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EFFECT OF PHOSPHORIC ACID (H_3PO_4) ACTIVATION ON THE PREPARATION OF ACTIVATED CARBON FROM *GYMNOSTOMA RUMPHIANUM* WOOD

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ABSTRACT

The utilization of Roya wood branches (RWB) as raw material for activated charcoal production has not yet been optimized in North Sulawesi, specifically in Wulauan Village, North Tondano District. This study aims to present the results of the analysis of the physical and chemical properties, X-Ray Diffraction (XRD), and Transmission Electron Microscope (TEM) of RWB-based activated charcoal. The production of activated charcoal was conducted in three stages: sample preparation, pyrolysis of RWB at a temperature of 365°C for approximately 3 hours, and activation of RWB charcoal using phosphoric acid (H_3PO_4) at concentrations of 1%, 2%, and 3% for 24 hours. The analysis of physical and chemical properties of both non-activated and H_3PO_4-activated RWB activated charcoal showed that the best quality was obtained at a 3% activator concentration, with a moisture content of 0.1683%, ash content of 2.3321%, volatile matter content (VMC) of 0.9429%, fixed carbon (FC) content of 96.7250%, and iodine adsorption capacity (IAC) of 10,535 mg/g, all of which meet the SNI 06-3037-1995 for activated charcoal. XRD analysis showed that non-activated RWB charcoal had three broad diffraction peaks in the ranges of 9–14°, 20–25°, and 35–50°, along with one sharp peak in the range of 25–30°. After activation with H_3PO_4, only three broad diffraction peaks were observed at 9–14°, 20–25°, and 35–50° for all three H_3PO_4 concentration variations.TEM analysis indicated that non-activated RWB charcoal had particle sizes ranging from 20–33 nm, while after activation with 1% H_3PO_4, the particle sizes ranged from 41–51 nm.

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INTRODUCTION

Indonesia possesses exceptionally high biodiversity, including many tree species with strong potential as biomass energy sources. One species that remains underexplored but holds significant promise is the mountain casuarina (*Gymnostoma rumphianum* (Miq.) L.A.S. Johnson), locally known as Roya wood. This species belongs to the Casuarinaceae family, which is characterized by hardwood properties, high durability, and suitability as a fuel source [1], [2].

Roya wood grows predominantly in tropical regions, particularly mountainous areas and rainforests [3], such as Sulawesi and Maluku [4]. In Marawas Subdistrict, Minahasa Regency, North Sulawesi

Province, local communities traditionally use Roya wood as a fuel for metal combustion, highlighting its high thermal potential. This finding aligns with previous studies reporting that Casuarinaceae family members exhibit low ash content and good combustibility even in a green condition, making them efficient fuel sources [2]. Despite this potential, the utilization of Roya wood is still limited to traditional charcoal production. Given its dense and durable structure, Roya wood shows considerable promise as a raw material for activated charcoal.

Activated charcoal is a porous material widely applied in water filtration, air purification, heavy metal adsorption, and the pharmaceutical and chemical industries. Its quality depends on the raw material type and processing conditions, with high-density and low-ash woods considered the most suitable feedstocks. According to [5], the production of activated charcoal generally involves three stages: drying, pyrolysis, and activation. The drying stage removes moisture, often under sunlight or with an oven, to prepare the raw material for pyrolysis. Pyrolysis, a thermochemical process conducted under high temperatures without oxygen, decomposes organic matter into solid, liquid, and gaseous products [6]. The final activation stage removes hydrocarbons from the charcoal surface to enhance porosity and adsorption properties [7]. This stage is commonly carried out through chemical activation using reagents such as H_3PO_4, KOH, $ZnCl_2$, or H_2SO_4.

Previous studies on other Casuarinaceae members, such as *Casuarina junghuhniana*, demonstrated that single and double activation with 2N KOH immersion for 1–5 days yielded iodine adsorption capacities of 317.25–507.60 mg/g and ash contents of 8–30%. These results did not meet the Indonesian National Standard (SNI 06-3730-1995), which requires a minimum iodine number of 750 mg/g and a maximum ash content of 10% [8]. Activation with HCl and NaOH for 24 hours resulted in moisture and ash contents of 2.97% and 3.92%, respectively [9]. Meanwhile, immersion in 6% CH_3COOH for 24 hours followed by heating at 105 °C produced charcoal with an iodine adsorption capacity of 761.58 mg/g [10].

