in various fields.

From Table-3, the proximate data of coconut shell and activated charcoal were obtained from carbonization at 650 ºC with chemical activator KOH. From these data, the results of this study are sufficient having better characterics with the lower volatile matter and higher content of fixed carbon. The moisture content, volatile matter and ash content decrease after carbonization and activation processes due to the breakdown of the biomass chemical bonds and impurities removal in the carbon pores therefore, carbon purity increases.

Ultimate analysis of activated charcoal: In this study, activated charcoal acquired from coconut shell was analyzed to determine nitrogen, carbon, hydrogen, sulphur and oxygen contents (Table-4). Its carbon contents increased from 85.17 to 88.39% with the increase of steam time. The oxygen and hydrogen content decreased from 11.26 to 9.36% and 1.92 to 1.36%, respectively. The increase in the carbon contents may

TABLE-3
PROXIMATE ANALYSIS OF COCONUT SHELL BASED ON [Ref. 18]

Samples	Moisture (%)	Volatile matter (%)	Ash content (%)	Fixed carbon (%)
Coconut shell	11.34	58.28	6.02	24.36
Coconut shell activated charcoal	2.96	14.29	2.48	80.27

TABLE-4
ULTIMATE ANALYSIS OF ACTIVATED CHARCOAL PRODUCED IN INDUSTRIAL SCALE

Samples	Sulphur	Carbon	Nitrogen	Hydrogen	Oxygen	Information
AK1	0.034	85.17	0.11	1.92	11.26	
AK2	0.028	85.61	0.22	2.06	10.23	
AK3	0.030	83.60	0.16	2.17	12.88	
AK4	0.038	85.10	0.17	1.85	11.45	
AK5	0.036	85.68	0.10	1.65	11.04	
AK6	0.038	88.22	0.24	1.45	9.25	
AK7	0.036	88.39	0.21	1.36	9.36	
Coconut shell activated charcoal	0.300	75.63	1.30	4.23	18.54	[30]
Coconut shell activated charcoal	–	84.00	1.70	0.03	14.27	[35,36]
Fox nutshell activated charcoal	0.150	64.85	1.24	4.52	29.24	[37]

result from the volatile matter removal during activation and open carbon pores.

Activation and carbonization lead to the increase and decrease in the carbon and oxygen amounts, respectively. For the adsorption capacity of activated charcoal, O/C ratios are the main parameters responsible and indicate surface oxidation [29]. In this study, the carbon and oxygen contents of activated charcoal are higher and lower, respectively, than those obtained in the previous research [30]. Thus, present activated charcoal can be employed as the material in the chemical industries such as adsorbents, energy and membrane filters.

Morphological properties: Morphological analysis of activated charcoal conducted using SEM (Fig. 2). It can be seen that the surface morphology of the raw material tends not to have many pores, with rough and irregular shape. After the carbonization and activation process, pore formation begins to occur due to the evaporation of volatile matter components, degradation of raw material components (such as cellulose), and decrease of hydrocarbon compound.

This process causes enlargement of pore and surface area so that the adsorption capacity increase. Moreover, it is observed that there are impurities which may close the pores and thus not adsorbing compounds such as iodine and nitrogen gas. This condition affects the adsorption and surface area (BET method) value.

Analysis of the surface area-specific and pore structure of coconut shell activated charcoal: The activated charcoal products of Nui Hia Prima were characterized using a surface area analyzer to determine their pore volume, surface area and pore diameter. The surface area and pores of materials were tested using the BET method through N_2 adsorption-desorption. The pore and surface area of the samples were compared (Fig. 3a and 3b).

Steam must be provided during the production of activate charcoal. In activated charcoal production, steam addition affects N_2 adsorption because it contributes to charcoal pore opening and expansion. Table-5 presents the BET analysis results of the raw materials and activated charcoal acquired using coconut shell in previous studies.

As a raw material, coconut shells show the surface area of 75 m^2/g. The surface area and pore diameter of the activated charcoal were 300-500 m^2/g and 2-2.5 nm, respectively. The treatment time and activation process considerably affect the surface area increase. These findings are similar to the results

