

SYNTHESIS AND CHARACTERIZATION OF ACTIVATED CARBON FROM BIOMASS WASTE AS A MICROWAVE ABSORBER MATERIAL

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Abstract. Excessive use of electronic technology can result in harmful radiation and electromagnetic interference, which pose risks to human health. To address this issue, researchers developed a material designed to absorb electromagnetic waves. The study focused on synthesizing and characterizing activated carbon derived from biomass waste, including water hyacinth, melinjo seed shells, and chicken eggshells, with the goal of reducing electromagnetic wave interference. The research process involved several key steps: washing the biomass materials, followed by carbonization, activation using a 65% KOH solution, and subsequent characterization of the material. The tests revealed that the activated carbon possessed a porous structure, which is essential for its absorption capabilities. The surface areas measured were 4.378 m²/g for water hyacinth, 2.518 m²/g for melinjo seed shells, and 2.992 m²/g for chicken eggshells. These surface areas are indicative of the material's potential effectiveness. Additionally, the microwave absorption capacities of the activated carbon were recorded as -18.342 dB for water hyacinth, -13.326 dB for melinjo seed shells, and -12.484 dB for chicken eggshells. These findings suggest that the activated carbons are highly effective as microwave absorber materials, with an absorption efficiency ranging between 94% and 98%.

Keywords: Absorbent, Water hyacinth, Melinjo seed shells, Chicken egg shells, Microwaves

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INTRODUCTION

Rapid advancements in electronic technology can simplify human life, as seen with the evolving smartphone industry in telecommunications over the years. However, excessive use of electronic technology can lead to serious electromagnetic radiation and interference. Overexposure to such radiation and interference can negatively impact human health [1]. To address this issue, researchers are competing to create materials that efficiently absorb electromagnetic waves. These materials, generally known as Microwave Absorbing Materials (MAM), have the property of dampening or eliminating electromagnetic waves by converting them into thermal energy [2]. MAM must have the capability to interact with the electric or magnetic fields of incoming electromagnetic waves. Therefore, MAM are classified into two categories, magnetic loss type and dielectric loss type [3]. MAM is characterized based on loss mechanisms and intrinsic electromagnetic properties such as permittivity and permeability. In the selection of absorbing materials, recent research shows that materials with porous structures can effectively attenuate electromagnetic waves. These pores provide a large surface area, allowing repeated reflection and scattering of electromagnetic waves between the pore walls [4][5]. These repeated reflections extend the path traveled by the electromagnetic waves, thereby increasing their absorption and attenuation by the absorbing material [6]. Activated carbon has a high surface area and a large volume, with the specific surface area of activated carbon reaching up to 3000 m²/g [5]. The research conducted by Negi P et al. in 2020, titled "Activated Carbon Derived from Mango Leaves as an Enhanced Microwave Absorbing Material" explains that porous activated carbon has the potential to be an effective material in the production of microwave-absorbing materials. This study utilized a chemical activation method by mixing a KOH (Potassium Hydroxide) solution with mango leaf carbon. The reflection loss value obtained from the mango leaves was -23.26 dB at a frequency of 17.68 GHz and a thickness of 1.50 mm. The microwave absorption properties were achieved through impedance matching and microwave attenuation due to dielectric loss, facilitated by the porous structure of the activated carbon [6].

In the use of biomass, water hyacinth, scientifically known as *Eichhornia crassipes*, belongs to the *Pontederiaceae* family, while melinjo seed shells, scientifically known as *Gnetum gnemon L.*, are part of the *Gnetaceae* family [7][8][9]. Both biomasses contain cellulose, hemicellulose, and lignin,

making them suitable as raw materials for the production of microwave-absorbing materials [10][11]. Additionally, chicken eggshells, scientifically known as *Gallus gallus domesticus* from the *Gallus gallus* family, contain approximately 98% calcium carbonate [12]. Research shows that chicken eggshells can be processed into calcium carbonate powder with various uses, such as in the food and pharmaceutical industries. Additionally, eggshells can also be used as an absorbent material [13]. The use of biomass waste, such as water hyacinth, is due to its nature as an aquatic weed with rapid growth. This rapid growth can cover water surfaces and cause environmental issues [14]. Additionally, biomass waste like melinjo seed shells and chicken eggshells, which are by-products of essential daily needs, are abundantly available but often underutilized, ending up as unprocessed waste [15][16].

