International Journal of Progressive Sciences and Technologies (IJPSAT)
ISSN: 2509-0119.

https://ijpsat.org/
Vol. 46 No. 2 September 2024, pp. 09-16

Modification of Rice Husk-based Activated Charcoal as Microwave Absorbing Material

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Abstract—Stealth technology is one of the technologies being developed to eliminate RADAR traces from the enemy. The spectrum used in this technology is in the microwave range (8-12 GHz). RAM (Radar Absorbing Material) is one of the techniques that can be used in the design of stealth technology. One material that has the potential to be used as RAM is activated charcoal as a dielectric material. In the manufacture of activated charcoal, a chemical activation method is used with an activator in the form of 5 M HCl to obtain good porosity properties in charcoal. Modification of active charcoal was also carried out using mechanical milling method with time variations of 50, 100, 150, and 200 minutes which were then characterized using X-Ray Diffraction (XRD), Scanning Electron Microscopy - Energy Dispersive Spectroscopy (SEM-EDS), and Vector Network Analyzer (VNA). Mechanical milling is proven to be able to change the shape of the charcoal structure from amorphous to semicrystalline with the crystalline phase formed in the form of C60-fullerene. Activated charcoal samples with a time variation of 100 minutes have the best absorbing ability with absorption reaching 78.22% and a reflection loss value of -25.96 dB at a frequency of 9.69 GHz with a frequency bandwidth of 3.68 GHz.

Keywords—stealth technology; rice husk activated charcoal; mechanical milling; microwave absorber.

I. INTRODUCTION

Technological developments related to the utilization of electromagnetic waves have now expanded in various fields including telecommunications, electronics, military, and civilian. In the field of military defense, RADAR (Radio Detection and Ranging) is used to detect enemy targets by reflecting signals coming from electromagnetic waves (Seo, et al., 2004). One of the technologies being developed to eliminate traces of the enemy is stealth technology. The implementation of this technology is usually used on fighter aircraft that have the ability to avoid enemy detection. The engineering in making this stealth technology is by designing the corners of the aircraft and by coating the aircraft parts using RAM (Radar Absorbing Material) (Wahyuni & Widyastuti, 2016).

Research conducted by Park, et al. (2006) is by making RAM from two main materials, namely dielectric material and magnetic material. If the two materials are combined it will produce the most optimum RAM (Syamsir & Astuti, 2012). Several studies have been conducted to make RAM, including using dielectric material (carbon) made from bamboo mixed with PaNi (Wu, et al., 2008), magnetic material (Fe3O4) made from Lumajang iron sand (Lestari, 2015), and a mixture of dielectric and magnetic materials made from iron sand combined with carbon from cassava skin (Yusro, 2016). As for improving the wave absorption performance, the material is designed to have strong absorption, wide absorption frequency band, low material density, and thin thickness of the absorbing material (Ibrahim, et al., 2020).

Carbon materials with various forms such as carbon nanotubes (CNT), carbon nanofiber (CNF), carbon foams, graphene, and activated carbon (activated charcoal) have been widely used as the main ingredient in the manufacture of microwave absorbing materials due to their conductivity loss characteristics (Zhang, et al., 2009). In addition, carbon materials have low density values, abundant raw material resources, easy preparation, and relatively low costs, which have received intensive attention (Du, et al., 2012). The existence of porous structures in carbon can also make wave absorption better because when there are microwaves passing through carbon materials, the waves are not directly reflected back, but are forwarded to enter the pores so that the waves weaken and run out of energy (Fang, et al., 2007).

Activated charcoal is a carbon material that has a porous morphological structure. The pores contained in activated charcoal make it applicable as an oil refiner, supercapacitor electrode, secondary battery electrode, and wave absorber (Zhang, et al., 2019). Activated charcoal can be made with an easy and cheap method, which comes from natural materials containing lignocellulose. Natural materials that can be used as raw materials for activated charcoal include wood, coconut shells, wood processing waste, coal waste, agricultural waste such as coffee fruit peels, chocolate fruit peels, straw, corn frond cobs, and rice husks (Asano, et al., 1999).