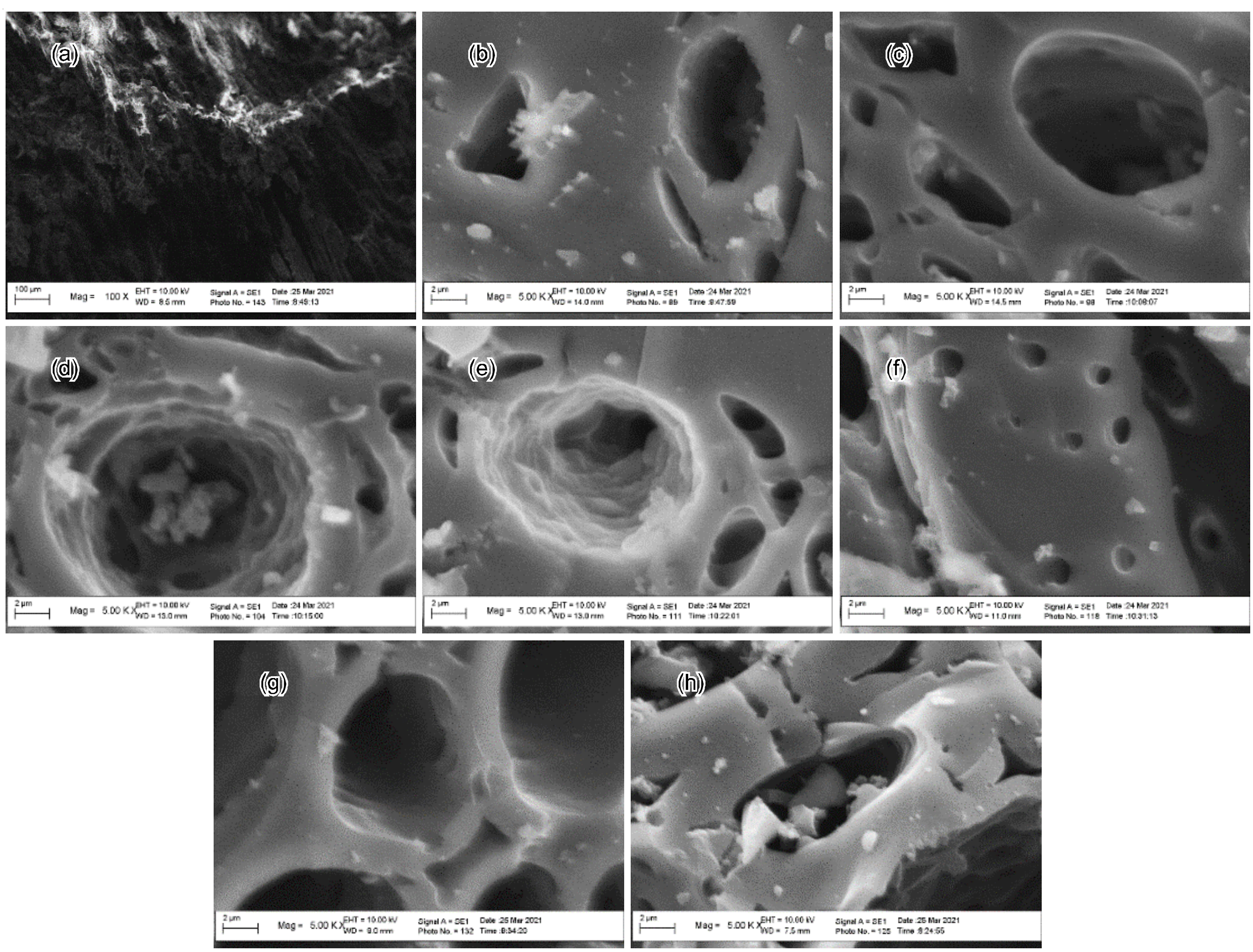

Fig. 2. Morphological analysis of activated charcoal produced in industrial scale; (a) coconut shell, (b) AK1, (c) AK2, (d) AK3, (e) AK4, (f) AK5, (g) AK6, (h) AK7

