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Chemical-Thermal Activation to Improve the Characterization of Sludge-Based Activated Carbon (SBAC) from Palm Oil Mill Sludge (POMS)

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ABSTRACT

The development of time and technology triggers significant challenges in preserving the environment. Large-scale industry that uses a large quantity of energy and clean water is the main reason environmental management due to industrial activities needs to be considered properly. In addition to liquid wastewater, waste in the form of sludge used in wastewater management is also a new problem that requires special attention. Sludge-based activated carbon (SBAC) is one of the options for utilizing WWTP sludge in low-cost adsorbent materials. The development of SBAC is a promising solution to solving two-way environmental problems. The most used activation method combines thermal and chemical compound impregnation. This paper aims to prove that Palm Oil Mill Sludge (POMS) can be applied as an adsorbent after chemical and thermal activation. KOH 4M was used with a ratio of 1:1 (weight/volume) with pyrolysis heating at 700ºC injected with nitrogen gas (N_2). Fourier Transform Infrared Spectroscopy (FTIR) test shows SBAC has hydroxyl and carboxyl functional groups, Pore Size Analyzer (PSA) classified SBAC as microporous with 1.7 nm size of SBAC, iodine number is about 821 mg/g, 23.63% of ash content, 62.96% of fixed carbon, 11.5% of water content, and 15.37% of volatile content.

Keywords**:** activated carbon, POMS, SBAC, sludge.

1. INTRODUCTION

The increased industrial development affects all sectors of life, including the environment. Various attempts are made to overcome environmental problems such as in the wastewater management industry [1]. Activated sludge has been a widely used technology since the early 20th century and has been improved continuously. The advantage of activated sludge is its ability to remove good pollutant loads with low operational and maintenance costs compared to similar technologies [2]. However, activated sludge produced sludge waste as much as 1-3% of the total activated sludge content. The presence of this sludge waste is considered more hazardous as it accumulates various contaminants that require special attention and special attention and effective [3,4]. One of the applications of sludge from biological treatment is to become sludge-based activated carbon. Sludge-based Activated Carbon (SBAC) holds promise as a sustainable water and wastewater treatment technology because it is carbon-rich, economical, easily accessible, and abundantly available [5].

Activated carbon derived from oil-containing sludge has a higher adsorption capacity than activated carbon from other sludge [6]. Adsorbent products from WWTP sludge can adsorb Mg^{2+} ions by 45% and Ca^{2+} by 35% with SBAC without chemical and thermal activation [7]. Color pollutants, oil & grease, and other organic compounds can be adsorbed by SBAC in the adsorption process [8].

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Many studies use SBAC to remove pollutants such as metals and heavy metals in wastewater. Heavy metals are minerals that are difficult to degrade or disappear completely from the environment and are uneasily destroyed which usually can only be transferred from one form to another [9]. It is according to the ability of pore and chemical interaction between SBAC surface with metal. SBAC has microporous pores and is highly recommended for adsorbing metals in water. Waters contaminated with heavy metals can accumulate in the bodies of animals, aquatic plants, and humans. Over time, heavy metals such as lead (Pb), iron (Fe), cadmium (Cd), and copper (Cu) can move from the liquid phase to the solid phase through the formation of deposits beneath the surface of water bodies that can last longer and become increasingly difficult to remove from the environment [9,10]. It is important to consider the presence of tertiary pollutants that have a major impact on piping systems and equipment, i.e. heavy metals, as they can cause scaling and corrosiveness.

Water sustainability is a complex issue that involves many sectors and challenges. This is due to the need to enlist the private sector, government, and communities to achieve sustainable clean water in industrialized areas. The Sustainable Development Goals (SDGs) continue to urge countries to start recycling wastewater and ensure that almost half of the wastewater is not released into the environment [11]. To achieve the SDGs target, the usage of SBAC became an appropriate activated carbon for solving environmental problems. Over the past 10 years, much research has been conducted on the utilization of sludge generated by wastewater treatment plants (WWTPs) from various industries and households to become valuable and powerful adsorbents for removing pollutants in water, such as color, COD, heavy metals, oil, and grease.

2. RESEARCH METHOD

The research was conducted at the Water Quality Laboratory, Environmental Engineering, Bandung Institute of Technology for 6 (six) months. Wastewater Treatment Plant (WWTP) sludge of PT X from Palm Oil Mill Industry in Kuantan Singingi Regency, Riau.

