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Effect of time and temperature on the physicochemical characteristics of bamboo-derived activated carbon

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DECLARATION

I declare that this thesis entitled “Effect of time and temperature on the physicochemical characteristics of bamboo-derived activated carbon” is the results of my own research except as cited in the references.

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Kesan masa dan suhu ke atas ciri fizikokimia karbon teraktif yang berasal dari buluh

ABSTRAK

Gel silika pengering di pasaran mengandungi bahan berbahaya dan menimbulkan bahaya lemas. Karbon teraktif boleh berfungsi sebagai penyelesaian alternatif untuk memasukkan sebatian berbahaya ke dalam bahan pembungkus makanan untuk menjerap dan mengeluarkan oksigen dan lembapan. Kajian ini mempunyai tiga objektif iaitu untuk menyediakan karbon teraktif menggunakan buluh (*Gigantochloa albociliata*) pada masa dan suhu yang berbeza. Untuk mengkaji kesan masa dan suhu terhadap ciri-ciri fizikokimia karbon teraktif terbitan buluh. Untuk mengkaji kesan masa dan suhu terhadap sifat morfologi karbon teraktif terbitan buluh. Buluh (*Gigantochloa albociliata*) dipotong menjadi kepingan 1.5-2 cm, dikeringkan dan dicampur dengan baik dengan larutan kalium karbonat (K_2CO_3). Suhu pengaktifan dalam relau dilakukan untuk 400°C, 600°C, dan 800°C, ia dikawal secara berterusan untuk tempoh masa tertentu, 1j, 1.5j, dan 2j. Analisis FTIR mengenal pasti puncak kekerapan setiap kumpulan berfungsi. Panjang gelombang AC 400 °C/1 jam telah diperiksa pada julat 1108.56 cm^{-1} hingga 3355.65 cm^{-1}, dan terdapat 6 sebatian berfungsi ditemui. Panjang gelombang AC 600 °C/1.5 jam telah diperiksa pada julat 533.71 cm^{-1} hingga 2359.35 cm^{-1}, dan terdapat 8 sebatian berfungsi ditemui. Panjang gelombang AC 800 °C/2 jam telah diperiksa pada julat 2082.31 cm^{-1} hingga 2322.95 cm^{-1} dan terdapat 2 sebatian berfungsi ditemui. Mengimbas mikrograf elektron (SEM) mengesahkan morfologi permukaan. EDX menentukan komposisi unsur karbon teraktif yang berasal dari buluh. Luas permukaan BET karbon teraktif diperoleh jumlah isipadu liang untuk AC 400 °C/1 jam ialah 3.498×10-3 cc/g manakala AC 800°C/2 jam menghasilkan 4.912×10^{-2} cc/g. AC 800 °C/2 jam menghasilkan nilai luas permukaan yang tinggi sebanyak 74.972 m^2/g. AC 400 °C/1 jam menghasilkan nilai luas permukaan yang rendah iaitu 1.962 m^2/g. Analisis XRD telah diperolehi dua puncak luas yang berbeza terletak pada sekitar 24° dan 43.6°, struktur kristal ditentukan di mana masing-masing adalah 29.8%, 26.9%, dan 28.2%. Karbon teraktif yang berasal dari buluh berpotensi untuk dipasarkan sebagai bahan pengering tumbuhan semula jadi pada masa hadapan.

Kata kunci: Buluh (*Gigantochloa albociliata*), pengaktifan, karbon teraktif, mengimbas mikrograf elektron (SEM), Brunauer-Emmett-Teller (BET)

Effect of time and temperature on the physicochemical characteristics of bamboo-derived activated carbon

ABSTRACT

Desiccant silica gel on the market contains dangerous ingredients and poses a suffocation hazard. Activated carbon can serve as an alternative solution for incorporating hazardous compounds into food packaging materials to adsorb and remove oxygen and moisture. This study had three objectives, namely, to prepare activated carbon using bamboo (*Gigantochloa albociliata*) at different time and temperature. To study the effect of time and temperature on the physicochemical characteristics of bamboo-derived activated carbon. To study the effect of time and temperature on the morphological properties of bamboo-derived activated carbon. The bamboo (*Gigantochloa albociliata*) was cut into 1.5-2 cm pieces, dried and thoroughly mixed well with a potassium carbonate (K_2CO_3) solution. The activation temperature in furnace was performed for 400°C, 600°C, and 800°C, it was controlled invariably for a given period of time, 1h, 1.5h, and 2hrs. FTIR analysis identified frequency peak of each functional group. The wavelength of AC 400 °C/1 hr was examined at range 1108.56 cm^{-1} to 3355.65 cm^{-1}, and there were 6 functional compounds found. The wavelength of AC 600 °C/1.5 hrs was examined at range 533.71 cm^{-1} to 2359.35 cm^{-1}, and there were 8 functional compounds found. The wavelength of AC 800 °C/2 hrs was examined at range 2082.31 cm^{-1} to 2322.95 cm^{-1} and there were 2 functional compounds found. Scanning electron micrograph (SEM) confirmed the surface morphology. EDX determined the elemental composition of bamboo-derived activated carbon. BET surface area of activated carbon was obtained total pore volume for AC 400 °C/1 hr was 3.498×10^{-3} cc/g while AC 800 °C/2 hrs yielded 4.912×10^{-2} cc/g. AC 800 °C/2 hrs produced a high surface area value of 74.972 m²/g. AC 400 °C/1 hr yielded a low surface area value of 1.962 m²/g. XRD analysis was obtained two distinct broad peaks located at around 24° and 43.6°, determined crystalline structure where were 29.8%, 26.9%, and 28.2%, respectively. Bamboo-derived activated carbon has the potential to be marketed as a natural plant desiccant in the future.

Keywords: Bamboo (*Gigantochloa albociliata*), activation, activated carbon, Scanning electron micrograph (SEM), Brunauer-Emmett-Teller (BET)

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing Material
BET	Brunauer – Emmett– Teller
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscope
AC	Activated Carbon
GAC	Granular Activated Carbon
PAC	Powder Activated Carbon
XRD	X-ray Diffraction
EDX	Energy dispersive X-ray

LIST OF SYMBOLS

cm	Centimeter
nm	Mircometer
%	Percentage
℃	Degree Celsius
m^2/g	the total surface area of a material per unit of mass
min	Minute
mL	Millilitre
K_2CO_3	Potassium carbonate
CO_2	Carbon dioxide
Mm	Millimetre

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Activated carbon has applications in a variety of sectors. The application used in municipal drinking water, industrial pollution control, odor removal, food and beverage processing. Activated carbons used in industrial wastewater treatment because have the capacity to adsorb many contaminants (hydroxyl derivatives, phenols, chlorine solvents, soluble dyes, detergents, aromatic component, and hydrocarbons). The significance of activated carbon in beverage and food processing for contaminant removal is considerable; applications include soft drinks, yeast, maple syrup, alcoholic beverages, fats and oil vegetables, sugars, honey, fruit juices, and sweeteners. The importance of activated carbon in pharmaceutical and chemical industries is considerable because it removes contaminants that are useful in quality control it able to removes toxic compounds (Soonmin et al., 2022).

In the food and beverage industry, activated carbon is used to improve the taste, smell, and purity of a variety of foods and beverages, including concentrated juice. Activated carbon can be used to decolorize sweeteners and remove undesirable by-products from them. Gases can be filtered using various types of activated carbon. Cloth, pelletized, and granular activated carbon are examples of the specific forms that can be utilised depending on the desired results. Applications for activated carbon in gas purification include filtering breathing air in air conditioners and eliminating hydrogen sulphide from natural gas. The recirculated air is cleaned of radon and odours in this secondary application of activated carbon. Gold recovery and other important metal recovery are two further uses for activated carbon in mining. Because of its adsorbent nature, activated carbon is used in the latter phases of mining to separate carbon from cyanide (Sodimate, 2022).

Carbonaceous source materials such as wood, coal, coconuts, peat is used to produced activated carbon. The primary source of raw materials for activated carbon might be any organic material with a high carbon amount. Soluble substance from the water is remove by the solid is the process adsorption. The solid state of active carbon in adsorption process. To create a very large interior surface (between 500 and 1500 m^2/g), activated carbon is generated particularly. Adsorption works best with active carbon because of its large interior surface. Granular activated carbon (GAC) and powder activated carbon (PAC) and are the two forms of active carbon available (Lenntech, n.d).

Bamboo botanical fragments are collected after they have grown for at least five years in order to make bamboo charcoal. Bamboo Charcoal goes through the same pyrolysis procedure to become "activated." There are two types of bamboo charcoal， raw bamboo briquette charcoal and bamboo charcoal (Newdirectionsaromatics, n.d). Modification of carbonaceous materials involves two types of activation; There are two types of activation: chemical and physical. The parameters that will be studied in this experiment are chemical activation with time, temperature, X-ray Diffraction (XRD), Fourier transformed infrared (FTIR) spectroscopy, scanning electron microscopy- energy dispersive X-ray analysis (SEM-EDX), Brunauer-Emmett-Teller (BET) analysis.

1.2 Problem Statement

In food packaging industry, the applications of package dry food use desiccants to maintain a constant relative humidity surrounding product. Silica gel is a desiccant that is placed in little packets packed with commercial products to avoid moisture damage. Desiccant silica gel is a choking hazard. Desiccant silica gel can also induce intestinal obstruction if consumed in high quantities. Manufacturers frequently label the packets with "Do not eat" or "Throw away after use". Other dangerous components, such as cobalt chloride and strong alkali, have been found in silica gel packets in uncommon situations. Symptoms include vomiting and stomach aches after eating silica gel (Khatri, 2023).

The problem faced by food packaging plants is that extended shelf-life foods are easily degraded by exposure to oxygen, moisture and other external factors. This may result in a reduced product's storage life, lowered quality, and more food waste. Odor and taste control is also the important problem faced in industry, some foods emit volatile compounds that can cause unpleasant odors and tastes inside the package. This can have a negative impact on the consumer's sensory experience. The human sense of smell and taste are inextricably linked, and the smell of food or beverages may affect consumer demand (Cilicant, 2021). Activated carbon can be incorporated into food packaging materials to adsorb and remove oxygen and moisture, thereby minimizing oxidation and microbial growth. This contributes to increasing the storage life of food products., maintain their freshness, also reduce food waste. High surface area and porosity of activated carbon enable it to effectively adsorb and trap volatile compounds responsible for undesirable odors and flavors. By incorporating activated carbon into packaging, it can help control and eliminate these odor-causing compounds, ensuring that the product retains its desired taste and aroma.

1.3 Objectives

The objectives of this study are:

i To prepare activated carbon using bamboo (*Gigantochloa albociliata*) at different time and temperature.

ii To study the effect of time and temperature on the physicochemical characteristics of bamboo-derived activated carbon.

iii To study the effect of time and temperature on the morphological properties of bamboo-derived activated carbon.

1.4 Scope of Study

Bamboo was cut into particle size to made activated carbon. In this study, several physicochemical characteristics of bamboo-derived activated carbon were examined using X-ray Diffraction (XRD), Fourier transformed infrared (FTIR) spectroscopy, Bruxnauer-Emmett-Teller (BET) analysis, scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDX). The experiment used chemical activation is repeated for different operating parameters activation time, activation temperature.

1.5 Significances of Study

The investigation of study preparation of condition bamboo-derived activated carbon in time and temperature influences the physiochemical properties. The findings of this study should be able prove that application of activated carbon to remove odor and control moisture content in food packaging industry. The activated carbon potential use to replace silica gel to absorbing significant quantities of moisture. The large surface area, strong surface reactivity, adsorption capacity, and favourable pore size of activated carbon are primarily responsible for its chemical and physical properties. These features enable the interior surface to be accessed, enhance adsorption rate, and improve mechanical strength. The characteristics of activated carbon are greatly influenced by the temperature and duration (time) of carbonization and activation. The initial porosity and carbon content are mostly created by the carbonization process, while the activation step contributes in the pore structure's development. (Negara et al., 2016).

CHAPTER 2

LITERATURE REVIEW

2.1 Bamboo

Bamboo (Figure 2.1) is subfamily from Bambusoideae, subfamily of tall tree like grasses of the family Poaceae, it grows in temperate and semi-tropical regions with open, well-drained environments. The three principal chemical components of bamboo are cellulose, hemicelluloses, and lignin, which are closely connected in a complicated structure. Bamboo is regarded as an alternative to wood due to its superior physical and mechanical properties. It matures in three years as opposed to nearly 20 years for wood. Bamboo has tensile strength comparable to mild steel after maturity (Intech, 2018).

Figure 2.1: Bamboo

(Source: UrbanMali, n.d)

Gigantochloa albociliata is a natural bamboo found in Thailand and Burma (Myanmar) (Pegu, Martaban, and Tenasserim). In the country's central and northern dry forest hills, it is widely spread. It has been brought to Indo-China (e.g., common in Laos) and India (e.g., Assam, West Bengal, Arunachal Pradesh). Densely clumps, clumps, evergreen or deciduous bamboo. The stalk is slender, arc-shaped, (5-)10(-16) meters high, 2-7 cm in diameter, 1 cm thick, bristles when young, gray-green with white stripes, internode length 15-60 cm, and the nodes are obvious.

Branches that grow from nodes, particularly in the top half, are typically upright and practically solitary, and they are almost as strong as the culm (Plantuse, 2019). Since ancient times, bamboo has been a vital resource for human civilization. It has helped people living in tropical and subtropical regions of the world, particularly in East and South Asia, with everything from amusement to survival needs and emotional support (Chongtham & Bisht, 2021). Bamboo is also used to make containers, chopsticks, woven mats, fishing poles, cricket boxes, handicrafts, and chairs. Culms are employed in construction as load-bearing elements, such as roofs, trusses, bridges, and towers (Akinlabi et al., 2017). Table 2.1 provides a summary of diversified bamboo utilisation.

Table 2.1: Some categorised uses of bamboo (Source: Akinlabi at el., 2017)

Use of bamboo as plant	Use of bamboo as material
Agro-forestry	**Chemical industries**
Plantations	Pharmaceutical industry
Natural stands	Biochemical products
Mixed agro-forestry systems	
Ecology	**Energy**
Uses on marginal land	Charcoal
Minimal land use	Gasification
Hedges and screens	
Stabilising the soil	
Ornamental horticulture	**Local industries**
	Kitchen utensils
	Houses
	Arts
	Nutritional industries
	Furniture
	Young shoots for human consumption

2.2 Activated carbon

Activated carbon, frequently referred to as activated charcoal, is an unprocessed version of graphite, the material used to make pencil leads. In contrast to graphite, its structure is exceedingly porous, irregular and random structure across various pore sizes, from obvious fractures and crevices to molecular levels. The unusually enormous carbon surface area is a result of the graphite structure, which also enables the carbon to adsorb a wide variety of chemicals. Activated carbon has the highest physical adsorption capacity or adsorption porosity of any material that is currently known to humanity. Activated carbon, sometimes referred to as activated charcoal has a surface area that can exceed 1000m^2/g. Thus, a football field's worth of surface area can be achieved with just 3g of activated carbon (chemviron, n.d).

