



7th EURASIA WASTE MANAGEMENT SYMPOSIUM

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Preparation and Characterization of Activated Carbon Derived From Municipal Wastewater Treatment Plant Sewage Sludge via Carbonization and Activation using Superheated Steam

*Lee Yang¹, Nordin Sabli, Halimatun Sakdiah Zainuddin, Yoshida Hiroyuki,
Shamsul Izhar^{1,2}*

Abstract

Sewage sludge was converted into activated carbon (AC) by reacting the sewage sludge with superheated steam via carbonization and activation processes in a rotary kiln. The produced AC were analyzed for their physical properties and adsorption capacity, and the effect of carbonization and activation temperature were studied. The sewage sludge was carbonized at 400°C and activated between 500°C to 900°C for 1h. The AC was characterized using proximate analysis, BET, FTIR, SEM, and XRD. Its performance as an adsorbent was conducted using methylene blue. Activation at 800°C gave the most positive characterization results. Its proximate analysis resulted in moisture, volatile matters, ashes, and fixed carbon of 0.89 wt%, 6.9 wt%, 80.5 wt% and 11.7 wt%, respectively. The BET surface area, micropore vol., total pore volume, and pore size were 50 m²g⁻¹, 0.0093 cm³g⁻¹, 0.45 cm³g⁻¹, and 17.95 nm, respectively. The FTIR analysis showed that AC had many O-containing functional groups. All analyses proved that the material produced was analogous to activated carbon derived from coconut husk. The methylene blue adsorption test showed AC activated at 800°C has the highest percentage methylene blue removal rate (97%) compared to those activated at other temperatures. The superior removal is due to superheated steam at 800°C stimulating the AC pore development, which excluded oxygen from the system to prevent unnecessary burnt offs. In short, carbonization and activation processes at 800°C using superheated steam produced enhanced physicochemical properties of AC and are very promising as a new adsorbent in attempting to reduce municipal sewage sludge.

Keywords: Wastewater Treatment Sewage Sludge, superheated steam carbonization and activation, activated carbon

¹ Dept of Chemical and Environmental Engineering, Faculty of Engineering, Universiti Putra Malaysia, UPM Serdang 43400, Selangor, Malaysia

² Corresponding author: shamizhar@upm.edu.my

1. INTRODUCTION

With increasing urbanization and industrial activities, the production of sewage sludge has significantly escalated. A typical wastewater treatment plant generates around 100,000 tons of sludge annually [1]. The projected yearly production from approximately 3 million wastewater treatment units was expected to reach 10 million tons by 2020, an increase from 6 million tons in 2015 [2]. The cost of managing this sludge is substantial for effective management [3]. Much of the generated sludge is still disposed of in landfills, posing environmental challenges and highlighting the need for sustainable management solutions. Given its complex composition, converting sewage sludge into value-added products is challenging, often resulting in inconsistent outputs [4].

Traditionally, landfilling has been the most common disposal method for sewage sludge. This has been supplemented by drying and incineration to reduce sludge volume [5]. However, these methods still require post-treatment disposal, and incineration produces harmful byproducts, such as ash and soot, that can degrade environmental quality. In this study, sewage sludge is converted into activated carbon (AC) using superheated steam during the pyrolysis process, eliminating oxygen from the system and preventing combustion, thus making the process environmentally friendly [6]. Unlike traditional methods, this process does not produce soot since there is no oxygen present to facilitate combustion [7]. Moreover, the residual ash will remain within the reactor due to its mass, ensuring a cleaner process.

AC is commonly known as activated charcoal, are highly porous, carbonaceous materials with an oxidized surface. Their quality is determined by their surface area, porosity, and functional groups, which make AC highly effective adsorbents. Globally, the demand for ACs continues to grow due to their wide range of applications, including water and air purification [8]. The production of AC requires high-temperature treatment, which significantly alters these parameters, thus requiring recalculation after processing.

Commercially, ACs are often produced from high-cost raw materials like wood and coal. The use of sewage sludge as a precursor for AC production offers a more sustainable and cost-effective solution, recycling the carbon content from waste [9]. The surface area of commercial ACs typically ranges between 950 and 2000 m²/g, and the quality of AC is often determined by its porosity. ACs have three types of pores: micropores (<2 nm), mesopores (2-50 nm), and macropores (>50 nm). For many applications, such as gas-phase adsorption, micropores are sufficient, but for the removal of larger molecules, mesopores and macropores are necessary. This study aims to produce AC with an optimized pore size distribution suitable for both small and large molecule adsorption, a critical factor for improving the utility of sludge-derived ACs.

To produce AC with a larger pore size, sewage sludge undergoes two key processes: carbonization and activation. Carbonization increases the fixed carbon content and creates new pores at a temperature of around 400°C. Activation, performed at temperatures up to 900°C, develops additional pores and enhances the porosity of the material [10]. The process parameters in this study, including temperature, residence time, and heating rate, are controlled to investigate the effect of activation temperature on AC quality. While carbonization primarily produces biochar, the activation process is essential to convert biochar into high-quality AC with greater surface area and porosity.

There are two main methods for activating carbon: physical and chemical activation. This study utilizes physical activation, where sewage sludge undergoes pyrolysis followed by thermal activation using superheated steam to open the pores [11]. In comparison to chemical activation, which involves the use of acids and alkalis and requires extensive washing to remove residual chemicals, physical activation is more environmentally friendly and cost-effective. Superheated steam, in particular, is chosen due to its high diffusion rate, allowing faster and more efficient pore development compared to other gases such as carbon dioxide or air [12]. This study focuses on the impact of activation temperature on the quality of the produced AC, aiming to demonstrate that sewage-sludge-based ACs are viable alternatives to commercially available products. This study also compared the quality of AC derived from sewage sludge with commercial alternatives to assess its potential as a high-quality, sustainable product.