Previous research on the manufacture of activated charcoal as a wave absorber has been done quite a lot. One of them is the research conducted by Widanarto, et al. (2022) who made active charcoal made from coconut shell. To get a high porosity value, the activated charcoal was modified using the mechanical milling method with a variation of time t = 50, 75, 100 in minutes. The results obtained show that the active charcoal formed is mesoporous with the best RL (reflection loss) value produced reaching -22 dB from the milling time variation of 100 minutes. The modification of activated charcoal with mechanical milling here makes the charcoal structure formed has a crystal phase in the form of C70-fullerene which indicates that absorption will be maximized by the formation of this phase.

II. Research Methods

This research aims to develop a microwave absorbing material based on activated charcoal made from biomass in the form of easily available rice husks. Activation of charcoal is done chemically using an activator in the form of HCl. Activated charcoal is then modified using the mechanical milling method with time variations of 50, 100, 150, and 200 minutes. The modified activated charcoal will be tested using X-Ray Diffraction (XRD) to determine the structural properties of rice husk activated charcoal, Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS) analysis to determine the morphology and constituents of rice husk activated charcoal, and Vector Network Analyzer (VNA) to determine the absorption of activated charcoal to microwaves. The results of this study are expected to show that activated charcoal from rice husk also has the potential to be used as a dielectric material in the manufacture of RAM.

III. Results and Discussion

A. X-Ray Diffraction (XRD) Characterization Results

X-Ray Diffraction (XRD) characterization of activated charcoal samples made from rice husk with milling time variations was carried out to determine the nature of the structure and crystal phase formed. XRD characterization results show data in the form of diffraction pattern peak intensity and diffraction pattern position (2θ angle) of the sample. The diffraction peaks refer to the International Center of Diffraction Data (ICDD) and Crystallography Open Database (COD). From the XRD test analysis data, the diffraction pattern results were obtained for each milling time variation.

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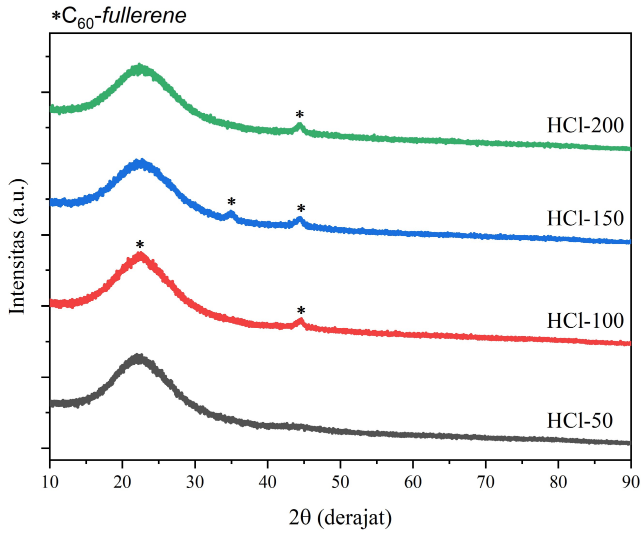

Fig. 1. Diffraction pattern of activated charcoal sample

The overall diffraction pattern of the activated charcoal sample by milling method starting at the 2θ angle position of 10° to 90° is shown in the graph above. The black graph is a graph of activated charcoal samples with milling variation (t = 50). In the sample does not form any phase. While the red graph is a graph of activated charcoal samples with milling time variation (t = 100), the blue graph is a graph of activated charcoal samples with milling time variation (t = 150), and the green graph is a graph of activated charcoal samples with milling time variation (t = 200). In each sample, the same phase is formed, namely C60-fullerene with a monoclinic crystal structure (COD 96-210-3201). This phase is formed at positions 2θ = 22.58° and 2θ = 44.46° for milling time variation (t = 100), at positions 2θ = 34.92° and 2θ = 44.34° for milling time variation (t = 150), and at position 2θ = 44.42° with cell parameters a = 13.2600 Å, b = 15.1770 Å, and c = 15.7640 Å and has lattice constants α = γ = 90° and β = 110.97°.