2.3 Mechanism of activated carbon

Adsorption is a physical process by which a solid surface concentrates fluid molecules (as contrasted with absorption, which describes the method by which fluid molecules are absorbed by a liquid and spread out across its surface). The adsorption of AC is influenced by numerous factors the adsorption mechanism of AC will be focused on its microspore and weak van der waals forces (Muzarpar et al., 2021).

The bond between each particle will be broken and replaced by a new bond, resulting in the porous nature of the material. However, because there are fewer microspores in the current spore, it is not as effective as activated carbon. In order to boost the effectiveness of the charcoal, another step was required, which was the activation process. During the activation phase, additional microspores are generated, increasing the surface area of AC. Microspores were extremely minute and could only be spotted under a SEM (Muzarpar et al., 2021). The microspore shown in Figure 2.2

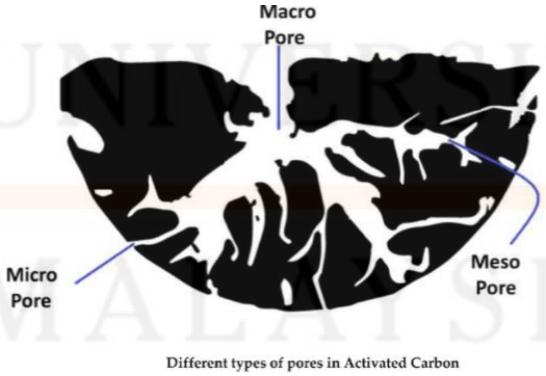


Figure 2.2: Microspore

(Source: Baby et al., 2019)

London dispersion force, a kind of van der Waals force generated by intermolecular attraction, is the fundamental force generating physical adsorption on activated carbon. Thus, when adsorption occurs, the adsorbate's and carbon's chemical compositions stay unchanged. In contrast, in the chemisorption process, molecules react chemically with the carbon surface (or else impregnant on the surface) and retained by chemical bonds that are significantly stronger forces than London dispersion forces. All molecules, polar and non-polar, interact with each other through a force known as the London dispersion force (Muzarpar et al., 2021)

Adsorption is the term for the process by which gas molecules adhere to or become trapped on the surface of activated carbon in applications for the purification, separation, and storage of gases. The pore volume, surface area and the distribution of pore sizes in the pores of activated carbon play a part in this procedure (Negara et al., 2016). Using activated carbons with different pore size distributions applied to the same adsorbate may result in different adsorption capacities. When utilised for different adsorbates, although different activated carbons have the same pore size distribution, they may have different adsorption capabilities. This is due to the fact that the adsorption capacity is influenced by the adsorbate utilised as well as the pore size distribution of the adsorbent, in addition to surface area and pore volume (Kupgan et al., 2017) .

2.4 Method preparation of activation method

Several methods can be used to the production activated carbon. There are two ways to activate, two types of activation: chemical and physical.

2.4.1 Chemical activation

Utilizing chemical reagents involves a part of chemical activation.For example, carbon can be activated using potassium hydroxide (KOH), sodium hydroxide (NaOH), hexafluorophosphate (H_3PO_4), and zinc chloride ($ZnCl_2$). Compared to physical activation, the chemical activation method has many of benefits, includes one-step procedure, better final carbon yields, often lower activation temperatures, and simpler porosity adjustment (Bergna et al., 2018). The mixture is then dried in an inert environment and heated at a temperature between 400°C and 700°C. The product is separated from the dried, slurry, and treated according to its application after the activating ingredient is removed by washing with water. Table 2.2 shows various chemical reagents used to AC prepared from different raw materials.

Table 2.2: Various chemical activating agents and precursors used for AC production (Source: Gottipati, 2012)

Activating agent	Material	Source
$ZnCl_2$	Coconut shells, corn cob, peanut hulls, hazelnut shells, rice husk, tamarind wood, cattle-manure, bagasse, sunflower seed hulls almond shells, apricot stones, pistachio-nut shells, macadamia nutshells	(Acharya et al., 2009; Ahmadpour and Do 1997; Aygun et al., 2003; Tsai et al., 1997, 1998; Girgis et al., 2002; Liou, 2010; Lua and Yang, 2005; Cronje et al., 2011; Sahu et al., 2010; Azevedo et al., 2007; Qian et al., 2007; Yalcin and Sevinc 2000).
KOH	Macadamia, rice straw, nutshells, corn cob, cassava peel, pine apple peel peanut hulls, cotton stalk, olive seed, rice straw, petroleum coke, coal,	(Ahmadpour and Do 1997; Stavropoulos and Zabaniotou 2005; Deng et al., 2010; Kawano et al., 2008; Sudaryanto et al., 2006; Oh and Park 2002; Girgis et al., 2002; Tsai et al., 2001; Tseng et al., 2008; Basta et al., 2009; Foo et al., 2011; Wu et al., 2010, 2011).
H_3PO_4	Pecan shells, corn cob, bagasse, sunflower seed hulls, lignin, almond shells, grain sorghum, rice straw, oak, sewage sludge, birch, chestnut wood, eucalyptus bark, rice hull, cotton stalk, jackfruit peel, peanut hulls, hemp	(Ahmedna et al., 2004; Deng et al., 2010; Diao et al., 2002; Fierro et al., 2010; Girgis et al., 2002; Gomez-Serrano et al., 2005; Patnukao and Pavasant, 2008; Klijanienko et al., 2008; Liou, 2010; Guo and Rockstraw, 2007; Montane et al., 2004; El-Hendawy et al., 2001; Prahas et al., 2008; Rosas et al., 2009; Wang et al.., 2011; Zuo et al., 2009).
K_2CO_3	Cotton stalk, corn cob, almond shell, pine apple peel, coconut shell, oil palm shell, pistachio shell, walnut shell, bamboo.	(Tsai et al., 2001; Adinata et al., 2007; Deng et al., 2010; Horikawa et al., 2010; Foo et al., 2011a,b; Hayashi et al., 2002).

2.4.2 Physical activation

The pyrolysis of carbonaceous materials in an inert atmosphere is called carbonization, which is the first heat treatment of the raw materials. The first approach includes pyrolyzing or carbonising the carbonaceous precursor at extremely high temperatures, usually between 700 and 900 °C, in an inert environment to prevent combustion of the carbonaceous component. The activation step, a second heat treatment, is required to eliminate the tar that is obstructing the pores as well as to perhaps generate a more pronounced porosity. This is accomplished through a reaction using oxidising reactants (steam, carbon dioxide, and combinations of them are most usually used) at temperatures between 800 and 1000 °C. As activating agents, air or oxygen may also be utilized (Rodríguez-Reinoso & Sepúlveda-Escribano, 2001). Table 2.3 shows various physical activating agents and precursors used for AC production.

Table 2.3: Various physical activating agents and precursors used for AC production (Source: Singh, 2018)

Activating agent	Material	Source
Steam	Sunflower shells, pinecone, rapeseed, cotton residues, olive-waste cakes, coal, rubberwood sawdust, fly ash, coffee endocarp, corn cob, olive residues, rice husk	(Baçaoui, 2001; Lazaro et al., 2007; El-Hendawy et al., 2001; Haykiri-Acma et al., 2006; Lu et al., 2010; Malik, 2003; Nabais et al., 2008; Prakash Kumar et al., 2006; Zhang et al., 2011)
CO_2	Rice straw, oak, coconut shells, corn stover, rice hulls, pecan shells, pistachio nutshells, coffee endocarp, sugarcane bagasse, corn cob, waste tyres, textile fibers, anthracite, corn hulls	(Ahmedna et al., 2000; Lua et al., 2004; Aworn et al., 2009; Betancur et al., 2009; Guo et al., 2009Nabais et al., 2008; Salvador et al., 2009; Yang and Lua, 2003; Zhang et al., 2004; Zhu et al., 2011)
Air	Coal, peanut hulls, almond shells, olive tree wood, almond tree pruning,	(Girgis et al., 2002; Ganan et al., 2006; Liu et al.., 2007; Marcilla et al., 2000; Ould-Idriss et al., 2011)

2.5 Structure of activated carbon

The structure of activated carbon highly impact adsorption capacity.

2.5.1 Porous structure

Porous properties including surface area, pore size distribution, and pore volume are which essentially determine activated carbon's enhanced adsorption capacity. Activated carbon contains up to 15% ash from mineral particles (Bansal et al., 1988). Every activated carbon has a porous structure. There are various types of activated carbon pore systems, and individual pore sizes and shapes may vary. Activated carbons have pores ranging from a few nanometers to thousands of nanometers. The average width of pores is used to classify them. The average width is typically defined as the radius of a cylindrical pore or the distance between the walls of a slit-shaped pore (Bubanale & Shivashankar, 2017). The International Union of Pure and Applied Chemistry (IUPAC) has formally adopted it. Figure 2.3 shows the typical distribution of pore sizes.

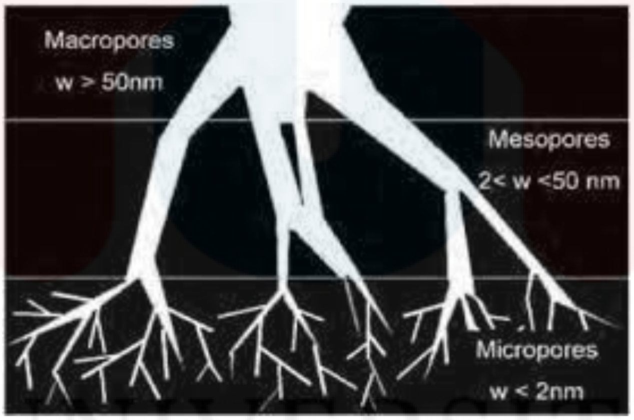


Figure 2.3: Graphical representation of pore structure on activated carbon

(Source: Bubanale & Shivashankar, 2017)

Porous structure affects the adsorption capacity. The porous structure of activated carbon also influences the accessibility and diffusion of molecules within the material. According to Lawtae and Tangsathitkulchai (2021) In the porous structure of activated carbon, the ratio of micropores and mesopores mainly affects the kinetics of methylene blue diffusion. A reduction in the number of mesopores tends to reduce the diffusion rate of methylene blue due to increased mass transfer resistance due to more micropores. Since adsorption on the carbon surface and transport of adsorbates through the pore network occur continuously, a decrease in the adsorption rate results in a decrease in the diffusion flux of dye molecules within the pores, which is caused by the smaller surface area of the larger mesopore volume.

2.5.2 Crystalline Structure

During the carbonization process, activated carbon forms a microcrystalline structure. Compared to graphite, activated carbon has a completely different structure. Graphite has a different interlayer gap than activated carbon. Graphite has an interlayer spacing of 0.335 nm, whereas activated carbon has an interlayer spacing of 0.34 to 0.35 nm. Activated carbon is divided into two categories based on its graphitizing ability: graphitized carbon and non-graphitized carbon. The graphene layers are parallel to each other as graphitized carbon. Because of the weak cross-linking between the neighbouring micro crystallites, the carbon produced was fragile and had a less developed porosity structure. Non-graphitizing carbons are certainly hard. In non-graphitizing carbons, strong cross-linking between crystallites reveals a well-developed micropore structure. When relevant oxygen is present or insufficient hydrogen is present in the early raw materials, non-graphitized structures with strong cross-links occur. (Muthaian Jaya & Clastin Indira, 2022). The structural differences between graphitizing and non-graphitizing carbons are shown in Figure 2.4.

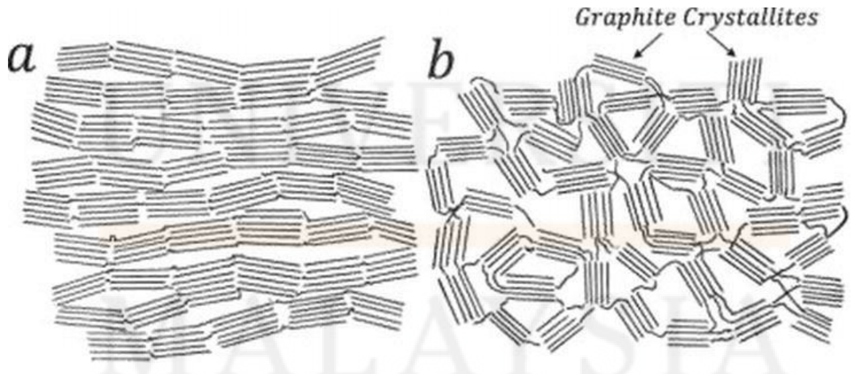


Figure 2.4: The structural difference between graphitizing (a) and non-graphitizing (b) carbons

(Source: Muthaian Jaya & Clastin Indira, 2022)

2.5.3 Chemical Structure

The structure of activated carbon can be both porous and crystalline. It also has a chemical structure correspondingly. The porous structure of activated carbon influences its adsorption capacity. However, it is greatly impacted by a relatively modest number of chemically linked heteroatoms, primarily oxygen and hydrogen. Due to changes in the electron cloud distribution within the carbon skeleton, the adsorption capacity of activated carbon changes, especially for polar compounds. These changes result in the creation of unpaired electrons and incompletely saturated valence states (Muthaian Jaya & Clastin Indira, 2022).

2.6 Classification of Activated Carbon

Activated carbons are complex products that are difficult to categorise based on surface properties, their behaviour, and other fundamental criteria. However, some generic classification is developed based on their size, preparation methods, and industrial applications.

2.6.1 Powered Activated Carbon

Powdered activated carbon (PAC) is generated from organic carbon-rich sources such as coal, wood, and lignite. PAC has an apparent density range of 23 to 46 lb/ft3 and a diameter of less than 0.1 mm. Powdered activated carbons have the advantage of cheaper processing costs and greater operational flexibility. Powder carbons are commonly used in brewing, sewage treatment, power plants, electroplating, and other sectors such as food, beverage, medicine, tap water, sugar, and oil (International, 2019). ASTM, previously known as the American Society for Testing and Materials, uses the term PAC or pulverised AC to describe particles that pass through an 80-mesh screen (0.180 mm). PAC contains small AC particles produced by milling or pulverising AC, with an average size of 0.075 mm and a mean particle diameter (MPD) of 0.045 mm (Ganjoo et al., 2023). The powered activated carbon shown in Figure 2.5

2.6.2 Granular Activated Carbon

Granular activated carbon (GAC) is also referred to activated carbon that is maintained on a 50-mesh filter. Their advantages are that they are tougher and more durable than powdered activated carbon (PAC), reactivated and reused numerous times, easier to handle, and capable of purifying vast amounts of gas or liquid of constant quality. Granular activated carbon finds extensive application in several industries such as gas purification, desiccant, brewing, industrial water treatment, decolorization, and waste gas treatment (Fatmarch, 2019). GAC has a smaller exterior surface than PAC because of its larger particle size. The 0.2 to 5 mm size range of the amorphous particles that make up GAC. While the water passes through a GAC filter, heat is applied to activate the carbon's surface area, removing some contaminants from the water that have been dissolved. (Ganjoo et al., 2023). The granular activated carbon shown in Figure 2.5

Figure 2.5: Powered activated carbon and granular activated carbon

(Source: Hangzhou Nature Technology Co., n.d)

2.7 Qualitative and quantitative analysis of bamboo-derived activated carbon

Quantitative analysis is frequently related with mathematical analysis, in which data are collected, identified, and then calculated for a desired result using a set of statistical methods. A huge sample from data is randomly selected followed by the data are examined. Qualitative analysis involves the examination of evidence that cannot be quantified. These data are about understanding and insights into the characteristics and properties of objects or participants. Qualitative methods are subjective, quantitative methods are objective (Madisha, 2018).

