

## CHARACTERIZATION OF ACTIVATED CHARCOAL FROM NUTMEG SHELL PYROLYSIS (*Myristica fragrans* hout) USING ACTIVATORS $\text{CaCl}_2$ AND $\text{ZnCl}_2$

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### ABSTRACT

A study was conducted on the characterization of activated carbon from pyrolyzed nutmeg shells (*Myristica fragrans* hout) using  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solution activators. This study aims to determine activated carbon's surface morphology and composition from nutmeg shells activated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions. The research stages include pyrolysis, activation, and characterization. The results showed that the pyrolysis of 6 kg of nutmeg shells produced a charcoal yield of 3.2 kg, liquid smoke of 1.1 L, tar of 65 g, and non-condensed gas. The pore diameter data using Scanning Electron Microscopy (SEM) of activated carbon activated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions were 3.72 and 3.02  $\mu\text{m}$ , respectively. In comparison, the pore diameter of nutmeg shell carbon before activation was 3.09  $\mu\text{m}$ . The composition data of activated carbon activated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions using Energy Dispersive Spectroscopy (EDS) showed the presence of Carbon, Oxygen, Aluminium, and Calcium elements, with the most significant composition being carbon at 58.36 % and 67.10 %. In comparison, nutmeg shell carbon before activation showed the presence of carbon of 64.39 %. The spectrum data of Fourier Transform-Infra Red analysis indicates that nutmeg shell carbon before activation and activated carbon activated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions show the presence of absorption areas of functional groups at relatively the exact wavenumber, namely O-H, aromatic C-H, aliphatic C-H, C=C, and C-O.

*Keywords:* activated charcoal, nutmeg shell, activators,  $\text{CaCl}_2$ ,  $\text{ZnCl}_2$ .

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### INTRODUCTION

Nutmeg (*Myristica Fragrans* Hout) is a significant crop in North Sulawesi, Indonesia, as its seeds and mace are exported commodities. Sitaro Regency (Siau, Tagulandang, and Biaro), with 4 471.76 hectares of nutmeg plantations and an annual production of 3 327.25 tons of nutmeg fruit, is a primary producer of nutmeg in Sulu and generates a substantial amount of nutmeg shell waste [1]. Approximately 5.1 % of the total nutmeg fruit, these shells remain underutilized [2, 3]. Locally, nutmeg shells are commonly used as cooking fuel, releasing smoke that pollutes the air and producing charcoal that

eventually turns ash. Pyrolysis offers a potential solution to mitigate these issues.

Pyrolysis is a thermochemical decomposition process of organic materials without air or oxygen. This technology holds promise for converting biomass into renewable fuels. The abundance of biomass and the relatively simple and environmentally friendly nature of pyrolysis technology make it a desirable option. Moreover, the process can yield various products, including liquid, solid (such as charcoal), and gaseous fuels, which can be used for renewable energy and other applications. Using biomass as an alternative energy source can help mitigate environmental issues such

as the increasing levels of CO<sub>2</sub> caused by fossil fuel consumption [4].

Pyrolysis is a thermochemical method that converts biomass waste into charcoal, which can be further processed into solid fuel (char) or activated carbon, syngas, and bio-oil, all without oxygen in a reactor [5]. Thermochemistry, involving high temperatures and pressures, breaks down organic materials into substances like charcoal, biofuels, and synthetic gas [6]. Biomass (such as wood) is typically pyrolyzed at temperatures between 300 - 600°C [7]. This process produces charcoal and a complex mixture of compounds, including those derived from the decomposition of cellulose, hemicellulose, and lignin. Hemicellulose pyrolysis yields furfural, furan, acetic acid, and their derivatives. Cellulose produces acetic acid and carbonyl compounds, while lignin decomposes into phenol, phenolic ethers, and their derivatives [8].

Activated carbon is produced by activating charcoal with an activating agent, increasing the surface area by opening closed pores and enhancing its adsorption capacity. Activated carbon has an amorphous structure composed of flat plates of carbon atoms covalently bonded in a hexagonal lattice [3]. Activated carbon is a commonly used adsorbent [9]. Its capacity to reduce wastewater makes it valuable in various industries, including beverages, water treatment, sugar processing, and as a catalyst [10]. To meet the growing demand for activated carbon, researchers have explored using agricultural and plantation waste as a source material. Activated carbon can be produced from any carbon-containing material with a porous structure.

The morphology and composition of activated carbon are typically characterized using Scanning Electron Microscopy - Energy Dispersive Spectroscopy (SEM - EDS). SEM is used to analyze particles' texture, size, and arrangement on a sample's surface, providing information about its micro and nanoscale morphology [11]. EDS, on the other hand, is employed to determine the elemental composition of a material [12]. This study investigated the pyrolysis of nutmeg shells to produce charcoal, followed by chemical activation using CaCl<sub>2</sub> and ZnCl<sub>2</sub> activators. The resulting activated carbon was characterized macroscopically (by determining moisture content, ash content, volatile matter content, and fixed carbon content) and microscopically (using SEM - EDS and FT - IR analysis).

## **EXPERIMENTAL**

The equipment used in this study was a set of pyrolysis equipment, oven, furnace, desiccator, analytical balance, porcelain crucibles, glassware, dropper pipettes, stirring rods, spatulas, pH meter, Whatman filter paper, 40-mesh sieve, mortar and pestle, crucible tongs, funnel, a set of SEM - EDS, and FT - IR equipment. The materials used in this study were nutmeg shells, CaCl<sub>2</sub>, ZnCl<sub>2</sub>, charcoal from the pyrolysis of nutmeg shells, and distilled water.