2.1. Material Preparation

The sludge was dried naturally in the sun for 2 x 24 hours. The sludge was then packed in airtight clear plastic bags and stored at room temperature. A 4 M KOH solution (weight/ volume ratio) was used as a chemical activation agent for the SBAC and a 3 M HCl solution (weight/volume ratio) was used as a washing agent for the SBAC to achieve neutral pH [8].

Laboratory equipment such as an oven was used in the drying process of POMS. Pyrolysis equipment with an operational temperature of 700^oC was supplied with nitrogen gas at a rate of 1.2 L/min for thermal activation of SBAC [12]. Fourier Transform Infrared Spectroscopy (FTIR) testing using a Shimadzu Prestige-21 type tool with an observation wavelength of 400-4000 cm^{-1}. Scanning Electron Microscope (SEM) testing using the SU3500 tool with 100x and 15,000x magnification was used to detect the surface characteristics of SBAC. BET surface area and Pore Size Analyzer (PSA) tests used a Quantachrome NovaWin 11.03 type tool with a test sample of 0.6526 grams. The test was conducted at a room temperature of 273.0 K. The testing of iodine number, ash content, moisture content, volatile matter content, and carbon content still refers to SNI 06-3730-1995.

2.2. Palm Oil Mill Sludge (POMS)

The sludge comes from the 7th pond or sedimentation pond. POMS contains protein content (11.35%), crude fiber or fiber (25.80%), cellulose (16.15%), and lignin (19.19%) [13]. The cellulose and lignin content indicates that Palm Oil Mill Sludge (POMS) can be produced into SBAC. Cellulose consists of carbon chains composed of -OH functional groups. Table 1 shows the characteristics of POMS from PT. X of Kuantan Singingi Regency. The high

C-organic content contained in POMS indicates that the material has the potential to be converted into activated carbon through an appropriate activation process.

Table 1. Characteristics of Sludge of PT. X of Kuantan Singingi Regency

Parameter	Value (Experimental data)	Value[a] (Theoretical data)
C-Organic (%)	63.32	37.5[a]
NTK	2.24	6.35 [a]
Moisture content (%)	27.95	68.46 [a]
pH	8.12	7.4 [a]
Ash content (%)	1.96	-
Volatile content (%)	67.45	52.67 [b]
Fixed carbon (%)	2.65	1.43[b]
Water content (%)	49.52	-
Heating value (kal/gr)	0.41 ± 0.01	

Source: [a]Ref. [14], [b]Ref. [15]

3. RESULT AND DISCUSSION

3.1. Activation of Sludge

3.1.1. Chemical Activation

KOH has a significant ability to improve the surface area of adsorbents from POMS. A large surface area of activated carbon is important for absorbing substances from its environment. Activated carbon has a complex and porous pore structure. A large surface area increases the absorption and adsorption capacity of the activated carbon to various chemical compounds, gases, and other substances. The activation process of SBAC using KOH (Figure 1) requires 16 hours soaking time with a ratio of 1:1 (weight/volume ratio) [8].

Because KOH is one of the strongest bases in the range of base-forming chemical compounds, SBAC requires a washing process using a strong acid, HCl 3 M, and additional distilled water until reaching the normal pH to prevent out-of-control reactions between KOH contained in SBAC. Before thermal activation, the sludge is dried again using a 105°C oven for six hours.

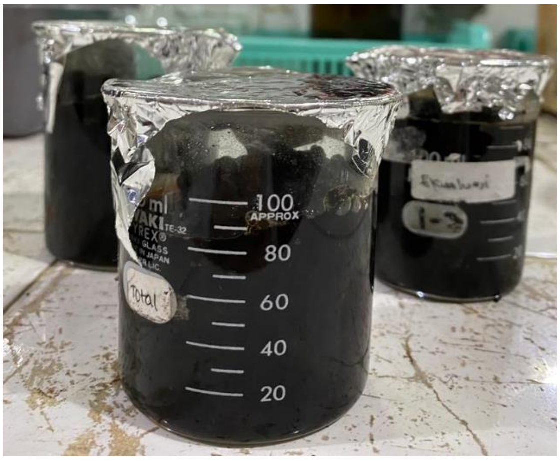

Figure 1. Chemical activation using 4M KOH for 16 hours.