Among the commonly used activating agents, H_3PO_4 is particularly effective in producing highly porous activated charcoal [11], compared to KOH. This effect is attributed to H_3PO_4 diffusion into the pores, which enhances acid hydrolysis and promotes pore development [12]. Furthermore, H_3PO_4 penetrates deeply into pore structures, retaining the primary framework while facilitating the removal of alkali, alkaline earth metals, and iron impurities [13]. Studies have shown that H_3PO_4 activation of jute-stick-based charcoal results in higher iodine adsorption capacity compared to activation with H_2SO_4 or $ZnCl_2$ [14]. Additionally, H_3PO_4 is considered more environmentally friendly, requiring lower energy input and yielding higher carbon recovery [15].

Based on these considerations and the limited scientific studies investigating the potential of Roya wood, this research seeks to evaluate the effect of H_3PO_4 activation on the properties of activated charcoal derived from Roya wood. The study supports utilizing local biomass resources and advances the development of environmentally friendly, high-performance activated charcoal materials.

METHODS

1. Materials and Equipment

This research was conducted at the Chemistry Laboratory, Faculty of Mathematics, Natural Sciences, and Earth Sciences, Manado State University, from November to December 2024.

The raw material used was Roya wood branches (RWB) collected from the forest area of Marawas Subdistrict, Minahasa Regency, North Sulawesi Province. Phosphoric acid (H_3PO_4, technical grade) at concentrations of 1%, 2%, and 3% served as the activating agent. A 0.1 N iodine (I_2) solution was used to determine iodine adsorption capacity, while 0.1 N sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$, Alba Chemicals) was employed as the titration agent. Potassium iodide (KI, Alba Chemicals) was used as the iodine dissolving agent, and distilled water was used throughout the testing process.

The main instruments included a pyrolysis reactor for carbonization, a furnace (Ceramic Muffle Furnace FNC 7) for ash and volatile matter determination, an oven (Memmert UN 160) for drying and moisture content analysis, a universal indicator (Nesco pH paper 0–14), and a 100-mesh sieve. Structural characterization was conducted using an X-ray diffractometer (Bruker D6 Phaser) with a Cu anode radiation source (λ = 1.5406 Å) to identify crystalline or amorphous phases and determine carbon purity. Particle size and d-spacing were analyzed using a Transmission Electron Microscope (TEM, JEOL JEM 1400) with an acceleration voltage of 40–120 kV and a LaB_6-cathode gun.

2. Sample Preparation

RWB samples were dried at room temperature, cut into approximately 5 × 7 cm pieces, and divided into four smaller parts. The bark was removed prior to pyrolysis.

3. RWB pyrolysis.

The cleaned RWB samples were loaded into the pyrolysis reactor and heated for approximately 3 hours at 365°C (the maximum temperature of the reactor without temperature control). After cooling, the resulting charcoal was ground with a mortar and pestle, then sieved using a 100-mesh sieve [16].

4. Activation of RWB charcoal

Four RWB charcoal samples, each weighing 15 grams, were prepared and placed into 125 mL Erlenmeyer flasks. Each charcoal sample was labeled as: without activation (WA), 1%, 2%, and 3%. Then, 100 mL of H_3PO_4 solution at concentrations of 1%, 2%, and 3% (v/v) (low concentrations to minimize the use of distilled water during neutralization) was added to each flask according to the concentration label. The same procedure was applied for the charcoal without activation, but distilled water was used as a substitute for H_3PO_4. All four samples were left to stand for 24 hours [16], followed by filtration until no water dripped. After drying, the activated charcoal samples treated with 1%, 2%, and 3% H_3PO_4 were washed with distilled water until reaching pH 7. Then, all four samples were dried in an oven at 110 ±1°C for 2 hours [16]. Once all activated charcoal samples were dry, physical and chemical properties were analyzed, which included: moisture content analysis using an oven at 105°C for 2 hours [17]; ash content analysis using a furnace at 650°C for 5 hours and 30 minutes [17]; volatile matter (VM) analysis using a furnace at 950°C for 7 minutes [5]; fixed carbon (FC) analysis calculated by subtracting the sum of ash content and VM from 100% [18]; and Iodine adsorption capacity (IAC) analysis using sodium thiosulfate titration [17]. The samples were then characterized using X-ray Diffraction (XRD) and Transmission Electron Microscope (TEM) instruments.