The nutmeg shells were dried under sunlight until dry. The dried nutmeg shells were then weighed and pyrolyzed. A 6 kg sample of dried nutmeg shells was placed into a pyrolysis reactor with a thermometer. The sample was heated for five h, starting from room temperature to 450°C [13]. The pyrolysis apparatus consists of a pyrolysis reactor equipped with a lid, thermometer, and barometer; a pyrolysis furnace equipped with a space for a gas stove; cover; barometer; thermometer; smoke outlet pipe; tar collection container; water container for cooling; spiral pipe/smoke conductor and cooler; support container; and liquid smoke collection container. After the pyrolysis process, the charcoal is allowed to cool for five h inside the reactor. Once cooled, the charcoal is removed from the pyrolysis furnace, ground into a fine powder using a mortar and pestle, and sieved using a 40 - mesh sieve [14]. Subsequently, the finely ground charcoal is activated using 2 M CaCl<sub>2</sub> and 2 M ZnCl<sub>2</sub> solutions.

Next, the pyrolyzed nutmeg shell charcoal, which has been ground and sieved, is soaked in CaCl<sub>2</sub> and ZnCl<sub>2</sub> solutions in a closed container for 24 h. The soaked charcoal is then filtered using Whatman filter paper and washed with distilled water until neutral, as indicated by a neutral pH of 7 for the washing solution. The washed and neutralized charcoal is dried in an oven at 120 °C for three h and then stored in a desiccator until stable. The resulting activated charcoal is then analyzed for its physical and chemical properties.

To determine the moisture content, 3 g of activated carbon is weighed and placed in a porcelain crucible [7]. It is then heated in an oven at 100°C for 2.5 h, cooled in a desiccator, and weighed again. The percentage of moisture content in the activated carbon is determined using the Eq. (1):

$$\text{Moisture Content (\%)} = \frac{w-d}{w} \times 100 \quad (1)$$

where: w = weight before heating, g, d = weight after heating, g. According to SNI 06 - 3730 - 1995, the moisture content of good quality technical activated carbon in powder form should not exceed 15 % [15].

The determination of ash content is carried out by weighing 3 g of activated carbon and placing it in a porcelain crucible [7]. It is then heated in an oven at 100°C for 2.5 h, cooled in a desiccator, and weighed again. The percentage of ash content in the activated carbon is determined using the Eq. (2):

$$\text{Ash content (\%)} = \frac{a-b}{a} \times 100 \% \quad (2)$$

where: a = weight of porcelain crucible + sample before ashing, g, b = weight of porcelain crucible + ash after ashing, g. According to SNI No. 06 - 3730 - 1995, the maximum ash content for good quality technical activated carbon should not exceed 10 % [15].

To determine the volatile matter content, 3 g of activated carbon with a known dry mass is weighed and placed in a porcelain crucible [4]. It is then heated in a furnace at 700°C for 10 min. Subsequently, the activated carbon is cooled in a desiccator for one h and weighed again. The percentage of volatile matter in the activated carbon is determined using the Eq. (3):

$$\text{Volatile matter content (\%)} = \frac{a-b}{a} \times 100 \% \quad (3)$$

where: a = weight of porcelain crucible + sample before heating, g, b = weight of porcelain crucible + residue after heating, g. According to SNI 06 - 3730 - 1995, the volatile matter content of technical activated carbon should be 25 % [16].

The determination of fixed carbon content can be calculated using Eq. (4) [17]:

$$\text{Fixed carbon content} = 100 \% - (\text{Volatile matter content} + \text{ash content}) \quad (4)$$

According to SNI 06 - 3730 - 1995, good quality powdered activated carbon should have a minimum carbon content of 65 % [15].

The activated charcoal obtained from activation with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions was characterized

microscopically using SEM - EDS and FT - IR [15, 18]. SEM - EDS analysis aims to determine the morphology, topology, and composition, while FT - IR analysis aims to determine the functional groups present in the activated carbon.

## RESULTS AND DISCUSSION

### Pyrolysis results of nutmeg husk

The nutmeg husk, used as the raw material, was dried under sunlight for three days. The drying process aimed to reduce the sample's moisture content. Subsequently, the sample was carbonized into charcoal through a pyrolysis process. Pyrolysis of 6 kg of dried nutmeg husk, heated from room temperature to 450°C, yielded 1.1 L of liquid smoke, 65 g of tar, 3.2 kg of charcoal, and uncondensed gases. The study showed that the pyrolysis of nutmeg husk waste produced liquid smoke that began to drip at a temperature of 195°C. The maximum heating temperature for liquid smoke formation was 450°C, and no further dripping occurred above this temperature. At 450°C, the process is no longer decomposing biomass components into organic compounds but rather the heating and cooking of charcoal or carbonization [2].

During pyrolysis, organic compounds decompose, forming hydrocarbons, steam, methanol, acetic acid, and tar, while non-carbon elements, oxygen, and hydrogen are removed from the carbon, which is converted into gas. Pyrolysis is the process of heating a substance with limited oxygen and decomposing its constituent components [19]. Pyrolysis combustion uses various biomasses from different plant sources [20]. Biomass is biological material derived from living organisms, consisting of carbon and a chemical mixture of organic compounds containing hydrogen, nitrogen, oxygen, and small amounts of other atoms and elements. Each biomass has different characteristics and compositions depending on its type and form. In pyrolysis, thermal energy drives oxidation, causing complex carbon molecules to decompose primarily into carbon or charcoal.