For qualitative analysis, Scanning Electron Micrograph (SEM) understanding pore development phenomena and qualitative data is possible regarding the microstructure of the specimen (Achaw, 2012). The Fourier-Transform Infrared (FTIR) spectra of the activated carbon sample show the presence of oxygen-containing surface functional groups, such as carboxyl, hydroxyl, and carbonyl groups. For quantitative analysis, The BET surface area technique Brunauer, Emmett, and Teller to calculate the activated carbon's interior surface area on pore surface area, pore structure, pore sizes, pore shapes, and pore size distribution. The most common application of XRD is in quantitative analysis. This method enables the direct calculation of the structural parameters (crystallite size and interplanar distance) of carbon materials from their X-ray diffraction patterns, which are the main determinants of their structure (Lee et al., 2021).

2.8 Application of activated carbon in food industry

Gaseous compounds, such as carbon dioxide, oxygen, and relative humidity or moisture must exist in the packing system for the food to be safe and of high quality. These variables affect the deteriorating effects of enzyme reactions, respiration, chemical reactions, microbiological growth, physical change, sensory qualities, the ripening process, and shelf-life on food goods, particularly fresh food that is still breathing. Activated carbon is used in food packaging's a versatile way to maintain or control the gaseous environment within the packaging. To remove important gas molecules from packing systems which are specifically harmful to the safety and quality of different food, activated carbon can be specially tailored for use in specific adsorption applications (Chaemsanit et al., 2018).

2.8.1 The vapor phase molecule scavenging of activated carbon

Veggies, fresh fruits, and things that don't absorb oxygen, like grains, meat, and processed foods are examples of food products that come in two different types of packaging depending on how they behave when they breathe. Fresh produce that is capable of respiration is most suited for passive modified atmosphere packaging (MAP), which creates and maintains the desired environment using the characteristics of the food product and the permeability of the packaging material. The respiration generates CO_2 and keeps this gas at a high level. Active MAP is a relatively new invention. The desired atmosphere is added after air has been removed to create a vacuum. This preserves the newly adjusted atmosphere in the package, together with additives and appropriate packing materials. This type of packaging is used for a variety of food products, including processed foods, medications, fresh produce that is breathing and non-respiring animal products (Trimble, 2022).

For products that respiration, oxygen is a crucial component of the respiration process, which results in the production of ethylene, water vapor, and carbon dioxide as byproducts. Many fresh items can have their postharvest life extended and their quality maintained by removing or inhibiting ethylene in storage conditions. For non-respiring products, oxygen and water vapors content (moisture content) play a significant role in the safety and quality of food products. high levels of oxygen speed up the oxidation process, which causes a change in colour, an off-odor, and nutrient loss. Furthermore, it creates favourable circumstances for the aerobic microorganisms that deteriorate food and cause foodborne sickness. Additionally, high moisture contents offer molecules of free water that might be employed in enzymatic processes, chemical processes, and microbiological expansion, leading to the degradation, spoiling, and decline in product quality. In addition, food's texture changes, becoming shrivelled or squishy. Consequently, the amounts of ethylene, oxygen, and relative humidity or water vapour should be decreased or managed in food packaging in order to maintain the quality and safety of the product. Additionally, the odor of the meal is a significant indicator of the sensory qualities and product's quality in specific products (Chaemsanit et al., 2018).

2.8.2 Adsorption of odor by activated carbon

Odour compounds have kinetic energy, which when adsorbed to the surface of the media is converted to heat. The kinetic energy of the molecules increases with increasing temperature of the odorous air, increasing the temperature of the media bed as a result. The effectiveness of odour elimination decreases as the bed temperature rises above 40 degrees C (Mark Ludwigson, 2023). Off-odors (off-flavors) and taints can seriously harm food, resulting in financial losses through product recalls, decreased consumer confidence, and possibly a tarnished brand image. Numerous in-depth reviews on this topic have been produced as a result of its significance to the food business. Foods can develop unpleasant smells due to oxidation, light-catalyzed processes, nonenzymatic browning, chemical reactions and interactions between food components, enzymatic reactions, and microbiological decomposition. The use of powder activated carbon in the packaging for irradiated ground beef could get rid of the odour that the irradiation procedure caused. Additionally, based on sensory evaluation, most consumers like irradiation ground beef preserved with activated carbon over normal beef (Chaemsanit et al., 2018). As shown in the table 2.4, some major volatile odour components in packaging containing activated carbon are reduced in both non-irradiated and irradiated samples.

Table 2.4: Volatile odour components in packaging containing activated carbon are reduced in both non-irradiated and irradiated samples (Source: Chaemsanit et al., 2018)

	Non-irradiated raw meat		Irradiated raw meat	
Compound	Without AC	With AC	Without AC	With AC
Pentane	42.50	31.87	84.47	66.00
3-Methylbutanal	25.37	-	72.23	50.07
2-Methylbutanal	28.77	-	55.17	45.73
Ethanol Benzene	31.23	15.67	-	-
Hexanal	222.07	227.10	297.33	384.33
Total volatile	551.13	565.04	747.93	738.71
Odor preference	2.25b	4.75a	3.25b	6.00a

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials and chemical

Bamboo (*Gigantochloa albociliata*) collected from Universiti Malaysia Kelantan, Jeli Campus). Preparation of bamboo raw were cut into suitable small particle sizes. Bamboo was collected the bamboo needed to carry out carbonization process and activation process to form bamboo-derived activated carbon. The chemicals and reagent needed for activation bamboo-derived activated carbon was outlined in detail with function in Table 3.1.

Table 3.1: List of chemicals used in the research

No.	**Chemical**	**Manufacturer**
1.	Potassium carbonate (K_2CO_3)	Sigma-Alorich

3.2 Apparatus and equipment

Apparatus used for this study are beaker (100 mL), plastic strainer, knife, spatula, cutting board. Equipment used for this study are high temperature tube furnace, oven, wood band saw, automated gas sorption analyzer (BET), scanning electron microscope (SEM), FTIR spectroscopy, X-ray diffractometer.

3.3 Research flowchart

The research flow chart presented in Figure 3.1 were divided into three stages. Stage 1 was preparation of bamboo; stage 2 was the carbonization and activation the bamboo using furnace and stage 3 was for analysis of the obtained experimental.

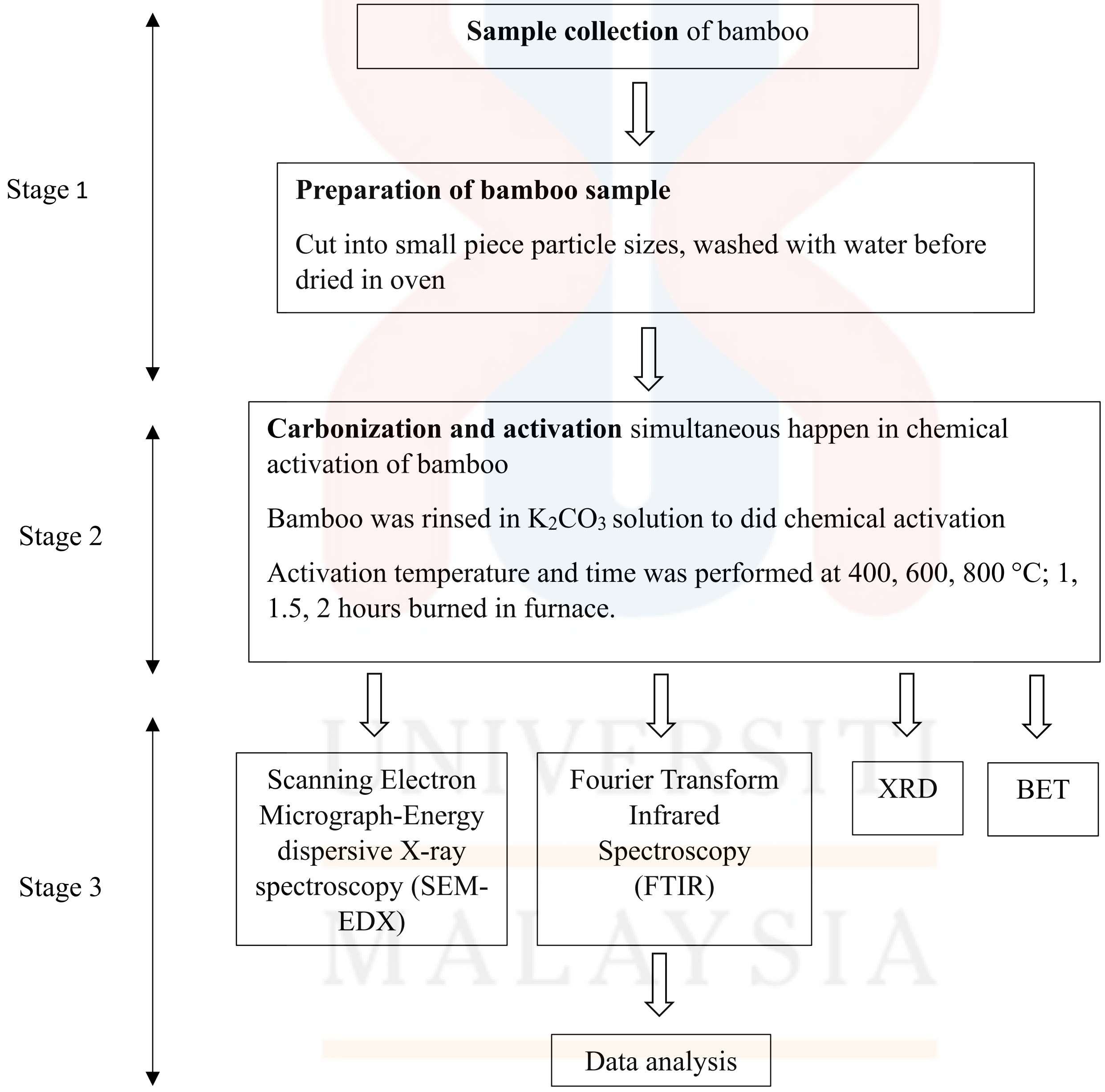


Figure 3.1: The research flowchart of the carbonization and activation the bamboo to activated carbon

3.4 Preparation of activated carbon

The bamboo samples were prepared to the desired size by using a wood band saw to cut the long bamboo into 1.5-2 cm pieces. The bamboo was cut in 1.5-2 cm by using a wood band saw. They were then washed with water and dried at a temperature of 70°C until the weight was constant. The dried bamboo was mixed well with a potassium carbonate (K_2CO_3) (powder mixed with distilled water into a solution) until the mixture was uniform, the bamboo was removed from the potassium carbonate solution and dried in an oven at 70°C for 24 hours. The mixture was heated with a furnace (1500°C) at a heat rate of 10/°C min, and the activation temperature used here was varied between 400°C, 600°C, and 800°C. When the activation temperature is reached, it is controlled constant for a given period of time, such as 1 hour, 1.5 hours and 2 hours. After the sample was cooled to room temperature and circulated with nitrogen, it was washed and filtered several times until the filtrate reached a neutral consistency. After the activation process, the activated carbon was washed with hot distilled water and filtered to eliminate any leftover activating agent. The sample was placed in a beaker and dried in a universal oven at 70°C for around 24 hours. Finally, the sample was converted into powder activated carbon using a plastic strainer

3.5 Chemical activation of adsorbent

Mixed activating agent. The impregnation process was utilized at a constant ratio of 1:1 (wt%). The definition of the impregnation ratio was the dry weight of the bamboo, W_B, divided by the dry weight of the activation agent, W_{K2CO3}. The reagents were thoroughly absorbed into the bamboo after the mixer was stirred until completely homogeneous and dried in a universal oven for 24 hours. The impregnation bamboo samples were heated in an oven at temperatures of 70°C to achieve good penetration of chemicals into the interior of the precursor. Bamboo 50 grams(g) of was mixed with Potassium carbonate 50 g of powder with added 500 mL of distilled water. It was subsequently cooled to consistent temperatures below 100° C.

3.6 Effect of carbonization temperature

The carbonization and activation processes happen simultaneously during chemical activation. Among the most important is the carbonization temperature it is important elements determining the development of porosity during the activation process. ACs were tested at temperatures ranging from 400 to 800 °C (400, 600, and 800 °C) for each chemical impregnation. The optimal carbonization temperature varies depending on the chemical used in impregnation.

3.7 Effect of Holding Time

Holding time was the duration of the sample kept at final carbonization/activation temperature. In order to examine the effect on porous characteristics, the holding period at the final temperature was adjusted throughout a range of 1-2 hours (1, 1.5, and 2 hours).

3.8 Characterization Method

3.8.1 Scanning Electron Micrograph-Energy dispersive X-ray spectroscopy (SEM-EDX) SEM-EDX analysis

The surface morphology of the bamboo-derived activated carbon will be analysed by using a scanning electron microscope (SEM) with an EDX spectrometer XT microscope software was used for data acquisition and analysis. The samples with average particle sizes of approximately 10 to 100 µm were used Software for microscopes was used to collect and analyse data.. Magnification that used are x100, x300, x500, x1000, x1500.

3.8.2 Brunauer-Emmett-Teller (BET) analysis

The automated gas sorption analyzer's nitrogen adsorption-desorption isotherms, which were obtained after nitrogen was degassed at 200°C for 3.1 hours and used as the adsorbate, determine the pore size distribution, specific surface area, and porosity of the materials. By physically adsorbing a gas to the surface of the solid, bamboo-derived activated carbon powder's specific pore size, surface area, and pore volume were measured. A monomolecular layer on the surface's corresponding amount of adsorbate gas was calculated. The main cause of physical adsorption was the relatively weak van der Waals forces between the test powder's adsorbent surface area and the adsorbate gas molecules. The measurement was performed at liquid nitrogen temperature.

