Indonesian Physical Review
Volume 07 Issue 02, May 2024
P-ISSN: 2615-1278, E-ISSN: 2614-7904

Synthesis and Characterization of Activated Carbon Prepared from Rice Husk by Physics-Chemical Activation

Anak Agung Gde Suyoga Wiguna[1*], Ida Bagus Putu Mardana[1*], Putu Artawan[1*]
[1] *Physics and Science Teaching Department, Faculty of Mathematics and Natural Science, Ganesha University of Education, Indonesia*

Corresponding Authors E-mail: anak.agung.gde@undiksha.ac.id, putu.mardana@undiksha.ac.id, artawan.putu@undiksha.ac.id,

Article Info

Article info:
Received: 16-02-2024
Revised: 16-05-2024
Accepted: 22-05-2024

Keywords:
Synthesis; characterization; activated carbon; Rice husk

How To Cite:
A. A. G. S. Wiguna, I. B. P. Mardana, P. Artawan, "Synthesis and characterization of rice husk activated carbon by HCl activation," Indonesian Physical Review, vol. 7, no. 2, p 281-290, 2024.

DOI:
https://doi.org/10.29303/ipr.v7i2.311

Abstract

Activated carbon is an amorphous carbon material predominantly composed of free carbon atoms with high adsorption capacity. The amorphous structure in activated carbon affects its adsorption capacity; the higher the percentage of amorphous carbon, the greater the adsorption capacity of the activated carbon. Activated carbon can be obtained from rice husk waste, which contains 30-40% C in cellulose, hemicellulose, and lignin. Therefore, this study was conducted to observe activated carbon's phases, morphology, elemental composition, and particle size. Activated carbon was synthesized using a physics-chemical activation method, which began with washing the rice husk samples, followed by air drying, carbonizing into charcoal, and grinding it finely. The rice husk charcoal powder was then physically activated at a temperature of 500°C. The physically activated product was then chemically activated using 6M HCl with a charcoal-to-activator ratio of 1:10 (m/V), stirred at 250 rpm for 1 hour, and then allowed to settle for 24 hours. It was then washed and dried to produce activated carbon powder. XRD test results showed diffraction peaks at $2\theta = 22.23°$ without sharp or pointed peaks, indicating that the activated carbon has an amorphous structure. SEM test results showed the morphology of rice husk-activated carbon with spherical particle shapes and a particle size distribution of 20-70 nm. EDX test results showed that the rice husk activated carbon is predominantly composed of C, O, and Si with respective percentages of 54.31%, 40.04%, and 2.49%.

Introduction

One sector that plays a crucial role in Indonesia's economic growth is industry. The industrial sector produces various products for human needs and exports. Adequate resources must support the sustainability of the sector in Indonesia. One abundant resource in Indonesia is its natural resources. Indonesia is an agricultural country where 40% of its population is involved in farming. One of the leading agricultural products in Indonesia is rice. This is because rice is a crucial commodity as a staple food for Indonesians. Based on data from the Central Statistics

Agency (CSA), 2022, rice production in Indonesia amounted to 54.75 million tons of paddy. The increase in rice production also impacts the rise in rice husk produced from the rice milling process. Rice milling produces 25% husk, 8% bran, 2% pollard, and 65% rice [1]. Based on this, about 20% of the GKG (Gabah Kering Giling) production is estimated to result in approximately 10.92 million tons of rice husk waste [2].

Rice husk is a byproduct of the rice milling process, consisting of a substantial accumulation of rice grain husks. Rice husk comprises 10% water, 40% cellulose, 30% lignin, and 20% ash [3]. The chemical compounds in rice husk include cellulose (38%), lignin (22%), hemicellulose (18%), and inorganic silica (17-20%). Based on its composition, rice husk has the potential for development in various industrial applications, such as being used as an adsorbent, filler material in rubber production, and as a green inhibitor. In the industrial sector, adsorption processes are widely utilized for gas separation, solvent purification, removal of organic pollutants from drinking water, and as catalysts [4].