Table. 1. Crystal phases in the modification of activated charcoal samples using mechanical milling method

Peak List			
Sample Variation	**Crystal Phase**	**Crystal Structure**	**2θ Position (°)**
HCl-100			22,58
			44,46
HCl-150	C_{60}-*Fullerene*	*Monoclinic*	34,92
			44,34
HCl-200			44,42

The diffraction pattern of rice husk-based activated charcoal indicates a partial crystal or semicrystalline structure, namely amorphous and crystalline. The crystalline part is shown on the graph with a sharp peak, while the amorphous part is shown on the part of the graph with a wide and sloping peak (Budianti, 2020). This can occur because the raw material for activated charcoal, namely rice husks, is a natural material. Natural materials contain lignin, cellulose, and hemicellulose. The cellulose content in this husk makes active charcoal have a crystalline form, while the lignin and hemicellulose contained make active charcoal have an amorphous structure (Rachmawati, 2018) (Yusro & Zainuri, 2016). Diffraction patterns from XRD results show that activated charcoal has a tendency to have an amorphous structure.

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Modifications in the manufacture of active charcoal using the milling method were carried out to determine the effect on the structure and morphology of the resulting active charcoal. XRD characterization results indicate that milling time variations result in changes in the structure of active charcoal from amorphous to crystalline form. Crystals are formed after milling at 100, 150, and 200 minutes due to the heating of the milling process (Yusro & Zainuri, 2016). C60-fullerene is a crystal phase formed based on matching using HighScore Plus software during data processing. Fullerene is a type of carbon allotrope with carbon elements bonded together as its constituent. In general, fullerenes consist of two types, namely cylindrical and spherical. C60-fullerene is one type of spherical fullerene which consists of 20 hexagonal rings and 12 pentagonal rings as the basis of a closed cage structure (Hill & Petrucci, 2002).

B. Scanning Electron Scanning Electron Microscopy - Energy Dispersive Spectroscopy (SEM-EDS) Characterization Results

Scanning Electron Microscopy (SEM) - Energy Dispersive Spectroscopy (EDS) characterization was carried out to determine the morphology and pore formation in rice husk activated charcoal samples through surface images using electron beams. The EDS tool in SEM also provides information related to the composition contained in the sample. Figure 2. is an image showing the morphology of the SEM test results with a magnification of 10,000x and a tool voltage of 15.0kV.