$$\text{Moisture Content (\%)} = \frac{a-b}{a} \times 100\% \quad [7]$$

$$\text{Ash Content (\%)} = \frac{a-b}{a} \times 100\% \quad [19]$$

$$\text{VMC (\%)} = \frac{W_1-W_2}{W_1} \times 100\% \quad [20]$$

$$\text{FCC \%} = 100\% - (\%Ash\ \text{content} + \%\text{VMC}) \quad [18]$$

$$\text{IAC (mg/g)} = A\,\frac{\frac{B.N\ Na_2S_2O_3}{N\ Iodium}}{a}\ 126{,}93\ df \quad [7]$$

Notation:

a/W_1	=	Sample weight before heating (g)
b/W_2	=	Sample weight after heating (g)
A	=	Volume of Iodine solution (mL)
B	=	Volume of Sodium thiosulfate used (mL)
df	=	Dilution factor
a	=	Mass of activated charcoal (g)
N ($Na_2S_2O_3$)	=	Concentration of sodium thiosulfate (N)
N (Iodine)	=	Concentration of Iodine (N)
126,93	=	Amount of Iodine in 1 mL of Sodium thiosulfate solution

RESULT AND DISCUSSION

1. Physical and Chemical Properties Analysis of RWB Activated Charcoal

a. Moisture Content

The moisture content analysis aims to determine the hygroscopic ability of activated charcoal based on the residual water retained in its pores after the pyrolysis and activation processes. The results of the moisture content analysis of RWB activated charcoal, both non-activated and H_3PO_4-activated, are presented in Table 1.

Tabel 1. Moisture Content of RWB-Based Activated Charcoal Without and With H_3PO_4 Activation.

Sample	Value Moisture Content (%)	Specification SNI 06-3730-1995
TA	0,0504	Maximum 15%
1	0,2631	
2	0,2275	
3	0,1683	

Based on Table 1, all unactivated samples and H_3PO_4-activated at concentrations of 1%, 2%, and 3%—met the maximum moisture content requirement of 15% according to SNI 06-3730-1995 for activated carbon. The values ranged from 0.0504% to 0.1683%, indicating good compliance with the standard. The relationship between moisture content and H_3PO_4 concentration is illustrated in Figure 1.

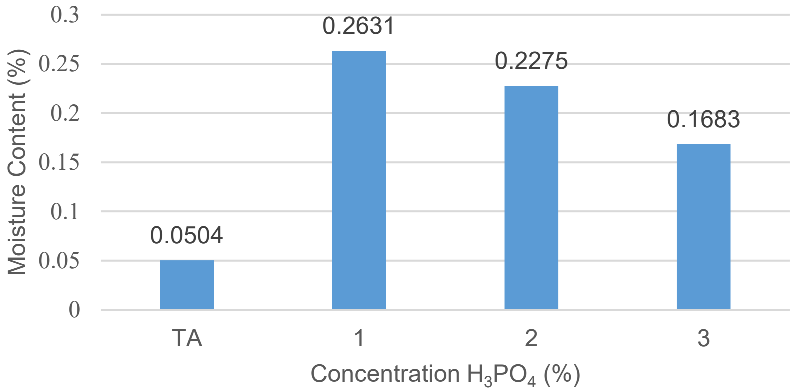


Figure 1. Moisture Content Analysis Percentage Graph

As shown in Figure 1, the moisture content of the unactivated RWB charcoal was lower than that of the H_3PO_4-activated samples. This increase can be attributed to introducing phosphate groups during activation, which render the surface of the charcoal more polar. Consequently, the surface exhibits a higher affinity for water molecules from the environment than unactivated samples [21].

