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RESEARCH PAPER

# Synthesis and characterization of activated carbon from biomass waste reject pulp as an adsorbent for wastewater treatment.

Sri Hilma Siregar[a]*, Hasmalina Nasution[a], Wirdati Irma[b], Dedi Suwito[a], Aulia Rizki Rahmadani[c], Sha-Ling Han[d]

[a] Study Program of Chemistry, Department of Education of Mathematics, Natural Science and Health, Universitas Muhammadiyah Riau, Jalan Tuanku Tambusai, Riau, Indonesia
E-mail: srihilma@umri.ac.id
[b] Study Program of Biology, Department of Education of Mathematics, Natural Science and Health, Universitas Muhammadiyah Riau, Jalan Tuanku Tambusai, Riau, Indonesia
[c] Postgraduate Chemistry, Department of Education of Mathematics, Natural Science, Universitas Riau, Jalan HR.Soebrantas, Riau, Indonesia
[d] Departement of Applied Chemistry, Chaoyang University of Technology, Taichung, Taiwan

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**Abstract:** The pulp and paper industry in Indonesia has developed rapidly. This industry produces a large amount of biomass waste that has the potential to pollute the environment. Different types of biomass available in pulp mills include black liquor, bark, wood residues, knots, reject pulp, and sludge cake. In this study, rejected pulp biomass waste can be used as an alternative material to reduce methylene blue dye. This study investigated the ability of carbonized pulp to reject carbon at a temperature of 450°C as an adsorbent in the adsorption of methylene blue dye by UV-Vis spectrophotometry method and characterization using X-ray diffraction (XRD). The variables used in this study were pH 3-11, contact time 15-90 minutes, and adsorbent mass 0.1-1.0 g. Optimum results in this study were obtained at pH 5 with a percentage of 57.34%, optimum contact time at 75 minutes with a percentage of 67.34%, the optimum mass of adsorbent 1.0 g pulp reject carbon at a temperature of 450°C as an adsorbent in the adsorption of methylene blue dye by UV-Vis spectrophotometry method, and characterization using X-Ray Diffraction (XRD). The variables used in this study were pH 3-11, contact time 15-90 minutes, and adsorbent mass 0.1-1.0 g. The optimum results in this study were at pH 5 with a percentage of 57.34%, optimum contact time at 75 minutes with a percentage of 67.34%, and optimum adsorbent mass of 1.0 g.

Keywords: adsorption, carbonization, reject pulp, methylene blue

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## INTRODUCTION

Indonesia's textile industry is growing rapidly and can cause environmental problems due to industrial waste pollution. One way to prevent water pollution is to neutralize industrial wastewater before it is discharged into waterways using the activated carbon adsorption method. The raw materials that can be made into activated carbon are all materials containing lignocellulose (lignin and cellulose) derived from plants, and several types of adsorbents have been developed to adsorb heavy metals, one of which is by using cellulose and lignin [1].

Recently, many alternative adsorbents have been developed, one of which is the development of cellulose-derived bio adsorbents. Cellulose is an abundant and renewable material. Unlike synthetic polymers, cellulose is a natural biopolymer, so the environment more easily degrades it. In addition, cellulose has significant potential due to its adsorption capacity [2].

Pulp is the result of the separation of fibers from fibrous raw materials. The pulp can be made from wood, non-wood, and recovered paper. Chemical pulp is wood pulp used as a basic ingredient in papermaking. Pulp raw materials usually contain three main components: cellulose, hemicellulose, and lignin. In general, the principle of pulping is the process of separating cellulose from the compounds contained in wood, including lignin. During the pulping process, the cooking solution breaks the lignin into smaller molecules that can dissolve in the black liquor. This process is called delignification [3]. The pulp and paper industry in Indonesia has developed quite rapidly. The pulp and paper industry is one of Indonesia's leading agro-industrial commodities. The pulp industry in Indonesia is competitive as it is ranked 8th in the world, and the paper industry is ranked 6th in the world. In 2019, the national paper production capacity was 10.1 million tons, and consumption was 6.3 million tons. Meanwhile, pulp is listed as a commodity with an export potential of 5.3 million tons. Despite being in the Covid-19 pandemic, the global demand for pulp and paper has increased by about 2.1 percent [4-5].