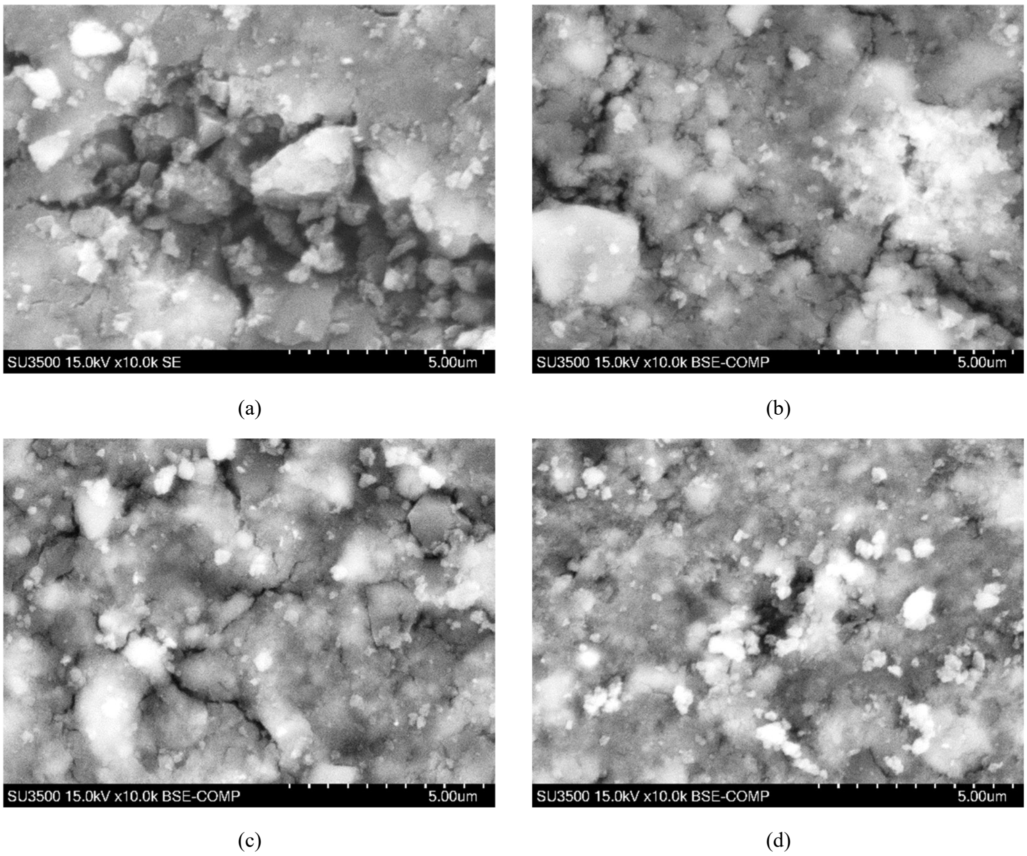

Fig. 2. SEM analysis results on the modification of activated charcoal samples using the mechanical milling method with variations of (a) 50 minutes, (b) 100 minutes, (c) 150 minutes, (d) 200 minutes.

Based on figure above, we can see the surface morphology produced for each variation of active charcoal from modified rice husk. The SEM test results here show that the surface of the active charcoal has a very large number of pores. The presence of pores on the active charcoal sample plays a role in microwave absorption (Meisrilestari, et al., 2013). The more pores formed, the better the absorption. Basically, charcoal is a material that already has pores. To make charcoal pores more and more then do the activation process. Activation is a process that makes the release of gas and decomposition of organic compounds so that it erodes the surface of the sample, then pores are formed. (Yusro, 2016). This study conducted chemical activation because it is easier to use than physical activation. HCl activator was chosen because it can dissolve impurities so that more charcoal pores will be formed and make the microwave absorption process more optimal (Rizky, 2015) (Nurhasni, et al., 2014).

Mechanical milling is a method used to reduce particle size. As the milling time increases, the surface area of the particles will also increase. The larger surface area occurs due to the smaller particle size. This shows that the mechanical milling method is one of the effective methods to reduce particle size in a material due to the collision that occurs between the ball mill and the material (Pangesthiaji & Purwaningsih, 2013) (Sari, et al., 2014). However, if the activation time is increased again, the pore size can again become larger. Therefore, this study has modified activated charcoal using mechanical milling method to make charcoal has a high porosity value in order to absorb microwaves well.

SEM testing not only provides information on the surface morphology of active charcoal samples, but also provides the percentage value of the distribution of active charcoal constituent atoms using the built-in analysis of SEM, namely EDS. EDS results show that the main elements contained in rice husk activated charcoal are C, O, and Si for each milling variation. The milling process in addition to reducing particle size can also reduce the oxygen contained in the active charcoal sample. The remaining elements are impurities that still exist in activated charcoal samples in small amounts, such as the Cl element which is the rest of the activation process using an activator in the form of HCl. The following table presents the percentage distribution of the constituent elements of activated charcoal obtained:

Table. 2. Elements contained in modified activated charcoal samples

Element (%)	Milling Time Variation			
	HCl-50	HCl-100	HCl-150	HCl-200
C	64,62	62,33	54,46	61,16
O	26,93	25,60	31,24	30,53
Si	8,13	11,50	13,57	7,91
Cl	0,07	0,12	0,16	0,10
Fe	0,05	0,15	0,20	0,10
Cu	0,11	0,18	0,21	0,11
Zn	0,09	0,13	0,16	0,08

C. Vector Network Analyzer (VNA) Characterization Results

Vector Network Analyzer (VNA) characterization of activated charcoal samples made from modified rice husk using mechanical milling method produces information related to microwave absorption. VNA characterization was carried out in the X- band region with a frequency width of 8-12 GHz which is microwave operation for military technology (Kholid, 2017) for each milling time variation, namely t = 50, 100, 150, and 200 minutes.

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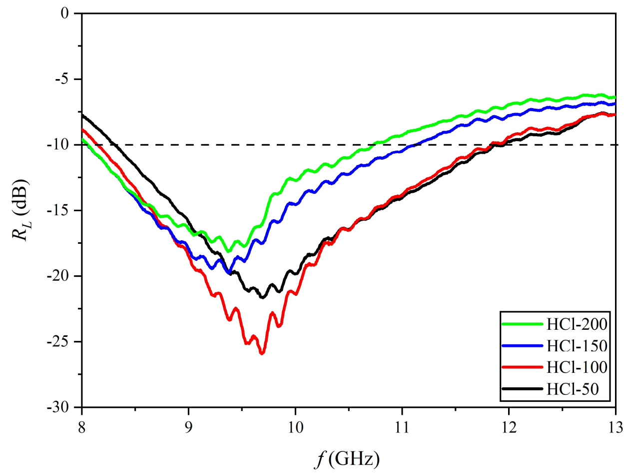