However, the moisture content decreased as the H_3PO_4 concentration increased from 1% to 3%. This trend suggests that stronger interactions between H_3PO_4 and water molecules facilitated more effective water removal from the pores during heating, ultimately resulting in larger pore diameters in the activated charcoal [22].

Indrayani et al. [23] reported that lower moisture content correlates with a higher surface area, enhancing adsorption capacity. Similarly, several studies confirm that low moisture content increases the adsorption efficiency of activated charcoal [24]–[27].

The moisture content obtained in this study was significantly lower than that reported by Hasanah et al. [16], who observed 12.63% in unactivated sugarcane bagasse, and by Prasetiyo et al. [6], who found 8.16% in unactivated banana stem charcoal. These differences can be attributed to variations in raw material type and structural composition.

After the pyrolysis or activation process, the ash content analysis aims to determine the presence of inorganic minerals or metal oxides in the charcoal. The results of the ash content analysis of RWB-based activated charcoal, both non-activated and H_3PO_4-activated, are presented in Table 2.

Table 2. Ash Content of RWB-Based Activated Charcoal Without and With H_3PO_4 Activation.

Sample	Value Ash Content (%)	Specification SNI 06-3730-1995
TA	2,4158	Maximum 10%
1	2,4625	
2	2,3981	
3	2,3321	

Based on Table 2, the ash content results for samples without activation and activated with 1%, 2%, and 3% H_3PO_4 have met the active carbon ash content standard of SNI 06-3730-1995, which has a maximum limit of 10%, with ash contents of 2,4158%-2,3321%, respectively. The relationship between the ash content of non-activated and H_3PO_4-activated samples is shown in the graph in Figure 2.

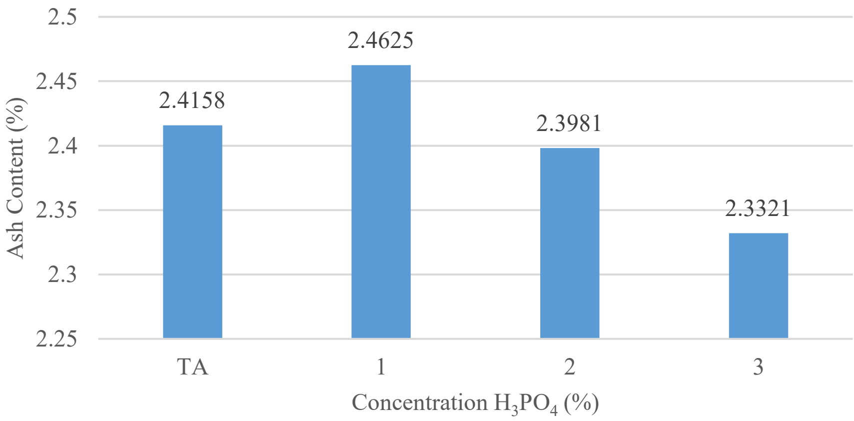


Figure 2. Graph of Ash Content Analysis Results.

Based on Figure 2, the ash content of unactivated charcoal slightly increased after activation with 1% H_3PO_4, but subsequently decreased with higher concentrations (2% and 3%). The initial increase in ash content at 1% H_3PO_4 is likely due to incomplete mineral removal during the washing stage, resulting in more residual inorganic oxides trapped within the pores of the charcoal. However, at higher concentrations of H_3PO_4, the activator more effectively dissolved these minerals, leading to a reduction in ash content. This trend is consistent with the findings of Prasetiyo et al. [6].