3.8.3 Fourier-Transform Infrared Spectroscopy (FTIR) analysis

The FTIR analysis approach scans test samples with infrared light to look for their chemical characteristics. In samples of activated carbon, it was utilized to determine the element and functional group.. FTIR spectra was computed by using FTIR (Thermo Fisher Scientific, USA). The FTIR spectra were recorded in the spectral range of 4000- 400 cm^{-1} at a resolution of 4 cm^{-1} and 254 scans. FTIR equipped with OMNI-sample Attenuated Total Reflection (ATR) smart accessory and coupled to a diamond crystal operating at 45° incidence angle A pinch of powdered bamboo activated carbon was placed in an FTIR spectrometer. The spectrometer lusters infrared light directly onto the sample and measures the frequency of the infrared beam absorbed by the sample. Subsequent, sample was identified through a reference database containing thousands spectrum.

3.8.4 X-ray diffractometer (XRD)

X-ray diffractometer was used to investigated the crystal phases and crystallite sizes of the three samples. Following initial concentration on the fixed sample, the X-rays were diffracted. Plotting the variations in diffracted X-ray intensity versus the rotation angles of the sample was done after measurements and records were made. Peak height or peak area was used to qualitatively analyze the resulting X-ray diffraction pattern. These were used to determine the degree of crystallization and the particle sizes.

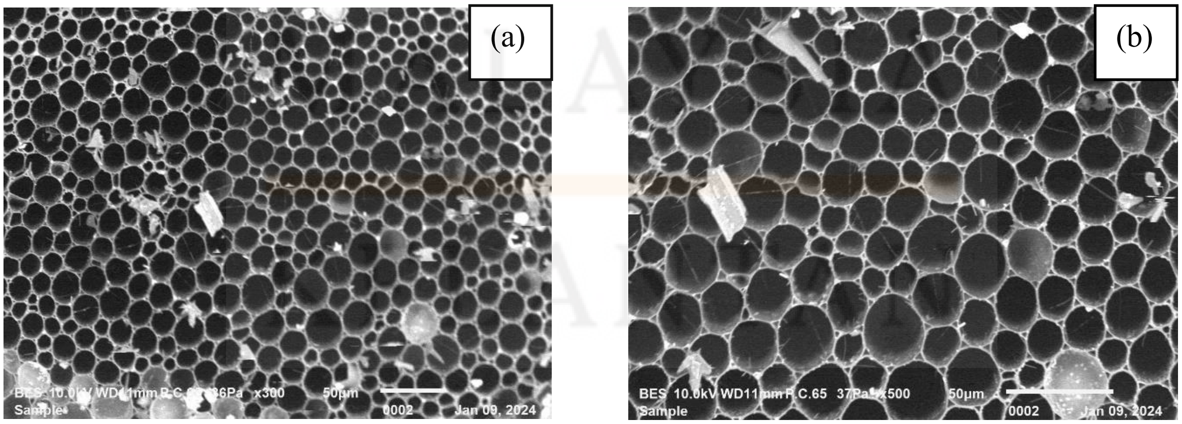
CHAPTER 4

RESULTS AND DISCUSSION

This chapter discuss the results of the Scanning Electron Micrograph-Energy dispersive X-ray spectroscopy (SEM-EDX) analysis, Brunauer-Emmett-Teller (BET) analysis, Fourier transformed infrared spectroscopy (FTIR) analysis, and X-ray Diffraction (XRD) for bamboo-derived activated carbon. SEM demonstrates that its surface features a considerable number of holes. The Activated carbon (AC) samples' BET surface area and pore size distribution were measured. FTIR was used to examine functional groups on the surface of samples of activated carbon. XRD on AC to determine crystallinity or amorphous nature.

4.1 Scanning Electron Micrograph (SEM) analysis

Surface morphology show in figure 4.1 the result of SEM imaging for bamboo-derived activated carbon activation temperature 400 °C and activation time 1 hour (AC 400 °C/1 hr) in 300x, 500x, 1000x and 1500x magnification.



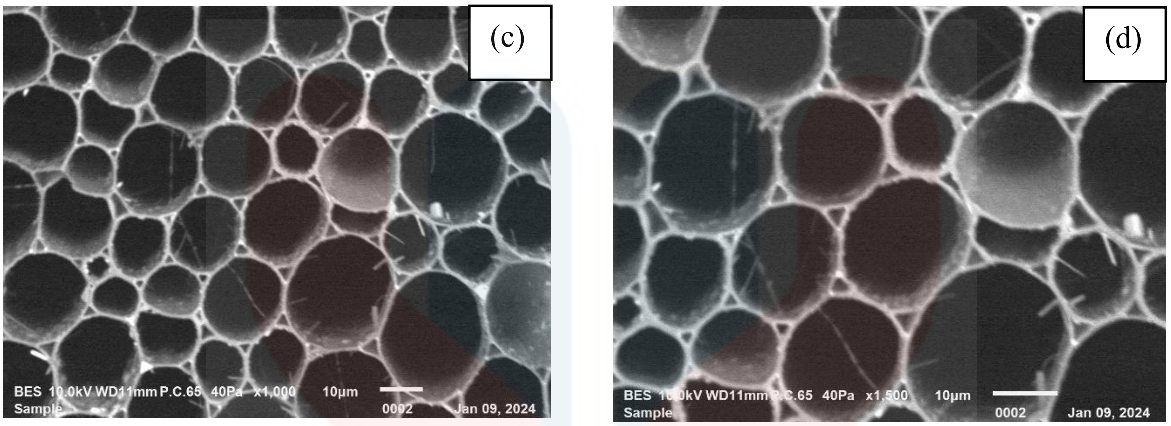


Figure 4.1: SEM image for bamboo-derived activated carbon (AC 400 °C/1 hr) at (a) 300x, (b) 500x, (c) 1000x and (d) 1500x magnification

In Figures 4.1 (a) and (b), images show the different pore spaces observed in bamboo-derived activated carbon. The perimeter of the void is clearly visible, no damage is seen and the pore size is almost identical. The pore space is clear, but the pore structure is better visualized and the pore size and adsorption capacity can be inferred. However, for the 1000x (c) and 1500x (d) magnifications there are finer details on the pore size, and good evidence of the development of the network and pore structure. The surface of activated carbon has well-developed pores with very little pore wall collapse which demonstrated well-developed longitudinal cavities and suggested a total internal volume, porosity, and high adsorption capacity, are the consequence of the increased magnification. The adsorption capacity of the sample increases. Accessible open pores indicate that these properties are critical for efficient adsorption of target molecules.

In figure 4.2 the result of SEM imaging for bamboo-derived activated carbon activation temperature 600 °C and activation time 1.5 hours (AC 600 °C/1.5 hrs).

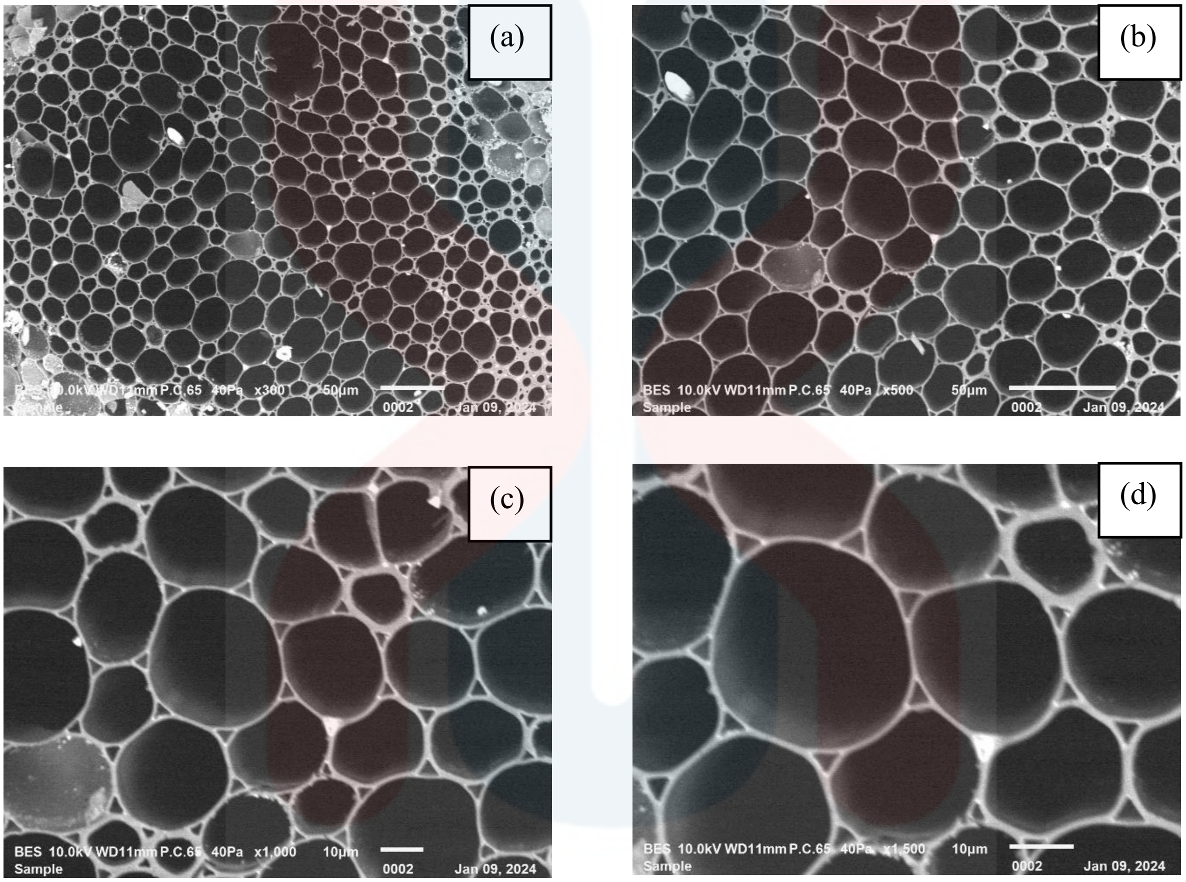


Figure 4.2: SEM image for bamboo-derived activated carbon (AC 600 °C/1.5 hrs) at (a) 300x, (b) 500x, (c) 1000x and (d) 1500x magnification

In figures 4.2 (a) and (b), images show the perimeter of the void has slightly collapsed, but is still clearly visible without damage. The pore sizes are inconsistent. There are obviously several large pores that are broken together by two pores, while other small pores seem to be pulled apart by the large pores, making them squeeze and become smaller. When comparing activated bamboo charcoal to regular bamboo charcoal, SEM pictures show a more even distribution of pores (Sucipta et al., 2021). This can be ascribed to the activation process, which causes more pores to develop and expand, enhancing the charcoal's surface area (Sucipta et al., 2021). For the 1000x (c) and 1500x (d) magnifications there are finer details which showed total internal volume and porosity, well-developed longitudinal cavities, inferring high adsorption capacity. The adsorption capacity of the sample increases. Accessible open pores indicate that these properties are critical for efficient adsorption of target molecules. These can be considered to have good pores and good adsorption potential.

In figure 4.3 the result of SEM imaging for bamboo-derived activated carbon activation temperature 800 °C and activation time 2 hours (AC 800 °C/2 hrs).

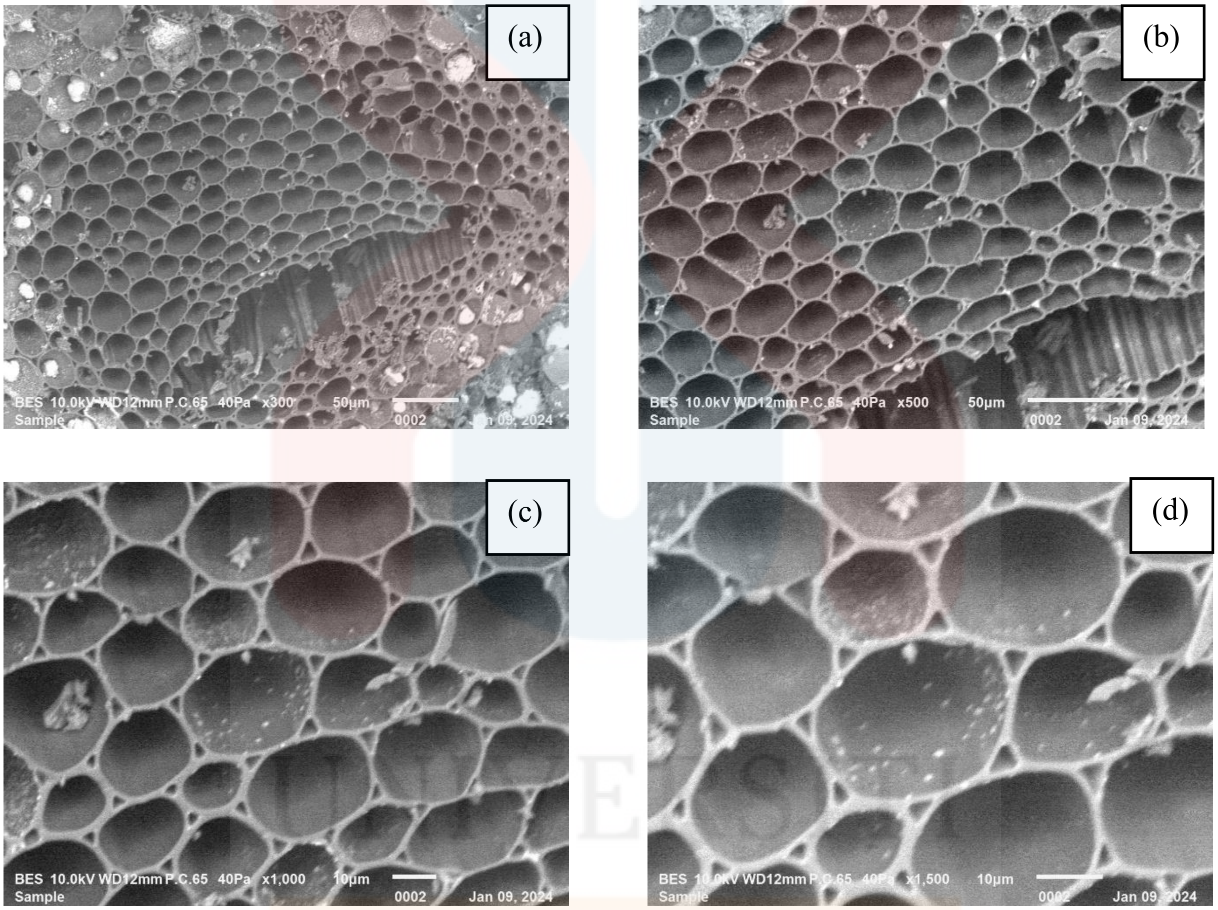
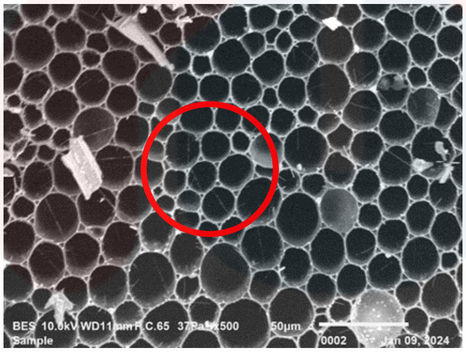
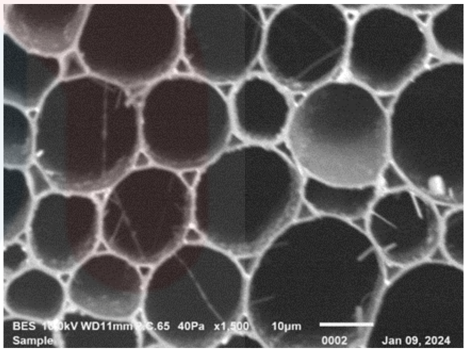
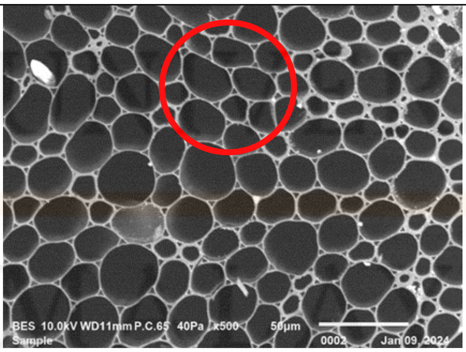
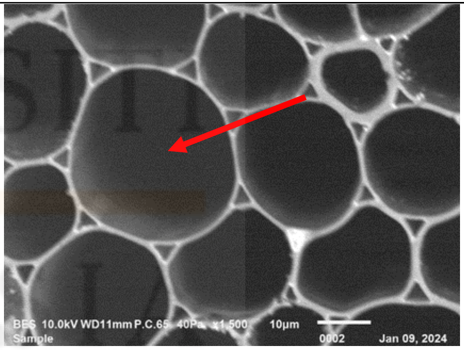