Fig. 3. Graph of reflection loss versus frequency of the modified activated charcoal sample

Figure on above shows the relationship between reflection loss and frequency of each modified activated charcoal sample variation. The black graph is a graph of the activated charcoal sample with milling time (t = 50). This sample has an RL value of -21.70 dB at a frequency of 9.70 GHz with a frequency bandwidth of 3.64 GHz. The red graph is a graph of the active charcoal sample with milling time (t = 100). This sample has an RL value of -25.96 dB at a frequency of 9.68 GHz with a frequency bandwidth of 3.68 GHz. The green graph is a graph of the activated charcoal sample with milling time (t = 150). This sample has an RL value of -19.77 dB at a frequency of 9.37 GHz with a frequency bandwidth of 3.07 GHz. The blue graph is a graph of the active charcoal sample with milling time (t = 200). This sample has an RL value of -18.16 dB at a frequency of 9.37 GHz with a frequency bandwidth of 2.67 GHz. The following table presents information related to the frequency, frequency width, and RL value of each sample.

Table. 3. Elements contained in modified activated charcoal samples

Sample	**Frequency (GHz)**	**Frequency Width (GHz)**	***Reflection Loss* (dB)**
HCl-50	9,70	3,64	-21,70
HCl-100	9,69	3,68	-25,96
HCl-150	9,37	3,07	-19,77
HCl-200	9,37	2,67	-18,16

The VNA test results produce reflection coefficients and transmission coefficients which are used to calculate the amount of absorption of each sample using Equation 2.3. The absorption values of the modified activated charcoal samples are presented in Table 4.4. In the sample with milling time variation (t = 50), the absorption value is 76.29%. In the sample with milling time variation (t = 100), the absorption value is 78.22%. In the sample with milling time variation (t = 150), the absorption value is 78.44%. In the sample with milling time variation (t = 200), the absorption value is 76.71%.

Table. 4. Absorbency value of modified activated charcoal sample

Sample	Reflection Coefficient (%)	Transmission Coefficient (%)	Absorbancy (%)
HCl-50	16,85	6,86	76,29
HCl-100	16,89	4,89	78,22
HCl-150	20,10	1,46	78,44
HCl-200	22,75	0,54	76,71

The VNA test results show that all activated charcoal samples can be applied as microwave absorbing materials in the military field, namely in the frequency range in the X-band. The sample with the largest absorption percentage is owned by the sample with milling time variation for 150 minutes. As for the best frequency bandwidth, it is not owned by the same activated charcoal sample, but by an activated charcoal sample with a milling time variation of 100 minutes. Effective absorbent materials are usually obtained from materials that have a large absorption value while having a wide frequency band range. In this study this did not happen because it is possible that not all microwaves that hit the sample will be absorbed, but some are also forwarded or reflected back. The sample with a milling time variation of 100 minutes is the best sample to be used as a microwave absorbing material because this sample has a wide range of frequency bands and high absorption. In addition, the amount of reflected waves is more than the waves that are forwarded.

Microwave absorption will increase in value along with the number of pores in the sample. The activation method has been carried out to open the pores that may be closed by impurities that are still attached after the carbonization process takes place. Then, the sample was modified using mechanical milling method to reduce the particle size. This reduction is done to produce a large surface area so that the porosity value increases. In this study, the pores formed cannot be analyzed further because no pore-related tests were carried out, so the shortcomings in this study will be a suggestion for future researchers.

IV. Conclusion

This study concluded that activated charcoal from rice husk was successfully made using chemical activation with 5 M hydrochloric acid. Modification was done through mechanical milling method with time variations of 50, 100, 150, and 200 minutes, and the mass ratio of ball mill to sample was 1:5. This method changes the structure of activated charcoal to semicrystalline with C60-fullerene phase. The variation of milling time affects the porosity of the activated charcoal, resulting in different absorbency in each sample. Activated charcoal with a milling time of 100 minutes showed the best absorption of 78.22% and a reflection loss value of -25.96 dB at a frequency of 9.69 GHz with a frequency bandwidth of 3.68 GHz.

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