Wood pyrolysis undergoes a stepwise decomposition: hemicellulose degrades at 200 - 260°C, cellulose at 240 - 350°C, and lignin at 280 - 500°C [21]. Hemicellulose pyrolysis produces furfural, furan, acetic acid, and its derivatives; cellulose produces acetic acid and carbonyl compounds, while lignin decomposes into phenol and

phenolic ethers and their derivatives. Pyrolysis is a thermochemical decomposition process that occurs in organic matter (biomass) through heating with little or no oxygen, where the raw material undergoes a breakdown of its chemical structure into a gas phase [22]. The pyrolysis process involves several phases: the drying phase at 200°C, the pyrolysis phase at 200 - 500°C, and the gas evolution phase at 500 - 200°C. Additionally, it is stated that pyrolysis combustion can produce primary products in the form of charcoal, liquid smoke (bio - oil), and gas. The resulting liquid smoke can be used as an additive or preservative for food or other products. The gas formed can be burned directly. Pyrolysis gas can be classified into non - condensable gases (CO, CO<sub>2</sub>, CH<sub>4</sub>, and others) and condensable gases (tar). Oil occurs during the condensation of the formed gas, also known as liquid smoke. The resulting charcoal is a high-calorie fuel or can be used as activated carbon. The characteristics of mixed pyrolysis display three types of pyrolysis characteristic graphs: mass, mass loss rate, and temperature [23].

The first stage is drying, characterized by a slow mass loss. The second stage is devolatilization, characterized by a very rapid mass loss. The third stage is carbonization, characterized by a slowing mass loss. Pyrolysis products generally contain three types: light gas (H<sub>2</sub>, CO, CO<sub>2</sub>, H<sub>2</sub>O, and CH<sub>4</sub>), tar, and char [22]. All pyrolysis products can be used as environmentally friendly fuels. In addition to these three types of products, pyrolysis technology also produces other products. Other pyrolysis products include charcoal (biochar), torrefied wood, activated carbon, charcoal briquettes, bio - oil, and syngas. The yield and composition of bio - oil depend on the biomass composition and process parameters such as temperature, reaction time, heating rate, airflow rate, the particle size of the material, and the chemical composition of the biomass, and the influence of these parameters on the yield and quality of bio - oil [24].

### **Activation of nutmeg shell charcoal**

The production of activated carbon from nutmeg shell charcoal involves carbonization and activation. In this study, carbonization was carried out through pyrolysis at room temperature to 450°C for five h, aiming to decompose organic materials into charcoal. Carbonization is decomposing organic cellulose from organic raw materials into carbon elements at a specific

temperature using a small amount of oxygen [20, 25]. During this process, organic compounds decompose, forming hydrocarbons, steam, methanol, acetic acid, and tar. At the same time, non-carbon elements, oxygen, and hydrogen are removed from the carbon, which is converted into gas.

The carbon produced from the carbonization of organic materials has a high adsorption capacity. During the carbonization process, pores begin to form, but the resulting carbon still has a low adsorption capacity due to impurities such as hydrocarbons, water, and oxides. Pores begin to form as the volatile matter is removed. Factors that affect the carbonization process include: 1) Carbonization time. A longer carbonization time results in a more complete carbonization reaction, producing less carbon product but increasing liquid and gas products. Carbonization time varies depending on the type and amount of material being processed; 2) Carbonization temperature. Carbonization temperature affects the carbon product because high carbonization temperatures produce less carbon but increase liquid and gas products due to the decomposition and vaporization of many substances. The pores that begin to form during the carbonization process are then activated to open the pores and increase the size of the tiny pores.

The second stage is the activation process, which is preceded by the grinding and sieving the charcoal using a 40 - mesh sieve. The grinding and sieving process aims to reduce the particle size and increase the surface area of the charcoal. In this study, chemical activation was carried out by immersing the ground charcoal in CaCl<sub>2</sub> and ZnCl<sub>2</sub> solutions. The chemical activation process aims to increase the pore surface area of the charcoal by oxidizing surface molecules using a chemical solution as an activator. Activation aims to enlarge the pores and form new ones by breaking hydrocarbon bonds or oxidizing surface molecules so that the resulting carbon can change its physical and chemical properties with a larger surface area and higher adsorption capacity [20]. Because of its undeveloped pore structure, the final carbonization product cannot be directly used as an adsorbent without activation. During the activation process, carbon oxide is dispersed on the carbon surface due to a reaction involving carbon and an oxidizing agent.

During activation, carbon is soaked to produce a large number or volume of pores and a large surface

area by eliminating or vaporizing volatile carbonization products. Activated carbon produced by chemical activation breaks carbon chains from organic compounds using chemicals as activators [26]. The chemical activation process involves several simultaneous steps. Chemical activation at lower temperatures produces a better pore structure in activated carbon. The chemicals will enter the activated carbon and open the carbon surface originally covered by tar deposits. The chemicals used as activating agents in this study were  $\text{CaCl}_2$  and  $\text{ZnCl}_2$ .