Ironically, the utilization of rice husks is still relatively low and suboptimal. Rice husk is considered agricultural waste in the form of unused material and remnants of processing. The natural decomposition process of rice husk waste is very dense, leading to waste piles that can disrupt the surrounding environment and have implications for human health [5]. Rice husk is commonly used as a growing medium for plants and decorative material or placed in animal pens [6]. The utilization of rice husk by burning or returning it to the soil is about 36-62%, with only 7-16% used by the industry [3]. Therefore, there is a need for innovation in processing rice husk waste. One solution is to turn it into a raw material to produce activated carbon [6].

Activated carbon is a compound that has enhanced adsorption capabilities through activation [7]. Activated carbon is often used as an adsorbent in adsorption processes because it exhibits superior adsorption capacity than other adsorbents [8] and has a large surface area for each gram of activated carbon weight. Essentially, activated carbon can be made from any carbon-containing material derived from plants, animals, or mineral resources, such as wood, rice husks, animal bones, banana peels, corn cobs, etc. The porosity of activated carbon depends on the process used and subsequent processes after combustion or carbonization [7]. The activation process increases the surface area of carbon because hydrocarbons that block the pores are released. Pore size is crucial as pores are adsorbate absorption sites [9]. Pores in activated carbon enhance its adsorption capability, as pores act as crevices that expand the surface area of the adsorbent. Smaller particle sizes increase the surface contact area, accelerating the adsorption process.

Research has been conducted on producing activated carbon from lignocellulosic materials such as corn cob waste using HCl as an activating agent. This resulted in activated carbon characterized by XRD, showing two broad peaks between angles of 20-30° and 40-45°, and SEM images revealing a honeycomb-like porous structure [10]. Similar studies using agro-waste and chemical activation with H3PO4 indicated an amorphous phase in the $2\theta = 20–30°$ range on XRD analysis [13]. Bakti et al. [23] reported that activated carbon from coconut shells using physical-chemical activation methods showed graphite phases at $2\theta = 36°$ and 44° on XRD analysis. Islam et al. synthesized activated carbon from jute sticks using H2SO4 and H3PO4, with XRD revealing amorphous silica structures at a 2θ peak of 22° and a very small peak at $2\theta = 44°$. Riyanto et al. [2] found that rice husk-derived activated carbon using NaOH activation exhibited a mixed amorphous and crystalline silica phase with a broad background and sharp peaks at $2\theta = 22°$ and 24°. SEM-EDX analysis of rice husk activated carbon (KASP

600) showed a carbon content of 28.17%, while the alkaline-treated rice husk carbon (KASPB 700) had a 27.01% carbon content. Furthermore, activated carbon from Ori bamboo using HCl as the activating agent resulted in carbon content between 74.75% and 84.491% [8]. Based on these previous studies, this research will synthesize and characterize activated carbon from rice husk lignocellulose using HCl as the activating agent. The addition of this activating agent aims to control the activation process better, resulting in high carbon content and smaller particle size (nanometer scale). This is because hydrochloric acid (HCl) acts as an activator and is hygroscopic, which helps reduce the water content in the produced activated carbon [8][12][25]. HCl has better ion adsorption capacity compared to other activators like H_2SO_4 and HNO_3, as it can dissolve more impurities, forming more pores and enhancing the adsorption process [12]

Experimental Method

A sample of 1 kg of rice husk was obtained from the rice barn in Br. Celuk, Buruan, Blahbatuh, Gianyar, Bali. 500 grams of rice husk was washed with regular water to remove any dirt adhering to it. Subsequently, the rice husk was air-dried for 5 hours [11]. The rice husk was then dried in an oven at 105°C for 1 hour to ensure complete dryness. The dehydration process aims to reduce the moisture content in the rice husk. The dried rice husk was carbonized until it turned into charcoal. Afterward, the rice husk charcoal was ground into powder using a blender and sieved with a 230-mesh sieve. The resulting charcoal powder was washed with distilled water and dried in an oven at 105°C for 1 hour.

In this research, the activation process is carried out through a physical-chemical method. A total of 27 grams of rice husk charcoal powder is heated in a furnace at 500°C for 1 hour. Subsequently, the charcoal is washed and dried in an oven at 105°C for 1 hour. The sample that has undergone physical activation is then further chemically activated. In the chemical activation process, the rice husk charcoal powder sample is activated with 6M HCl [11], with a charcoal mass to activator volume ratio of 1:10 (m/V) and soaking time of 24 hours [12]. This process produces activated charcoal. The activated carbon is filtered using filter paper and washed with distilled water until reaching a neutral pH (pH=7). The activated charcoal is dried in an oven at 105°C for 1 hour.