Based on the review outlined previously, this study involves the synthesis and characterization

of activated carbon materials derived from water hyacinths, melinjo seed shells, and chicken eggshells. This study employs the chemical activation method using a KOH solution. It has been reported that using potassium hydroxide (KOH) as an activating agent, carbon can achieve a surface area of up to 3000 m²/g [17]. The synthesized materials will be characterized using SEM to observe the morphology and microstructure of the materials, BET Surface Area Analyzer to assess the pore surface area of the materials before and after activation, and VNA to examine the microwave absorption properties of the activated carbon materials. The study concludes that utilizing biomass waste for the production of activated carbon not only offers a new innovation but also contributes to maintaining environmental cleanliness.

EXPERIMENTAL SECTION

Chemicals and Instruments

The synthesis of this activated carbon was conducted at the Integrated Laboratory Center (PLT), Syarif Hidayatullah State Islamic University (UIN) Jakarta. The study utilized three primary components for its production: water hyacinths, melinjo seed shells, and chicken eggshells. Additionally, chemical reagents were employed for carbon activation, including a 65% KOH solution, as well as distilled water and a 2M HCI solution as washing or neutralizing agents for the activated carbon. The material was then characterized using the SAA Quantachrome Novatouch LX-4, SEM-EDS Hitachi Flexsem 100, and VNA Anritsu MS46322A instruments, conducted in several locations.

Synthesis of Activated Carbon

Activated Carbon Water Hyacinths and Melinjo Seed Shells

Water hyacinths and melinjo seed shells were washed and cut into small pieces, then carbonized using a furnace at 300°C for 3 hours [18] and at 400°C for 15 minutes [11]. The resulting carbon weights were 37.1 grams for the water hyacinths sample from an initial weight of 83.3 grams and 31.5 grams for the melinjo seed shells sample from an initial weight of 60.7 grams, as summarized in Table 1. Each type of carbon was ground using a mortar and pestle and sieved with a 200-mesh

sieve, resulting in 30 grams of material for each sample after sieving. Subsequently, 5 grams from each sample were set aside as non-activated samples. The remaining 25 grams of each carbon sample were activated using a 65% KOH solution with a 4:1 weight ratio for 24 hours. The weights of the activated carbon obtained were 16 grams for water hyacinths and 23 grams for melinjo seed shells, as shown in Table 2. The activated carbon samples were stirred using a magnetic stirrer at 200 rpm and 90°C for 4 hours for the water hyacinths sample [18], and at 110 rpm and 110°C for 5 hours for the melinjo seed shells sample [11]. After stirring, the samples were filtered using filter paper and rinsed with distilled water until neutral. Once neutral, the activated carbon samples were dried in an oven at 120°C for 4 hours for water hyacinths [18] and at 110°C for 2 hours for melinjo seed shells [11].

Activated Carbon Chicken Eggshells

Chicken eggshells were washed and then dried in an oven at 110°C for 1 hour. Subsequently, the eggshells were ground using a mortar and pestle until they became small-sized particles and were sieved using a 200-mesh sieve. The eggshells were then carbonized in a furnace at 600°C for 2 hours [19], resulting in a final weight of 30.9 grams from an initial weight of 32.6 grams, as shown in Table 1. Subsequently, 5 grams of the carbonized eggshell sample were set aside as non-activated carbon. The remaining 25 grams were activated using a 65% KOH solution with a 4:1 ratio for 24 hours. After the activation process, the sample weighed 12 grams, as shown in Table 2. The activated carbon sample was stirred using a magnetic stirrer at a speed of 110 rpm and a temperature of 110°C for 5 hours. After stirring, the sample was filtered using filter paper and washed with distilled water and 2M HCI until neutral. The use of HCI was necessary because the chicken eggshell sample was highly alkaline. Once the activated carbon was neutralized, it was dried in an oven at 110°C for 3 hours [20].

