

Reduction of BOD, COD, and TSS in Textile Wastewater Using Bentonite Activated Charcoal Adsorbent

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Abstract

Wastewater generated by the textile industry contains a variety of hazardous contaminants, including complex organic compounds, synthetic dyes, surfactants, heavy metals, and critical pollution indicators such as biochemical oxygen demand (BOD), chemical oxygen demand (COD), and total suspended solids (TSS). This study evaluates the efficacy of a dualadsorbent system comprising hydrochloric acid-activated bentonite and phosphoric acid-activated carbon for the removal of BOD, COD, and TSS from textile effluents. The activation processes significantly altered the physicochemical properties of both adsorbents. The moisture content of activated carbon decreased from 18% to 3.10%, while ash content was reduced from 15% to 3.05%. The iodine number, indicative of adsorption capacity, increased from 650 mg/g to 810 mg/g. Additionally, the fixed carbon content improved from 55% to 80%. Surface characterization via Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) confirmed substantial structural modifications, which enhanced the adsorption performance of the materials. Application of the combined adsorbent system resulted in substantial pollutant removal efficiencies, with reductions of 70.23% in BOD, 70.11% in COD, and 74.88% in TSS. These findings demonstrate that acidactivated bentonite and phosphoric acid-treated activated carbon represent a promising and efficient adsorptive treatment strategy for the remediation of textile wastewater.

Keywords: bentonite, activated carbon, textile wastewater, BOD, COD.

1. INTRODUCTION

The textile industry is pivotal in the global economy, particularly in developing countries such as Indonesia. This sector contributes significantly to the Gross Domestic Product (GDP) and provides substantial employment opportunities. However, along with its rapid expansion, the textile industry has emerged as a major contributor to environmental pollution, primarily through the wastewater generated during production. Improper disposal of untreated textile wastewater can result in severe impacts on aquatic ecosystems and overall environmental health. This wastewater often contains a variety of harmful substances, including complex organic compounds, synthetic dyes, surfactants, heavy metals, as well as key pollutants such as Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), and Total Suspended Solids (TSS), posing serious risks to environmental integrity and public health[1–3].

High concentrations of BOD and COD can deplete dissolved oxygen levels in water, creating hypoxic conditions that threaten aquatic life. Toxic substances such as dyes and heavy metals present in textile wastewater can accumulate through the food chain, disrupting reproductive systems and causing genetic mutations in aquatic organisms. Furthermore, elevated TSS concentrations can disturb the osmotic balance of aquatic species, adversely affecting their physiological functions and survival. Heavy metals, including lead, chromium, and cadmium, frequently found in textile wastewater, are neurotoxic and carcinogenic. These contaminants can cause kidney dysfunction, developmental issues in children, and an increased risk of cancer. Direct exposure to contaminated water can also lead to dermatological problems, such as irritation and allergic reactions. The use of contaminated, subsequently entering the human food chain, thus broadening the potential long-term health impacts[3,4]

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Despite the widespread application of conventional wastewater treatment technologies, such as coagulationflocculation, activated sludge processes, and membrane filtration, these methods still face significant challenges. These include high operational costs, substantial energy consumption, limited effectiveness in removing recalcitrant compounds, and the risk of secondary pollution. Consequently, there has been an increasing interest in exploring more efficient, sustainable, and cost-effective alternatives, one of which is adsorption[5,6]

Adsorption has gained considerable attention due to its simplicity, relatively low cost, and proven effectiveness in removing a wide range of pollutants[7,8]. Natural adsorbents such as bentonite and activated carbon have been extensively studied for their efficiency in adsorbing both organic and inorganic compounds from wastewater[9,10]. Bentonite is known for its high surface area and cation exchange capacity, while activated carbon is renowned for its complex pore structure and strong affinity for dyes and organic pollutants[11–15]

Several studies have demonstrated that combining bentonite and activated carbon can produce a synergistic effect, enhancing overall pollutant removal efficiency. [16] and [17]Research has shown that the combination of these adsorbents outperforms their individual use. [18] and [19]Moreover, utilizing low-cost, locally available materials for wastewater treatment has been emphasized, particularly in developing countries, to ensure both sustainability and economic feasibility.