The activated carbon is further characterized using XRD and SEM-EDX. X-ray Diffraction (XRD) is a technique used in material analysis to gather detailed atomic-scale information about crystalline and non-crystalline (amorphous) materials. XRD produces a diffraction pattern formed by a series of diffraction peaks with varying relative intensities at specific 2θ values [22]. Scanning Electron Microscopy (SEM) is a tool that scans an object using a high-energy electron beam and produces images of the object. Energy Dispersive X-ray Spectroscopy (EDX) is a technique used for elemental analysis of a sample. When the electron beam in SEM interacts with the sample, it ejects electrons from the inner shells of the atoms in the sample. This process generates X-rays with characteristic energies, allowing for the identification and quantification of elements in the sample. SEM-EDX characterization is used to observe the morphology and elemental composition of the activated carbon.

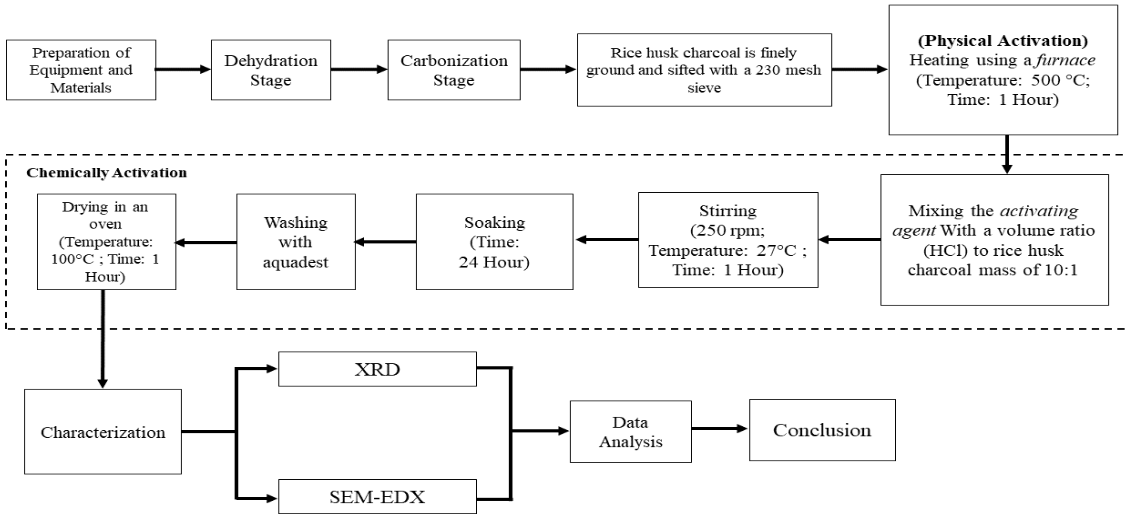


Figure 1. Flowchart Experiment

Result and Discussion

XRD Analysis

The characterization test of the sample using X-ray diffraction (XRD) aims to determine the phases and crystal or amorphous structures present in the sample. Crystal phases are indicated by the presence of sharp peaks, while amorphous phases are characterized by the formation of broad peaks or humps with low intensity [10]. XRD characterization in Figure 2 shows the X-ray diffraction pattern of rice husk-activated carbon activated with HCl.