2. METHODS

2.1. Materials

Sewage sludge samples were sourced from the treatment plant and subsequently divided into 200 g portions, each stored in separate airtight bags. These bags were clearly labeled according to the experimental conditions, as outlined in Table 1. To minimize carbon contamination, the samples were kept frozen until needed for analysis [13]. Before analysis, the frozen sludge was dried to eliminate moisture content from the rapid growth of molds and fungus. This is to preserve the integrity of the samples during the drying process. Methylene blue (Quality Reagent Chemical), acetone and other chemicals (R&M Chemicals, Analytical (A.R.) grade) and a purity of 95% were used in this work. The physical properties

of activated carbon produced from the sewage sludge were compared to the commercial coconut husk-based activated carbon (ComAC) obtained from Ki Carbon Solutions Sdn Bhd.

Table 1: Abbreviation of the raw sludge and activated carbon (AC) treated with superheated steam at various temperatures

Abbrev.	Type	Temp (°C)
RAW	Raw Sewage Sludge	-
BC400	Biochar	400
AC500	AC	500
AC600	AC	600
AC700	AC	700
AC800	AC	800
AC900	AC	900

2.2. Experimental Setup

The rotary kiln furnace (Tanaka Tech, Japan) was utilized for the carbonization and activation processes equipped with the evaporator and furnace and a condenser system. During the process of the sewage sludge, the superheated steam was injected into the kiln body of the furnace. The evaporator pump would be set at a constant flow rate of 3 ml/min by a peristaltic pump. The evaporator converted water into superheated steam at 300°C. The superheated steam produced by the evaporator would be sent into the furnace kiln. The carbonization and activation processes took place in an inert environment within the kiln. The rotary kiln furnace's heating process allows the volatile matter to evaporate and then quenched by a condenser. The remaining solid products in the kiln after treatment were collected and analyzed.

2.3. Activation by superheated steam

The quality of the ACs was analyzed by varying the activation temperature at 600°C, 700°C, 800°C and 900°C, after a constant carbonization at 400°C. Both carbonization and activation processes were 60 min after the kiln's temperature had reached the target temperature. The four processes: drying, carbonization, activation and cooling, were conducted in the rotary kiln furnace. The experiment started with the drying process. During the drying process, the temperature raised from 25°C to 110°C with a heating rate of 10°C/min for 9 min. The drying process of the sewage sludge continued at a constant 110°C for 40 min. After that, the temperature raised to 400°C with a heating rate of 10°C/min for 30 min. At this temperature, the carbonization process occurs for 60 min. Next, the temperature was raised to 800°C with a heating rate of 10°C/min for 40 min. The activation process was conducted for 60 min. After that, the rotary kiln furnace automatically stopped and was allowed to cool down for 24 hours before the ACs could be taken out.

The non-condensable gases were collected by immersing the collector pipeline in a bucket of water. [8] Bergna et al. [8] asserted that temperature higher than 900°C prefers carbon monoxide. However, the experimental temperatures were not exceeding 900°C. Besides, based on the trial of the experiment, most volatile matters were condensed, leaving out a negligible amount of uncondensed gases, which were not significant to cause pollution. Therefore, it was concluded that the amount of uncondensed gases produced would not give a significant analysis.

2.4. Product analysis

The mass yield (Y_M), defined as the percentage of ACs produced per initial mass of the sewage sludge after the carbonization and activation processes was calculated by dividing the mass of ACs after the carbonization and activation processes with the initial mass of the sewage sludge [14].

$$Y_M = \text{Mass of ACs produced} / \text{Initial mass of sewage sludge} \times 100\% \quad (1)$$

The thermal drying method was used to measure the moisture content (MC) of the ACs produced [15]. The drying process was conducted in an oven heated at 110°C and weigh until the difference of the consecutive mass was less than 5 mg [16]. The moisture content was calculated according to the wet basis.

Therefore, the moisture content was the difference between the initial and final masses of the sample divided by the initial mass of the sample.

The volatile matters (*VM*) of the samples were determined after the moisture content experiment. 1 g of sample was placed into crucibles and were placed into a muffle furnace at 950°C for 7 min. The short heating time was sufficient for all the high boiling points volatile matters to evaporate. Although this high temperature was enough to burn the samples into ashes, the time was too short for the ashes to form. Next, the crucibles were removed immediately from the muffle furnace once the timer reached 7 min mark. The crucibles were then placed into the desiccator and cooled down. After the crucibles reached room temperature, the crucibles were weighed. The volatile matter content was calculated by dividing the mass loss due to volatile by the initial mass of the sewage sludge sample, subtracting by the moisture content percentage.

The ash content of the samples was determined using the previous samples that had undergone the volatile matter experiment. 1.0 g of each sample were put into crucibles. Without covering the crucibles with their lids, the crucibles were placed into the muffle furnace at 500°C for 1 hour. The temperature was increased from 500°C to 750°C and heated for 2 h. The crucibles were taken out from the muffle furnace and placed into the desiccator to cool down. The crucibles were weighed as soon as they reached room temperature. The ash content of the samples was calculated by dividing the mass of residue left on the crucibles by the initial mass of samples. Fixed carbon (*FC*) content was the ordinary pure carbon content of the samples. *FC* is the total percentage of the *MC*, ash content, and volatile matters (*VM*) deducted from 100 percent as represented by Equation 2.

$$FC = 100\% - (ASH + VM + MC) \quad (2)$$

The FTIR analysis was conducted using a FTIR spectrometer (Nicolet 6700). The BET method surface area was based on Wang [17](2010) and Hellack et al. [18] by the BET Analyzer (Micromeritics 3Flex). The SEM analysis (HITACHI S3400-N) was conducted to visually compare the pore size and structure between the biochar (before the activation process) and the AC (after the activation process). The images' magnifications were as 10000x, 3000x, 2000x, 1200x and 700x. XRD analysis was conducted using a Shimadzu 6000 XRD Diffractometer to determine the curve trend of the produced ACs. The 2 theta range was set between 5 to 70 degrees [19][20][21].