One type of solid waste produced by pulp mills is rejected pulp, which is pulp that is not fully cooked during the processing of wood chips into pulp [6]. Based on the data that 7000 tons of pulp are produced daily, 160 tons of reject pulp is also produced. Reject pulp can usually be re-cooked or repulped, whereas traditionally, this reject pulp is disposed of in landfills [7]. Biomass can be used as an adsorbent material; in the pulp industry, acacia trees are a reliable raw material for paper pulp due to their high cellulose content. Currently, biosorption technology using plant biomass as a biosorbent is an alternative technology that has the potential to be developed [8]. It is the main attraction for researchers to develop it for various applications, especially in the industrial sector, to reduce factory effluents. Many studies on synthesizing activated charcoal from biomass have been successfully carried out on organic compounds such as dye waste. This rejected pulp is also expected to degrade methylene blue dye waste, a dye used by the textile and paper industry. The results of this study are expected to provide an alternative for development on an industrial application scale. It will provide a waste management solution in addition to alternative use of biomass waste (reject pulp), a by-product of pulp and paper mill processing.

## MATERIALS AND METHODS

**Instrument.** The tools used in this study are analytical balance, oven, furnace, desiccator, glass beaker, funnel, filter paper, porcelain cup, magnetic stirrer, 100 mesh screen, storage container, UV-Vis spectrophotometer, X-ray diffraction (XRD).

**Material.** The materials used in this study were Karbon reject pulp, Methylene Blue 1000 ppm, and Aquadest.

### Methods

### Preparation and Activation of Reject Pulp

**Reject pulp waste is taken in a storage area at** pulp making 9; reject pulp is then carried out to produce activated carbon. Reject pulp is taken to be washed and screened to get the fiber. Next, the fiber from the washing is squeezed and dried using an oven at 105°C for 4 hours, removed, and put into a desiccator until room temperature. Prepare the dried reject pulp for burning at 450°C for 1 hour. The result of carbonization was cooled to room temperature. Then the rejected pulp carbon is put into a reagent bottle which is tightly closed and ready for use. Characterize the carbon for moisture content and ash content [9].

### Waste methylene blue degradation

The activated carbon produced was tested for the degradation of methylene blue color solution. Put the methylene blue solution into a beaker glass as much as 50 ml with a concentration of 20 ppm set at the optimum pH. Add 0.5 g activated carbon. Stir using a magnetic stirrer with time according to the variables 15, 30, 45, 60, and 90 minutes at room temperature. Then filtered, the filtrate was analyzed using a UV-Vis spectrophotometer [10].

## RESULTS AND DISCUSSION

To determine the moisture content, 1.0005 grams of rejected carbon pulp was dried in an oven at 105°C for 2 hours in an open cup to a constant weight. Meanwhile, the ash content (unburned carbon) is determined as follows: 3.2112 grams of carbon are heated in an oven at 450°C for 1 hour. The percentages of water and ash content are given in Table 1.

Table 1. Results of Carbon Pulp Reject Characters

| Parameter | SNI Quality No.06-3730-1995 | Analysis results |
|---|---|---|
| Yield | - | 17.54 % |
| Water content | Max 15 % | 2.98 % |
| Ash Content | Max 10 % | 2.37 % |

The adsorption of methylene blue by carbon reject pulp depends on the pH of the solution, where pH affects the surface charge of the adsorbent. A pH variation was carried out to determine the optimum pH conditions for the adsorption of methylene blue dye. This study used a variation of pH 3, 5, 7, 9, and 11 with a contact time of 15 minutes and a dose of 0.5 grams of adsorbent. The results of the adsorption percentage at different pH are shown in Table 2.

Table 2. Adsorption at pH Variations

| pH | Initial concentration (ppm) | Concentration (ppm) | Adsorption % |
|---|---|---|---|
| 3 | 20 | 8.693 | 56.54 |
| **5** | **20** | **8.532** | **57.34** |
| 7 | 20 | 8.974 | 56.99 |
| 9 | 20 | 9.472 | 52.64 |
| 11 | 20 | 9.848 | 50.76 |

The adsorption process at different contact times used variations of 15, 30, 45, 60, 75, and 90 minutes with a dose of 0.5 grams of adsorbent at the optimum pH. The variation of the contact time between the adsorbent and the adsorbate is carried out to determine the optimum time for the adsorbent to adsorb the adsorbate. The optimum contact time is achieved when the increase in the percentage of adsorbate adsorbed reaches a maximum point. The results of varying the contact time between methylene blue and carbon adsorbents are shown in Table 3.

Table 3. Adsorption at Variations in Contact Time

| Time (Minutes) | Initial concentration (ppm) | Concentration (ppm) | Adsorption % |
|---|---|---|---|
| 15 | 20 | 8.558 | 57.21 |
| 30 | 20 | 8.177 | 59.11 |
| 45 | 20 | 7.554 | 62.23 |
| 60 | 20 | 6.645 | 66.77 |
| **75** | **20** | **6.532** | **67.34** |
| 90 | 20 | 6.723 | 66.39 |

The mass variation of the adsorbent was 0.1, 0.3, 0.5, 0.7, and 1.0 g; then, the solution was adjusted to the best pH conditions. Stir using a magnetic stirrer using the best time variable obtained previously (see Table 4).