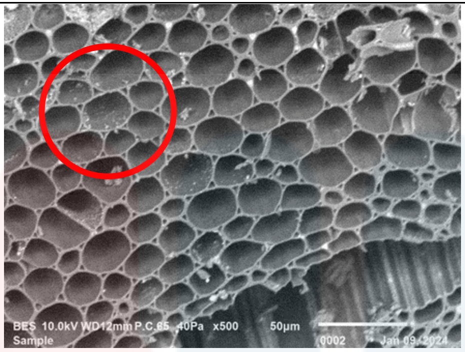
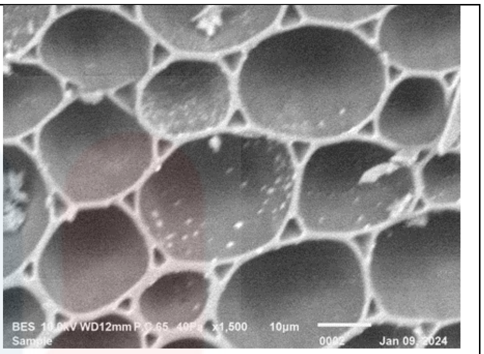
Figure 4.3: SEM image for bamboo-derived activated carbon (AC 800 °C/2 hrs) at (a) 300x, (b) 500x, (c) 1000x and (d) 1500x magnification

Overall, it looks like an oval-shaped pore with irregular sizes and a bit of collapse show in figures 4.3 (a) and (b). A magnified view in figure 4.3 (c) and (d) however, demonstrates that the impacts of the activation procedure they went through are responsible for the enormous pore size and spacing, microstructure, and well-distributed network. Obvious ash residue produced by AC due to high-temperature combustion can be seen inside pore walls. The activation process uses potassium carbonate to promote the formation of micropores and produce. Impregnated K_2CO_3 limits tar formation and enhances dehydration and cross-link formation (Chen et al., 2017). When the activation temperature reaches 800°C, obvious cracks and collapse of the pore structure appear, this is because the high activation temperature led to an excessive etching reaction. (Gao et al., 2021). The adsorption capacity of the sample is relatively low. Accessible open pores indicate that these properties are critical for efficient adsorption of target molecules. These can be considered to have porosity and adsorption potential.

Comparison morphology of bamboo-derived activated carbon in different temperature 400, 600, and 800 °C and magnification 500x and 1500x was shown in Table 4.1.

Table 4.1: Comparison morphology of bamboo-derived activated carbon in different temperature and magnification

Temperature (°C)	**500x**	**1500x**
400 °C		
600 °C		

800 °C		

From the figure 500x magnification of activated carbon at 400 °C. The pores of AC are regular and round in shape, with mainly small pores. When the temperature increases to 600°C, the holes begin to deform and take on irregular shapes, and oval-shaped microspores appear. A clear difference can be seen from the circled area. When the temperature reaches 800°C, the entire AC gradually burns into ash powder. From the 1500x magnification of 800°C it can be seen that there is residual ash powder in the wells compared to the results from the other two temperatures. Morphological observation based on microstructure, the optimal temperature for making activated carbon is 400 °C, which depends on the structural pores formed after the entire carbonization and activation process. At 400°C, AC has perfect pores shape minimal pore wall collapse which can better exert its ability to adsorb or adsorb substances.

4.1.1 EDX (energy dispersive X-ray spectroscopy) analysis

EDX spectra elemental content percentage for carbon (C), oxygen (O_2), silica (Si), and potassium (K) was shown in Figure 4.4.

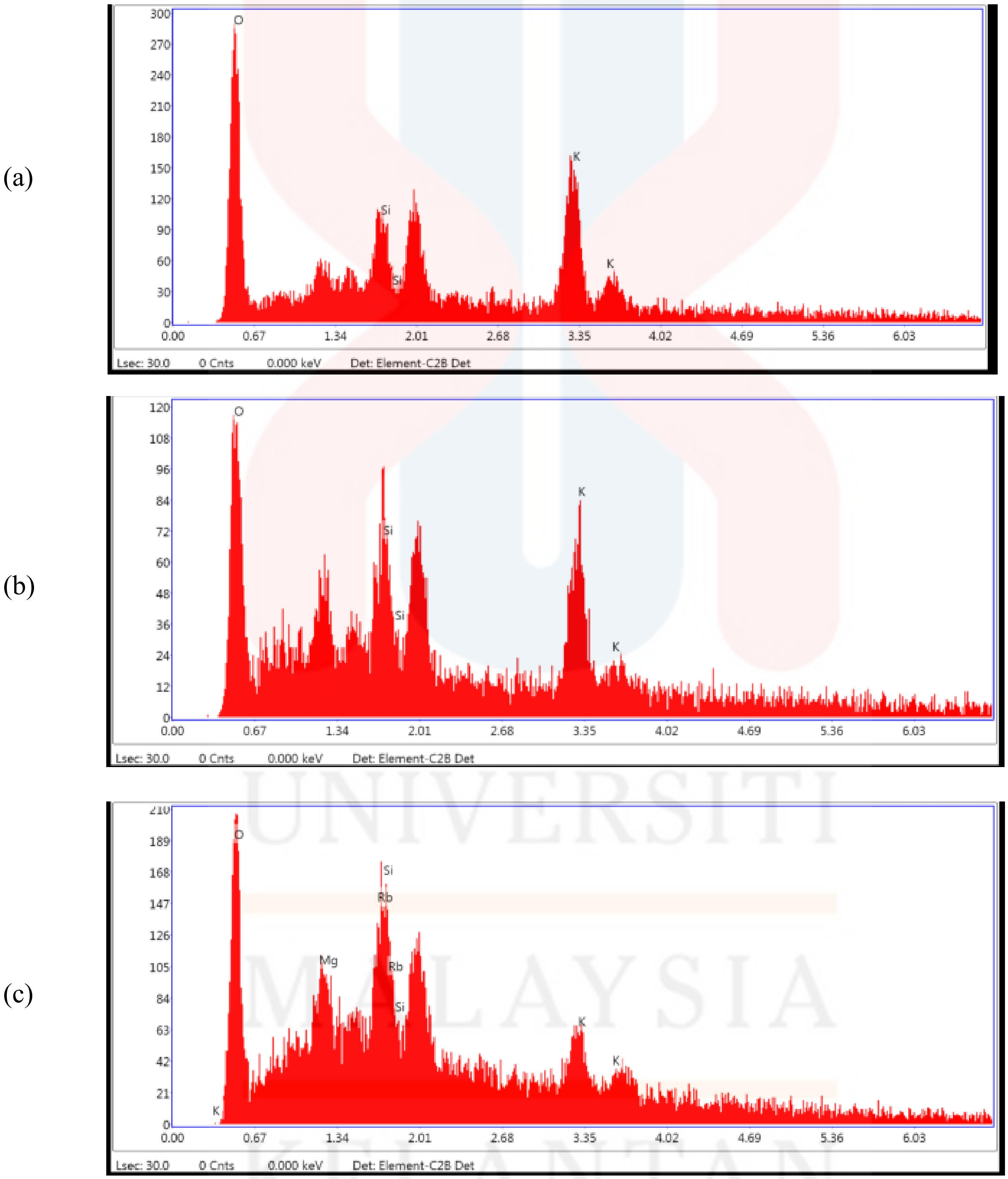


Figure 4.4: EDX spectra of (a) AC 400 °C/1 hr, (b) AC 600 °C/1.5 hrs, (c) AC 800 °C/2 hrs

The analysis of elemental composition and chemical characterization of samples was shown in Table 4.2.

Table 4.2: The elemental composition of bamboo-derived activated carbon

Element contents	AC 400 °C/1 hr		AC 600 °C/1.5 hrs		AC 800 °C/2 hrs	
	Weight (%)	Atoms (%)	Weight (%)	Atoms (%)	Weight (%)	Atoms (%)
Carbon	0.01	0.03	0.04	0.08	0.05	0.13
Oxygen	36.20	56.89	27.65	46.35	27.24	54.13
Silicon	8.22	7.36	14.70	14.04	0.02	0.02
Potassium	55.56	35.72	57.62	39.53	32.10	26.10

There are elements contained oxygen, carbon, potassium and also silicon in bamboo-derived activated carbon. At the same time, the formation of oxygen was caused by the creation of bonds between oxygen and carbon during the physical activation of CO_2, while the presence of silicon element is related to its appearance in biomass (Farma et al., 2021). The element potassium belongs to potassium carbonate (K_2CO_3) activators for chemical activation. This content is excessive compared to other elements in the three samples. All AC samples was not dominant composition contribute element carbon but relative increased carbon content with increasing time and temperature. Higher carbon content is generally associated with greater adsorption capacity. AC 800 °C/2 hrs has greater adsorption capacity. The more severe breakdown of heteroatom such as oxygen functions at higher temperatures was attributed to the reduction in heteroatom contents with increasing temperature (Shi et al., 2019).

4.2 Brunauer-Emmett-Teller (BET) analysis

The Brunauer-Emmett-Teller (BET) theory explains the fundamental analytical method for figuring out the specific surface area of different materials is based on the physical adsorption of gas molecules on solid surfaces. The BET surface areas corresponding to the chemical activation with different activation temperature and time was shown in Figure 4.5.

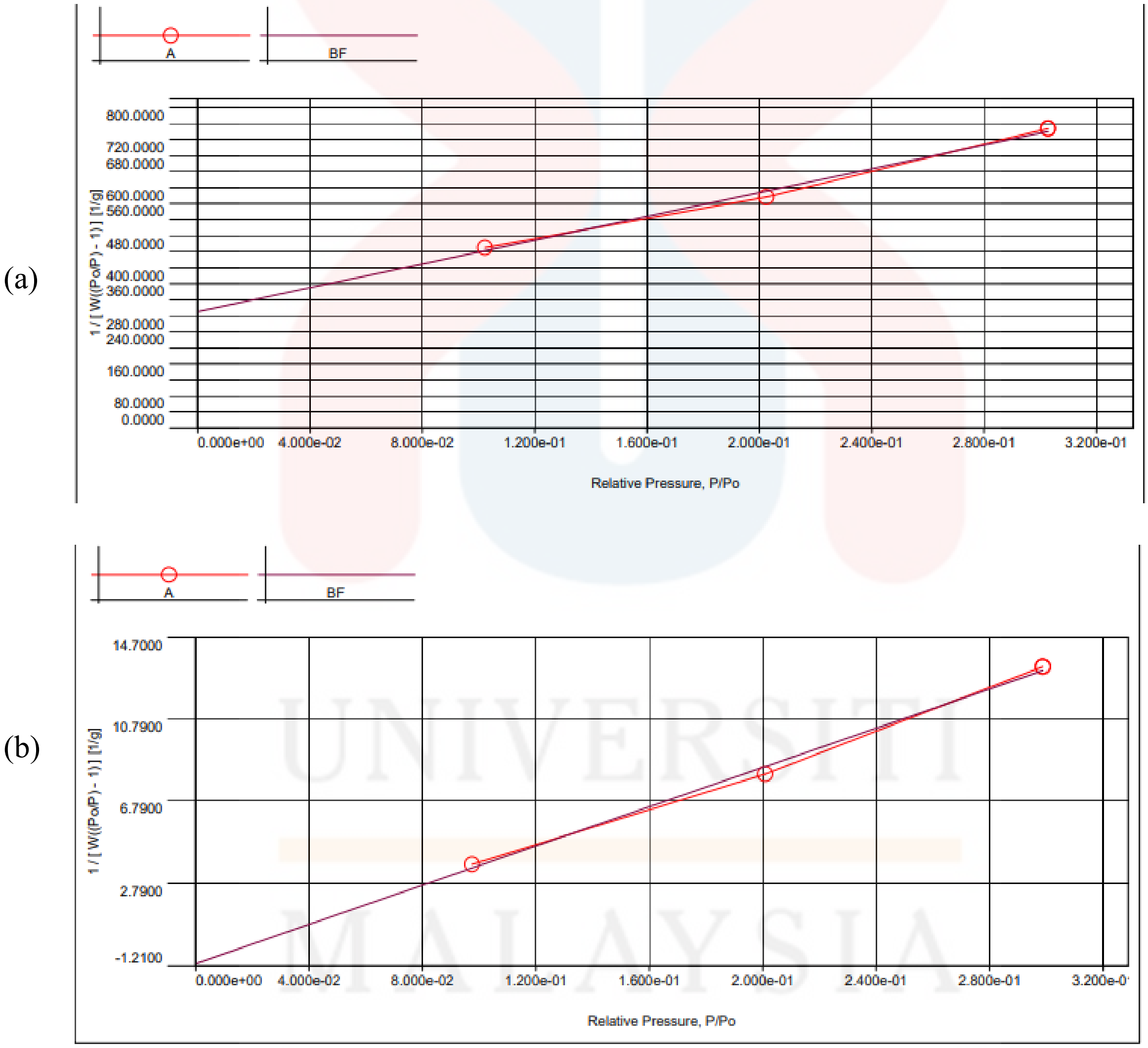


Figure 4.5: BET surface area (a) AC 400 °C/1 hr, (b) AC 800 °C/2 hr

Table 4.3: BET surface area, average pore size and total pore volume

Sample code	**BET surface area (m^2/g)**	**Average pore size (Å)**	**Total pore volume (cc/g)**
AC 400 °C/1 hr	1.962	6.83818×10^{2}	3.498×10^{-3}
AC 800 °C/2 hrs	74.972	6.30878×10^{2}	4.912×10^{-2}

The BET surface areas corresponding to the chemical activation with different activation temperature and time was shown in Figure 4.5. The BET surface area of activated carbon is critical because, like other physicochemical properties, it may strongly influence the behavior and reactivity of activated carbon. The current results, as tabulated in Table 4.3 suggested that the modified AC possess greater surface area and also pore volume. Total pore volume for AC 400 °C/1 hr is 3.498×10^{-3} cc/g while AC 800 °C/2 hrs yielded 4.912×10^{-2} cc/g.