Previous studies using H_3PO_4 activation, such as those conducted by Adawi et al. [28] and Dwityaningsih et al. [29], reported the opposite trend, where ash content increased with increasing H_3PO_4 concentration. In contrast, the present study shows decreased ash content at higher activator concentrations. According to Rini et al. [18], H_3PO_4 plays an important role in corroding metallic elements within the charcoal, producing activated carbon with lower ash content. This is supported by Tiwow et al. [30], who found that inorganic minerals such as Fe, Mg, Ca, Al, and K often cover the pores of charcoal, thereby increasing the ash content.

The differences in trends between this study and earlier works are likely due to variations in the raw materials used. For example, research by Wijaya et al. [31] using teak wood–based activated charcoal also demonstrated a decreasing ash content trend. Lower ash content is generally considered advantageous, as it indicates reduced inorganic residue and enhances the effectiveness of activated charcoal as an adsorbent [20].

b. Volatile Matter Content

The volatile matter content (VMC) analysis was conducted to identify the presence of non-carbon compounds that remain attached to the activated charcoal after the pyrolysis and activation processes. The results of the VMC analysis of RWB-based activated charcoal, both untreated and H_3PO_4-activated, are presented in Table 3.

Table 3. Volatile Matter Content of RWB-Based Activated Charcoal Without and With H_3PO_4 Activation.

Sample	Value VMC (%)	Specification SNI 06-3730-1995
TA	0,9260	Maximum 25%
1	0,8108	
2	1,0435	
3	0,9429	

As shown in Table 3, both unactivated and H_3PO_4-activated samples met the SNI 06-3730-1995 standard for volatile matter in activated carbon, which requires a maximum value of 25%. The VMC values obtained ranged from 0.8108% to 1.0435%, well below the permissible limit. The relationship between untreated and activated samples is shown in Figure 3.

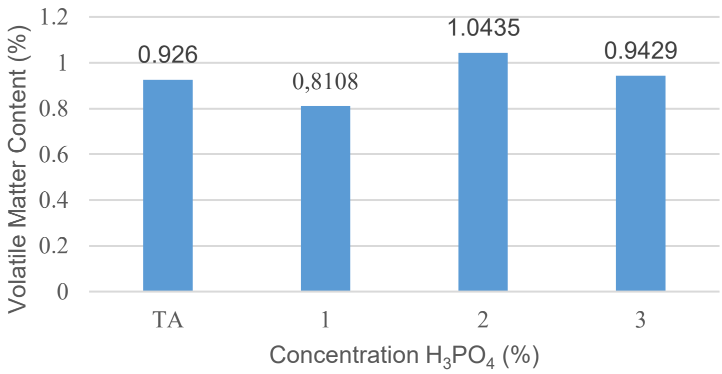


Figure 3. Percentage Graph of Volatile Matter Content Analysis Results: Kadar ZMM.

Based on Figure 3, the volatile matter content of RWB-based activated charcoal exhibited slight fluctuations after activation. The sample activated with 1% H_3PO_4 showed a decrease in VMC compared to the untreated sample. This reduction is likely associated with the relatively higher moisture content at this concentration, which evaporated during heating at 950°C, thus lowering the final VMC.

The VMC increased slightly at higher concentrations (2% and 3% H_3PO_4). This trend may be attributed to the role of H_3PO_4 as an activating agent that provides partial protection against thermal decomposition, thereby reducing the release of volatile elements such as sulfur and nitrogen during heating. Similar findings were reported in previous studies, which indicated that activated charcoal generally contains 87–

97% carbon, with the remainder composed of moisture, ash, sulfur, and nitrogen [18].

The VMC values obtained in this study were significantly lower than those reported by Prasetiyo et al. [6], where unactivated banana stem–based charcoal exhibited a VMC of 53.29%. This large difference highlights the influence of raw material type on volatile matter content. As noted in earlier works, high or low VMC indicates the extent of non-carbon compounds remaining on the surface of the activated charcoal, which can directly affect its adsorption performance.

c. Fixed Carbon Content

The fixed carbon (FC) content analysis determines the amount of pure carbon after subtracting the ash and volatile matter contents. The results of the FC content analysis of RWB-based activated charcoal, both untreated and activated with H_3PO_4, are presented in Table 4.