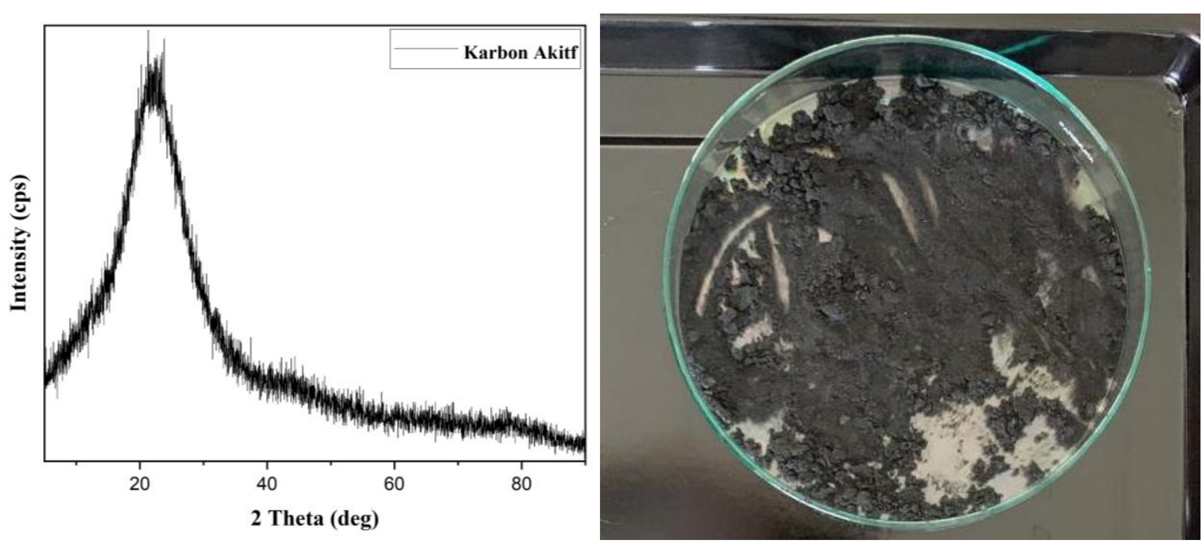


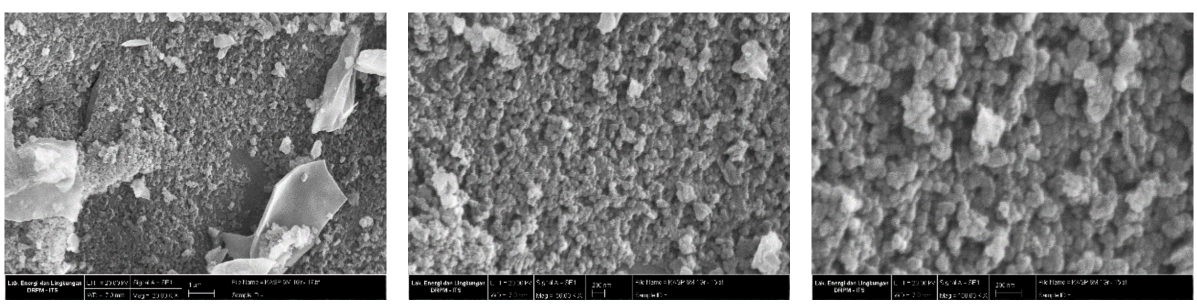
Figure 2. X-Ray Diffraction Pattern of Activated Carbon

The diffraction pattern indicates the presence of carbon with an amorphous structure [13][14][15], making it impossible to determine the crystal phase of the formed activated carbon [16]. The formation of the amorphous carbon phase can also be observed through the irregular spectrum and the broad peak shapes. Carbon with an amorphous compound structure has a turbostratic structure originating from layers of graphite with a microcrystalline network. According to Hashemian et al. in Perdani et al., [17] the turbostratic model assumes that the sample is made up of microcrystalline graphite-like layers, constrained by a cross-linking network, consisting of several layers similar to graphite, stacked almost parallel and equidistant, with each layer having a random orientation.

The XRD results of the activated carbon in this research reveal broad peaks at 2θ = 22.23°, consistent with the graphite structure where diffraction peaks are observed at angles 2θ = 21°-23° [16]. These findings are supported by several other studies; for instance, Saban et al. [10] obtained two broad peaks between angles 20°-30° and 40°-45° for HCl-activated corn cob-activated carbon. Wazir et al. [15], using pyrolysis, carbonization, and activation methods, demonstrated an amorphous carbon structure at 2θ = 22°. A very small peak is also visible at 2θ = 44°, corresponding to the (100) plane, indicating the formation of turbostratic amorphous carbon. Garg et al. [25] reported that the XRD pattern for activated carbon with NaOH and KOH treatment shows a diffraction peak at 2θ= 26.28°confirms the graphitic structure of the activated carbon. Furthermore, Islam et al. [18], in their research on activated carbon from ramie sticks, found XRD results indicating an amorphous silica structure at the peak 2θ = 22°, with an additional small peak at 2θ = 44°.