Sample Characterization

The samples were characterized using non-destructive testing, including Scanning Electron Microscope – Energy Dispersive Spectroscopy (SEM-EDS) with the Hitachi Flexsem 100. This test aimed to observe the microstructural morphology of the three activated carbons using the secondary electron mode. The analysis employed a magnification of 2,500× with an accelerating

voltage of 8.00 kV for the water hyacinths sample and 20.0 kV for the chicken eggshells and melinjo seed shells samples. Visual observations of the three activated carbons are shown in Figure 2. Additionally, the EDS feature was utilized to identify and determine the chemical element composition of the three activated carbons, as summarized in Table 4. Surface Area Analyzer (SAA) characterization was conducted using the Quantachrome Novatouch LX-4. This test aimed to determine the surface area of the three samples by flowing nitrogen gas and performing degassing at 300°C for 7 hours. Finally, testing was performed using the Vector Network Analyzer (VNA) Anritsu MS46322A to measure the microwave absorption level within the x-band frequency range of 8–12 GHz.

RESULTS AND DISCUSSION

Mass Changes in Samples

In the synthesis of activated carbon, the process generally involves preparation, carbonization, activation, and characterization stages. However, during the synthesis of activated carbon, the samples undergo changes in mass, particularly during the carbonization and activation stages. Carbonization is the process of converting a material into carbon or transforming organic substances into carbon-containing residues during charcoal production [21].

The carbonization process involves the removal of non-carbon elements, such as hydrogen and oxygen, in the form of volatile gases through pyrolytic decomposition, leaving behind only carbon elements with a porous structure that remains imperfect [22]. This carbonization process results in mass changes in each sample, as shown in Table 1.

Sample —	Carbonizatio	Percentage	
	Before	After	Reduction
Water Hyacinths	83.3	37.1	55%
Melinjo Seed Shells	60.7	31.5	48%
Chicken Eggshells	32.6	30.9	5%

Table 1. Sample Mass Before and After Carbonization

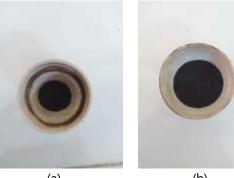
The mass before and after carbonization was measured for the three samples. The water hyacinths sample experienced a 55% mass reduction, from an initial weight of 83.3 grams to 37.1 grams, under carbonization conditions of 300°C for 3 hours. The melinjo seed shells sample showed a 48% mass reduction, from an initial weight of 60.7 grams to 31.5 grams, under carbonization conditions of 400°C for 15 minutes. Meanwhile, the chicken eggshells sample experienced only a 5% mass reduction, from an initial weight of 32.6 grams to 30.9 grams, under carbonization conditions of 600°C for 2 hours. The higher carbonization temperature for the chicken eggshells sample is due to the absence of lignocellulose content, which is typically found in plant-derived materials, making a higher temperature necessary for carbon formation. After the carbonization can be categorized into three types: chemical activation, physical activation, and chemical-physical activation. In this study, chemical activation was performed by mixing the samples with a 65% KOH solution at a ratio of 4:1 by weight (in grams). Chemical activation involves breaking carbon chains in organic compounds with the aid of chemical agents [23]. The mass before and after activation is detailed in Table 2.

