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Synthesis of Bamboo-based Activated Carbon through Physicochemical Activation for Coal-runoff Wastewater Treatment

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ABSTRACT

Bamboo-based activated carbon (BAC) has been successfully synthesized through a physico-chemical technique. The characteristics of BAC were investigated using Scanning Electron Microscopy (SEM), Fourier Transform Infra-Red (FTIR), and X-Ray Diffraction (XRD). Evaluating BAC performance on coalrunoff wastewater was carried out by varying contact times and adding low-level alum on remediation of pH and Total Suspended Solid (TSS). The results confirm that BAC performs well in stabilizing pH, indicated by the neutral pH, after 10 minutes of interaction. The TSS with BAC treatment and 1% BAC-Alum combination reduced the TSS value from 880 mg/L to levels below the threshold for wastewater quality standards, 387 mg/L and 73 mg/L, respectively. This combination can be an alternative coalrunoff wastewater treatment technique for better environmental quality.

1. INTRODUCTION

Coal, carbon-based energy, is still the world's primary energy source to support human life today. A common problem in coal mining is acid-mine water released by rainwater combined with sulfite sedimentation. Acid-mine water is generally formed in high levels of acidity, sulphate content, heavy metals (such as Fe and Mn), and total suspended solids (TSS), which can present danger to the environment, so it must be treated according to quality standards before released into the environment [1-3].

The general method in treating coal acid-mine water is neutralization by adding alkaline chemicals such as $Ca(OH)_2$, CaO, NaOH, Na₂CO₃, and NH₃. However, this method is relatively expensive and requires more than simple apparatus [4]. Another standard method is alum $(Al_2(SO_4)_3)$ powder coagulation. *Alum* is a coagulant material that effectively accelerates the removal of suspended solids in wastewater. However, excessive use of alum can cause the release of aluminium ions (Al^{3+}) at confident levels which can be toxic and harmful to the environment [5].

Activated carbon is a material that is widely used in wastewater treatment as an adsorbent for catching pollutants [6]. The advantages of using activated carbon in wastewater treatment are high adsorption capacity, easy application, and relatively low-cost are the advantages of using activated carbon [7]. Various sour ces can be used as precursors in producing activated carbon, such as coal, lignite, peat, and agro-industrial waste. Agro-industrial waste is a potential precursor because it has

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high cellulose content, abundant availability, inexpensive, biodegradable, non-toxic, and thermalmechanically stable to support clean and sustainable development [8].

Bamboo industrial waste is one of the agro-industrial wastes that can be converted into activated carbon. Moreover, the primary use of bamboo has been limited to building materials and handicrafts. However, bamboo is a potential raw material for activated carbon because of its fast growth, low cost, and good mechanical properties. In addition, activated carbon produced from bamboo exhibits good surface characteristics and porosity [9].

Commonly, the synthesis of activated carbon consists of two steps, carbonization and activation. Carbonization uses controlled pyrolysis/gasification at high temperatures (400-1000°C) to remove volatile matters [10]. Then, the activated stage can be through physical, chemical, or physicochemical activation. The activation process causes an increase in the volume and pore diameter, and surface area by breaking the hydrocarbon bonds or oxidizing the surface molecules, which impacts the adsorption capacity [11]. Several types of chemicals are commonly used as activators, such as H₃PO₄ [12], ZnCl₂ [13], KOH [14], etc. Santana *et al.* [12] reported that combining physical and chemical activation could produce bamboo-based activated carbon with a high surface area and excellent adsorption capacity for pesticide waste in surface water.

Activated carbon derived from bamboo has become an essential and promising adsorbent in wastewater treatment. Several previous studies have reported the synthesis of bamboo-based activated carbon and reported good performance for the removal of various contaminants in wastewater, such as pesticides [12], heavy metals [15-17], and dyestuffs [18-19]. The use of bamboo-based activated carbon in coal runoff wastewater has yet to be found, so this research studied the synthesis of activated carbon using bamboo industrial waste materials and its effectiveness against several parameters of coal wastewater quality standards.