SEM-EDX Analysis

The structure of rice husk-activated carbon was tested using a scanning electron microscope (SEM) on the samples. This study performed SEM-EDX tests at magnifications of 20,000 times, 50,000 times, and 100,000 times. The SEM test results can be seen in Figure 3.



(a) (b) (c)

Figure 3. Rice Husk Activated Carbon Morphology (a) 20.000 times magnification, (b) 50.000 times magnification, (c) 100.000 times magnification.

Based on the SEM results in Figure 3, it is evident that the surface of rice husk-activated carbon has particles with various round shapes. Similar results were also obtained in the study by Riyanto et al., stating that activated carbon has a round-shaped structure [19]. The particle size distribution of activated carbon was analyzed using ImageJ and Origin 2023 software. Data were obtained by marking the particles visible in the images using ImageJ, representing the diameters of the activated carbon particles. The data were then plotted in Origin software and analyzed using Gaussian fit. The results of this analysis produced a histogram of the particle size distribution of rice husk-activated carbon, as shown in Figure 4. The analysis results indicate that rice husk-activated carbon has a particle size distribution of 20-70 nm with an average of 40.48 nm. Similar results were obtained in the study by Nurcahyo & Wibawa, where the average diameter of activated carbon was 50 nm [20]. The study by Yu et al. reported a size of 70 nm for activated carbon nanoparticles [21]. The survey by Riyanto et al. obtained diameter sizes with an average of 31.58 – 35.19 nm [19]. Based on these results, rice husk activated carbon can be classified as activated carbon nanoparticles.

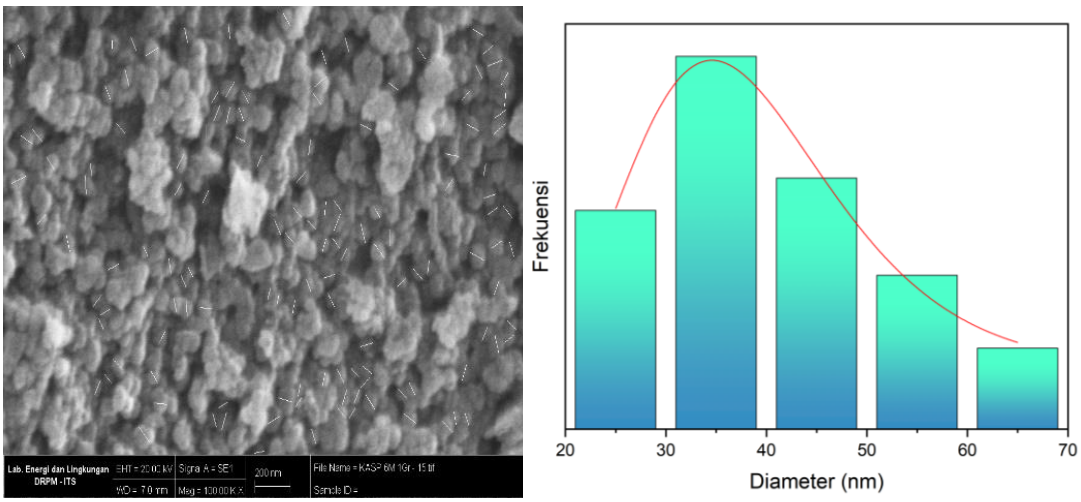


Figure 4. Morphology and Particle Size Distribution of Activated Carbon

The EDX results of rice husk-activated carbon can be seen in Figure 5.