Chemical activation has the following advantages: 1) more products are produced in chemical activation; 2) Lower temperatures are used; 3) Using chemical solutions affects the formation and development of pores in activated carbon; 4) The carbonization and activation processes are carried out simultaneously (after the carbonization process, the activation stage is carried out immediately) [27]. Calcium chloride and zinc chloride have dehydrating properties and are suitable activators in producing activated carbon from lignocellulosic raw materials. Calcium chloride and zinc chloride as activating agents can act as catalysts in bond cleavage, hydrolysis, dehydration, and condensation, accompanied by cross - linking reactions between alkali ions (Ca) and Zn with the charcoal surface. The reaction between charcoal and the activator begins during mixing. The ions produced in the  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions will attack the active sites on the charcoal surface.

The effect of using  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions is expected to bind impurities (tar) so that they will be released through the pores of the activated carbon. This causes the pores of the activated carbon to increase and

have a large surface area, so the adsorption capacity will also increase. The activation process is a physical change where activated carbon's surface area and adsorption capacity increase because the hydrocarbons contained in the carbon are removed [28]. The characteristics of activated carbon can be determined through several quality tests based on the Indonesian National Standard No. 06 - 3730 - 1995, including moisture content, ash content, volatile matter content, fixed carbon content, and iodine adsorption capacity.

The results of the moisture content analysis of activated charcoal activated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions are presented in Table 1. Table 1 shows that the average moisture content of activated charcoal activated with  $\text{CaCl}_2$  was 0.533 %, while the charcoal activated with  $\text{ZnCl}_2$  had a moisture content of 0.438 %. These results of the moisture content analysis indicate that the moisture content of the activated nutmeg shell charcoal activated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions has met the quality standard for activated carbon, as stated in the Indonesian National Standard (SNI) 06 - 3730 - 1995, which is a maximum of 15 % for powdered activated carbon. Moisture content is one of the properties of activated carbon that significantly influences the quality of the activated carbon itself [12]. Through the moisture content test, it can be determined how much water can be evaporated so that the water bound in the activated carbon does not block the pores of the activated carbon itself. The loss of water molecules in the activated carbon causes the pores on the surface of the activated carbon to open wide.

The results of the ash content analysis of activated nutmeg shell charcoal treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$

Table 1. Moisture content percentage of charcoal soaked in  $\text{CaCl}_2$  and  $\text{ZnCl}_2$ .

Treatment soaking	Moisture content, %				SNI 06-3730-1995 Max. 15 %
	W1	W2	W3	Average, %	
$\text{CaCl}_2$	0.56	0.60	0.50	0.533	
$\text{ZnCl}_2$	0.54	0.36	0.55	0.483	

Table 2. Ash content percentage of activated charcoal treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions.

Treatment soaking	Ash content, %				SNI 06-3730-1995 Max. 10 %
	W1	W2	W3	Average, %	
$\text{CaCl}_2$	0.67	0.63	0.66	0.653	
$\text{ZnCl}_2$	0.39	0.40	0.41	0.4	

solutions are presented in Table 2. Table 2 shows that the average ash content of activated charcoal treated with  $\text{CaCl}_2$  solution was 0.653 %, while the charcoal treated with  $\text{ZnCl}_2$  solution had an ash content of 0.4 %. These results of the ash content analysis indicate that the ash content of the activated nutmeg shell charcoal treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions meets the quality standard for activated carbon, as specified in the Indonesian National Standard 06 - 3730 - 1995, which sets a maximum of 10 % for powdered activated carbon. Before activation, the charcoal had a relatively higher content of metal oxides due to the lack of chemical treatment, resulting in many metal oxides trapped in the charcoal pores [29]. The ash content value affects the yield of the produced charcoal. As the activation time increases, the ash content also increases because the content of organic compounds in the activated carbon decreases. The ash content influences activated carbon's reactivity as an adsorbent. Ash content is considered an impurity in activated carbon, so the lower the ash content, the better the quality of the activated carbon.

The determination of volatile matter content aims to determine the content of compounds that can evaporate at a temperature of 600°C for 30 min. The results of the volatile matter content analysis of activated nutmeg shell charcoal treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions are presented in Table 3. Table 3 shows that the average volatile matter content of activated charcoal treated with  $\text{CaCl}_2$  is 0.406 %, while the charcoal treated with  $\text{ZnCl}_2$  has a volatile matter content of 0.403 %. These data on volatile matter content meet the standard for activated carbon as specified in SNI 06 - 3730 - 1995, which sets a maximum of 25 %.

Fixed carbon content represents the amount of pure carbon present in activated carbon. Determining fixed carbon content aims to determine the value or magnitude of the pure carbon content in activated carbon. The results of the fixed carbon content analysis of activated charcoal treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  are shown in Table 4. Table 4 shows that the average fixed carbon content of charcoal treated with  $\text{CaCl}_2$  is 70.70 %, while the charcoal treated with  $\text{ZnCl}_2$  has a fixed carbon content of 71.13 %. Based on the data obtained in this study, the fixed carbon content of activated nutmeg shell charcoal treated with  $\text{CaCl}_2$  is slightly lower than that of activated carbon treated with  $\text{ZnCl}_2$ . However, both meet the quality standard of SNI 06 - 3730 - 1995 for powdered activated carbon, which requires a minimum carbon content of 65 %.

#### Morphology analysis of activated nutmeg shell charcoal surface

Morphology and elemental composition analyses were conducted on pyrolyzed nutmeg shell charcoal and activated nutmeg shell charcoal treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions. The results of the morphology analysis of the surface of pyrolyzed nutmeg shell charcoal before and after activation with 2 M  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  using SEM are shown in Fig. 1. Scanning Electron Microscopy (SEM) is a type of electron microscope that uses an electron beam to image the surface profile of an object. SEM is commonly used to analyse the morphology and topography of a sample. Energy Dispersive Spectroscopy (EDS) is an analytical technique used to determine the elemental composition of a sample by directing an electron beam from the SEM

Table 3. Volatile matter content percentage of charcoal soaked in  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions.