The adsorption capacity of the ACs was analyzed using methylene blue. The adsorption experiment was conducted via two trials. The first trial was conducted by referring Zainal et al. [22]. A stock solution of 500 mg/L was prepared. The ACs were ground using a set of pestle and mortar, and were screened using a 300 µm sieve, thus, having a particle size lesser than 300µm. 15 mg of ACs with a dosage of 0.6 g/L was mixed with 25 ml of prepared 500 ppm methylene blue solution.

3. RESULTS AND DISCUSSION

3.1. Mass Yield (*YM*)

Figure 1 shows the mass yield trend of different AC samples. The mass yield decreases from 8.6% to 4.5% for the respective AC samples. The increasing activation temperature reflects the decreasing mass yield trend. The higher the activation temperature, the more moisture content and volatile matters are eliminated, leading to higher purity of carbon content of the ACs. The graph shows a less significant mass yield difference between the AC samples. This is because most of the moisture content and volatile matters are eliminated in the carbonization process at 400°C, leaving a small portion of the moisture content and volatile matters at higher activation temperatures [23]. Since the residence time, heating rate and the superheated steam flow rate are set constant in this study, the AC samples' mass yields depend on the activation temperatures. the mass yield of ACs could achieve decent to high yield. Tinnirello et al. [24] asserted that their sludge based activated carbon (SBAC) mass yield achieved more than 50% and could reach a maximum of 97%. Besides, Gong [25] also stated that his mass yield of SBACs was around 34.2% - 38.5%. Betsholtz et al. [26] reported that his SBACs had a mass yield ranging from 37.1% - 50.5%. The difference between the results could be due to the utilization of different types of sewage sludge. The literature results could be based on dried sewage sludge, where most moisture contents were eliminated beforehand. It also could be due to the difference in experimental procedures, where the initial mass of the sewage sludge is taken after the drying process instead of being taken before the drying process, which is what the current study has conducted. In this study, the drying process is combined with the carbonization and activation processes to produce AC. the initial mass of the sewage sludge has included the raw water content in the sewage sludge.

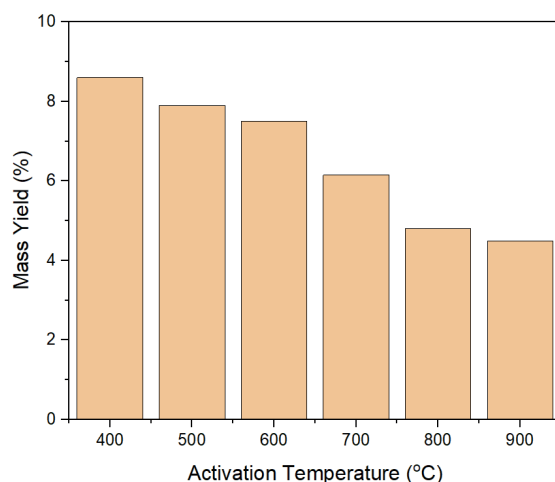


Figure 1: Mass yield as a function of activation temperatures

3.2. Moisture Content and Mass Changes of Raw Sewage Sludge

Figure 2 illustrates the MC and the mass loss data in 10 minutes intervals during the drying process. The mass of raw sewage sludge is decreasing over time, signifying that the moisture content in the raw sewage sludge is also decreasing. Moreover, both curves would increase or decrease until a certain point where they start to converge to form a constant line. The constant line represents that the wet raw sewage sludge's moisture content is getting lower to the minimum, where if the difference of the consecutive mass of the wet raw sewage sludge was less than 5 mg [16], the drying process is considered completed. Wakari [27] stated that the raw palm oil empty palm bunch would take 2 hours to complete the drying process. Since the raw empty fruit bunch has less free and vicinal water than the raw sewage sludge, it can be fully dried in a shorter time.

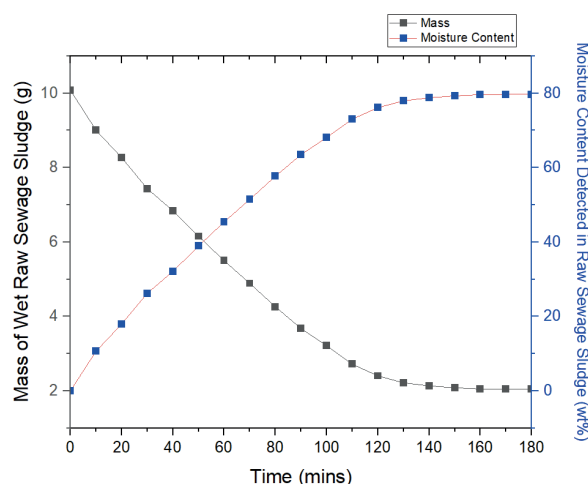


Figure 2: The curve of the moisture content detected in the raw sewage sludge and its mass loss over time during drying at 110°C

3.3. Characterization of Raw Sewage Sludge and AC

ACs are often characterized by proximate analysis, which includes parameters such as fixed carbon (FC), volatile matter (VM), moisture content (MC), and ash content [11]. Figure 3 illustrated the proximate analysis for the raw sewage sludge (RAW), BC400 and ACs activated at various temperatures. The RAW has a higher moisture content than its VM, ash content and FC content. MC was 79.7%, which is in the range of 73 wt% to 84 wt% as reported in the literature [28]. Its FC content is less than 1%, which is low due to a large amount of water coverage of the raw sewage sludge.