3.1.2 Thermal Activation

In this study, the heating temperature used was 700°C for 1 hour [4]. Three stages occur during the pyrolysis process, (i) the dehydration stage, which releases water molecules bound in organic matter, (ii) many volatile components are dissolved in the pyrolytic zone, and (iii) the pyrolysis process continues and occurs slowly. Temperature is crucial during the formation and removal of oxygen functional groups on the adsorbent [12]. The adsorbent material in the form of POMS is put into a porcelain cup before being put into the pyrolysis heating chamber. Various devices can be used for pyrolysis, such as a muffle furnace, tube furnace, or a pyrolysis device assembly as shown in Figure 2. During the combustion process, the POMS samples were heated at 700°C for 1 hour and the injection of nitrogen gas (N_2) with a flow rate of 1.2 L/min directly to create conditions without oxygen (inert conditions).

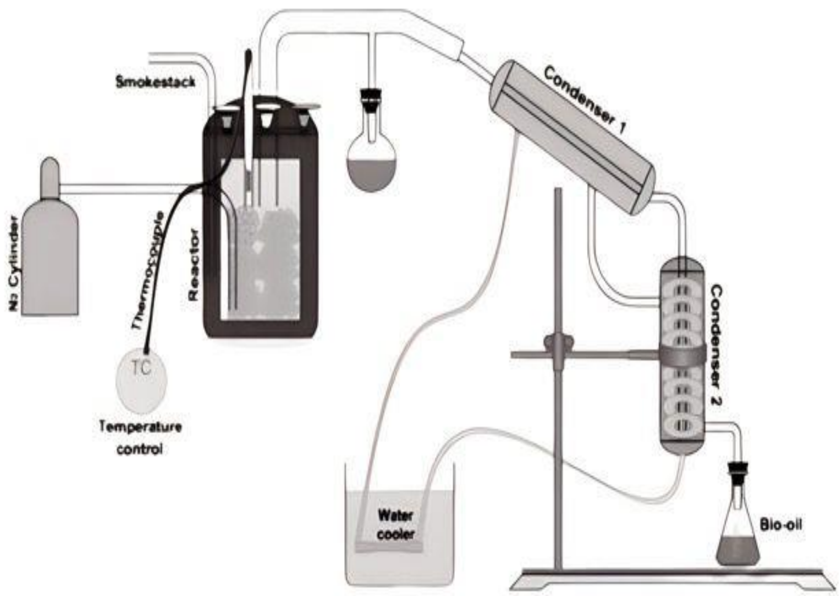

Figure 2. Pyrolysis Equipment with Nitrogen Gas Injection at 1.2 L/minute [16].

3.2. Characterization of Sludge-Based Activated Carbon

SBAC characterization determines the optimum initial conditions of activated carbon before adsorption (Figure 3). The characterization of FTIR test, BET test, Pore Size Analyzer (PSA), and SEM-EDS test are expected to identify the performance of activated carbon in adsorbing metals.

3.2.1. Fourier Transform Infrared Spectroscopy (FTIR) Test

The potential use of an activated carbon obtained from natural materials is seen from the presence of oxygenated functional groups such as hydroxyl and carboxyl on the surface of the adsorbent [17]. Compared to commercial activated carbon, SBAC has better removal performance because its adsorbent material comes from POMS that contain carbon composed of cellulose molecules and lignin, thus making it adsorb heavy metals better. Metal ion adsorption using SBAC has been widely tested in recent years, and it shows that POMS activated with chemical compounds has greater sorption ability than physical activation. The main mechanism between SBAC and metals could be detected by the presence of functional groups that can be known through FTIR testing. FTIR classifies the chemical bonds formed to determine the suitability of an adsorbent in the sorption of certain pollutants. Hydroxyl bonds (O-H) that appear in the FTIR testing peaks indicate the ability of an adsorbent to adsorb metal ions, in this case, heavy metals. The hydroxyl group plays a role in forming acidic sites on the surface of activated carbon, which can increase the ability to adsorb heavy metal ions. Other bonds, such as carboxyl functional groups (-COOH) can provide the ability to bind to heavy metals through ionic bonds or covalent bonds. The carboxylic group will experience deprotonation due to the presence of hydroxyl groups so the carboxylic group will become the negatively charged -COO- which is indicated to be very reactive in binding to metal ions [18]. Figure 4 shows the results of FTIR testing of activated SBAC with observations of 400-4000 cm^{-1}.