The surface areas of porous activated carbon AC 400 °C/1 hr and AC 800 °C/2 hrs are 1.962 cm^2/g and 74.972 m^2/g, respectively. The result indicates that surface areas of porous activated carbon AC 800 °C/2 hrs with 2 hours and 800 °C activation are higher than 1 hour and 400 °C activation. The higher surface area may be due to the use of higher activation temperatures to open restricted pores. Activation produced carbon with a higher surface area due to the microporous structure was significantly developed. The pore sizes of AC 400 °C/1 hr and AC 800 °C/2 hrs were mainly between 630 Å and 690 Å. The average pore sizes of were 6.83818×10^{2} Å and 6.30878×10^{2} Å.

4.3 Functional group determination using Fourier Transform Infrared Spectroscopy (FTIR) analysis

Fourier Transform Infrared Spectroscopy (FTIR) is an effective analytical technique for rapidly identifying the chemical family of a substance. The sample's absorbance of infrared light is measured and a spectrum is generated based on the functional groups in the material. Studying the peak between the frequencies where specific bond vibrations occur. An infrared optical table, a computer, and a printer comprise the basis for the FTIR working concept. In this study, bamboo was activated to produce activated carbon was analyzed by using FTIR for to identify functional group of each compound.

Studies of FTIR spectroscopy in bamboo-derived activated carbon powder were conducted between wavelengths ranged from 4000 cm^{-1} to 400 cm^{-1}. Figure 4.9 shows the FTIR spectra of bamboo-derived activated carbon powder with activation temperature 400 °C and activation time 1 hour (AC 400 °C/1 hr). Based in Figure 4.6, the absorption band occur at 3355.65 cm^{-1} , 2114.03 cm^{-1} , 1574.73 cm^{-1} , 1373.39 cm^{-1} , 1108.56 cm^{-1}

Figure 4.6: FTIR spectra of bamboo-derived activated carbon powder 400 °C/1 hour

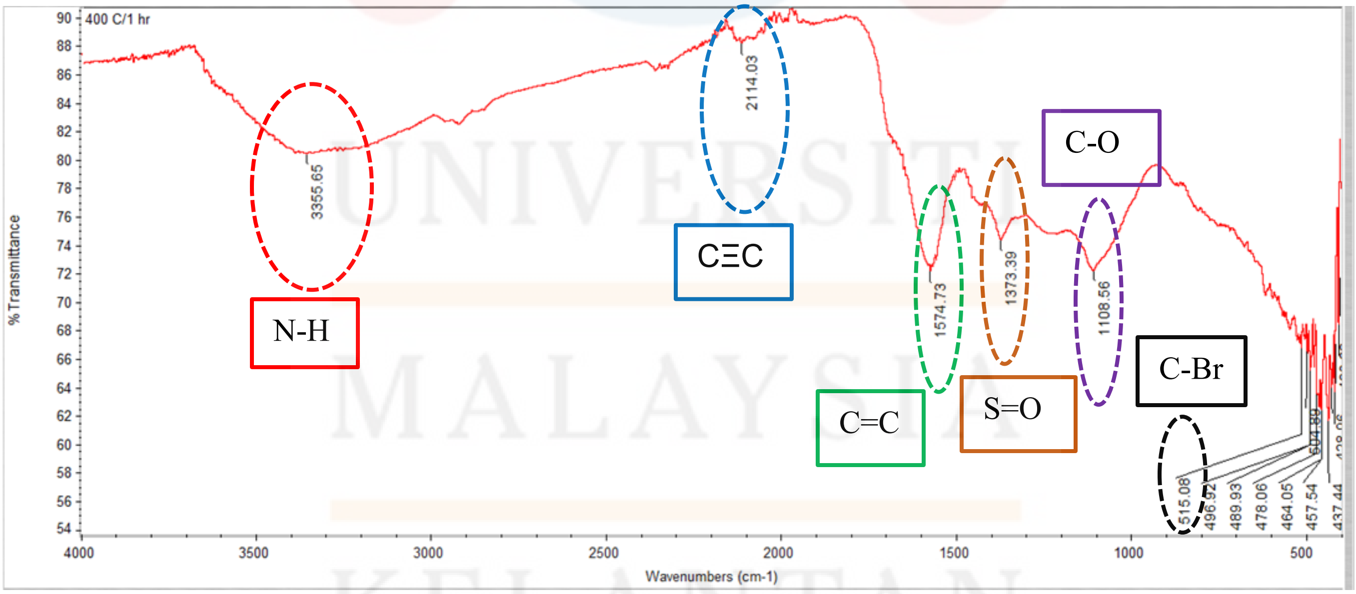


Table 4.4: Functional compounds of bamboo-derived activated carbon powder, 400 °C/1 hour analyzed by using FTIR (Chart, n.d)

No.	Wavelength (cm^{-1})	Type of vibration	Functional compound
1.	515.08	C-Br stretching	Halo compound
2.	1108.56	C-O stretching	Secondary alcohol
3.	1373.39	S=O stretching	Sulfonate
4.	1574.73	C=C stretching	Cyclic alkene
5.	2114.03	CΞC stretching	Alkyne
6.	3355.65	N-H stretching	Aliphatic primary amine

The purpose of the FTIR test was to determine the functional group of activated carbon. there are different spectral peaks. Figure 4.6 was in wave length range of 515.08 cm^{-1} to 3280.59 cm^{-1}, and there were 6 functional compounds found (refer Table 4.4). The band at 2114.03 cm^{-1} and 3355.65 cm^{-1} would be related to alkyne or to aliphatic primary amine group. Besides, the band at 1574.73 cm^{-1} would be related with stretching vibrations of C=C. Carbon has a C=C bond, but it did not appear after activation. At wave number 1573 cm^{-1}, a new peak of activated carbon showed evidence of C-C stretching as a result of newly produced aromatic rings. This provided more evidence that the creation of new pores led to the development of activated carbon (Qanytah et al., 2020). The peaks below 1500 cm^{-1} indicate the samples' fingerprint. Aromatic substitution was shown to be linked with the absorbance at wavenumbers 500 to 800 cm^{-1}. The presence of C=O (aldehydes) was indicative of hemicellulose, whereas the significant absorption of -OH and C-O indicated cellulose (Qanytah et al., 2020). The presence of strong C-Br peak band was observed at 515.08 cm^{-1}, which is due to the bromine impurity introduced into the sample in the form of KBr during the particle formation of the FTIR process (Chigova & Mudono, 2022). Peaks at nearly 1100 cm^{-1} are attributed to the C-O stretching in range 1124-1087 cm^{-1} at the band 1108.56 cm^{-1}.

Studies of FTIR spectroscopy in bamboo-derived activated carbon powder with 600 °C for the activation temperature and 1.5 hours for the activation duration (600 °C/1.5 hrs) were conducted between wavelengths ranged from 4000 cm^{-1} to 400 cm^{-1}. In this study, FTIR analysis was performed, increasing the temperature to 600 °C and extending the activation time to 1.5 hrs to obtain optimal parameters for comparison with the first sample. Based in Figure 4.7, the absorption band occur at 2359.35 cm^{-1} , 2117.56 cm^{-1} , 1996.69 cm^{-1} , 1895.19 cm^{-1} , 1559.36 cm^{-1} , 1077.76 cm^{-1} , 872.71 cm^{-1} and 533.71 cm^{-1} .

Figure 4.7: FTIR spectra of bamboo-derived activated carbon powder 600 °C/1.5 hours

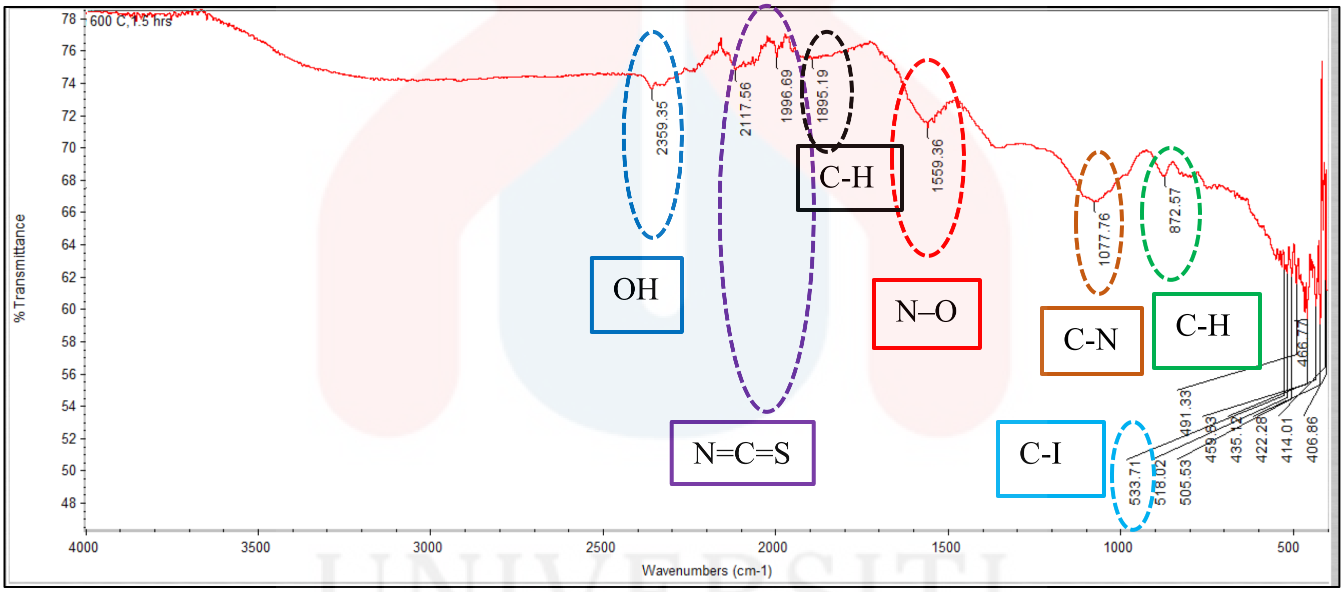


Table 4.5: Functional compounds of bamboo-derived activated carbon powder, 600 °C/1.5 hours analyzed by using FTIR (Chart, n.d)

No.	Wavelength (cm^{-1})	Type of vibration	Functional compound
1.	533.71	C-I stretching	Halo compound
2.	872.57	C-H bending	1,2,4-trisubstituted
3.	1077.76	C-N stretching	Amine
4.	1559.36	Assumed N–O stretching	Assumed aromatic nitro compounds
5.	1895.19	C-H bending	Aromatic compound
6.	1996.69	N=C=S stretching	Isothiocyanate
7.	2117.56	N=C=S stretching	Isothiocyanate
8.	2359.35	OH stretching	Alcohols & Phenols

Figure 4.7 the functional groups (AC 600 °C/1.5 hrs) was shown in the wave length range of 533.71 cm^{-1} to 2359.35 cm^{-1}, and there were 8 functional compounds found (refer Table 4.10). The presence of broad bands at 2359.35 cm^{-1} can be attributed to OH stretching. The hydroxyl group which is attached O-H represents the functional group on the activated carbon were observed at range 2359.35-2926.78 cm^{-1}, the band at 2359.35 cm^{-1} (Thirumaran & Prakash, 2015). It is mainly attributed to the stretching of -OH groups caused by the existence of molecules of water (Chigova & Mudono, 2022). Regarding the C-H bond's symmetric and asymmetric stretching vibration, the peak in the AC spectrum at 1895.19 cm^{-1} indicates the presence of organic remains from the precursor. In the AC sample, The peak for C=O bonds disappears because the activation process is carried out at 973 K, consequently, there is significant decomposition of the organic materials (Arias-Niquepa et al., 2019). Small peaks obtained at 2117.56 cm^{-1} and 1996.69 cm^{-1} are the characteristics of N=C=S stretching. The peaks at 533.71 cm^{-1} are assigned to C-I stretching halo-compound. The presence of band was observed at 872.57 cm^{-1} which confirms of C-H bending. The presence of medium bands at 1077.76 cm^{-1} can be attributed to stretching of C-N. The absorption band at 1559.36 cm^{-1} could not be found, but it is close to the assumed peak

assigned to 1555-1485 cm^{-1} (aromatic nitro compounds) related to N-O stretching (Akinbile et al., 2023).