The MC drops significantly from 2.4 wt% to 0.2 wt% for ACs as compared between RAW and BC400. This is because most of the moisture content was eliminated during the drying process at 110°C, and even

more moisture content excluded above 400°C. There are *MC* in BC400 and the AC samples when they have already undergone a heating process. The reason could be some of the bound and interstitial water was trapped in the solids. The heating process might cause some of the bound water and the interstitial water to turn into steam, but the trapped steam was unable to be eliminated, and therefore, condensate during the cooling process to form back into liquid water. Kadir et al reported that sewage sludge consists of a lot of silica [29], and the high heat resistance property of the silica [30] would prevent some portion of water from forming steam. The *MC* in AC900 is the lowest, representing it is one step nearer to proving it is the better quality of AC. Although the *MC* of the BC400 and the AC samples are not significant, long-term adverse effects on the quality of the materials. According to Loh [31], during storage, the *MC* contained in the materials would cause fungus growth and the development of the biological activity of the microorganisms, which would gradually reduce the quality of the materials due to the reduction of the fixed carbon content.

The volatile matter (*VM*) trend decreases over activation temperature as shown in Figure 3. *VM* decreased from 31.6 wt% to 0.2 wt% for BC400 and the AC samples. The percentage of *VM* increases between RAW and BC400. This is due to the dramatic drop of *MC* at 400°C. Since most of the significant amount of *MC* has been eliminated, the volatile matters are the next to be released to increase the ash and *FC* content. The *VM* percentage tend to reduce with increasing temperature. The loss of volatile matters was also due to a reaction between the carbon surface and the superheated steam. This reduction could be attributed to CO₂ and CH₄ formation, which are parts of the *VM* that escaped into the atmosphere during reaction the reaction in the carbonizer. CO₂ was not produced as the activation temperatures did not exceed 900°C. The lower the *VM* content, the better the quality of the materials because there would be less contamination on the materials. Therefore, AC800 and AC900 that has the lower *VM* content (< 10%) is a more desirable as adsorbent material.

The ash content increases with the activation temperatures. The trend increases from 62.5 wt% to 83.7% for BC400 and the respective AC samples. The loss of *MC* and *VM* percentage has led to the increase of ash content. The ashes are mostly inert and have high heat resistance [32], causing them difficult to be burnt off. The increasing ash content trend follows the trend stated in [33]. However, the increase in ash content would cause fouling, slagging and agglomeration [34]. On the other hand, the ash content, which contains some metal compounds or metals, would tend to improve the heating efficiency of the carbonization and activation, in which it acts as a heating carrier. However, a lower ash content is more desirable because these metals that functioned as heating carriers are not significant for AC formation.

The fixed carbon content (*FC*) increases with the activation temperature. The RAW has only 0.6 wt% *FC* before the reaction. It increased from 3.4 wt% to 15.9 wt% for BC400 and the respective AC samples. This increase could be due to the relatively higher rate of elimination of the *VM* and *MC* to the usage of the *FC* for the reaction. However, the *FC* content was very low when compared to plant-derived AC. The *FC* content for oil palm empty fruit bunch (EFB) derived AC is reported to be 62.9 wt% to 71.6 wt% when activated with superheated steam between 400–800°C. This is because the oil palm EFB already has high *FC* content (63.33 wt%) in the raw state [35], whilst the raw sewage sludge only has approximately 30.82 wt% *FC* content [36] and 0.6 wt% in this study. Furthermore, sewage sludge contains higher percentage of *MC* than oil palm EFB, in which a large fraction of water content covers the presence of the *FC*.

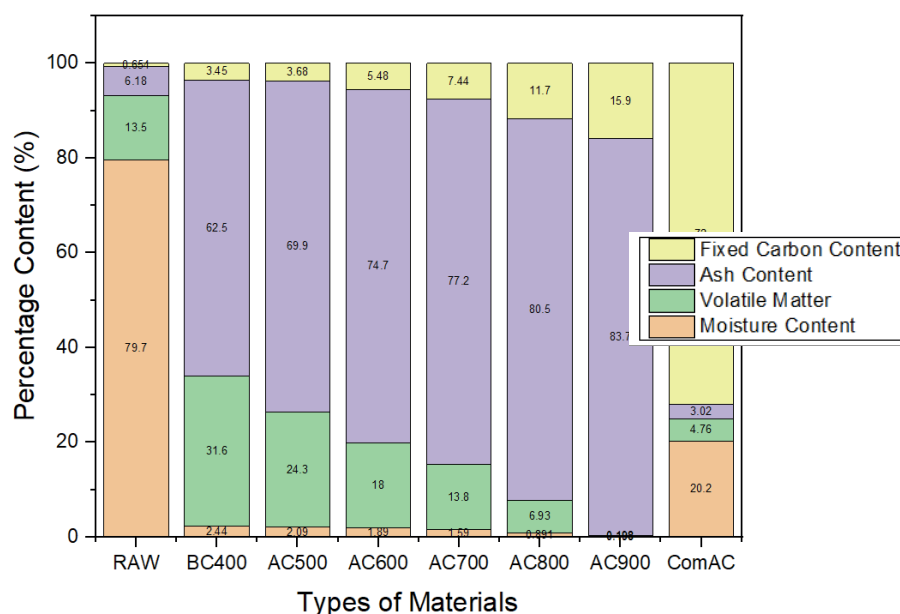


Figure 3: Proximate analysis for the raw sewage sludge, BC400 and ACs activated at various temperatures

3.4. Comparison of Raw Sewage Sludge from Other Sludge Types

Table 2 shows that most of the sludge sources analyzed by the literature had decent high *FC* contents. Paper waste sludge, biological sludge and dewatered sludge would have higher *FC* contents than municipal aerobically digested sewage sludge. Since the sludge type used in this study has a very low percentage *FC* content, the ACs it produces would not be suited to be used as a good adsorbent, but it can be reused back in the sewage sludge treatment plant to treat back the sewage.