SBAC has an O-H functional group characterized by an elongated and widened stretch from the range of 2800-3700 with a peak at 3448.72 cm^{-1}. O-H stretch indicates that SBAC can trap metal ions. The hydroxyl group also plays a role in forming the acidic sites on the surface of activated carbon, increasing the ability to adsorb metal ions. The O-H (hydroxyl) group found in the FTIR spectrum proves that SBAC derived from POMS does contain lignin and cellulose. Cellulose and lignin are composed of long threaded chains with many -OH groups that will be able to react with positively charged metal ions [19].

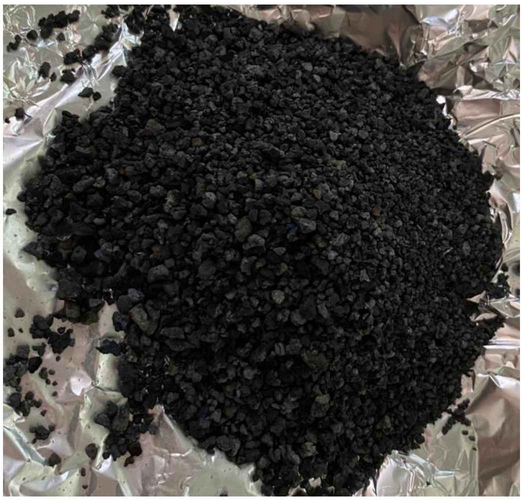

Figure 3. Sludge Based Activated Carbon (SBAC) from Palm Oil Mill Sludge (POMS).

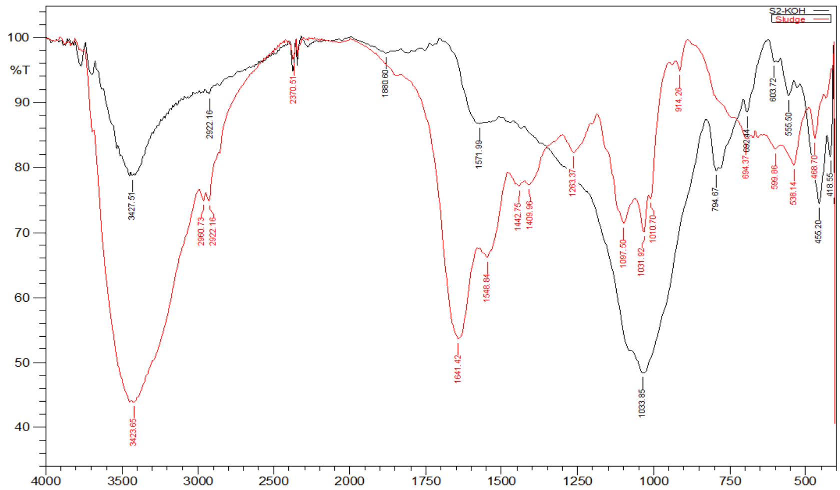

Figure 4. FTIR Testing Results of Raw Sludge and Sludge-Based Activated Carbon.

An adsorption peak was also found at 1085.92 cm^{-1}, which indicates that SBAC has a C-O group, meaning that the material is acidic and is indicated to be able to absorb positively charged ions [20]. The carboxylic acid in SBAC shows that the material has a -COOH functional group which can provide a negative charge to the molecule. This carboxylate ion ($-COO^{-}$) can interact with positive cation ions. The fingerprint area is a range of wavelength between 1500-500 cm^{-1} It represents the characteristic of active carbon. The presence of hydroxyl and carboxyl groups indicates that the dissociated SBAC has a negatively charged surface, which will form electrostatic bonds with metals and heavy metals that have positive ions so that an indicated adsorption event can occur.

The FTIR testing for POMS is also shown in Figure 4. It can be seen that the O-H strain formed in POMS is similar to the functional groups in SBAC. The peak of the O-H group is formed at 4423.66 cm^{-1}. In the range 1250-750 cm^{-1}, no –COOH functional group was found, which in the SBAC FTIR results was found with a peak of 1085 cm^{-1}. Apart from the presence of the O–H group, the –COOH group is also a characteristic that indicates the possibility that metal can be adsorbed on the adsorbent.

The presence of the –COOH cluster indicates that activation carried out on POMS has a significant role in exposing this cluster to SBAC.

3.2.2. Scanning Electron Microscopy-Energy-dispersive X-ray spectroscopy (SEM-EDS) Test

Scanning Electron Microscopy (SEM) was carried out to see the surface structure and predict the pore size of SBAC-KOH. Figure 5 shows the results of SEM tests carried out at two different magnifications. Figure 5a is SBAC with 100x magnification, showing that SBAC has a rough surface structure with several visible open pores due to the execution of chemical activation and pyrolysis processes. The asymmetrical and non-uniform pore diameter of SBAC is theorized to come from the chemical-physical modification of natural materials. This shape can provide sufficient diffusion space for contaminants adsorption in the SBAC during the adsorption process.