However, research on the simultaneous reduction of BOD, COD, and TSS using a combination of bentonite and activated carbon in textile wastewater treatment remains limited. Therefore, this study aims to evaluate the performance of activated bentonite and activated carbon as a combined adsorbent for the reduction of BOD, COD, and TSS in textile industry wastewater. It is anticipated that the findings of this research will contribute to the development of cost-effective, environmentally friendly, and practical wastewater treatment technologies. These outcomes will also support efforts to mitigate the environmental and public health risks associated with textile industrial waste.

2. METHODS

This laboratory-scale experimental study aims to evaluate the effectiveness of a combined treatment using activated bentonite and activated carbon in reducing BOD, COD, and TSS levels in textile industry wastewater.

2.1 Materials

The primary materials utilized in this study included bentonite (sourced from PT Mineral Alam Lestari, Medan), activated carbon (PT Karbon Mandiri Sejahtera, Jakarta), hydrochloric acid (HCl, Merck), phosphoric acid (H₃PO₄, Merck), and distilled water. Textile wastewater samples were directly collected from an operational textile manufacturing facility located in the industrial zone of Deli Serdang Regency, North Sumatra, Indonesia.

2.2 Instruments and Equipment

The instruments employed in this study include a digital balance (±0.001 g, Sartorius), beaker glassware (Pyrex), Erlenmeyer flasks (Pyrex), funnel (Pyrex), magnetic stirrer, hot plate (Thermo Scientific), digital pH meter (Hanna Instruments), Whatman No. 42 filter paper, drying oven (Memmert), BOD incubator, UV-Visible spectrophotometer (Shimadzu), micropipette (Eppendorf), digital DO meter (YSI) for BOD analysis, closed reflux titration system (Metrohm) for COD analysis, centrifuge (Hermle), gravimetric equipment for TSS determination, Fourier-transform infrared spectroscopy (FTIR) (Perkin Elmer), and Scanning Electron Microscope (SEM) (JEOL).

2.3 Experimental Procedures

2.3.1 Activation of Bentonite

One hundred grams of bentonite were immersed in a 3 M HCl solution with a solid-to-liquid ratio of 1:10 (100 g: 1000 mL) and stirred intermittently for 24 hours. After activation, the bentonite was filtered, thoroughly rinsed

with distilled water to reach neutral pH, and oven-dried at 105°C for 12 hours. The dried bentonite was stored in an airtight container prior to application.

2.3.2 Activation of Activated Carbon

A similar procedure was applied to 100 g of activated carbon, using 3M H₃PO₄ at a 1:10 ratio for 24 hours. Subsequently, the carbon was filtered, washed to neutral pH, and oven-dried at 105°C for 12 hours before being stored in sealed containers.

2.3.3 Preparation of Adsorbent Mixture

Equal portions of the activated bentonite and activated carbon (5 g each) were weighed and homogenized using a ceramic mortar and pestle. A total of 10 g of the composite adsorbent was utilized per experimental run.

2.3.4 Adsorption Process

A total of 100 mL of textile wastewater was transferred into a 250 mL beaker, followed by the addition of 10 g of an adsorbent mixture consisting of bentonite and activated carbon. The mixture was stirred using a magnetic stirrer for 60 minutes at room temperature (~27°C). After the adsorption process, the mixture was filtered using Whatman No. 42 filter paper. The resulting filtrate was collected in a labeled clean container and stored for further analysis

2.3.5 BOD Determination

BOD levels were measured using the standard five-day incubation method. Initial dissolved oxygen (DO) concentrations were determined using a calibrated digital DO meter. The samples were then incubated at 20°C for 5 days. Final DO levels were recorded, and BOD was calculated as follows: BOD₅ (mg/L) = DO initial – DO final

2.3.6 COD Determination

COD analysis was carried out using the closed reflux method with potassium dichromate ($K_2Cr_2O_7$) as the oxidizing agent under acidic conditions. Samples were refluxed and subsequently titrated using ferrous ammonium sulfate [Fe(NH₄)₂(SO₄)₂], employing ferroin as an indicator. The COD concentration was derived from the volume of titrant consumed and expressed in mg/L O₂ equivalents.