Table 4. Fixed Carbon Content of RWB-Based Activated Charcoal Without and With H_3PO_4 Activation.

Sample	Value FC (%)	Specification SNI 06-3730-1995
TA	96,6582	
1	96,7267	Maximum 65%
2	96,5584	
3	96,7250	

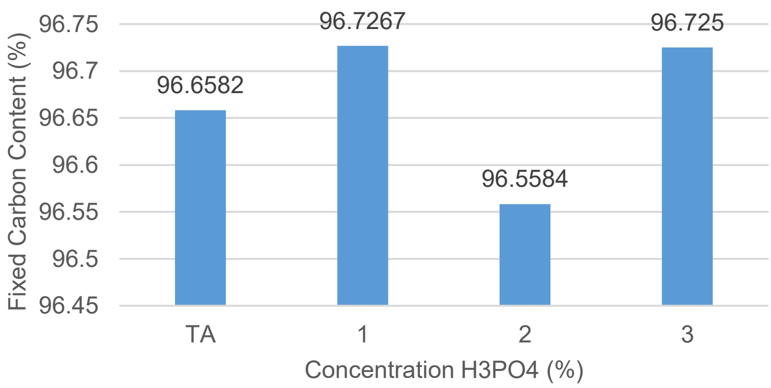


Figure 4. Percentage Graph of Fixed Carbon Content Analysis Results.

Based on Table 4, FC results for samples without activation and activated with 1%, 2%, and 3% H_3PO_4 have met the SNI 06-3730-1995 standard for activated carbon, which requires a minimum of 65%, with FC values of 96,6582%-96,7250%, respectively. The relationship between the FC of samples without activation and those activated with H_3PO_4 is shown in the graph in Figure 4. Based on Figure 4, an increase and a decrease in the fixed carbon (FC) content of RWB-based activated charcoal were observed. The high and low FC content of activated charcoal is influenced by the volatile matter (VM) and ash content. Furthermore, according to Hanavia et al. [32], the lower the VM and ash content, the higher the pure carbon content and the better the quality of the activated charcoal. According to Adawi et al. [28], a higher pure carbon content results from a higher cellulose content in the raw material used to produce activated charcoal. A higher FC value indicates the activated charcoal's adsorption capacity [5].

d. Iodine Adsorption Capacity

The Iodine adsorption capacity analysis aims to determine the ability of activated carbon to adsorb Iodine. The results of the iodine adsorption capacity (IAC) analysis for RWB activated carbon without activation and activated with H_3PO_4 are shown in Table 5.

Table 5. IAC Results of RWB-Based Activated Charcoal Without and With H_3PO_4 Activation.

Sample	Value IAC (mg/g)	Specification SNI 06-3730-1995
TA	8885	Maximum 750 mg/g
1	9266	
2	9520	
3	10535	

Based on Table 5, IAC results for samples without activation and activated with 1%, 2%, and 3% H_3PO_4 have met the SNI 06-3730-1995 standard for activated carbon, which requires a minimum of 750 mg/g, with IAC values of 8885 mg/g-10535 mg/g/g, respectively. The relationship between the IAC of samples without activation and those activated with H_3PO_4 is shown in the graph in Figure 5.

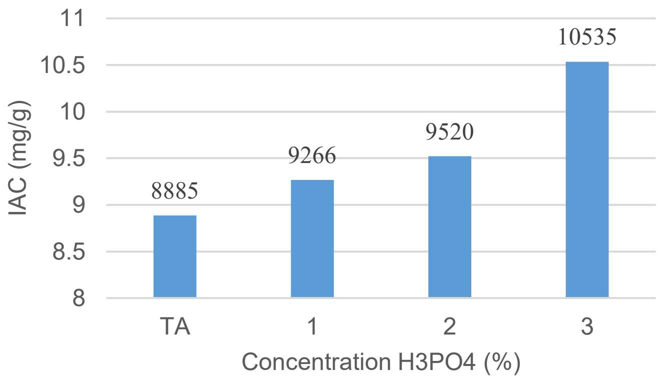


Figure 5. Percentage Graph of Iodine Number Analysis Results

Figure 5 shows that the higher the concentration of H_3PO_4, the greater the iodine adsorption value (IAC), due to a higher number of carbon atoms forming hexagonal crystalline carbon, which results in the formation of larger pores between the hexagonal crystal layers [33]. According to Saitun et al. [22], the increase in IAC is caused by removing impurities that previously blocked the pores, thus expanding the surface area of the activated charcoal and allowing more iodine to be adsorbed.