Treatment soaking	Volatile matter content, %				SNI 06-3730-1995  Max. 25 %
	W1	W2	W3	Average, %	
$\text{CaCl}_2$	0.42	0.41	0.39	0.406	
$\text{ZnCl}_2$	0.38	0.40	0.43	0.403	

Table 4. Fixed carbon content percentage of activated charcoal treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions.

Treatment soaking	Fixed carbon content, %				SNI 06-3730-1995  Max. 65 %
	W1	W2	W3	Average, %	
$\text{CaCl}_2$	71.54	71.06	69.51	70.70	
$\text{ZnCl}_2$	68.72	70.86	73.81	71.17	

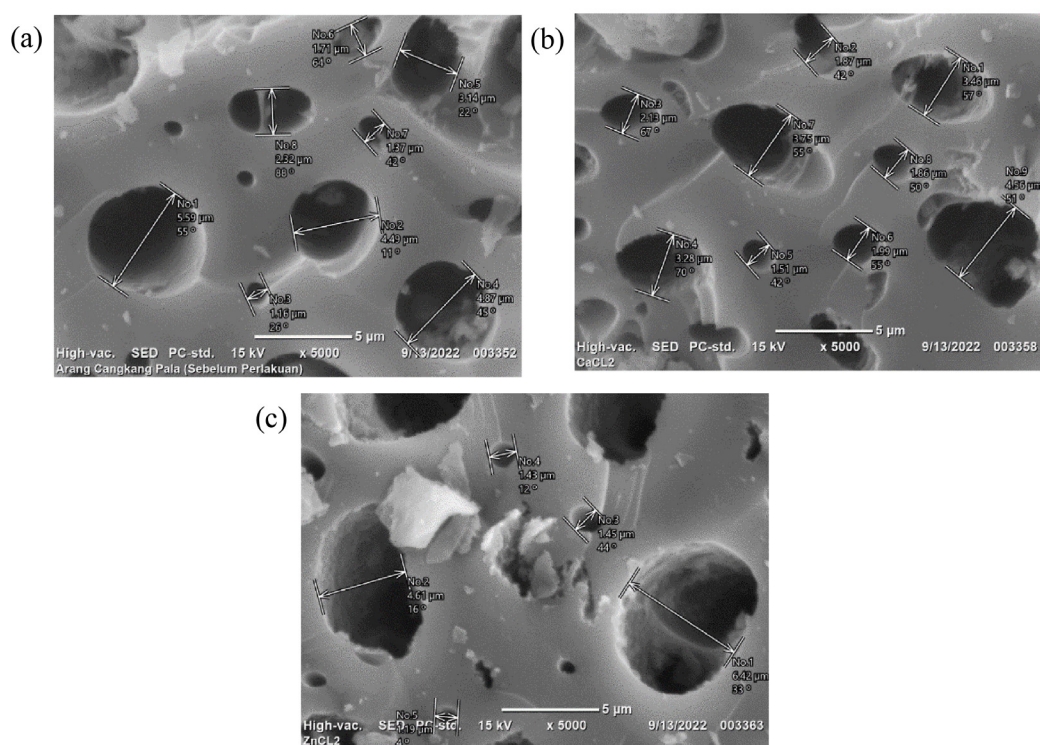


Fig. 1. SEM surface morphology of charcoal from the pyrolysis of nutmeg shells charcoal (a) before activation, (b) treated with  $\text{CaCl}_2$ , (c) treated with  $\text{ZnCl}_2$ .

onto the specimen.

Additionally, EDS in an SEM can be performed on a specific point or a selected area and can be used to map the distribution of elements within the sample. The ability of SEM - EDS to perform characterization is based on the fundamental principle that each element has a unique atomic structure, allowing X-rays to uniquely identify the characteristics of an element's atomic structure. SEM characterization aims to determine particle morphology and pore diameter [14, 30]. At the same time, EDS analysis is used to identify the elemental composition of the pore constituents in activated carbon [31]. Morphological characterization using SEM is an analytical technique used to determine the texture, size, and arrangement of constituent particles on the surface of a layer [11, 32]. The results of SEM analysis on nutmeg shell charcoal, both unactivated and activated with 2 M  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions at 5000 $\times$  magnification, reveal extensive morphological features, including surface area, pore size, and distribution.

The SEM image in Fig. 1 (a) shows that the surface morphology of nonactivated nutmeg shell charcoal exhibits pore diameters with the following

measurements: 5.59  $\mu\text{m}$ , 4.49  $\mu\text{m}$ , 1.16  $\mu\text{m}$ , 4.87  $\mu\text{m}$ , 3.14  $\mu\text{m}$ , 1.17  $\mu\text{m}$ , 1.37  $\mu\text{m}$ , and 2.32  $\mu\text{m}$ , as presented in Table 5. The average pore diameter of charcoal obtained from the pyrolysis of nutmeg shells is 3.09  $\mu\text{m}$ . Fig. 1 (b) shows that the SEM surface morphology of the activated nutmeg shell charcoal sample treated with  $\text{CaCl}_2$  solution exhibits pore diameters of 3.46  $\mu\text{m}$ , 1.87  $\mu\text{m}$ , 2.13  $\mu\text{m}$ , 3.28  $\mu\text{m}$ , 1.51  $\mu\text{m}$ , 1.99  $\mu\text{m}$ , 3.75  $\mu\text{m}$ , 1.86  $\mu\text{m}$ , and 4.56  $\mu\text{m}$ . The average pore diameter of the activated nutmeg shell charcoal sample is 2.72  $\mu\text{m}$ , as shown in Table 5.