2.3.7 TSS Determination

TSS was determined gravimetrically. Samples were filtered through pre-weighed and pre-dried filter paper. After filtration, the filter papers were re-dried and reweighed. TSS values were calculated using the following formula: TSS (mg/L) = (Final filter mass – Initial filter mass) \times 1000 / Sample volume (mL)

2.4 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was performed to identify the functional groups present on the surface of bentonite and activated carbon before and after the adsorption process. The spectra were recorded within the range of 4000–400 cm¹ to observe changes that may indicate interactions between the adsorbents and pollutants.

2.5 Scanning Electron Microscopy (SEM)

SEM was used to examine the surface morphology and pore structure of the adsorbents, both before and after adsorption. This analysis helped to visualize physical changes and pore distribution, providing insight into the adsorption mechanism.

3 RESEARCH RESULTS

3.1 Characteristics of Textile Industry Wastewater

The wastewater generated from the textile industry contains levels of BOD, COD, TSS, and pH that exceed the limits established by the Ministry of Environment and Forestry Regulation of the Republic of Indonesia No. P.16/MENLHK/SETJEN/KUM.1/4/2019. Characteristics of the textile wastewater are presented in Table 1.

Table 1. DOD, COD, 155; and pri Levels in Textile industry wastewater				
Parameter	Measured Value (mg/L)	Regulatory Limit (mg/L)	Compliance Status	
BOD	215	30	Non-compliant	
COD	542	100	Non-compliant	
TSS	430	50	Non-compliant	
pH	10,2	7,0	Non-compliant	

Table 1. BOD, COD, TSS, and pH Levels in Textile Industry Wastewater

3.2 Characterization of Activated Carbon Before and After Phosphoric Acid Activation

The physicochemical properties of activated carbon were evaluated before and after activation with 3M phosphoric acid (H₃PO₄). Parameters assessed included moisture content, ash content, iodine number, and fixed carbon, by the Indonesian National Standard (SNI 06-3730-1995). The results are summarized in Table 2. **Table 2**. Characteristics of Activated Carbon Before and After Activation

Parameter	Before Activation	After Activation	SNI Standard Requirement
Moisture Content (%)	18.00	3.10	≤ 15%
Ash Content (%)	15.00	3.05	$\leq 10\%$
Iodine Number (mg/g)	650	810	\geq 750 mg/g
Fixed Carbon (%)	55.00	80.00	\geq 750 mg/g

The activation process significantly enhanced the adsorption capacity and overall quality of the activated carbon.

3.3 Effect of Combined Bentonite and Activated Carbon Adsorbents on BOD, COD, and TSS Reduction An adsorption treatment was conducted using a combination of acid-activated bentonite and activated carbon, with a total mass of 10 grams in 100 mL of textile wastewater for 60 minutes. The treatment resulted in substantial reductions in BOD, COD, and TSS concentrations, as presented in Table 3.

Parameter	Initial Concentration (mg/L)	Final Concentration (mg/L)	Removal Efficiency (%)
BOD	215	64	70.23
COD	542	162	70.11
TSS	430	108	74.88

The significant reduction in all three parameters demonstrates the effectiveness of the combined bentonite and activated carbon adsorbents in treating textile industry wastewater.

3.4 Characterization of activated charcoal before and after activation

Figure 1 presents the FTIR spectra of activated charcoal before and after chemical activation. The spectral differences indicate significant changes in the surface functional groups following the activation process. The FTIR analysis shows that activation introduces oxygen-containing groups, with dominant peaks at 3400 cm⁻¹ (O-H) and 1600 cm⁻¹ (C=O), improving the adsorption capacity of the activated charcoal.



Figure 1. FTIR spectra of activated charcoal before and after chemical activation

3.5 Characterization of bentonite before and after activation.

FTIR analysis of bentonite before and after activation shows significant changes in surface functional groups, particularly an increase in hydroxyl (O-H) groups and modifications in the silicate structure (Si-O). These changes enhance the material's adsorption capacity, improving its effectiveness in applications like wastewater treatment.