2. *X-Ray Diffraction* (XRD) Analysis

XRD analysis was conducted to identify the crystalline or amorphous phases, carbon purity, and d-spacing (interplanar distance) of RWB-based activated charcoal, untreated and activated with H_3PO_4. The XRD diffractograms of RWB activated charcoal without and with H_3PO_4 activation are shown in Figure 6.

The XRD results for both untreated and H_3PO_4-activated samples reveal broad and non-sharp peaks, indicating the presence of an amorphous carbon structure. Similar findings were reported by Argianti et al. [34], who observed broad and non-distinct peaks in coconut shell–based charcoal, characteristic of a predominantly amorphous structure. The amorphous phase is more advantageous than the crystalline phase for adsorption or filtration applications. The crystalline phase is more ordered and structured, leaving limited space for adsorbate molecules. In contrast, the amorphous phase has a disordered structure, which can generate a wider range and larger pore sizes, thereby facilitating better adsorption of molecules.

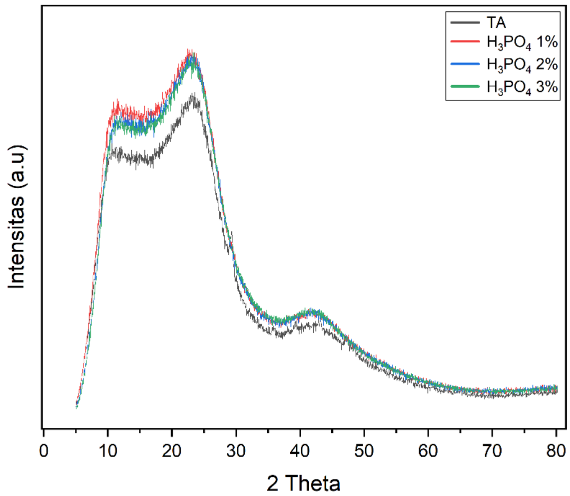


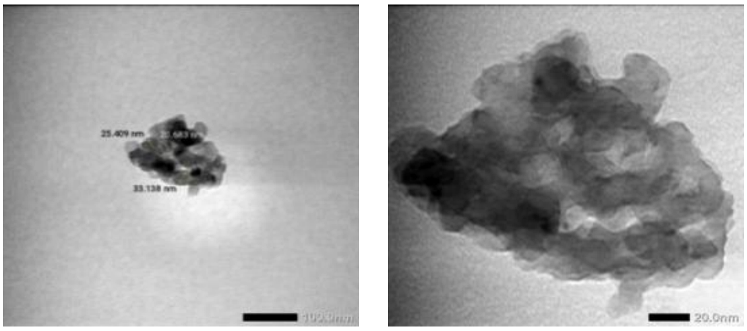
Figure 6. XRD Diffractograms of RWB-Based Activated Charcoal Without Activation (WA) and Activated with H_3PO_4.

In the untreated RWB charcoal sample, three broad diffraction peaks were observed within the ranges of 9–14°, 20–25°, and 35–50°, along with one sharp peak in the 25–30° range. The sharp peak is suspected to originate from residual ash or mineral content from pyrolysis, as also noted in the study by Saban et al. [9]. After activation with H_3PO_4, the diffractograms of all three concentrations showed only three broad peaks within 9–14°, 20–25°, and 35–50°, indicating that the addition of H_3PO_4 at varying concentrations had no significant influence on the diffraction pattern of the RWB activated charcoal. This may be due to the relatively small difference in the concentration variations applied.