2. EXPERIMENTAL METHOD

2.1. Materials

The chemicals used in this experiment were HNO_3 and H_3PO_4 pro analysis from Merck, distilled water, commercial $Al_2(SO_4)_3.18H_2O$ (alum) and CaO (quicklime). All the reagents were used without further purification. The raw material of bamboo charcoal is resulted from bamboo handcraft waste by PT. Hanan Alam Lestari, a small business assisted by PT. Bukit Asam, Tbk.

2.2. Synthesis of Bamboo Activated Carbon

Bamboo charcoal removed the moisture content in the material using the universal aging oven at a temperature of 105°C. Then, the bamboo charcoal was crushed using a grinder and sieved to obtain a size of 10 mesh. Activation of bamboo charcoal is carried out physic (heating) and continued by chemical using H₃PO₄. Bamboo charcoal is heated at 700 °C for 1 hour in a furnace. The obtained materials are cooled and separated from the ash formed during heating. The activation process was continued by soaking 45 g of bamboo charcoal in 70 mL of 10% H₃PO₄ solution for 24 hours. Furthermore, the activation results were separated by filtering and followed by washing until the pH was neutral. The results were dried in an oven at 105 °C for 1 hour. The resulting bamboo-based activated carbon (BAC) was stored in a desiccator [20].

2.3. Characterization

Bamboo charcoal before (BC) and after activation (BAC) were characterized to determine the properties. The surface morphology was analyzed by Scanning Electron Microscope (SEM). Functional group analysis was performed using Fourier Transform Infra-Red Spectroscopy (FTIR), and crystallinity was analyzed using X-Ray Diffraction (XRD).

2.4. Performance test of BAC on wastewater

The BAC performance test in wastewater was investigated by varying the contact time on the pH and TSS parameters of the wastewater. The liquid waste interacted with BAC at a ratio of 1:20 (w/v) accompanied by stirring using a shaker at a speed of 150 rpm with variations in

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immersion time of 10, 20, 30, 40, 50, and 60 min. The mixture was decanted to continue measuring the pH and turbidity level (TSS) as BAC performance parameters in wastewater [21-22].

2.5. Study of BAC implementation

Applying BAC in wastewater treatment ponds is designed using a flow method approach using filtration techniques. In addition, palm fibre is used as a supporting material for BAC to keep it in place. Figure 1 shows a BAC-based filtration structure design for coal runoff wastewater treatment. This study was also conducted to optimize the combination of BAC with the use of low levels of alum.



Figure 1. The structure of BAC-based filtration equipment for coal runoff wastewater treatment.

3. RESULT AND DISCUSSION

3.1. Characteristics of BC and BAC

The surface morphology of AB and BAC is displayed in Figure 2, showing distinctive bamboo charcoal pores. The porous surface structure of bamboo charcoal enables absorption interactions with pollutants (cations, anions, and suspended solid substances/TSS) in wastewater. Bamboo charcoal exhibits different physical characteristics compared to charcoal from other raw materials due to its unique lignin composition. Furthermore, the BAC material appears to have a smoother and more even surface than AB. This is attributed to the activation process, which removes organic substances or impurities on the surface and pores of bamboo charcoal. Figure 2a also reveals numerous images of residue from the charcoal-making carbonization process. These residues may result from the agglomeration of organic volatile compounds on the carbon surface [23]. The physical-chemical activation process can enhance the availability of interaction sites on the surface of activated carbon.

The functional groups in BC and BAC are confirmed by FTIR spectra, as shown in Figure 3a. The FTIR spectra patterns of BC and BAC are similar but different in intensity. The detected functional groups in both materials are not different. However, there are differences in absorption intensity, particularly in the hydroxyl functional group at around 3400 and 1600 cm⁻¹, which shows relatively lower intensity in BAC. The intensity decrease is attributed to the dehydration process during both physical and chemical activation, releasing hydroxyl groups from the bamboo charcoal structure. Besides, C-H functional groups from methyl or methylene compounds do not show a decrease in intensity at the wavenumber around 2900 cm⁻¹. FTIR characterization results show that the activation process can remove volatile compounds covering the active sites on the surface of bamboo charcoal, which is more optimally functional.

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Figure 2. Surface morphology graph using SEM of (a) BC and (b) BAC.