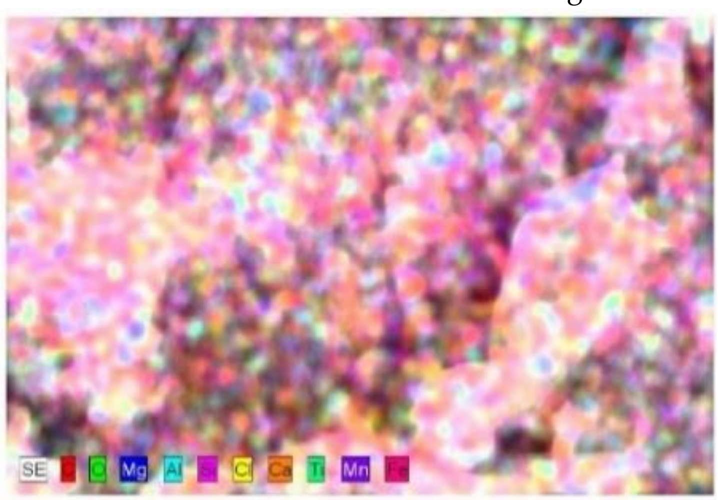


Figure 5. Mapping of the Distribution of Elements in Rice Husk Activated Carbon

Table 1. Distribution Percentage of Rice Husk Activated Carbon Elements

Sample	Concentration (%)								
	C	O	Si	Fe	Mn	Ca	Cl	Mg	Ti
Rice husk-activated charcoal	54.31	40.04	2.49	0.03	0.02	0.03	0.03	0.04	0.01

The EDX results in Figures 5 and Table 1 show the dominance of constituent elements in rice husk activated carbon in this research, sequentially, as C (54.31%), O (40.04%), and Si (2.49%). These results indicate that the carbonization process has proceeded effectively, converting almost all organic compounds in rice husk into carbon. Rice husk predominantly consists of cellulose, hemicellulose, lignin, and silica, with carbon being the primary constituent. During carbonization, organic components such as cellulose and hemicellulose decompose, leaving behind a carbon-rich residue. Additionally, the high silica content in rice husk contributes to silica in the activated carbon samples. The oxygen detected likely originates from residual oxygen-containing functional groups and silica and other inorganic compounds in the rice husk [26].

These findings are supported by previous studies, such as the research by Saban et al., [10] which obtained EDS results for corn cob activated carbon activated with HCl: C (13.13%), O (1.28%), and Si (0.77%). The research by Riyanto et al, [2] showed that rice husk activated carbon (KASP 600) consists of the elements O (46.44%), C (28.17%), P (17.48%), Si (6.81%), and Na (1.10%) [2]. Perdani et al., [17] obtained EDX results for cassava peel-activated carbon activated with H_3PO_4, revealing a porous surface with the dominant constituent elements in the following order: C (55.20%), O (28.86%), N (8.00%), P (6.22%), and Na (1.72%). Garg et al. [25] reported that the EDX result of rice husk-activated carbon with NaOH and KOH treatment shows elements C (80%), O (17.71%), Na (0.54%), and Si (1.45%).

Carbon (C) in activated carbon primarily provides this material's basic structure and unique adsorption properties. Oxygen (O) in activated carbon is generally present as oxides or oxygen functional groups attached to the carbon structure. Additionally, silicon (Si) in activated carbon allows for the silica (SiO_2) content in the activated carbon. The silica content in activated carbon can reduce the number of pores formed from the activated carbon produced. The silica content found in rice husk activated carbon is obtained from carbonization because rice husks will produce silica when burned at high temperatures (500-600) °C [12].

Conclusion

Activated carbon from rice husk has been successfully synthesized using the physic-chemical activation method with HCl. XRD results indicate that rice husk-activated carbon has a graphite structure with the highest diffraction peak appearing at $2\theta = 22.23°$, which is amorphous in nature. SEM results show the morphology of activated carbon with spherical particle shapes ranging in size from 20 to 70 nm, with an average diameter of 48 nm. EDX results indicate the dominance of constituent elements in rice husk-activated carbon in this study, with carbon (C) at 54.31%, oxygen (O) at 40.04%, and silicon (Si) at 2.49%.

Acknowledgment

This research was supported by the thesis supervisor, the Inorganic Chemistry Laboratory at Ganesha Education University, the State University of Malang laboratory, and the National Research and Innovation Agency.