Figure 2. FTIR spectrum of bentonite before and after activation

3.6 Characterization Based on SEM Morphology

Chemical activation of the bentonite and activated carbon composite resulted in significant changes to the surface morphology, characterized by an increased number of pores and a larger surface area. These modifications enhance the material's adsorptive capacity, thereby improving its efficiency in reducing BOD, COD, and TSS in textile industry wastewater.



Figure 3a. SEM image of the bentonite and activated carbon composite before chemical activation. Figure 3b. SEM image of the bentonite and activated carbon composite after chemical activation.

4. Discussion

4.1 Moisture Content

Moisture content analysis is carried out to determine the hygroscopic properties of activated carbon, or its ability to absorb water. Figure 4 shows the moisture content before and after activation using H_3PO_4 . Before activation, the activated carbon had a moisture content of 18%, which is higher than the maximum limit of 15% according to the Indonesian National Standard (SNI 06-3730-1995). After activation with 1M H_3PO_4 , the moisture content dropped to 8.35%, and with 3M H_3PO_4 , it decreased further to 3.10%. This reduction is due to the ability of H_3PO_4 to remove water and open the pores of the carbon material. Lower moisture content is beneficial because it increases the adsorption capacity of activated carbon. High moisture can block pores and reduce effectiveness. These results are in line with previous studies by [20] dan [21], which also found that higher concentrations of H_3PO_4 reduce moisture levels. The values obtained in this study meet the standard requirements of SNI 06-3730-1995.



Figure 4. Moisture content of activated carbon before and after activation.

4.2 Ash Content

The ash content determination aims to evaluate the residual metal oxides in activated carbon. Ash is the residue left after combustion or carbonization. Figure 5 shows the ash content of activated carbon before and after activation with H_3PO_4 . Unactivated carbon has an ash content of 15%, exceeding the SNI standard limit of 10%. After activation with H_3PO_4 , the ash content decreased significantly. Activation with H_3PO_4 reduced the ash content to 7.20%, and further reduced it to 3.05% with a 3M concentration. This decrease is due to H_3PO_4 role in

removing metal oxides, opening pores, and increasing surface area. Low ash content is critical for enhancing adsorption capacity, as high ash can block pores. These results align with previous studies by [20] and [22]confirming that higher H_3PO_4 concentrations reduce ash content. This study complies with the SNI 06-3730-1995 standard[23].



Figure 5. Ash content of activated carbon before and after activation.

4.3 Iodine Adsorption Capacity

The determination of iodine adsorption capacity aims to evaluate the ability of activated carbon to adsorb dissolved substances. As shown in Figure 6, unactivated carbon exhibited a low iodine number of 650 mg/g, which falls below the minimum standard requirement of 750 mg/g. This is attributed to insufficient pore development and limited surface area, resulting in reduced adsorption performance. Following chemical activation with H₃PO₄ at concentrations of 1M and 3M, the iodine adsorption capacity increased to 770 mg/g and 810 mg/g, respectively. The activation process enhances pore development, removes impurities, and expands the surface area of the activated carbon, thereby improving its adsorption capability. These findings are consistent with previous studies by [20], [21], and [22], which reported that H₃PO₄ activation significantly improves the porosity and the number of active sites on activated carbon. Consequently, this leads to a higher capacity for adsorbing dissolved substances.



Figure 6. Iodine adsorption capacity of activated carbon before and after activation

4.4 Fixed Carbon Content

The determination of fixed carbon content measures the amount of pure carbon in charcoal after carbonization and activation. This is influenced by moisture, ash, and iodine adsorption. The results are shown in Figure 6. Figure 6 shows that unactivated charcoal has a low fixed carbon content of 55%, below the SNI standard of 65%. This is due to low porosity and surface area, which reduces adsorption. After activation with 1M H₃PO₄, the carbon content increased to 70%, and 80% with 3M H₃PO₄. This increase is due to improved porosity, removal of impurities, and larger surface area. These results are consistent with studies by [20][21] and [22], which found that H₃PO₄ activation increases fixed carbon content by opening pores and removing contaminants. The carbon content meets the SNI 06-3730-1995 standard.