The commercial activated carbon (ComAC) has *MC* of 20.2 wt% as shown in Fig. 3. Compared to other AC samples, it has a relatively larger moisture content. This could be due to ComAC has absorbed a lot of humid water vapour from the atmosphere after it was manufactured. In this study, the AC samples were freshly produced before the proximate analysis was conducted, which is why their moisture content is very low. Moreover, ComAC has a relatively lower *VM* (4.76 wt%) and ash content (3.02 wt%) and higher *FC* content (71.98 wt%). Since the differences between these three components are large, it reflects that the produced AC samples are unsuitable for practical applications.

In short, based on the proximate analysis results, AC800 is selected to be the expected most promising material among other materials due to its low moisture content, volatile matter content and high fixed carbon content. Too many ashes would degrade the material. However, if the ashes are consisted of large volume of silica groups, the higher the ash content, the better the quality of the sewage sludge-based AC. This would be explained in detail in the XRD analysis. Therefore, the ash content of the AC800 is considered acceptable if it is correlated with its other proximate analysis results.

Table 2: Comparison of proximate analysis results of different raw sludge types

Sludge Type	Moisture Content (wt%)	Volatile Matter Content (wt%)	Ash Content (wt%)	Fixed Carbon Content (wt%)	Reference
Paper Mill Sludge			20	34	[37]
Paper Mill Sludge	0.33		36.4	44	[38]
Biological Sludge			30	36.5	[39]
Viscous Liquid Sludge			22	39.4	[40]
Municipal Dewatered Raw Sludge (dry basis)		65.9	20.4	41	[41]
Municipal Dewatered Undigested Sewage Sludge	54.95	27.3	14.7	3.1	[42]
Anaerobic Digested Biological Sludge			34.6	32	[39]
Normal Wastewater Treatment Plant Sludge	2.32	53.2	43.9	2.81	[43]
Municipal Aerobically Digested Sewage Sludge	79.7	13.5	6.2	0.654	This study

3.5. FTIR Analysis

Figure 4 shows the FTIR has wavenumber from 400-1500 cm^{-1} is for the single bond adsorption of carbon with other elements, except for hydrogen; 1500-2000 cm^{-1} is for double bond adsorption; 2000-2500 cm^{-1} is for triple bond adsorption; 2500-4000 cm^{-1} is specifically single bond adsorption of hydrogen with other elements. The wavenumber, 400-700 cm^{-1} , is the fingerprint region, which only applies to unique compounds that are rarely used [44].

BC400 has the steepest curve, followed by AC500 and AC600. From AC700 onwards, the steepness of the FTIR spectra curve has dramatically decreased. According to Jamari & Howse [45], the higher the activation temperature, the surface functional groups of the materials would be damaged, and some would be destroyed. BC400 has amines and ammonium functional groups [46][47], representing the characteristics of a typical sewage sludge sample. This is because common faeces by a human would have a high content of ammonium compounds [48], which would then be treated in a sewage treatment plant to produce sewage sludge. Besides, BC400 also contains siloxane and silanol functional groups [49], which have already been analyzed in the XRD analysis silica groups are the main components of the sewage sludge. Besides these functional groups, BC400 contains oxygen functional groups such as phenol, glycerine, peptide, phenol and silanol stretching bonds, reflecting that the materials produced from sewage sludge have great potential in the adsorption process.

The AC samples have almost the same surface functional groups as BC400, but as the activation temperature increases, some additional surface functional groups appeared. Only AC500 has the aliphatic chloro compound functional groups and methyne groups [50], which could represent that this functional group's amount should be very little. Besides, AC500 and AC600 have additional alkane or methoxy groups, which the biochar does not have. AC700 and AC800 contain most of the oxygen functional groups. They contain additional aliphatic nitro compounds, nitrate compounds and carboxylic acid stretching as compared to BC400. This means that as the activation temperature increases, more oxygen functional groups were yielded on the AC samples. Although the surface functional groups on AC700 and AC800 are lesser than BC400, AC500 and AC600, the variety of oxygen functional groups would aid in attracting the heavy metals during the wastewater treatment process. S. Khalili et al. [51] asserted that oxygen functional groups are good adsorbers of cadmium, and the presence of carboxylic functional groups could enhance the adsorption performance of the SBACs. Therefore, it can be concluded that AC700 and AC800, which have the most oxygen functional groups and have carboxylic functional groups, are expected to perform well in the adsorption process. On the other hand, AC900 has significantly decreased in its surface functional groups due to the burnt-off of the surface functional groups during very high activation temperatures.