Fig. 1 (c) shows the SEM surface morphology of activated charcoal samples derived from nutmeg shells treated with  $\text{ZnCl}_2$ . The pore diameters of the activated charcoal are 6.42  $\mu\text{m}$ , 4.61  $\mu\text{m}$ , 1.45  $\mu\text{m}$ , 1.43  $\mu\text{m}$ , and 1.19  $\mu\text{m}$ . The average pore diameter of the activated charcoal sample is 3.02  $\mu\text{m}$ , as shown in Table 5. Based on Fig. 1 and the data in Table 5, there are apparent differences in the morphology and pore structure of the charcoal before and after activation. The average pore diameters are as follows: 3.09  $\mu\text{m}$  for nonactivated nutmeg shell charcoal, 2.72  $\mu\text{m}$  for nutmeg shell charcoal activated with  $\text{CaCl}_2$ , and 3.02  $\mu\text{m}$  for nutmeg shell charcoal activated with  $\text{ZnCl}_2$ .

Table 5. Pore diameter of charcoal from the pyrolysis of nutmeg shell.

Pore diameter, $\mu\text{m}$		
Before activation	Treated with $\text{CaCl}_2$ solution	Treated with $\text{ZnCl}_2$ solution
5.59	3.46	6.42
4.49	1.87	4.61
1.16	2.13	1.45
4.87	3.28	1.43
3.14	1.51	1.19
1.71	1.99	
1.37	3.75	
2.32	1.86	
	4.56	
Average pore diameter, $\mu\text{m}$		
3.09	2.72	3.02

In the activated carbon treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$ , the pores appear more numerous and form deeper and larger pore cavities than in nutmeg shell charcoal before activation. This is due to the carbonization and chemical activation process, where the presence of calcium and zinc ions enhances the formation of pores in the activated charcoal. In contrast, nonactivated nutmeg shell charcoal undergoes only the carbonization process, which results in fewer and less developed pores. The activation process with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions reduces the presence of minor elements such as Na and Al in nutmeg shell charcoal. The shape of the pore surface is one of the key factors influencing the ability of an adsorbent to adsorb substances. Pores in activated carbon enhance their adsorption capacity as they create gaps that expand the surface area of the material.

The pore size characteristics of activated carbon can be analysed through SEM imaging [17]. Activated carbon derived from activated charcoal has a larger pore surface and greater pore size than charcoal, which has not undergone physical or chemical activation. Therefore, activated carbon's surface area and pore size characteristics play a crucial role in its adsorption efficiency. Activated carbon with high adsorption capacity typically possesses a large pore surface area [5].

#### Energy Dispersive Spectroscopy (EDS) analysis of elemental composition

EDS characterization is an analytical technique used

to determine the elemental composition of a material [32]. The Energy Dispersive Spectroscopy (EDS) test data for nutmeg shell charcoal, both before and after activation with  $\text{CaCl}_2$ , are presented in Fig. 2. The data in Fig. 2 show the percentage content of various elements. Unactivated nutmeg shell charcoal (Fig. 2 (a)) contains 58.36 % carbon. In addition to carbon, there are impurity elements such as oxygen (15.13 %), aluminium (1.89 %), and calcium (24.63 %).

The EDS data for activated nutmeg shell charcoal treated with  $\text{CaCl}_2$  solution (Fig. 2 (b)) show a carbon content of 64.39 %, an increase compared to the nutmeg shell charcoal before activation. In addition to carbon, oxygen (20.13 %), aluminium (1.01 %), and calcium (14.46 %) are present. Fig. 2 (c) shows that the activated charcoal treated with  $\text{ZnCl}_2$  contains 67.10 % carbon, further increasing compared to the charcoal activated with  $\text{CaCl}_2$  and before activation charcoal. Along with carbon, oxygen (16.22 %), aluminium (1.75 %), and calcium (12.89 %) are present. These impurities may originate from mineral additives used in the charring process and the activator compound.

Based on the EDS analysis data in Table 6, the percentage of carbon elements in activated charcoal treated with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions increases. This variation is attributed to differences in the activating agents, where Ca and Zn metals significantly enhance the carbon and oxygen content while concurrently reducing the concentrations of Al and Ca elements [5, 6].

#### Fourier Transform Infrared (FT - IR) analysis

The FT - IR spectral data of nutmeg shell charcoal, both before activation and after activation with  $\text{CaCl}_2$  and  $\text{ZnCl}_2$  solutions, are presented in Fig. 3. Based on the FT - IR spectrum data of activated charcoal before activation, as shown in Fig. 8, various functional groups are identified in the adsorption region, as listed in Table 7. The functional groups of nutmeg shell charcoal obtained from pyrolysis without activation exhibited adsorption regions at the following wave numbers:  $3446.79\text{ cm}^{-1}$ ,  $2920.23\text{ cm}^{-1}$ ,  $1608.63\text{ cm}^{-1}$ ,  $1105.21\text{ cm}^{-1}$ , and  $875.68\text{ cm}^{-1}$ . These correspond to the adsorption of O-H, =C-H aromatic, C-H aliphatic, C=C, and C-O functional groups, respectively. Additionally, the bending adsorption of C=C aromatic groups is observed at  $875.68\text{ cm}^{-1}$ .