The adsorption of methylene blue by carbon pulp reject in solution depends on the pH of the solution because pH affects the surface charge of the adsorbent. The adsorption of methylene blue in this study used a variation of pH 3, 5, 7, 9, and 11 with a solution concentration of 20 ppm and 0.5 g of carbon reject pulp which was stirred with a magnetic Figure 2 Adsorption of Methylene Blue at Variations in Contact Time stirrer for 15 minutes. The absorbance was then measured using a UV-Vis spectrophotometer at a maximum wavelength of 666 nm. Based on the results, the absorbed methylene blue dye concentration decreased with increasing pH. At pH 3, 56.54% was adsorbed, and at pH 5, 57.34% was adsorbed, but after pH 5, the concentration of adsorbed methylene blue continued to decrease up to pH 11.

Table 4. Adsorption on mass variations

| Mass (g) | Initial concentration (ppm) | Concentration (ppm) | Adsorption % |
|---|---|---|---|
| 0.1 | 20 | 12.195 | 39.03 |
| 0.3 | 20 | 9.511 | 52.45 |
| 0.5 | 20 | 6.515 | 67.42 |
| 0.7 | 20 | 5.320 | 73.40 |
| 1.0 | **20** | **5.108** | **74.46** |

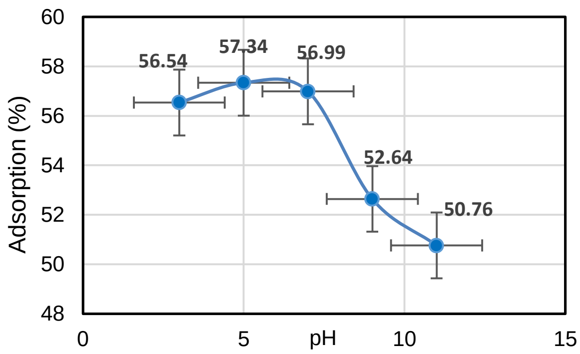


Figure 1 Adsorption of Methylene Blue at pH Variations

It shows the relationship between pH and the compound being adsorbed, methylene blue dye. According to Anselm et al. [11], methylene blue is protonated by $H^+$ ions at an acidic pH, which causes a change in the structure of the compound in methylene blue, making it less colorful. Under these conditions, H+ ions will interact with $Cl^-$ ions in methylene blue and facilitate interaction with hydrophilic active groups (lignin, cellulose, and hemicellulose). The adsorption capacity of methylene blue tended to be greater in low-pH media than in high-pH media [12]. Adsorption at high pH decreased because the surface area of the adsorbent was more protonated, and adsorption competition occurred between H+ ions and free methylene blue ions, and $OH^-$ ions for their binding sites. Therefore, $H^+$ ions react with anionic functional groups on the surface of the adsorbent, reducing the amount of methylene blue ions that can be bound. One of the variables that can determine the amount of adsorbate that the adsorbent can adsorb is contact time. In this study, contact time variations of 15, 30, 45, 60, 75, and 90 were used at a concentration of 20 ppm methylene blue, adjusted to the optimum pH obtained, and the absorbance was measured using a UV-Vis spectrophotometer at a wavelength of nm.