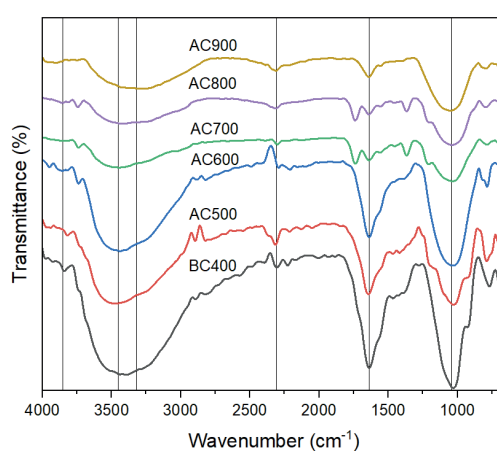


Figure 4: The FTIR combined spectrums results for BC400 and ACs activated at different temperatures

3.6. SEM Analysis

Figure 5 shows SEM analysis results for BC400, AC800 and AC900 at magnifications of 700x, 1200x, 2000x. BC400 has a segregated, non-uniform pore distribution. At 400°C, where the sewage sludge was not activated, the pore development and enhancement were insignificant, as the carbonization process only initiates and creates new pores. The porosity started to form after the carbonization process compared to the SEM diagram of raw sewage sludge. AC800 shows a uniform pore distribution, and the pore sizes are quite even. The pores tend to shrink or, in other words, have developed into well-structured pores. Compared to BC400 and AC800, the AC800 has a better pore structure in terms of the uniformity of the pores. The shrinking of the pores is due to the sintering process of the silica groups, which are the main compounds in the sewage sludge or the sewage sludge itself. As the fire temperature increases, the occurrence of the sintering process of silica groups increases [52]. In this context, the silica groups pull the sewage sludge structure inwards, causing the pores to be pulled inwards. According to Nayak & Bera [53], materials with smaller particle sizes and pores prefer a rapid sintering process. This means that AC800, which has a finer particle size, would favour the sintering process of silica groups. In this context, the particle size of the materials tends to decrease with the increase in activation temperature [54].

Furthermore, AC900 has a rough, waved surface, and its pore distribution is unpredictable. Its pore size enlarges until a certain point that the pores can be seen as hollow. Hollow pores are due to the excessive burnoffs and the amalgamation of mesopores into macropores or macropores into hollow holes [55]. The hollow holes would eventually ease the splitting process of the particle size, causing the particle size of AC900 to decrease. To compare these three materials, AC800 stood up to have a better overall structure. The pore sizes can be determined using the scaling ruler in the SEM diagram. However, many pores have sizes in the nanoscale, making the scaling ruler impossible to determine their sizes. Therefore, the pore sizes cannot be analyzed via SEM analysis, but it can be done via BET surface area analysis.

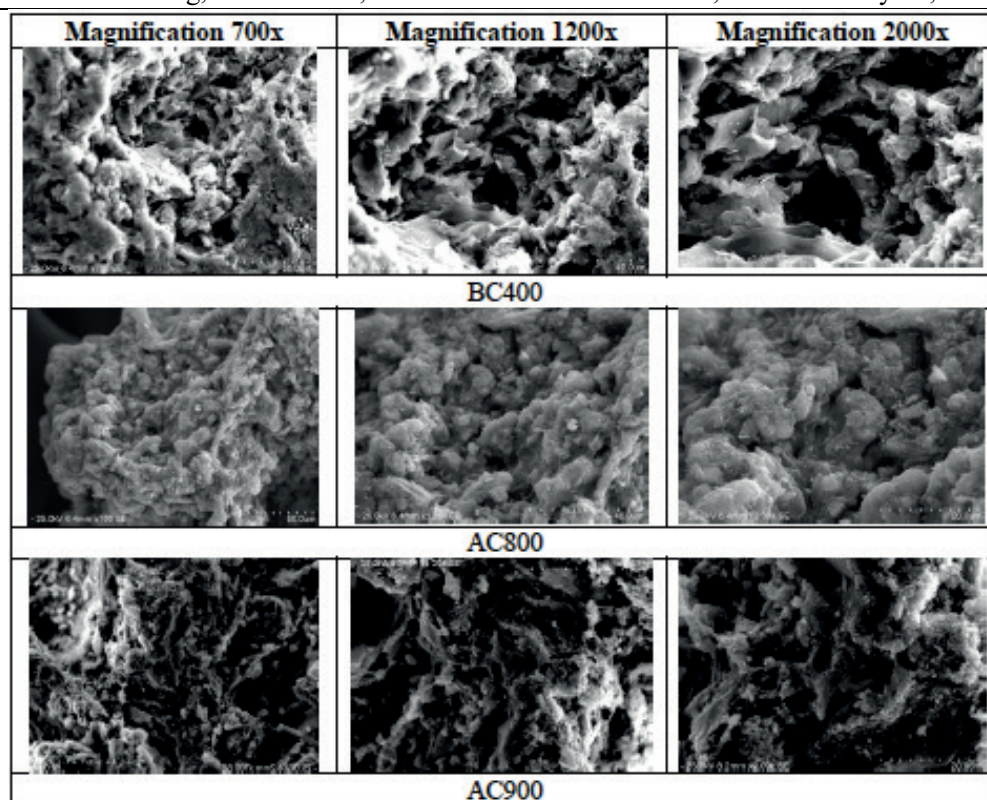


Figure 5: SEM analysis results for BC400, AC800 and AC900 at magnifications of 700x, 1200x, 2000x