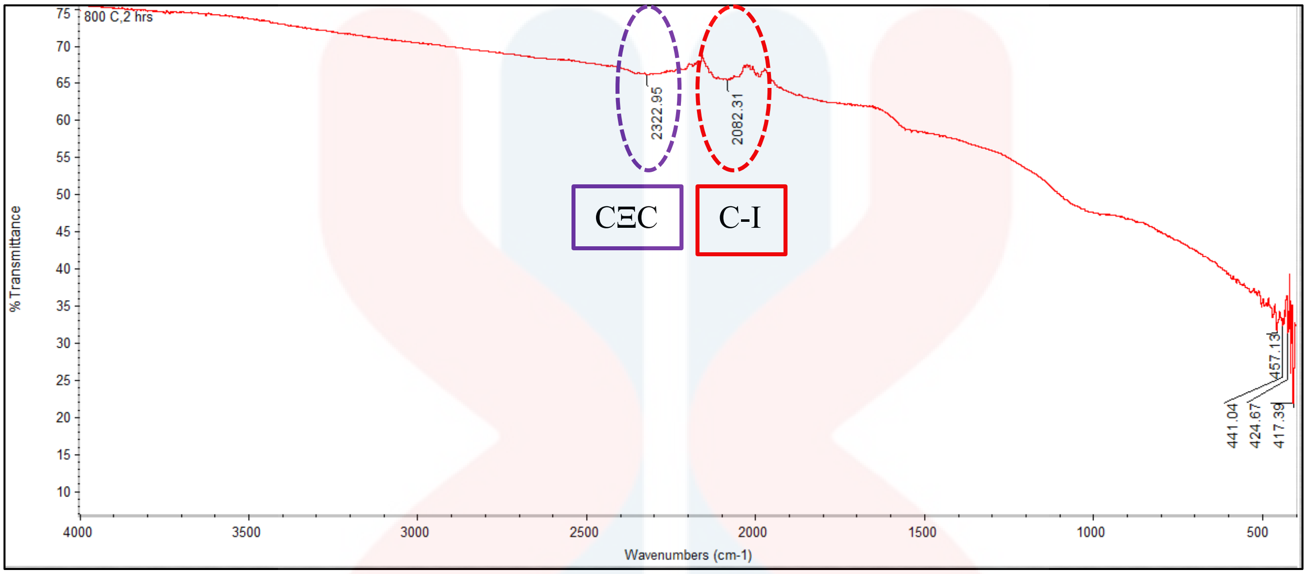


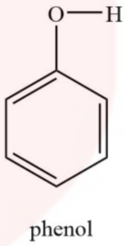
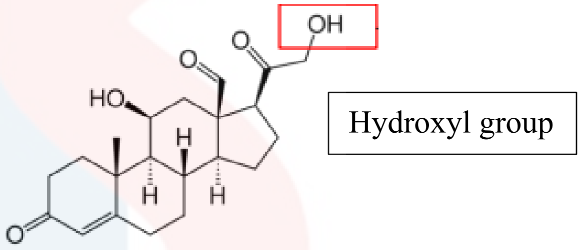
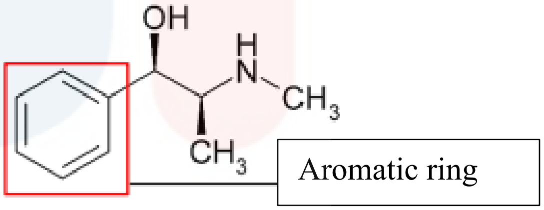
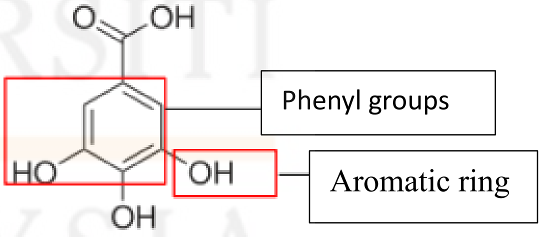
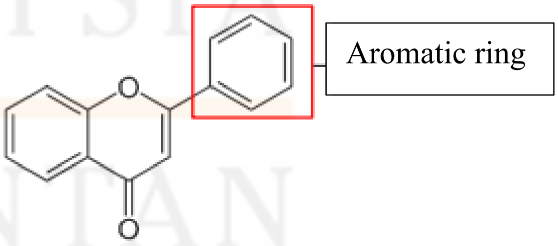
Figure 4.8: FTIR spectra of bamboo-derived activated carbon powder 800 °C/2 hours

Table 4.6: Functional compounds of bamboo-derived activated carbon powder, 800 °C/2 hours analyzed by using FTIR (Chart, n.d)

No.	**Wavelength (cm^{-1})**	**Type of vibration**	**Functional compound**
1.	2082.31	C-I stretching	Halo compound
2.	2322.95	CΞC stretching	Alkyne

Figure 4.8 was in wave length (AC 800 °C/2 hrs) range of 2082.31 cm^{-1} to 2322.95 cm^{-1}, and there were 2 functional compounds found (refer Table 4.6). The absorption band at 2322.95 cm^{-1} could not be found, but it is close to the assumed peak assigned to 2260-2100 cm^{-1} would be related to alkyne group. The presence of strong to medium intensities band was observed at 2082.31 cm^{-1}.

Table 4.7: Active components present in FTIR analysis of bamboo-derived activated carbon powder with the structure and functional group

Active component	Structure and functional group
Ethanol	Alcohols & Phenols  an alcohol  phenol
Steroid	
Alkaloid	
Tannins	
Flavonoids	

FTIR spectroscopy were compared in bamboo-derived activated carbon with difference activation temperature and activation time, 400 °C/1 hour, 600 °C/1.5 hours and 800 °C/2 hours. It was conducted between wavelengths ranged from 4000 cm^{-1} to 500 cm^{-1}. Figure 4.12 shows the comparison of FTIR spectrum.

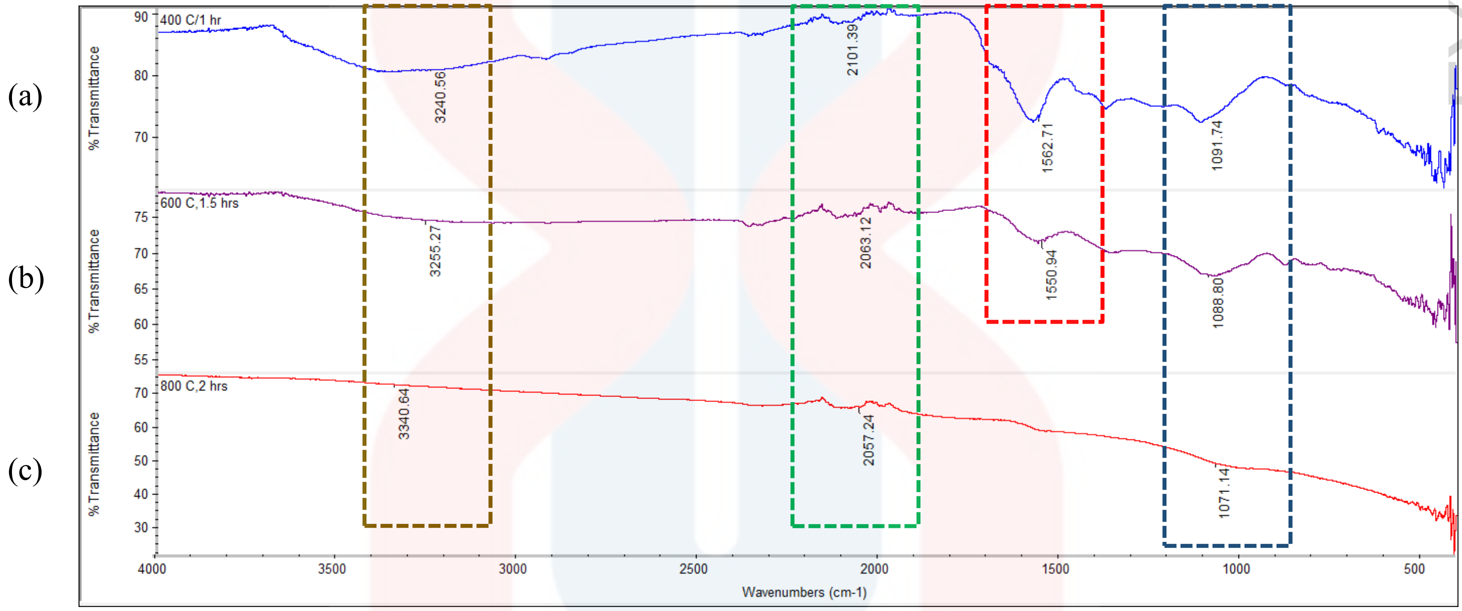


Figure 4.9: Comparison of FTIR spectrum of (a) Bamboo-derived activated carbon powder (AC 400 °C/1 hr), (b) Bamboo-derived activated carbon powder (AC 600 °C/1.5 hrs) and (c) Bamboo-derived activated carbon powder (AC 800 °C/2 hrs)

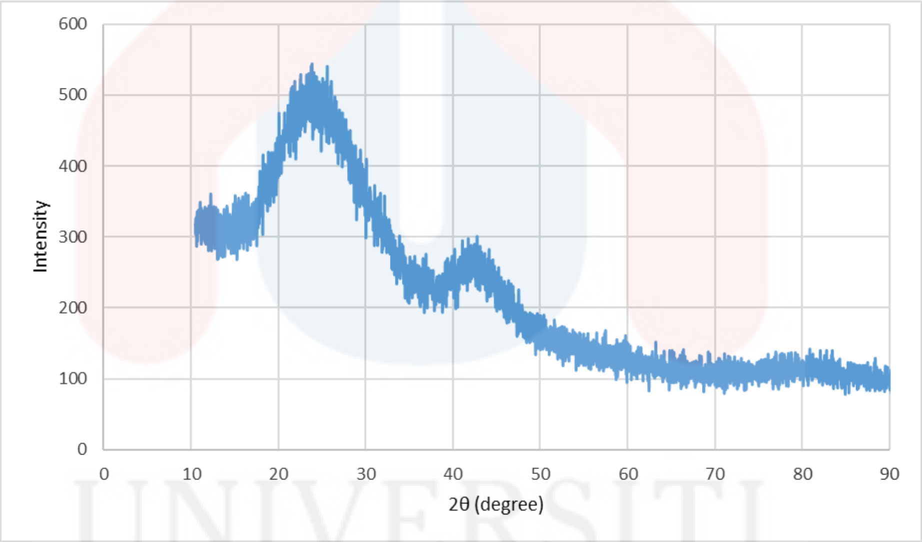
Based on Figure 4.9, by comparing the FTIR results of the samples the obtained infrared spectra (IR) of samples with different parameters were essentially indistinguishable with only some band intensity differences. These spectra also have similar intensity and wavenumber characteristics of certain absorption bands. By referring Figure 4.9, major peaks were observed between 3200 cm^{-1} and 3550 cm^{-1} that could be specified as O-H stretch or hydroxyl group. OH group influence the adsorption kinetics and equilibrium behavior of adsorbates by increasing the surface wettability of activated carbon. Peaks occur at band between 2100 cm^{-1} and 2260 cm^{-1} in (a), (b) and (c) which attribute to alkyne group.

Besides, medium band between 1566 and 1650 cm^{-1} in (a) and (b) would be related with stretching vibrations of C=C, in contrast to the others (a) and (b), there is no peak when bamboo was burned in a furnace at extreme temperatures. Carbohydrate regions from 1200 to 900 cm^{-1} are present in (a), (b) and (c) due to stretching of aliphatic compounds.

4.4 X-ray Diffraction (XRD)

XRD pattern of low angle peak intensity increases with activation temperature, supporting high-density micropores as shown in Figure 4.10.

(a)



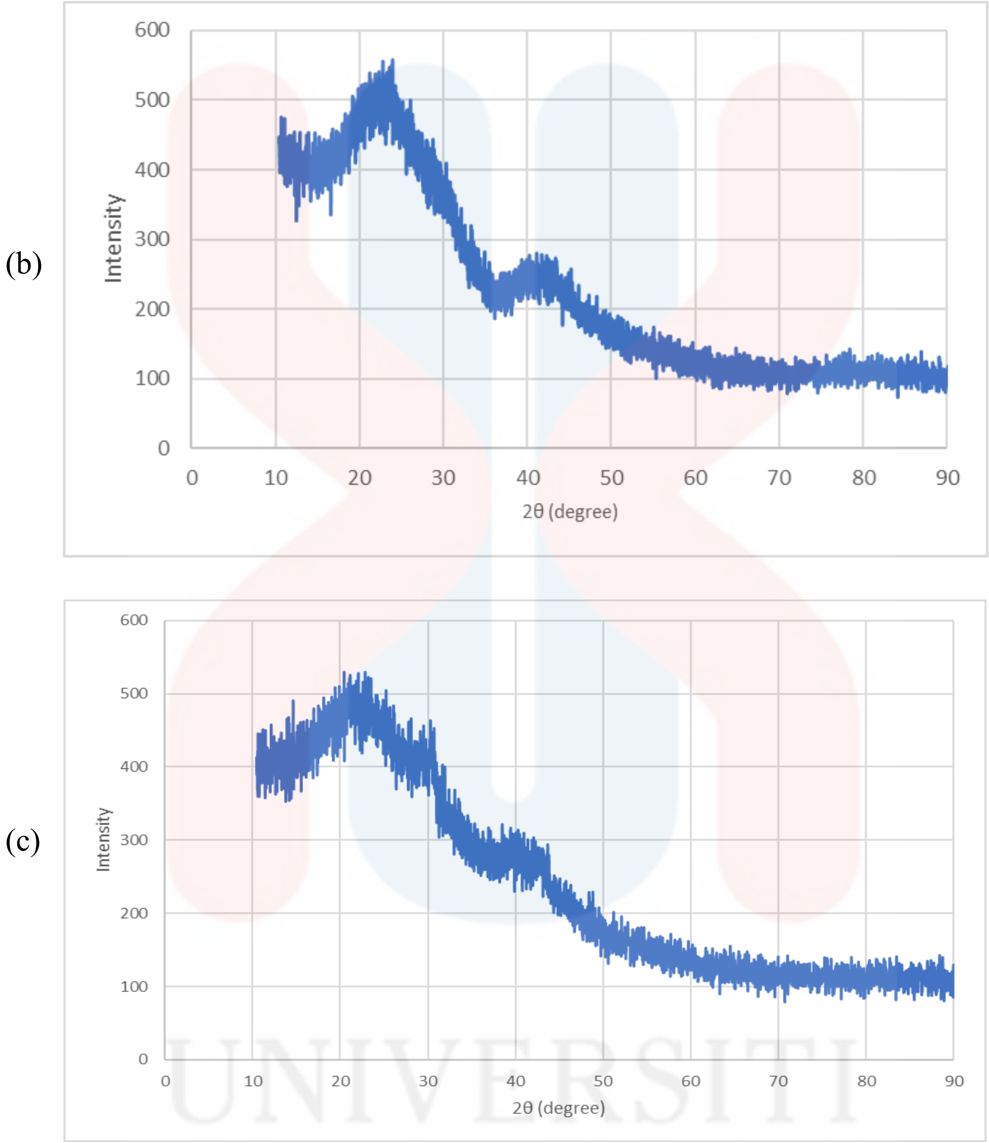


Figure 4.10: XRD pattern of (a) Bamboo-derived activated carbon powder (AC 400 °C/1 hr), (b) Bamboo-derived activated carbon powder (AC 600 °C/1.5 hrs) and (c) Bamboo-derived activated carbon powder (AC 800 °C/2 hrs)

Two distinct broad peaks located at around 24° and 43.6°, the graphitic carbon facets (002) and (100) or (100/101) are indicated by the peaks at 24° and 43.6° accordingly (Kar, 2021). Furthermore, The 002-reflection plane was shifted to the lower angle from 25.50 to 22.33 when the carbonization temperature was raised from 600°C to 800°C, suggesting a better developed pseudo-crystallite structure (Kar, 2021). This assumption is proved by the largest surface area exhibited by (a) and the smallest is seen in (c). As the activation temperature rose, the peaks shifted slightly to a narrower angle, signifying a greater distance between layers. This could be linked to the rise in faults produced throughout the activation procedure.