References

[1] D. E. G. Sofhia, W. Nurhasanah, and J. M. Munandar, " Pemanfaatan limbah sekam padi menjadi produk arang sekam untuk meningkatkan nilai jual di desa gunturmekar, kabupaten sumedang," Jurnal Pusat Inovasi Masyarakat, vol. 2, no. 4, p. 679-684, bulan tahun. Doid:

[2] C. A. Riyanto, E. Kurniawa, and N. R. Aminu, "Pengaruh NaOH dan suhu aktivasi terhadap karakteristik karbon aktif sekam padi teraktivasi H3PO4," Rafflesia Journal of Natural and Applied Sciences, vol. 1, no. 2, p. 59-68, Oktober 2021.

[3] Ferdiansyah, A. S. A. Premesti, A. R. Fathichin, B. M. G. Ariani, A. H. Fahmim and Y. W. Mirzayanti. Review Studi: analisa pemanfaatan limbah sekam padi sebagai bahan material maju. Prosiding Seminar Nasional Teknologi Industri Berkelanjutan III, March 4, 2023.

[4] Shofa, "Pembuatan karbon aktif berbahan baku ampus tebu dengan aktivasi kalium hidroksida" Depok, Indonesia University, July 4, 2012.

[5] I. Listiana, R. Bursan, R. A. D. Widyastuti, A. Rahmat, and H. Jimad, "Pemanfaatan limbah sekam padi dalam pembuatan arang sekam di pekon bulurejo kecamatan gadingrejo kabupaten pringsewu," Jurnal Pengabdian Masyarakat, vo. 3, no. 1, p. 1-5. 2021.

[6] Hendriyana, "Kajian awal pemanfaatan sekam padi menjadi karbon aktif," Jurnal Teknik, vol. 10, no. 2, p. 83-89, November 2011.

[7] I. F. Anggraini, E. Kusniawati, and M. Mayanagsari, "Pemanfaatan tongkol jagung pada pembuatan karbon aktif dengan menggunakan aktivator (Na2CO3) serta pengaruhnya terhadap sampel air sumur gali menggunakan parameter ph, turbidity, total suspended solid (TSS) & total disolved solid (TDS)," Jurnal Cakrawala Ilmiah, vol. 2, no. 5, p.2261-2271, 2023.

[8] S. Huda, R. D. Ratnani, and L. Kurniasari, "Karakterisasi karbon aktif dari bambu ori (bambusa arundinacea) yang di aktivasi menggunakan asam klorida (HCl)," Inovasi Teknik Kimia, vol. 5, no. 1, p.22-27, April 2020.

[9] Wardalia, "Karakterisasi pembuatan adsorben dari sekam padi sebagai pengadsorpsi logam timbal pada limbah cair," Jurnal Integrasi Proses, vol. 6, no. 2, p.83-88, 2011.

[10] A. Saban, Jasruddin, and Husain, "Pengaruh konsentrasi aktivator (NaOH dan HCl) terhadap karakteristik karbon aktif dari tongkol jagung menunjukkan karbon aktif tongkol jangung," Junal Sains dan Pendidikan Fisika (JSPF), vol. 19, no. 2, p. 219-228. August 2023. Avaliable: http://ojs.unm.ac.id/jsdpf.

[11] R. Dwityaningsih, T. E. P. S. Rahayu, M. Handayani, and M. Nurhilal, "Pengaruh variasi konsentrasi H3PO4 sebagai zat aktivator terhadap karakteristik karbon aktif dari sekam

padi," Infotekmesin, vol. 14, no. 1, p. 98-104, January 2023. DOI: 10.35970/infotekmesin.v14i1.1641.

[12] L. I. Wirani, "Aktivasi karbon dari sekam padi dengan activator asam klorida (HCl) dan pengaplikasiannya pada limbah pengolahan baterai mobil untuk mengurangi kadar timbal (Pb)," Medan, North Sumatera University, January 26, 2017.

[13] J. B. Njewa, E. Vunain, and T. Biswick, "Synthesis and characterization of activated carbons prepared from agro-wases by chemical activation," Journal of Chemistry, p.1-13 March, 2022. Avaliable : https://doi.org/10.1155/2022/9975444.

[14] E. H. Sujiono, D. Zabrian, Zurnansyah, Mulyati, V. Zharvan, Samnur, and N. A. Humairah, "Fabrication and characterization of coconut shell activated carbon using variation chemical activation for wastewater treatment application,"Result in Chemistry", p. 1-10, January 2022. Available: https://doi.org/10.1016/j.rechem.2022.100291.