3.7. BET Surface Area Analysis

Table 3 shows the surface area, micropore area, micropore volume, total pore volume and pore size of the biochar and different AC samples. The surface area has a decreasing trend, where it starts to decrease significantly when it reaches AC800 and AC900. The surface area of AC800 is $50 \text{ m}^2\text{g}^{-1}$, while the surface area of AC900 is $1 \text{ m}^2\text{g}^{-1}$. The decreasing trend of the surface area shows that it was affected by the increase in activation temperature. To further justify this statement, the sewage sludge does not have the lignocellulose structure, like the plant-based ACs, to withstand its shape during high-temperature activation, causing it to break apart easily. At higher activation temperature, the AC becomes finer, causing the surface area to decrease due to the decrease in particle size in comparing one particle with another. If the comparison is based on a per mass basis, the surface area of 1g of materials increases with the increment in particle sizes. However, the AC particles become too fine to build up new pores to compensate for the rate of the decrement of particle size. The surface area of the AC samples is a function of the porosity, in other terms, pore volume. Higher pore volume means the material has a large pore size and number of pores. The pore volume does not have a significant impact on the surface area, as it has no significant changes for the increasing activation temperature. The decrement of the surface area of the materials is explained by giving an example of a fine particle having a large pore size, but since it has a lesser number of pores, it has a lower surface area [56]. Besides, AC900 has the least surface area because of the amalgamation of macropores to hollow pores, which causes the particles to be broken down into very fine particles that can hardly generate new pores.

For the micropore area and its volume, their trend decreases significantly from AC700 ($96.42 \text{ m}^2\text{g}^{-1}$ and $0.041 \text{ cm}^3\text{g}^{-1}$, respectively) to AC800 ($21.10 \text{ m}^2\text{g}^{-1}$ and $0.0093 \text{ cm}^3\text{g}^{-1}$, respectively) and AC900 ($0.65 \text{ m}^2\text{g}^{-1}$ and $0.00028 \text{ cm}^3\text{g}^{-1}$, respectively). Their decreasing trends are due to the amalgamation of micropores into mesopores and macropores. Since the presence of the micropores decreases with the increasing activation temperature, the micropore volume would also tend to decrease. Based on Table 3, the surface areas of the materials decrease with the increase in pore sizes. This is due to the internal pore strain, which is caused by the incorporation of existing heat resistance particles that could enlarge the pore diameter of the materials [57]. The silica groups and sewage sludge ashes are heat resistance particles in this context. At higher temperatures, sewage sludge and silica groups' sintering processes occur rapidly [58], causing the entire particle to shrink. However, during the shrinking process, some pores would remain their size, as the unshrinkable silica groups and some inorganic compounds resist the pores from getting smaller.

The decrement in surface areas of the materials is due to the drop in the pore volume, but the pore volume trend shown in Table 3 slightly increases with the increase in activation temperature, which ranged from $0.28 \text{ cm}^3\text{g}^{-1}$ to $0.50 \text{ cm}^3\text{g}^{-1}$ for BC400 to AC900. This could be due to two contexts. First, the number of pores increases with the increase in activation temperature, but since the surface area also decreases, the pores might be blocked or incorporated with the ashes and the silica groups, causing the pore volume to increase. The second context is that the number of pores decreases as more hollow pores are generated, leading to a slight increase in pore volume, depending on the particle sizes, which also means that the slight increase in pore volume could be due to the presence of macropores. In terms of the physical properties of the silica groups, their melting point is more than 1600°C , and their sintering temperatures are from 1000°C to 1400°C [59][60][61]. However, some studies did their research on the sintering process of the silica at less than 1000°C and showed the results as stated that the sintering of the silica will affect the porosity of the ACs [58][62]. Therefore, the sintering of silica groups could occur during the activation process by superheated steam.

For the pore size of the materials, its trend increases with the increase in activation temperature. It increases significantly when it reaches AC800 and AC900. The pore sizes increase significantly from 4.7 nm to 18.0 nm and 611.5 nm. The pore size of AC900 is not logical due to the resulted pore size cannot be represented by its actual small particle size, and its resulted pore size is a lot larger than the ordinary macropores, which has a size of more than 50 nm. Thus, it can be justified that the pore size of AC900 is based on the long hollow pores, which causes the pore size to increase significantly. On the other hand, AC800 has a logical value of its pore size.

Its pore size represents it consists of many mesopores, which have a range of sizes between 2 nm to 50 nm. Table 4 compares BET analysis results of different SBACs samples from the literatures and the current study. AC800 is used as a reference for comparison. Based on Table 3, the BET analysis results of BC400, AC500, AC600 and AC700 show that they have decent pore properties compared to the literature, but they are still considered lower than the results reported by other investigators.

The pore properties of a good AC can be proven by the surface area, micropore volume, pore volume and pore size. Comparing the SBACs produced using steam at 800°C for 1 hour [63] and the current study, the differences in surface area and pore size of the current study are significantly lower, while the difference in pore size is higher. These differences could be caused by the utilization of different types of reactors. The rotary kiln in this study could cause decent ACs to burn off and break into finer particles at 800°C , signifying that it has low heat loss and high thermal efficiency, as it could also contribute to similar results at 500°C as shown in Table 3. The adsorption performance of the ACs is not dependent on their surface area, but the pore volume and pore size, as the adsorption of materials are based on how big the pores could contain. If the pores are saturated with contaminants, the surface area would now be important, as the contaminants would tend to cling onto the surface functional groups. Based on Table 3, the trend of pore properties of BC400, AC500, AC600 and AC700 show that the activation temperature did not significantly impact them. Only AC800 starts to show significant changes in pore properties' values, which is why it is presumed to have a good adsorption performance in the methylene blue adsorption test in the next section.

Table 3: BET surface area analysis results for BC400 and ACs activated at various temperatures and Comparison of BET surface area analysis results of different sludge based activated carbon (SBAC) prepared by various methods.