Comple	Activation	Mass Daduction	
Sample —	Before	After	— Mass Reduction
Water Hyacinths	25	16	36%
Melinjo Seed Shells	25	23	8%
Chicken Eggshells	25	12	52%

Table 2.	Sample	Mass	Before	and	After	Activation
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The mass before and after activation was measured for the three samples. The water hyacinths sample showed a 36% reduction, decreasing from an initial weight of 25 grams to 16 grams. The melinjo seed shells sample experienced an 8% reduction, from an initial weight of 25 grams to 23 grams. The chicken eggshells sample exhibited a 52% reduction, from an initial weight of 25 grams to 12 grams. The mass reduction occurred due to several factors during the activation process,

including the formation of gases from the decomposition of organic materials, the loss of volatile compounds, and the reduction of water molecules bound to the carbon [24]. Figure 1 illustrates the appearance of the samples after undergoing the synthesis process.



(a)

(b)

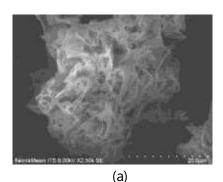


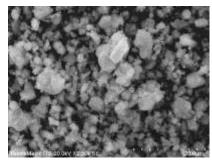
Figure 1. Physical characteristics of activated carbon samples: (a) Activated carbon from water hyacinth, (b) Activated carbon from melinjo seed shells, (c) Activated carbon from chicken eggshells

After the samples underwent the synthesis process, they were characterized, and three characterizations were performed, including Scanning Electron Microscope – Energy Dispersive Spectroscopy (SEM-EDS) using the Hitachi Flexsem 100, Surface Area Analyzer (SAA) using the Quantachrome Novatouch LX-4, and Vector Network Analyzer (VNA) using the Anritsu MS46322A. In this study, to assess the quality of the three activated carbons in microwave absorption, the microstructural morphology and chemical composition were identified, the surface area was analyzed, and the microwave absorption level was measured.

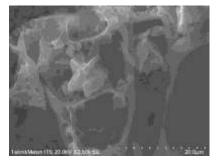
SEM-EDS Microstructure Morphology Results

To analyze the microstructure and morphology of activated carbon, Scanning Electron Microscope – Energy Dispersive Spectroscopy (SEM-EDS) testing was conducted. In this measurement, a magnification of 2,500× and an accelerating voltage of 8.00 kV were used for the water hyacinths sample, and 20.0 kV for the chicken eggshells and melinjo seed shells samples. The observation of the microstructure of the activated carbon surface in Figure 2 shows that the activated carbon particles have a porous structure. The pores on the carbon surface result in a large specific surface area, which plays a role in microwave absorption. These pores are formed due to the activated carbon process. During this process, gases such as CO and CO_2 are released, diffusing onto the activated carbon surface and eroding it, thereby forming the pores [25].









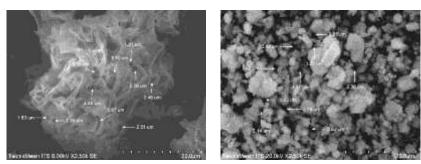
(C)

Figure 2. (a) Morphology of activated carbon from water hyacinth, (b) Morphology of activated carbon from chicken eggshells, (c) Morphology of activated carbon from melinjo seed shells.

From the morphological analysis, pore size measurements were performed using ImageJ software. The measurement involved taking 10 pore diameter measurements to obtain an average result for each sample, as shown in Figure 3. The largest average pore diameter was found in the water hyacinths sample at 2.85 μ m, followed by the chicken eggshells sample with a pore diameter of 2.09 μ m, and the melinjo seed shells sample with a pore diameter of 1.93 μ m. Grain size analysis was also conducted using ImageJ software, with 10 grain size measurements taken to calculate the average result for each sample, as shown in Figure 4. The average grain sizes obtained were 4.32 μ m for the water hyacinths sample, 3.66 μ m for the chicken eggshells sample, and 3.44 μ m for the melinjo seed shells sample. The average grain size and pore diameter are summarized in Table 3.

Sample	Average			
Sample	Grain Size (µm)	Pore Diameter Size (µm)		
Water Hyacinths	4.32	2.85		
Melinjo Seed Shells	3.66	2.09		
Chicken Eggshells	3.44	1.93		

Table 3. Table of the average grain size and pore diameter of activated carbon.