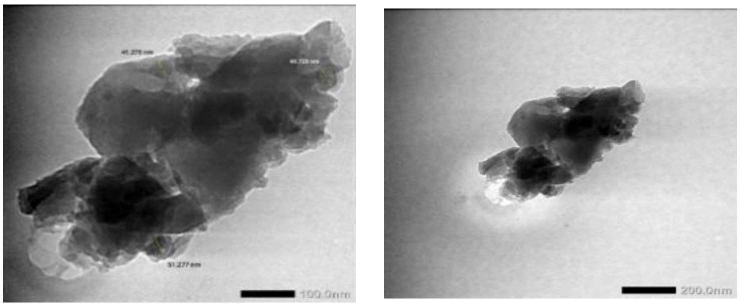
The diffraction peak in the 9–14° range of RWB-activated charcoal treated with H_3PO_4 shifted, became broader, and showed increased intensity (d-spacing value from 8.3570 Å to the range of 7.3563–7.8063 Å), indicating an increase in atomic layer disorder. At another peak in the 20–25° range (with a slight decrease in d-spacing from 3.7374 Å to the range of 3.6782–3.9032 Å) and 35–50° (a significant decrease in d-spacing from 2.9547 Å to the range of 2.1236–2.2535 Å), specific changes were observed, with peaks becoming broader and intensities increasing. This confirms that the structure became more amorphous, forming new pores on the surface of the RWB-activated charcoal. The formation of a more amorphous structure results in a wider range of pore sizes. These changes observed in the diffractogram of RWB activated charcoal indicate an increase in porosity and surface area, which has implications for improved adsorption performance. These results align with the findings of Argianti et al. [34], who reported a similar diffraction pattern.

3. *Transmission Electron Microscope* (TEM) Analysis

The purpose of TEM analysis is to observe the morphological distribution and particle size of RWB activated charcoal without activation and activated with 1% H_3PO_4. The TEM images of RWB activated charcoal without and with 1% H_3PO_4 activation are shown in Figure 7.



a.



b.

Figure 7. TEM Images of RWB Activated Charcoal (a) Without Activation (b) Activated with 1% H_3PO_4.

CONCLUSION

The results of the physical and chemical property analyses of RWB activated charcoal, both non-activated and activated with 1%, 2%, and 3% H_3PO_4, showed moisture content of 0.0504%, 0.2631%, 0.2275%, and 0.1683%; ash content of 2.4158%, 2.4625%, 2.3981%, and 2.3321%; volatile matter content of 0.9260%, 0.8108%, 1.0435%, and 0.9429%; fixed carbon content of 96.6582%, 96.7267%, 96.5584%, and 96.7250%; and Iodine adsorption capacity of 8885 mg/g, 9266 mg/g, 9520 mg/g, and

10535 mg/g, respectively. All values meet the SNI 06-3730-1995 for activated charcoal.

The XRD analysis showed that RWB activated charcoal samples, both non-activated and activated with 1%, 2%, and 3% H_3PO_4, exhibited amorphous characteristics. The non-activated RWB charcoal displayed three broad diffraction peaks within the ranges of 9–14°, 20–25°, and 35–50°, and one sharp peak around 25–30°. After H_3PO_4 activation, only three broad peaks remained at 9–14°, 20–25°, and 35–50° for all three H_3PO_4 concentration variations. The XRD patterns of the H_3PO_4-activated RWB charcoal showed increased intensity and broader peaks, indicating improved porosity and surface area. The d-spacing values of RWB activated charcoal for peak one were 2.9547 Å (Without activated), 2.1375 Å (1%), 2.1236 Å (2%), and 2.2535 Å (3%); for peak two, 3.7374 Å, 3.7023 Å, 3.6782 Å, and 3.9032 Å; and for peak three, 8.3570 Å, 7.4045 Å, 7.3563 Å, and 7.8063 Å, respectively.

The TEM analysis showed that the particle size of non-activated RWB charcoal ranged from 20–33 nm, and increased to 41–51 nm after activation with 1% H_3PO_4. The increase in particle size after H_3PO_4 activation indicates an enhancement in the porosity of the RWB activated charcoal.

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