The crystal structure of BC and BAC was characterized using X-Ray Diffraction (XRD). The diffractograms of both are presented in Figure 3b. The XRD patterns of BC and BAC show similarities, with two peaks at 2θ around 23° and 43° . The broadening of the diffraction peaks indicates that bamboo charcoal has an amorphous structure, meaning it has an irregular arrangement (amorphous). The diffraction peak at around 23° represents the (002) plane, while the peak at 43° represents the presence of the (100) or (101) planes [24]. There is no difference in crystal structure between bamboo charcoal before and after activation. This is because the activation process only involves the removal of organic compounds and impurities from the surface and pores of bamboo charcoal, not the rearrangement of the crystal structure.



Figure 3. (a) FTIR Spectra and (b) diffractogram of BC and BAC.

3.2. The Effect of Contact Time on pH and TSS

The activity test of BAC on coal mine runoff wastewater is evaluated with varying contact times between BAC and wastewater, observing changes in pH and Total Suspended Solids (TSS), which are indicators of wastewater suitability. Figure 4a exhibits the influence of interaction time against wastewater pH after treatment. The initial pH of the wastewater sample is 6.6, which increases to 7.4 after interacting with BAC for 10 minutes and no significant changes to 60 minutes. The variation in contact time does not significantly impact the pH value of the wastewater, indicating that BAC works rapidly in stabilizing wastewater pH. This is caused by oxygen functional groups on BAC that FTIR reveals. BAC can interact with acidic groups in

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wastewater, including H^+ and other cations. The binding of acidic groups by BAC leads to the disappearance of acid donors, increasing pH. Hence, BAC can stabilize pH, which is usually managed by a neutralization process using quicklime which can be accumulated and poses environmental risks.

Total Suspended Solids (TSS) are a visible indicator of wastewater quality. Higher TSS levels usually correspond to more turbid wastewater. Figure 4b displays the influence of contact time on TSS in wastewater. TSS values decrease with increasing contact time. The most significant reduction occurs after 60 minutes of contact, with 800 mg/L TSS value. BAC can contribute to reducing TSS through a similar mechanism to pH stabilization. Alum stabilizes ion charges to facilitate precipitation, whereas BAC can also engineer wastewater ions through interactions. Active sites on the surface of bamboo charcoal can bind cations and anions in the wastewater, role as a coagulant.



Figure 4. The Effect of BAC Contact Time on (a) pH and (b) TSS of Wastewater.

3.3. The Effect of BAC and Alum Combination on pH and TSS

The optimization of BAC performance is achieved by combining it with a low alum dosage at 1%, and its effects on pH and TSS parameters are presented in Figure 5. Figure 5a demonstrates that BAC and the BAC-alum combination treatments can stabilize pH. The initial (pH of 6.6) increases to a pH of 7.5 after BAC interaction and pH of 7.4 with the BAC-alum combination treatment. Figure 5b demonstrates the results of the TSS measurement of wastewater using BAC and BAC-alum. This result confirms that the BAC-alum exhibits a significant reduction in TSS values, approximately twice as much as the reduction achieved with BAC without alum, with a TSS value of 73 mg/L. This value suits the regulatory limit for TSS in wastewater (<400 mg/L). This combination approach can reduce the usage of alum at a low dosage and eliminates the application of quicklime, which presences a more eco-friendly treatment. Moreover, BAC can be produced from various waste materials, including bamboo; this fact supports one of the principal green chemistry applications by reducing potentially hazardous substances released into the environment. Based on the good results of the BAC-based filtration optimization method, a wastewater treatment system is proposed and illustrated in Figure 6.



Figure 5. The Influence of BAC and alum combination on (a) pH and (b) TSS of wastewater.

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Figure 6. Proposed design of BAC implementation in wastewater treatment ponds.

4. CONCLUSION

Bamboo-based activated carbon (BAC) has been successfully produced from bamboo waste. BAC's surface morphology is smoother than BC, with evenly distributed pores on its surface. The physicochemical activation enhances the availability of interaction sites on the surface of BAC by removing volatile compounds, as evidenced by a decrease in the intensity of hydroxyl functional groups in the FTIR spectra. BAC can rapidly neutralize the pH of wastewater within 10 minutes. However, achieving a significant reduction in TSS using BAC requires a relatively longer contact time. Combining BAC with low-dosage alum at 1% can reduce TSS levels becoming 73 mg/L. This combination can reduce the usage of quicklime and alum, providing an eco-friendly alternative in wastewater management.

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