Charcoal activated with  $\text{CaCl}_2$  solution displayed adsorption regions at wave numbers:  $3479.58\text{ cm}^{-1}$ ,

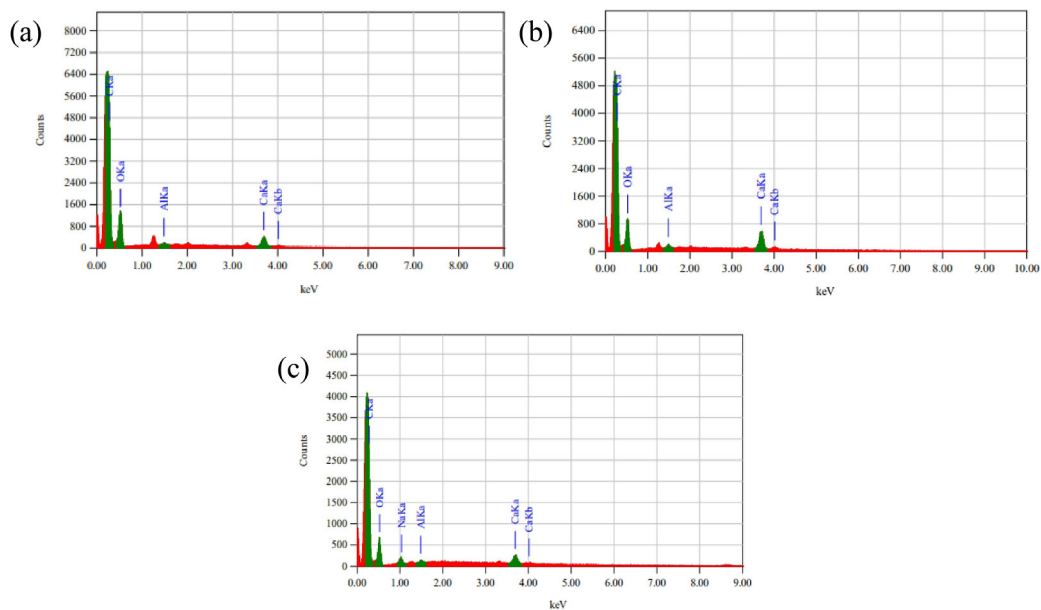


Fig. 2. EDS spectrum of nutmeg shell charcoal (a) before activation, (b) treated with  $\text{CaCl}_2$ , (c) treated with  $\text{ZnCl}_2$ .

Table 6. EDS data of the elemental composition of charcoal from the pyrolysis of nutmeg shells.

Elements	Atom %		
	Before activation, %	$\text{CaCl}_2$ , %	$\text{ZnCl}_2$ , %
C	58.36	64.39	67.10
O	15.13	20.13	16.22
Al	1.89	1.01	1.75
Ca	24.63	14.46	12.89

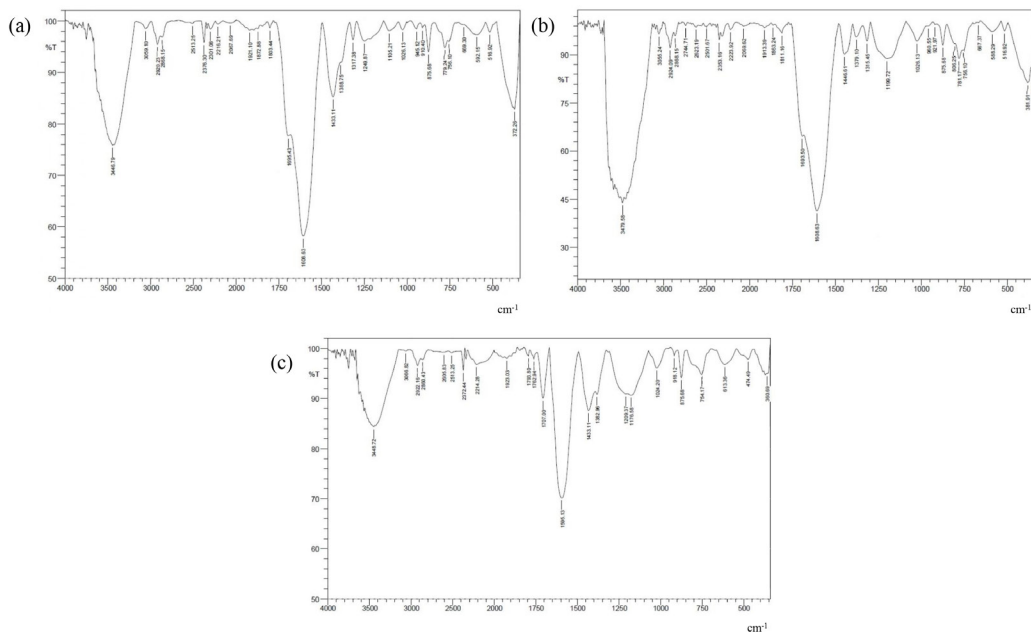


Fig. 3. FT - IR spectrum of charcoal derived from the pyrolysis of nutmeg shell (a) before activation, (b) treated with  $\text{CaCl}_2$ , (c) treated with  $\text{ZnCl}_2$ .

Table 7. FT-IR data of charcoal samples derived from the pyrolysis of nutmeg shell.