Table 4.8: Crystalline structure of XRD for Bamboo-derived activated carbon powder (AC 400 °C/1 hr), Bamboo-derived activated carbon powder (AC 600 °C/1.5 hrs) and Bamboo-derived activated carbon powder (AC 800 °C/2 hrs)

Crystallinity	**AC 400 °C/1 hr**	**AC 600 °C/1.5 hrs**	**AC 800 °C/2 hrs**
Crystallinity - From	10.502	10.502	10.502
Crystallinity -To	90.558	90.558	90.558
%-Crystallinity	29.8	26.9	28.2
%-Amorphous	70.2	73.1	71.8

Based on Table 4.8, the crystallinity pattern of AC 400 °C/1 hr, AC 600 °C/1.5 hrs, and AC 800 °C/2 hrs ranged from 10.502 to 90.558. In percentage terms, the crystallinity of each AC was recorded 29.8%, 26.9%, and 28.2%, respectively. AC 400 °C/1 hr it exhibits a high degree of crystallinity and is not affected by etching, resulting in a significant reduction in the number of micropores (Zhong et al., 2023). While the amorphous pattern was found of each AC to be 70.2%, 73.1%, and 71.8% for the study. The structural stability and mechanical strength of activated carbon can be enhanced by increasing crystallinity. Crystallinity reduction It is feasible to increase the surface area and pore volume of activated carbon by systematically reducing its crystallinity and increasing its amorphous content.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In conclusion, activated carbon was successfully developed using bamboo (*Gigantochloa albociliata*) as raw material in this study. The purpose of this study was successfully achieved with the following objectives. Firstly, to prepare activated carbon using bamboo (*Gigantochloa albociliata*) at different time and temperature. Secondly, to study the effect of time and temperature on the physicochemical characteristics of bamboo-derived activated carbon. Last objective is to study the effect of time and temperature on the morphological properties of bamboo-derived activated carbon.

Besides, FTIR analysis identified frequency peaks for each functional group such as carbon, oxygen, silicon and potassium of three bamboo-derived activated carbons at different temperatures and times. The wavelength of AC 400 °C/1 hr was examined at range 1108.56 cm^{-1} to 3355.65 cm^{-1}, and there were 6 functional compounds found. The wavelength of AC 600 °C/1.5 hrs was examined at range 533.71 cm^{-1} to 2359.35 cm^{-1}, and there were 8 functional compounds found. The wavelength of AC 800 °C/2 hrs was examined at range 2082.31 cm^{-1} to 2322.95 cm^{-1} and there were 2 functional compounds found. Scanning electron micrograph (SEM) confirmed the surface morphology.

In addition, scanning electron micrograph (SEM) confirmed the surface morphology. 3 samples activated carbon (AC) showed well-developed pore space on the surface activated carbon. EDX determined the elemental composition of bamboo-derived activated carbon. BET surface area of activated carbon was obtained total pore volume for AC 400 °C/1 hr was 3.498×10^{-3} cc/g while AC 800 °C/2 hrs yielded 4.912×10^{-2} cc/g. AC 800 °C/2 hrs produced a high surface area value of 74.972 m^2/g. AC 400 °C/1 hr yielded a low surface area value of 1.962 m^2/g. XRD analysis was obtained two distinct broad peaks located at around 24° and 43.6°, determined crystalline structure where were 29.8%, 26.9%, and 28.2%, respectively.

5.2 Recommendations

The following suggestions for future work are recommended for future development:

1. Thermogravimetric analysis is used to characterize bamboo-derived activated carbon. TGA is a technique that evaluates a sample's weight change as a function of temperature, yielding information on its thermal stability and composition. TGA is performed to study the weight loss or gain of a sample when the temperature is increased in a controlled manner. This can help identify the different stages of decomposition and evaluate the thermal stability of activated carbon.

2. Through energy dispersive X-ray spectroscopy (EDX) measurement, it was found that the carbon content was too low and was inconsistent with the main components of activated carbon. To increase carbon content in chemical activation. Consider pre-treating the precursors to increase their carbon content prior to activation. This could include pre-carbonization or other pre-treatment procedures to eliminate contaminants and boost carbon yield. Optimize EDX analysis parameters including beam energy and acquisition time to achieve accurate and reliable results. This will aid in identifying and quantifying carbon content in activated carbon.

3. Gas Chromatography-Mass Spectrometry (GC-MS) is used to identify and quantify volatile organic compounds (VOCs) that have been desorbed from activated bamboo carbon. Analyse the gases produced during thermal desorption or under other relevant conditions using GC-MS. This can reveal the types and concentrations of adsorbed organic molecules on the activated carbon.

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APPENDIX A

Figure A.1: Sample collection of bamboo (*Gigantochloa albociliata*)

Figure A.2: Bamboo that has been washed and dried in the oven

Figure A.3: 14” Wood band saw used to cut bamboo into small sizes

Figure A.4: 50 g dried bamboo was mixed well with 50 g of potassium carbonate (K_2CO_3) powder mixed with 500 mL distilled water into a solution with ratio 1:1 bamboo and chemical

Figure A.5: Furnace 1500°C (Carbolite Gero Sheffield, United Kingdom) for activation bamboo

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Figure A.6: Quantachrome Autosorb iQ3 (Interscience Sdn. Bhd) for BET analysis

APPENDIX B

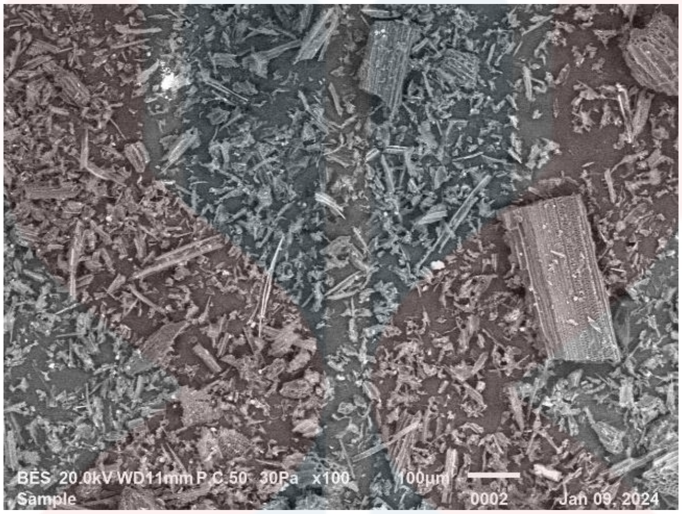

Figure B.1: SEM image for powdered bamboo-derived activated carbon (AC 400 °C/1 hr) at magnification 100x

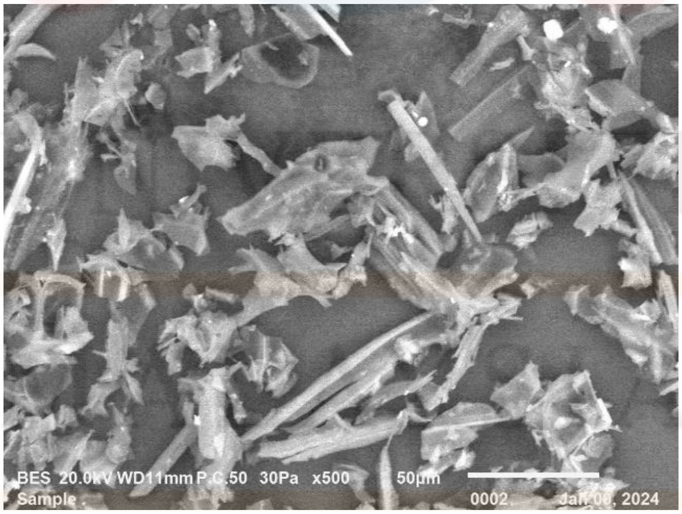

Figure B.2: SEM image for powdered bamboo-derived activated carbon (AC 400 °C/1 hr) at 500x magnification

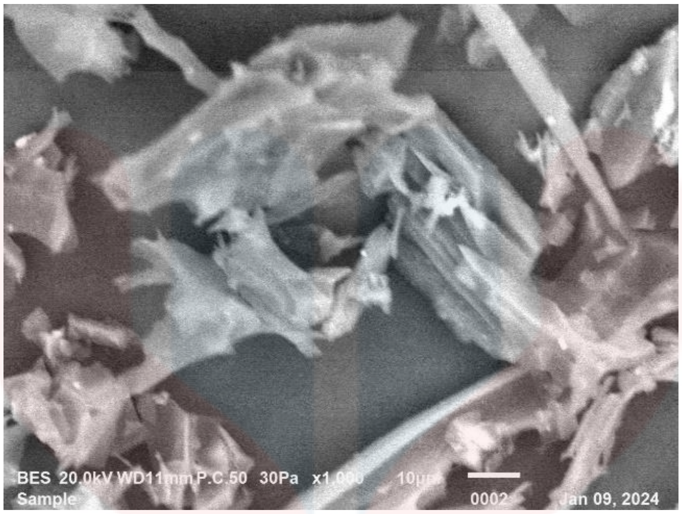

Figure B.3: SEM image for powdered bamboo-derived activated carbon (AC 400 °C/1 hr) at 1500x magnification

Figure B.4: SEM image for powdered bamboo-derived activated carbon (AC 600 °C/1.5 hrs) at 100x magnification

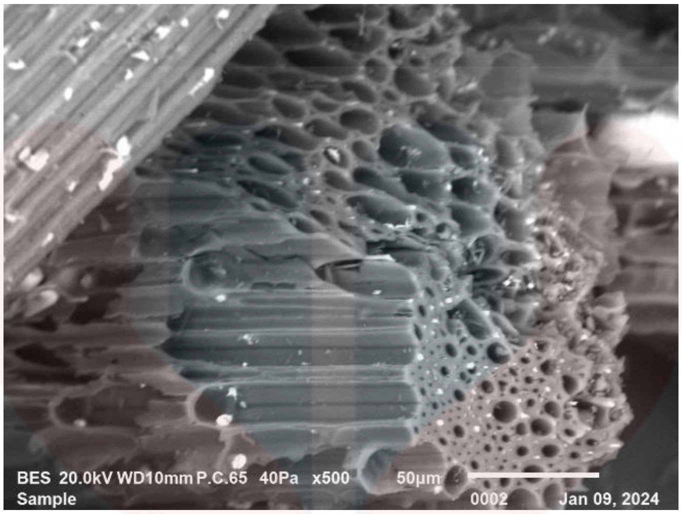

Figure B.5: SEM image for powdered bamboo-derived activated carbon (AC 600 °C/1.5 hrs) at 500x magnification

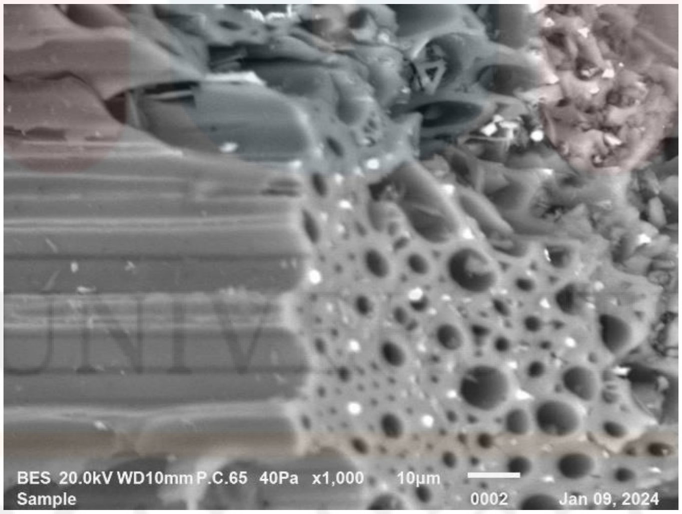

Figure B.6: SEM image for powdered bamboo-derived activated carbon (AC 600 °C/1.5 hrs) at 1500x magnification

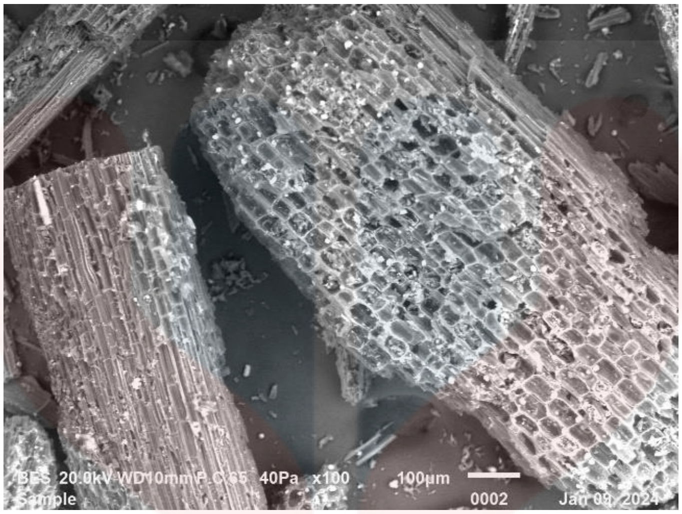

Figure B.7: SEM image for powdered bamboo-derived activated carbon (AC 800 °C/2 hrs) at 100x magnification

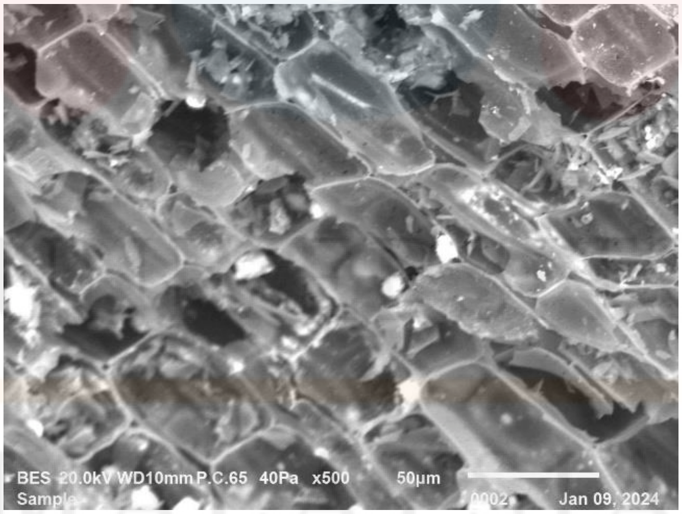

Figure B.8: SEM image for powdered bamboo-derived activated carbon (AC 800 °C/2 hrs) at 500x magnification

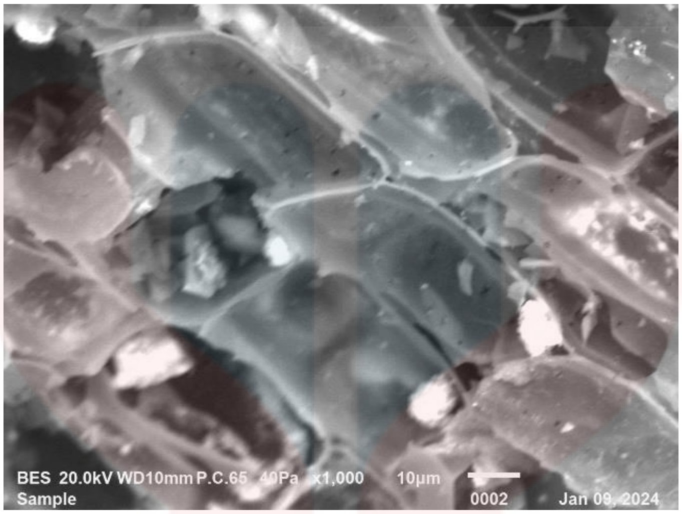

Figure B.9: SEM image for powdered bamboo-derived activated carbon (AC 800 °C/2 hrs) at 1500x magnification