(a)

(b)

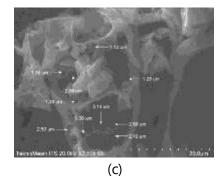
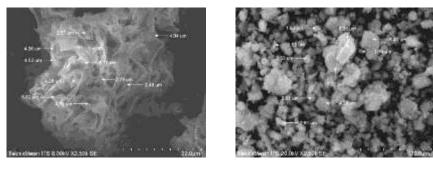


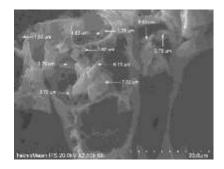
Figure 3. (a) Pore size distribution of water hyacinths activated carbon, (b) Pore size distribution of chicken eggshells activated carbon, (c) Pore size distribution of melinjo seed shells activated carbon

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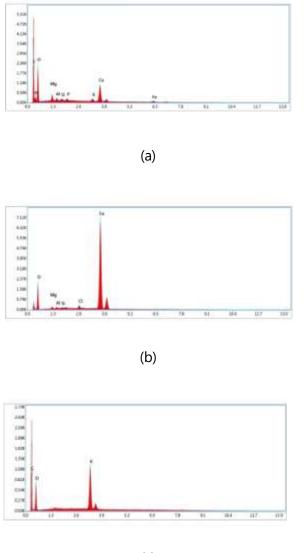
(a)





(c)

Figure 4. (a) Grain size of water hyacinths activated carbon, (b) Grain size of chicken eggshells activated carbon, (c) Grain size of melinjo seed shells activated carbon



(C)

Figure 5. (a) EDS spectrum of water hyacinths activated carbon, (b) EDS spectrum of chicken eggshells activated carbon, (c) EDS spectrum of melinjo seed shells activated carbon

In its production, activated carbon is made through two stages: the carbonization stage and the activation stage [26]. Carbonization is a process used to increase the calorific value of biomass through heating or combustion. The result of carbonization is black charcoal containing carbon [27]. After the carbonization process, the next stage is activation, which is a treatment of the carbon aimed at enlarging the pores by breaking hydrocarbon bonds or oxidizing surface molecules. In general, carbon can be activated in three ways: chemical activation, physical

activation, or a combination of both, which can produce activated carbon [28]. Activated carbon is carbon that has undergone activation, resulting in a larger surface area due to an increased number of pores [29]. This indicates that carbon that has gone through the activation process can be classified as activated carbon. In the chemical composition analysis using EDS (Energy Dispersive Spectroscopy), it was observed that the water hyacinths and melinjo seed shells samples contained the element C (Carbon) with wt% (mass percentage) and at% (atomic percentage) values of 34.14% and 42.28% for the water hyacinths sample, and 42.23% and 53.87% for the melinjo seed shells sample, as shown in Table 4. This test demonstrated that the water hyacinths and melinjo seed shells samples had successfully transformed into carbon. However, the carbon content in the chicken eggshells sample was not detected. There may be several factors contributing to the failure of carbon formation in the chicken eggshell sample, one of which could be an insufficiently high temperature during the carbonization process.

Element	Water H	Water Hyacinth		Melinjo Seed Shells		Chicken Egg Shells	
Liement	wt%	at%	wt%	at%	wt%	at%	
Fe	0,78	0,21	-	-	_	-	
Ca	5,94	2,21	-	-	45,64	25,58	
К	0,72	0,27	16,25	6,37	-	-	
Р	0,47	0,23	-	-	-	-	
Cl	-	-	-	-	1,03	0,65	
Si	0,46	0,24	-	-	0,37	0,30	
Al	0,67	0,37	-	-	0,61	0,51	
Mg	1,91	1,17	-	-	1,11	1,03	
0	39,92	37,11	41,52	39,76	51,24	71,94	
Ν	14,99	15,91	-	-	-	-	
С	34,14	42,28	42,23	53,87	-	-	
Tot	tal			100			