Before activation	Wavenumbers, cm <sup>-1</sup>		Alleged functional group
	Activation of CaCl <sub>2</sub>	Activation of ZnCl <sub>2</sub>	
3446.79	3479.58	3448.72	O-H (stretching)
3059.10	3055.24	3066.82	=C-H Aromatics (stretching)
2920.23	2924.29	2922.16	C-H Aliphatic (stretching)
1608.63	1608.63	1595.13	C=C (stretching)
1105.21	1199.72	1176.58	C-O (stretching)
875.68	875.68	875.68	C=C (bending)

3055.24 cm<sup>-1</sup>, 2924.29 cm<sup>-1</sup>, 1608.63 cm<sup>-1</sup>, 1199.72 cm<sup>-1</sup>, and 875.68 cm<sup>-1</sup>. These regions indicate the presence of the same functional groups as those observed in the unactivated charcoal. On the other hand, activated charcoal obtained through activation with ZnCl<sub>2</sub> showed FT - IR adsorption regions at wave numbers 3448.72 cm<sup>-1</sup>, 3066.82 cm<sup>-1</sup>, 2922.16 cm<sup>-1</sup>, 1595.13 cm<sup>-1</sup>, 1176.58 cm<sup>-1</sup>, and 875.68 cm<sup>-1</sup>, which are similar to those observed in both the unactivated charcoal and the charcoal activated with CaCl<sub>2</sub>. These findings suggest the presence of the same functional groups.

The adsorption regions of the functional groups in unactivated nutmeg shell charcoal and charcoal activated with CaCl<sub>2</sub> and ZnCl<sub>2</sub>, where the coconut shell activated carbon functional groups show adsorption regions at wave numbers 3414.73 cm<sup>-1</sup> (O-H functional group), 1561.27 cm<sup>-1</sup> (C=C functional group), 1223.75 cm<sup>-1</sup> (C-O functional group), and 885.27 cm<sup>-1</sup> (C=C functional group) [33]. The wave numbers associated with the activated carbon derived from nutmeg shell include 3401.7 cm<sup>-1</sup> for the O-H functional group, 1584.2 cm<sup>-1</sup> for the C=C functional group, 1201.2 cm<sup>-1</sup> for the C-O functional group, and 740.05 cm<sup>-1</sup> for the C=C functional group. The adsorption regions of the resulting activated carbon functional groups are consistent with the findings in [34], where the 3200 - 3600 cm<sup>-1</sup> absorption band corresponds to the O-H functional group, the 1500 - 1600 cm<sup>-1</sup> band corresponds to the C=C functional group, and the 1050 - 1300 cm<sup>-1</sup> range corresponds to the C-O functional group.

The functional groups in coconut shell activated carbon include C=O groups at 1751.24 cm<sup>-1</sup>, C=C (stretch) groups at 1542.95 cm<sup>-1</sup>, C-C groups at 1155.28 cm<sup>-1</sup>, and C=C (bending) groups at 885.27 cm<sup>-1</sup> [27]. The functional groups in animal bone activated carbon include O-H stretching vibrations at 3431.05 cm<sup>-1</sup>, C=O

stretching vibrations between 1800 and 1650 cm<sup>-1</sup>, and C-O stretching vibrations between 1600 and 1400 cm<sup>-1</sup> [31].

FT - IR spectroscopy is commonly used to identify and analyse the characteristics of these functional groups. FT - IR spectroscopy is a highly valuable analytical technique widely employed in analysing various food products. This is due to its relatively fast analysis time, accurate measurement results, simple preparation, and the ease with which individuals can perform it without requiring specialized skills [8]. FT - IR spectroscopy can differentiate the spectra of two distinct samples based on the characteristics of their intramolecular structure.

The ability of a compound to absorb light varies depending on its physicochemical properties, the bonds between atoms, and the characteristics of its functional groups. The complexity of IR spectrum patterns can make direct and visual interpretation challenging [35]. The characterization of activated carbon derived from coconut shell charcoal and hazelnut shell charcoal using FT - IR results in the identification of the same functional groups, namely O-H, C=C, C-H, and C-O, although the wave number adsorption regions differ slightly [36].

## CONCLUSIONS

The pyrolysis of 6 kg of dried and heated nutmeg shells, ranging from room temperature to 450°C, resulted in a charcoal yield of 3.2 kg, liquid smoke pyrolysate of 1.1 L, tar of 65 g, and non - condensed gas. Activation of the charcoal derived from the pyrolysis of nutmeg shells resulted in activated charcoal with distinct characteristics. The average pore diameter of the charcoal before activation from nutmeg shells was 3.09 μm. In comparison, the activated charcoal treated with CaCl<sub>2</sub> solution had an average pore diameter of 2.72 μm,

and the activated charcoal treated with  $ZnCl_2$  solution had an average pore diameter of 3.02  $\mu m$ . The activated charcoal from nutmeg shells contains the elements C, O, Al, and Ca, with carbon being the predominant element in the composition. FT - IR analysis of the charcoal from the pyrolysis of nutmeg shells before and after activation with  $CaCl_2$  and  $ZnCl_2$  solutions revealed similar adsorption patterns at nearly the same wave numbers. The functional groups identified include -OH, aromatic C-H, aliphatic C-H, C=C, and C-O.

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### Authors' contributions

*J.Z.L.: Conceptualization, Methodology, Analysis FT - IR, Writing oroginal draft; M.J.R.: Investigation, Data collection, Analysis SEM - EDS, Review; V.A.T.: Review, Editing, Visualization, Resources.*

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