[15] A. H. Wazir, I. U. Wazir, and A. M. Wazir,"Preparation and characterization of rice husk based physical activated carbon,"Energy Sources, Part A: Recovery, Utilization, And Environmental Effects, p.1-12. DOI: 10.1080/15567036.2020.1715512.

[16] C. Bijang, M. F. J. D. P. Tanasale, D. Sri, Tahril, L. A. Kadir, and T. Azis, "Synthesis and characterization of activated carbon from waste compedak fruit (artocarpus champeden) activated h3po4 as adsorbent of methylene blue," Jurnal Akademika Kimia, vol. 11, no. 1, p. 56-63, February 2022. DOI: 10.22487/j24775185.2022.v11.i1.56-63.

[17] F. P. Perdani, C. A. Riyanto, and Y.Martono, "Karakterisasi karbon aktif kulit singkong (manihot esculenta crantz) berdasarkan variasi konsentrasi h3po4 dan lama waktu aktivasi," Indonesian Journal of Chemical Analysis, vol. 4, no. 2, p. 72-81, September 2021. DOI: 10.20885/ijca.vol4.iss2.art4.

[18] M. N. Islam, J. Sraker, A. Khatton, S. M. M. Hossain, H. A. Sikder, R. Ahmed, and A. M. S. Chowdhury, "Synthesis and characterization of activated carbon prepared from jute stick charcoal for industrial uses," Scholars International Journal of Chemistry and Material Sciences, vol. 5, no. 3, p. 33-39, March 2022. DOI: 10.36348/sijcms.2022.v05i03.003.

[19] C. A. Riyanto, E. Prabalaras, and Y. Martono, "Karakterisasi nanopartikel karbon aktif dari daun eceng gondok (eichhornia crassipes) berdasarkan variasi suhu dan waktu aktivasi," Jurnal Kimia dan Kemasan, vol. 42, no. 2, p. 85-92, September 2020. DOI: 10.24817/jkk.v42i2.5633.

[20] R. Y. Nurcahyo, and P. J. Wibawa, "Fabrikasi ramah lingkungan komposit nano karbon aktif-partikel perak dan uji aktifitas antibakterinya," Greensphere: Journal of Environmental Chemistry, vol. 2, no. 1, p. 31-37, May 2022. DOI:10.14710/gjec.2022.14827

[21] L. Yu, I. D. Tatsu, and T. Kondo, "Preparation of carbon nanoparticles from activated carbon by aqueous counter collision," Journal of Wood Science, vol. 68, no. 29, p. 1-8, May 2022. Avaliable: https://doi.org/10.1186/s10086-022-02036-3.

[22] N. Masta," Buku materi pembelajaran scanning electron microscopy", Jakarta: Universitas Kristen Indonesia, 2020.

[23] A. I. Bakti, M. M. Lemembang, and Jumriadi," Karakterisasi karbon aktif yang terbuat dari tempurung kelapa menggunakan Teknik pirolisis dengan aktivasi fisika dan

kimia,"Jurnal MIPA, vol.12, no.2, p.56-60, August 2023. DOI: https://doi.org/10.35799/jm.v12i2.46160.

[24] A. Kwaghger, and J. S. Ibrahim,"Optimization of conditions for the preparation of activated carbon from mango nuts using HCl," American Journal of Engineering Research, vol. 2, no. 7, p. 74-85, 2013.

[25] R. Garg, A. Gaur, and R. P. Chauhan," Synthesis and characterization of rice husk activated carbon by torrefaction, desilication, and alkalies based approach," Indian Journal of Engineering & Materials Sciences, vol. 30, p. 567-572, August 2023. DOI: 10.56042/ijems.v30i4.3744.

[26] Z. Sitorus, Halimatuddahliana, E. Sembiring, and R. F. Y. Butar-butar," Synthesis of activated carbon from rice husk as a filter for iron (Fe) and copper (Cu) in well water," International Conference on Physics and Applied Physics, **2733** 012007, 2024. DOI:10.1088/1742-6596/2733/1/012007.