Table 4.	The Composition	of Elemental Constituents
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BET-SAA Surface Area Results

In the surface area analysis of the three samples, surface area characterization was conducted using a Surface Area Analyzer (SAA) with a Quantachrome Novatouch LX-4 device. This instrument is used to directly determine the surface area of the resulting activated carbon. Before measuring the surface area, the sample mass was weighed to determine the surface area per gram of sample. The analysis was performed using a degassing temperature of 300°C for 7 hours. The purpose of the degassing temperature is to remove impurities from the sample being measured. During the measurement, nitrogen gas (N₂) at a temperature of 77.35°K was passed over the surface of the activated carbon, filling the pores formed by the activated carbon. The results obtained from this BET characterization include surface area (m²/g), pore volume (cc/g), and pore size (nm). Adsorption-desorption isotherms using N₂ gas at 77.35°K were obtained from the water hyacinths, melinjo seed shells, and chicken eggshells activated carbon samples, measured to determine the porosity and surface area of the samples. From the isotherm curve in Figure 6, it can be seen that at a relative pressure in the range of P/Po < 0.4, nitrogen adsorption is relatively low, which may be due to a lack of micropores in the sample [6].

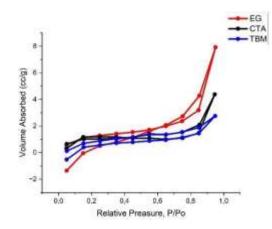


Figure 6. Activated carbon isotherm curve

However, as the P/Po value (relative pressure) increases, nitrogen adsorption continues to rise, with adsorption at P/Po = 0.94 indicating that the formed pores are mesopores in the sample. This is also supported by the hysteresis curve generated at relative pressures from 0.4 to 0.94, which shows that the sample has mesoporous characteristics. It can also be seen from Table 5 that

the pore sizes of the three samples are above 2 nm. The surface area, pore volume, and pore size of the activated carbon produced from the three materials can be seen in Table 5.

Type of Biomass	Surface Area (m^2/g)	Pore Volume (<i>cc</i> /	Pore Size (nm)
Mator Hussinths	4 270	<i>g</i>)	E COC
Water Hyacinths	4.378	0.012	5.606
Melinjo Seed Shells	2.518	0.004	3.394
Chicken Eggshells	2.992	0.007	4.533

Table 5. Surface area of activated biomass carbon

A large surface area indicates that the carbon can absorb optimally. The larger the pore surface area on the adsorbent, the higher its absorption capacity [26]. As shown in Table 5, the surface area of the activated carbon from water hyacinths is $4.378 \text{ m}^2/\text{g}$, the surface area of the chicken eggshells is 2.992 m²/g, and the surface area of the activated carbon from melinjo seed shells is 2.518 m²/g. Among the three samples, the activated carbon from water hyacinths has the largest surface area compared to the other activated carbons.

Table 6. Surface Area Before and After Activation

Turne of Pierman	Surface Ar	Surface Area	
Type of Biomass	Non-Activation	Activation	Changes (ΔA)
Water Hyacinths	1.363	4.378	3.015
Melinjo Seed Shells	1.829	2.992	1.163
Chicken Eggshells	0.241	2.518	2.277

The measurement of surface area changes before and after activation was also conducted, as shown in Table 6. The table shows an increase in surface area after the activation of biomass carbon. This increase is due to the activation process, which can cause changes in the pore structure, leading to the expansion of the carbon surface. The surface area results obtained from the samples were very small. It is possible that the presence of impurities in the samples is still high. Higher moisture content, volatile substances, and ash content can reduce the surface area of activated carbon because the pores in the activated carbon become clogged [24]. This is supported by the particle size values, which are still larger than the pore sizes in the SEM testing, as shown in Table 3. It can be seen that the particle size values range from $3.43\mu m$ to $4.32\mu m$, while the pore diameter ranges from $1.93\mu m$ to $2.85\mu m$ in the three samples. This information is supplemented by the EDS test, as shown in Table 4, which indicates that the mass percentage of impurities, when summed, still exceeds 50% of the adsorptive elements.