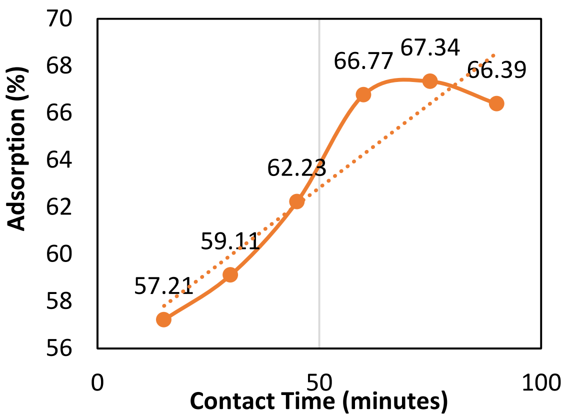


Figure 2 Adsorption of methylene blue for different contact times

Figure 2 illustrates that from 15 minutes to 75 minutes, the adsorption percentage continued to increase. At 75 minutes, the percentage of adsorbate adsorbed was 67.34%. After 75 minutes, the percentage of adsorption tended to decrease. At 90 minutes, the percentage was 66.39%, so during contact, the optimum occurs at 75 minutes. The optimum time is reached when the increase in the percentage of adsorbate adsorbed reaches a maximum point so that the addition of contact time does not significantly reduce the levels of dye in the sample. Contact time is very important in the adsorption process. The longer the contact time, the greater the possibility of the adsorption process. However, when equilibrium conditions are reached, the adsorbent tends to release (desorb) the dye so that the adsorption percentage decreases, indicating that the rejected pulp is experiencing saturation time. Generally, the adsorption rate increases at the beginning of the process and gradually decreases until equilibrium is reached. It happens because many pores are still available for adsorbate entry at the beginning of the process, and the number decreases with time (Indah et al., 2019). The optimal contact time obtained in this study is the same as that obtained by other studies, which is 75 minutes [12].

The adsorbent mass of 0.1 - 1.0 grams showed an increase in adsorption percentage from 39.03 to 74.46%. It is due to the increase in total surface area and the number of pores used to bind the adsorbate in the adsorption process by adding the adsorbent dose [10]. An increase in adsorbent mass leads to an increase in color removal due to the larger surface area and the availability of more adsorption-active sites [11]. At a lower adsorbent mass, the surface of the adsorbent becomes saturated with methylene blue adsorbate. At the same time, the remaining dye concentration in the solution is still high, so the removal is relatively low.

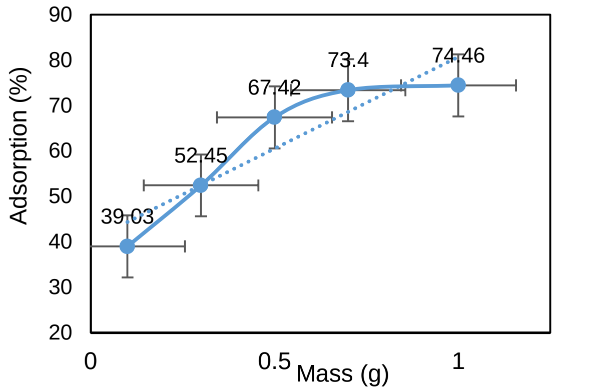


Figure 3 Adsorption of Methylene Blue Dye on Mass Variation of Adsorbent

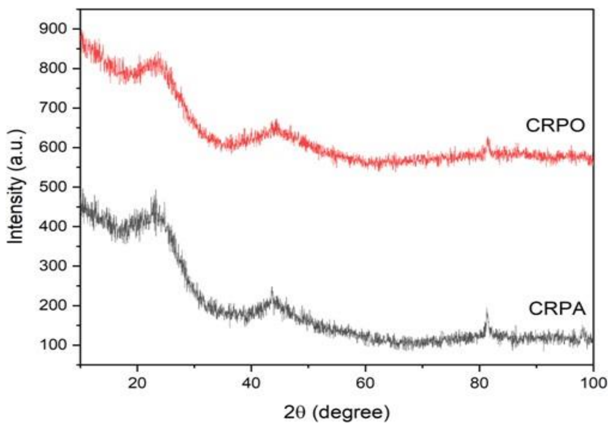


Figure 4 Results of XRD analysis of carbon reject pulp before (CRPO) and after adsorption (CRPA)

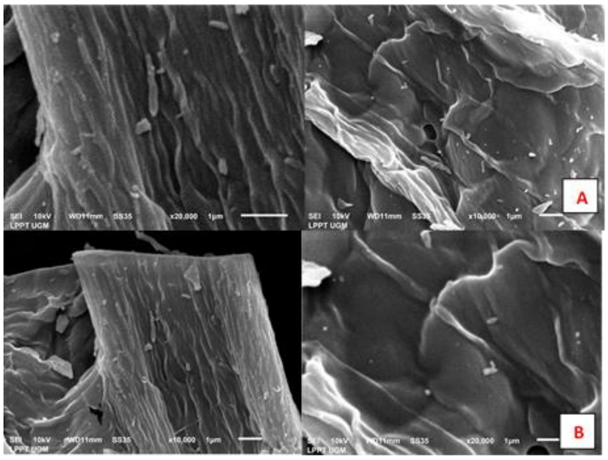


Figure 5. Comparison of SEM (Scanning Electron Microscope) 10000 magnification and 20000 magnification of carbon reject pulp (CRP)

Diffraction peaks at two theta, 22.73°, 43.58°, and 81.42°, explain the presence of cellulose in the rejected pulp. The diffraction peaks at 2θ around 20 - 22° are cellulose [6]. Broad peaks with large full width at half maximum FWHM) values indicate the atomic arrangement of the solid cellulose material, so it can be predicted that the irregular arrangement of atoms is present in the structural arrangement of the isolated cellulose [13-14]. However, the presence of fields with (good crystallinity can also be found in the cellulose results by showing peaks on a narrow diffractogram with a small full width at half maximum (FWHM) value in the diffraction angle region around 35-70º. Figure 4 shows a decrease in intensity that occurs after the adsorption process. Figure 5 explains that the morphology of CRP has very clear and very dense fibers so that separation and defibering are not visible. The morphology of this rejected pulp carbon also shows a hollow structure, indicating that CRP has an asymmetric pore.