Figure 6. Fixed carbon content of activated carbon before and after activation.

4.5 FTIR Spectrum Analysis of Activated Charcoal Before and After Activation

Based on Figure 1, the FTIR Spectrum of Activated Charcoal before activation (red line), a broad absorption band observed around 3420 cm¹ corresponds to O–H stretching vibrations, indicating the presence of hydroxyl groups. The peak near 2920 cm¹ is associated with C–H stretching of aliphatic compounds. A peak around 1620 cm⁻¹ suggests the presence of C=C stretching vibrations from aromatic rings, while the absorption in the range of 1100–1030 cm⁻¹ is attributed to C–O stretching. Post-activation (green line), the absorption bands become more pronounced and well-defined. This enhancement signifies that phosphoric acid activation effectively removed impurities and introduced or enhanced active functional groups on the charcoal surface. The process also reinforced the aromatic structure and expanded the surface area, thereby improving the adsorption performance of the activated charcoal.

4.6 FTIR Spectrum Analysis of bentonite before and after activation.

Figure 2 presents a comparison of the FTIR spectra of bentonite before and after activation. The pre-activation spectrum (blue line) shows absorption peaks around 3400 cm⁻¹ (O-H), 1030 cm⁻¹ (Si-O), and 500 cm⁻¹ (Al-O). After activation (red line), changes are observed in the O-H and Si-O peaks, indicating an increase in porosity and adsorption capacity of the bentonite. Acid activation opens the pores of bentonite, enhancing its adsorption capacity and increasing functional groups such as -OH and -COOH, thereby improving its efficiency. These findings are consistent with the results of [24], which demonstrated that bentonite activation enhances its adsorption capabilities.

4.7 Surface Morphology Based on SEM Analysis

Based on Figures 3a and 3b, chemical activation of the bentonite and activated carbon composite resulted in a marked transformation of the surface morphology. Before activation (Figure 3a), the adsorbent surface appeared dense, irregular, and exhibited relatively few pores. Following the activation process (Figure 3b), the surface became noticeably rougher, more porous, and featured more open cavities. These morphological changes indicate an increase in specific surface area, which enhances the adsorptive capacity of the material. Consequently, the chemically activated composite demonstrates improved potential for the removal of pollutants such as BOD, COD, and TSS from textile industry wastewater.

4.8 Purification of Textile Industry Wastewater Using a Combination of Bentonite and Activated Charcoal Adsorbents Before and After Activation

The combination of acid-activated bentonite and activated carbon effectively reduced BOD, COD, and TSS concentrations in textile wastewater, with removal efficiencies of 70.23% for BOD, 70.11% for COD, and 74.88% for TSS (Table 3). The reduction in BOD indicates the successful adsorption of biodegradable organic materials, while the decrease in COD and TSS suggests that other organic compounds and suspended solids were also effectively adsorbed. Acid-activated bentonite adsorbs larger organic molecules, while activated carbon captures smaller dissolved organic compounds and other substances. Together, they reduce the organic content in the wastewater, contributing to the reduction in both BOD and COD. The reduction in TSS is attributed to bentonite's ability to adsorb larger particles, while activated carbon captures finer particles, leading to a decrease in suspended solids in the wastewater. These findings are consistent with the study by Pratama et al. (2020), who also used a combination of bentonite and activated carbon to reduce COD and TSS in textile wastewater, although their BOD removal efficiency was lower. The research by Wijaya et al. (2021) further supports these findings, highlighting the influence of activated carbon surface area on its ability to adsorb TSS and COD. Overall, the combination of acid-activated bentonite and activated carbon presents an effective and environmentally friendly solution for textile wastewater treatment.

5. Conclusion

The combination of acid-activated bentonite and activated carbon proves to be effective in reducing BOD, COD, and TSS levels in textile wastewater. Adsorption treatment conducted for 60 minutes using 10 grams of adsorbent in 100 mL of wastewater resulted in a removal efficiency of 70.23% for BOD, 70.11% for COD, and 74.88% for

TSS. These findings demonstrate that the synergistic effect of the two adsorbents has significant potential as an environmentally friendly and cost-effective solution for textile wastewater treatment.

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