VNA Microwave Absorption Results

In measuring the microwave absorption rate, the samples were characterized using the Vector Network Analyzer (VNA) Anritsu MS46322A. The data obtained from this characterization includes the reflection coefficient, which is used to create the reflection loss (RI) curve. This reflection loss curve indicates the material's ability to absorb electromagnetic waves.

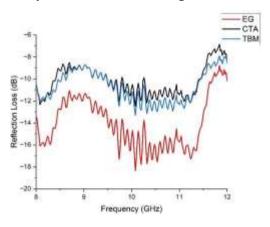


Figure 7. Microwave absorption curve for activated carbon samples

The samples were tested in the X-band frequency range, which is 8-12 GHz, and the Reflection Loss (RL) data were obtained from the reflection coefficient. Among the three samples, namely water hyacinths, chicken eggshells, and melinjo seed shells, it was found that the activated carbon from water hyacinths had the highest microwave absorption capability compared to the other samples. This can be seen from the curve in Figure 7, where the activated carbon from water hyacinths has a much deeper dip compared to the other samples.

Comula	Frequency	Reflection Loss	Through	Reflection
Sample	(GHz)	(dB)	Power (%)	Coefficient (F)
Water Hyacinths	10.08	-18.342	98.54	0.121
Melinjo Seed Shells	10.08	-13.326	95.35	0.216
Chicken Eggshells	10.08	-12.484	94.36	0.238

Table 7. Electromagnetic wave absorption data

Overall, all three samples showed excellent electromagnetic wave absorption capabilities, as indicated by the VNA data summarized in Table 7. The reflection loss values for each sample ranged from -12.484 dB to -18.342 dB. The smaller the reflection loss value, the better the material's performance in absorbing microwave waves. The Through Power values, which indicate the maximum wave adsorption strength at a frequency of 10.08 GHz, ranged from 94.36% to 98.54%, and the reflection coefficient values, indicating the amount of reflected waves, are shown in Table 7. This high absorption is due to the porous structure of the surface area produced by the activated carbon. However, in this study, while the eggshell sample exhibited a large surface area compared to the melinio seed shells, its microwave absorption was very low. This suggests that the eggshell sample has not fully converted to carbon, and thus, a higher temperature and longer carbonization process are needed. The porous structure of the carbon in the water hyacinths and melinjo seed shells samples allows them to exhibit high absorption capacity. In the absorption mechanism, the waves entering the pores in the carbon get trapped, leading to a phenomenon of multiple reflections. This high absorption is caused by the porous structure and the large interface of the activated carbon, which effectively weakens the incoming microwave through mechanisms such as interfacial polarization, space charge polarization, and relaxation loss. Additionally, the oxygen-containing functional groups on the surface of the activated carbon serve as active sites for various types of polarization and contribute significantly to the absorption of microwave energy [4] [30].

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CONCLUSION

Abundant biomass can be utilized as a material for microwave wave absorption. In this study, the biomass used includes water hyacinth, melinjo seed shells, and chicken egg shells. The biomass was processed into activated carbon using 65% KOH. This activation process was used to enhance the absorption quality of the biomass. The characteristics of the activated carbon from the biomass tested showed a porous structure. The surface areas produced by each biomass were 4.378 m²/g for water hyacinths, 2.992 m²/g for melinjo seed shells, and 2.518 m²/g for chicken egg shells. The microwave absorption levels for each biomass were -18.342 dB for water hyacinths, -13.326 dB for melinjo seed shells, and -12.484 dB for chicken egg shells, with absorption efficiency ranging from 94% to 98%. This study proves that biomass-derived absorbents can be used as materials for microwave wave absorption. However, the treatment of biomass absorbents needs to be further improved using other variables.

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