## CONCLUSION

The properties and characteristics of carbon reject pulp can be used as an adsorbent for the adsorption of dye waste. The data shows that CRP can adsorb up to 74.45% of methylene blue dye.

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## REFERENCES

[1] Kunle, O. F., Egharevba, H. O., & Ahmadu, P. O. (2 1. Jasmal, Sulfikar, Ramlawati. 2015. Kapasitas Adsorpsi Arang Aktif Ijuk Pohon Aren (Arenga Pinata) Terhadap $Pb^{2+}$. Jurnal Sainsmat. 4(1), 57 – 66.

[2] Chakravarty, S., Pimple, S., Chaturvedi, H. T., Singh, S., & Gupta, K. K. (2008). Removal of copper from aqueous solution using newspaper pulp as an adsorbent. Journal of hazardous materials, 159(2-3), 396-403.

[3] Ratna, A. S., Ghosh, A., & Mukhopadhyay, S. (2022). Advances and prospects of corn husk as a sustainable material in composites and other technical applications. Journal of cleaner production, 133563.

[4] Van Dijk, M., & Bell, M. (2007). Rapid growth with limited learning: industrial policy and Indonesia's pulp and paper industry. Oxford Development Studies, 35(2), 149-169.

[5] Siregar S. H, Syafri, R. Prasetya, Nasution. H., Ramadhanti, A. R. K. (2023). Pulp synthesis using bamboo raw materials through unbleached and bleached processes. Acta Chimica Asiana 6(1), pp 247-252 DOI: 10.29303/aca.v6i1.135

[6] Aulia. A. A., Erdawati, Suhartono. 2019. Pengaruh Massa Bio-Char Kayu Akasia (acacia mangium) dan Waktu Perendaman Bio-Char Terhadap Adsorpsi Sebum Buatan. Jurnal Riset Sains dan Kimia Terapan. 8(1).

[7] Sumathi, S., & Hung, Y. T. (2004). Treatment of pulp and paper mill wastes. In Handbook of industrial and hazardous wastes treatment (pp. 507-555). CRC Press.

[8] Wahi, R., Abdullah, L. C., Mobarekeh, M. N., Ngaini, Z., & Yaw, T. C. S. (2017). Utilization of esterified sago bark fibre waste for removal of oil from palm oil mill effluent. Journal of environmental chemical engineering, 5(1), 170-177.

[9]. Christy, E. O., Soemarno, S., Sumarlan, S. H., & Soehardjono, A. (2020). Pilot study on low-density binderless bark particleboards manufacture from gelam wood (Melaleuca sp.) bark. BioResources, 15(4), 7390.

[10] Yeni. R. W., Mardhiyyatin. N., Devy. N., Abdul. A., Suparno. 2020. Penggunaan Arang Aktif Berbahan Dasr Limbah Biomassa Pada Proses Filtrasi Air Groundtank Ditinjau dari Uji TDS, Uji Kejernihan dan Uji Fe. Jurnal Teori dan Aplikasi Fisika. 8(2).

[11] Anselmus. B, B., Hildegardis. M. 2020. Kajian Kinetika Adsorpsi Metilen Biru Menggunakan Adsorben Sabut Kelapa. 5(2), 76 – 85.

[12] Indah. R., Nimatul. F., Suwardiyono. 2019. Adsorpsi Zat Warna Methylene Blue Menggunakan Abu Alang-Alang (Imperata cylindrica) Teraktivasi Asam Sulfat. Jurnal Teknik Kimia. 4(2), 6 – 11.

[13] Phanthong, P., Reubroycharoen , P., Hao, X., Xu, G., Abudula, A., & Guan, G. (2018). Nanocellulose: Extraction and application. Carbon Resources Conversion 1, 32–43.

[14] Thakur, V., Guleria, A., Kumar, S., Sharmad, S., & Singh, K. (2021 2). Recent advances in nanocellulose processing, unctionalization and applications: a review. Material Advances, 1872 - 1895.