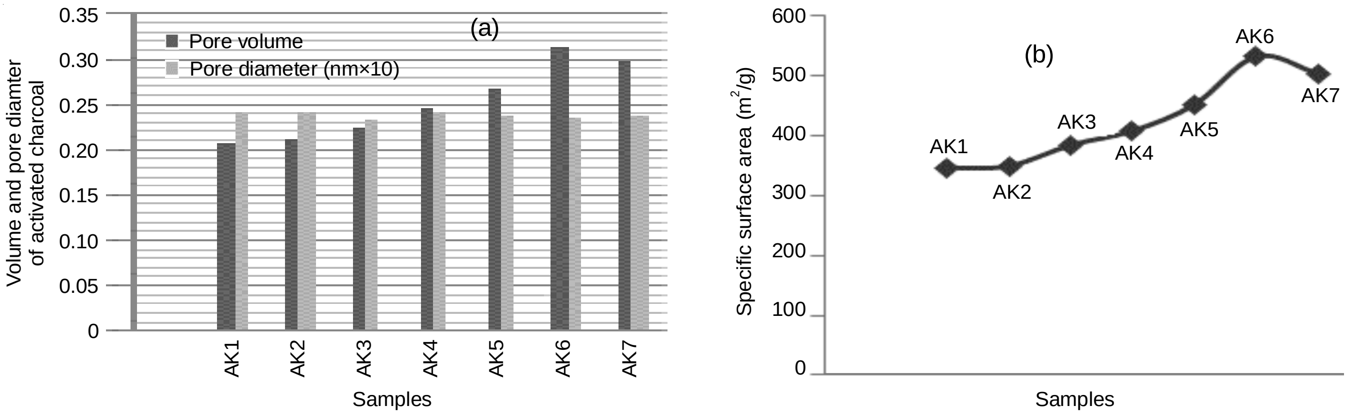

Fig. 3. Porosity analysis of PT Nui Hia Prima's coconut shell activated charcoal (a) pore volume and diameter (b) specific surface area using surface area analyzer

TABLE-5
SPECIFIC SURFACE AREA AND PORE PROPERTIES OF COCONUT SHELL ACTIVATED

Samples	SBET (m^2/g)	Pore volume (cm^3/g)	Pore diameter (nm)	Activation	Ref.
Coconut shell	75.71	0.0260	2.230	–	[15]
Activated charcoal	1203.613	0.6710	2.229	KOH	[15]
AC800	365	0.1480	–	Physical	[32]
AC-S-0-CO_2	2342	1.1630	–	Physical	[38]

of Freitas *et al*. [31] and higher than those of Purnomo *et al*. [32]. Freitas *et al*. [31] reported the surface area for the activated charcoal prepared using coconut shells of 560 m^2/g at the activation temperature of 800 ºC under carbonization of 550 ºC by using CO_2. The surface area of our activated charcoal is smaller than previous studies, which is > 1000 m^2/g [15]. Therefore, further improvement is required to enhance activated charcoal quality.

The application of activated charcoal is influenced by its surface area. The larger is the surface area, the better is adsorption capacity of activated charcoal. Activated charcoal should exhibit large surface areas to attain a high adsorption capacity. The adsorption power of activated charcoal is increased through carbonization and activation. During activation, from the carbon surface, gases, hydrogen, and water are eliminated. Hence, the surface undergoes physical changes. Activation occurs because of interactions among atoms such as oxygen, nitrogen and free radicals present on the carbon surface. Moreover, new pores were formed during activation because of carbon atom erosion caused by heating or oxidation. With the increase in activation time with steam, the pore volume of the activated charcoal increased to 0.313 cm^3/g. The pore size of activated charcoal was 2-2.5 nm, which is similar to that pore reported value [33], which found narrow micropores and mesopores.

Fig. 4 illustrates the curve of adsorption-desorption isotherm for the activated charcoal samples of coconut shells with different treatments. The curves of nitrogen adsorption-desorption isotherm are similar to the type-1 isotherm. The N_2 adsorption sharply increased at a low relative pressure (p/p_0 = 0-0.1) and then became stable at a high relative pressure (p/p_0