Before SBAC undergoes the adsorption process, the pores of this sludge-based activated carbon have pores as mesoporous because they are 2-50 nm [5]. Based on Figure 5b, it can be seen that SBAC has 0.5-1 µm sized pores, and is aligned with the previous research that has proven mesoporous. The EDS test results are shown in Table 2, where it can be seen that the elements Mg and Ca were detected at 0.62% and 0.71%. The other elements such as O, C, Na, and Fe were found to be 31.43%, 62.92%, 0.05%, and 0.44%, respectively. The C (%) value detected on the surface of activated carbon indicates that POMS was successfully activated using chemical activation and thermal pyrolysis. In addition, the potential absorption of magnesium and calcium by SBAC is evidenced by the small presentation of other impurities detected on the surface of the activated carbon.

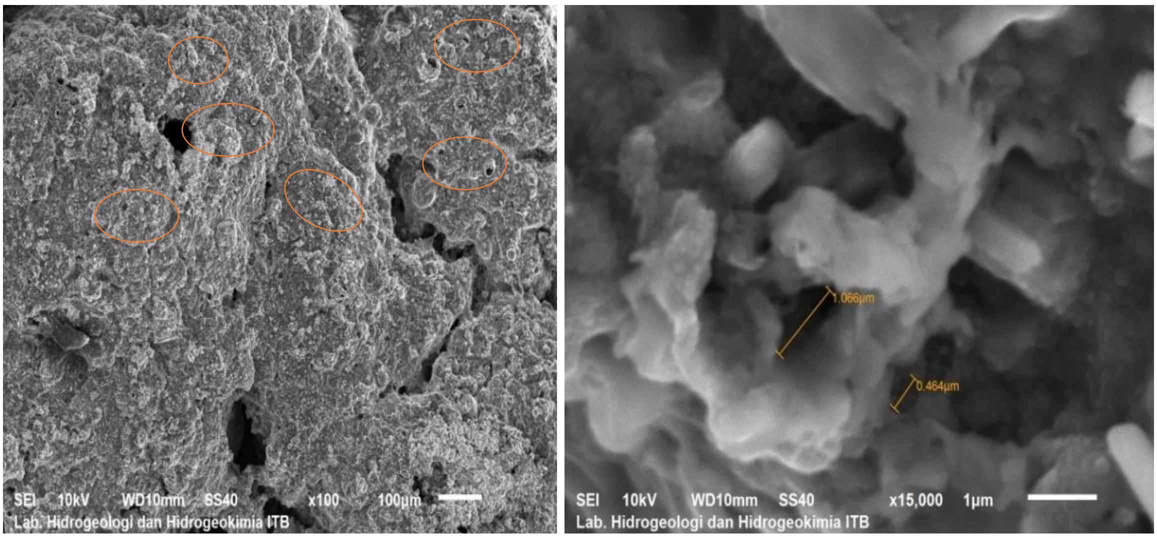

Figure 5. (a. left) SEM SBAC-KOH test results with 100x magnification; (b. right) SEM SBAC-KOH test results with 15,000x magnification

Table 2. SEM-EDS test results of SBAC.

Element	Mass (%)	Atom (%)
Oxygen (O)	35.07	31.43
Carbon (C)	52.70	62.92
Magnesium (Mg)	1.04	0.62
Calsium (Ca)	1.98	0.71
Natrium (Na)	0.08	0.05
Iron (Fe)	1.71	0.44
Silica (Si)	5.20	2.65
Aluminium (Al)	2.21	1.18
Total	**100**	**100**

3.2.3. Technical Activated Carbon Standardization Test

Sludge-based activated carbon qualification test refers to SNI 06-3730-1995, which regulates the standardization of technical activated charcoal. According to SNI, there are several qualification parameters such as iodine number (mg/g), water content (%), ash content (%), volatile content (%), and fixed carbon (%). Regarding the SNI No. 06-3730-1995 requirement for the technical activated charcoal in Indonesia, SBAC from POMS requires several tests such as iodine absorption (I_2), water content (%), ash content (%), volatile content (%), and fixed carbon (%). These are the reference standards for the quality of activated carbon created from alternate materials to match commercial activated carbon. Table 3 shows the characterization of the SBAC.