Sample	Surface area (S_{BET}) (m ² /g)	Micropore area (S_{MICRO}) (m ² /g)	Micropore vol. (V_{MICRO}) (cm ³ /g)	Total pore vol. (V_{TOTAL}) (cm ³ /g)	Pore size (nm)	Ref
BC400	166	116.16	0.047	0.28	3.41	This study
AC500	153	110.00	0.045	0.32	4.15	This study
AC600	183	131.09	0.054	0.29	2.77	This study
AC700	168	96.12	0.041	0.30	4.66	This study
AC800	50	21.10	0.0093	0.45	17.95	This study
AC900	1	0.65	0.0003	0.50	611.5	This study
AC using CO ₂ at 800°C	7	-	0.02	-	-	[64]
AC using Steam at 800°C for 1h	226	-	0.083	-	-	[40]
AC using N ₂ as 950°C for 0.5 h	125	-	0.049	-	-	[65]
AC using FeCl ₃ and N ₂ at 1000°C for 1h	468	-	0.16	0.56	-	[66]
AC using Steam at 800°C for 1h	135	-	0.02	0.17	63	[67]
AC using Steam at 750°C for 1h	95	-	0.04	0.09	87	[67]

3.8. Methylene Blue removal

Figure 6 shows the percentage of removal of methylene blue versus commercial activated carbon (ComAC), sludge carbonized at 400°C (BC400), and activated carbon activated at various temperatures. The superheated steam, as an activator, generates good-quality activated carbon. Activation by superheated steam at 800°C (AC800) is a more promising adsorbent among all material samples due to its high adsorption capacity (97.12%). AC800 comprises 80.50 wt% ash content, mainly containing silica groups, and 11.68 wt% fixed carbon content, which is comparatively higher than other material samples. The high silica content in the AC800 proves that sewage-sludge-based AC has the potential to be a promising adsorbent. Moreover, there are various oxygen functional groups in AC800, making it a high-quality adsorbent to adsorb multiple contaminants.

The effect of the activation temperature showed as the activation temperature increases, the percentage moisture content, percentage volatile matter content, pore volume, surface area, micropore volume, and micropore area of the ACs would decrease, while the percentage ash content, percentage fixed carbon content, pore size, adsorption capacity and variety of surface functional groups of the ACs would increase. Temperatures beyond 800°C would cause the ACs to break down and decrease their adsorption capacity. Consequently, this study confirms that carbonization and activation processes at 800°C using superheated steam would produce enhanced physicochemical properties of an AC and are very promising as a new adsorbent to reduce municipal sewage sludge.

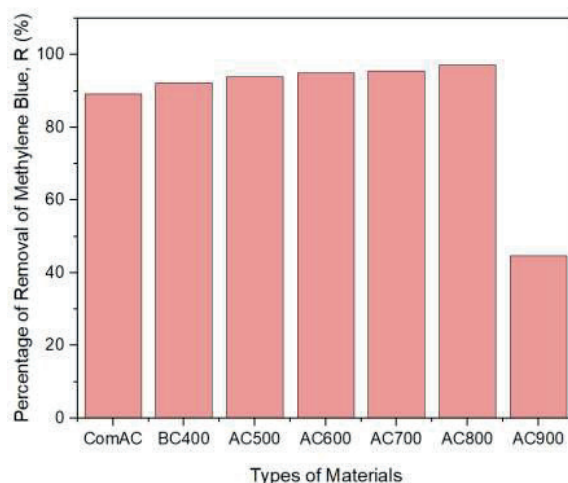


Figure 6. Percentage of removal of methylene blue versus commercial activated carbon (ComAC), sludge carbonized at 400°C (BC400), and ACs activated at various temperatures.

4. CONCLUSION

This study proves that the physical activation process, which uses superheated steam as its activator, could generate good quality activated carbon. This study also proves that activated sewage sludge activated at 800°C by superheated steam, is the more promising adsorbent among all material samples due to its high adsorption capacity (97.12%). Also, it comprises 80.50 wt% ash content, mainly containing silica groups, and 11.68 wt% fixed carbon content, which is comparatively higher than other material samples. The high silica content in the AC800 proves that sewage-sludge-based AC has the potential to be a promising adsorbent. Moreover, there are various oxygen functional groups in AC800, making it a high-quality adsorbent to adsorb a variety of contaminants. In terms of the effect of the activation temperature, as the activation temperature increases, the percentage moisture content, percentage volatile matter content, pore volume, surface area, micropore volume, and micropore area of the ACs would decrease, while the percentage ash content, percentage fixed carbon content, pore size, adsorption capacity and variety of surface functional groups of the ACs would increase. Temperatures beyond 800°C would cause the ACs to break down and decrease their adsorption capacity. Consequently, this study confirms that carbonization and activation processes at 800°C using superheated steam would produce enhanced physicochemical properties of an AC and are very promising as a new adsorbent to reduce municipal sewage sludge.

ACKNOWLEDGMENT

The authors are grateful to Indah Water Konsortium Sdn Bhd company (Num: IWRC/TIS/LOA22/001) for supplying the raw sewage sludge and Universiti Putra Malaysia for providing the research grant (UPM.RMC.800-3/3/1/GP-IPS/2021/9701300).

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BIOGRAPHY



Shamsul IZHAR works as a lecturer at Universiti Putra Malaysia in the Dept of Chemical and Environmental Engineering.

Shamsul received his BSc in Chemical Engineering in 1998 and his Ph.D. in Chemical Engineering in 2008 from Tokyo Univ of Agriculture and Technology, Japan. He is on Hazardous Waste Management at Yildiz Technical University Environmental Engineering Department.

Shamsul is a member of the Board of Engineers, Malaysia.

He may be contacted at shamizhar@upm.edu.my.