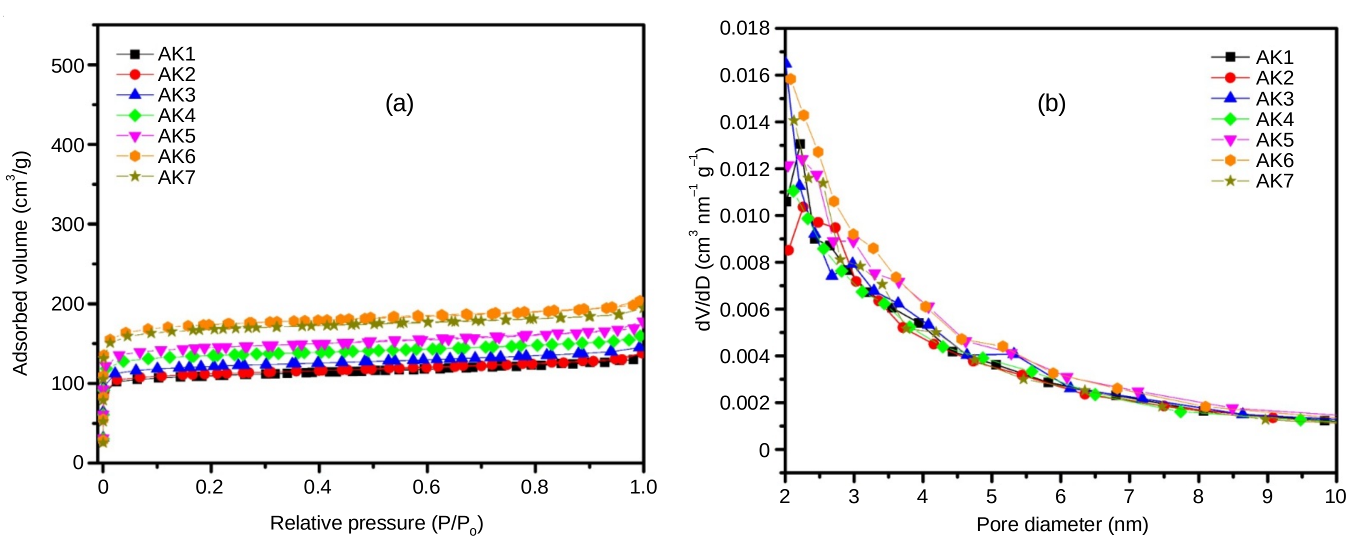

Fig. 4. (a) Adsorption-desorption isotherm of activated charcoal, (b) pore size distribution of activated charcoal

= 0.1-0.99). In the adsorption-desorption pattern, this curve showed no hysteretic loop in. This result revealed that the activated charcoal formed the micropore structure. For the seven samples, the adsorption isotherm gradually shifted upward with the increase in the vapour time after activation, which indicated the total pore volume increase.

These results agreed with the findings of Sun *et al*. [35] and Fang *et al*. [34], who have reported micropores for activated charcoal created from coconut shells by using the BET analysis. Near the axis, the curve is concave (Fig. 4a). This curve is obtained from small external areas (marked with the flat nearly horizontal curve) and small porous (micropore) adsorbents and indicates monolayer adsorption.

In this study, the samples AK1 and AK7 of activated charcoal were improved at the activation temperature of 800 ºC with the steam time increase to 60, 90 and 120 min (Fig. 5). After treatments, the product quality enhanced. Moreover, for AK7 and AK1 samples, the products of activated charcoal exhibited the iodine adsorption capacity of 723-973.9 mg/g with the steam time increase. With the increase in the steam time, the quality of the product increased and nearly satisfied the SNI requirements. Longer time allows an activator to come in contact with charcoal for satisfactory reaction results and react to a higher extent. The AK1 and AK7 samples subjected to steam had better quality than the samples without steam treatment (Fig. 1). The adsorption value of methylene blue on activated charcoal was 70-124 mg/g. This value increased with the increase in the treatment time. Steam is an activator which affects methylene blue adsorption, opens charcoal pores and can adsorb pollutant dyes. During activation and carbonization, the methods, activators, operating conditions and technology used lead to different qualities of activated charcoal. A few activators, including KOH, H_3PO_4 and steam, were used for the production of biomass activated charcoal.

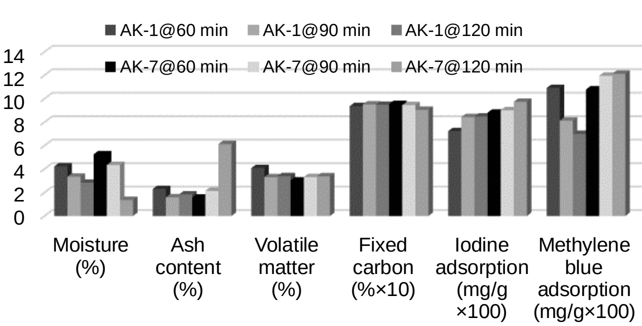

Fig. 5. Proximate analysis of activated charcoal after additional steam treatment with various contact time

Conclusion

The activated charcoal acquired from coconut shells could be produced on the industrial scale by employing steam activators meet the AWWA standards and Indonesian National Standard for technical activated charcoal and water purification, respectively and provide satisfactory results. The fixed carbon content and iodine adsorption of our samples increased to 95% and from 500 to 973.9 mg/g, respectively. The ash, moisture and volatile matter contents decreased. Additional modifications are required, specifically for instrument reactor enhancement or steam technology, to increase the surface area and adsorption capacity of activated charcoal. Studied activated charcoal can compete with the products of other activated charcoal and fulfill the market standards. Furthermore, it can be used in various fields as per the requirement. Although physical activators, including steam time increase, are highly economical for long-term production on the industrial scale, equipment operation stability and temperature control are required to maintain the product quality. This work can be continued in the future to obtain highly beneficial methods for economic growth of the community and company.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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