About 1 gram of sample was tested for water, ash, and volatile content. According to SNI 06-3730-1995, a 1-gram sample was first used to test the water content using an oven at 105°C for two hours and then weighed using an analytical scale. The same sample for the water content test was used for the ash content test using a furnace under 600°C for one hour.

The sample was then weighed as well as the water content test. For volatile content, use the same sample after the ash test and use the furnace under 950°C for 7 minutes. Fixed carbon is obtained from the percentage between the weight of the sample, the ash content, water content, and volatile content.

Table 3. SBAC Characterization Based on Technical Activated Carbon Standardization.

No	Parameter	Value
1	Iodine number (mg/g)	821
2	Water content (%)	11.5
3	Ash content (%)	23.63
4	Volatile content (%)	15.37
5	Fixed carbon (%)	62.96

The SBAC test for iodine (I_2) represents the ability of activated carbon to adsorb chemical compounds, in this case, iodine. The higher the iodine number of an activated carbon, the greater the active carbon's ability to absorb other pollutants. According to existing standards, the threshold value of activated carbon for large usage is >750 mg/g. Meanwhile, water content (%) has a maximum value of 15%, where the water content of SBAC (11.5%) is below the active carbon quality threshold. Activated carbon with high water content needs to be avoided because it can fill the pores of the activated carbon and decrease the adsorption capacity. This is also a reason why the ash content (%) in activated carbon cannot exceed 10%, This test showed that the ash content of SBAC was 23.63%. Ash content values exceeding existing standards can be influenced by the raw materials of SBAC that originated from POMS which is rich in Mn and Zn [14]. An excessive ash content can insult the pore and ash will clog the pores and inhibit the adsorbate in attaching the adsorbent surface. Meanwhile, the percentage of volatile substances in SBAC is 15.37% and below the maximum standard value of 25% as described in the SNI. Through these tests, the content of fixed-carbon contained in SBAC is determined to be 62.96%, and slightly below the 65% minimum standard. This can serve as a reference for the ability of SBAC to absorb pollutants.

3.2.4. BET Surface Area and Pore Size Analyzer Test

Brunauer, Emmet, and Teller (BET) Surface area method aims to determine the effective surface area of SBAC activated carbon. According to John et al. [21], the relationship between adsorption capacity and adsorbent surface area is linear. The high surface area will allow the adsorption capacity of the adsorbent to increase as well. BET surface area is characterized by using a test device of the *Quantachrome NovaWin* 11.03 type with a test sample of 0.6526 grams. The test was conducted at room temperature 273.0 K, so the surface area measurement results were 320.69 m^2/g.

The pore size of activated carbon plays an important role in the adsorption process of adsorbate. SBAC from POMS has an average diameter pore size of 17.07 Å or 1.7 nm. Pore Size Analyzer (PSA) testing used a dispersed sample in an ethanol solution. This preparation was done to help separate the SBAC particles so that the pore distribution could be read clearly. The test results show that SBAC is classified as an adsorbent with a microporous pore size. Based on research conducted by Liu et al. [5], SBAC is classified as mesoporous (2-50 nm) because it has an average pore diameter of 3.68 nm with a surface area of 289.58 m^2/g.

The difference in surface area and pore size with SBAC in this study can occur because the origin of the sludge source used is different. In addition, SBAC has oil content and has the potential to form micro-sized pores [22]. In this study, the size of SBAC products was 1.69 nm with a pore volume of 1.98 cm^3/g. This is affected by the activation method and the structure of a sludge that contains cellulose and lignin.

4. CONCLUSION

Future developments of this study are the optimization of SBAC as an adsorbent for metal ions, not only heavy metals. The application of SBAC from wastewater treatment plant sludge has an opportunity to produce low-cost adsorbents while minimizing environmental pollution. The FTIR test shows that the functional groups that form SBAC consist of hydroxyl (O-H) and carboxyl (-COOH), which indicates that SBAC from POMS) can adsorb metal ions and heavy metals, and also color, COD, and other pollutants. SBAC is included in the microporous category because it has a pore range of 0.5-1 micron based on SEM testing. BET surface area and PSA show that SBAC has 320.69 m^2/g and is classified as microporous with a pore size of 1.7 nm. The values of iodine value, water content, ash content, volatile content, and fixed carbon of SBAC are 821 mg/g, 11.5%, 23.63%, 15.37%, and 62.96%, respectively.

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