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Research Article

Utilization of Bottom Ash as an Adsorbent for Color and COD Removal for Textile Industry Waste

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Abstract

This study investigates the use of bottom ash as an adsorbent with and without chemical activation treatment. This study also determines the effect of pH and stirring speed on the efficiency of bottom ash in the adsorption of color and COD. Bottom ash was ground and sieved by 100 mesh sieves and was activated by hydrochloric acid 2%. The experiments were conducted at pH variations of 6, 8, 10; contact time variations of 30, 60, and 90 minutes; and stirring speeds of 30 and 60 rpm. The most effective adsorption by non-activated adsorbents occurred at the combination of pH six and stirring speed of 60 rpm with the removal efficiency for color 66.30% at a contact time of 90 minutes and for COD 31.55% at a contact time of 30 minutes. While for activated adsorbent, the highest removal for color (77.44%) and COD removal (40.54%) were at pH 6, stirring speed 30 rpm, contact time 30 minutes. The bottom ash was potentially used as an adsorbent for treating wastewater containing high concentrations of color and COD.

Keywords: Adsorbent; bottom ash; COD; color

1. Introduction

Fast-growing urbanization and industrialization have resulted in the rapid development of the textile industry in West Java Province, Indonesia; based on data from the Central Bureau of Statistics, the growth of the textile industry has increased by 9.53% by the end of 2019 (BPS, 2020). In specific, the Ministry of Industry has stated that the textile and clothing industry is among the five manufacturing sectors that are prioritized and prepared to support the 4.0 era in Indonesia. The textile and clothing industries have achieved the highest growth, accounting for 15.08% in the third quarter of 2019. These industrial sectors have provided an opportunity to develop national economic growth (Hermawan, 2011). Moreover, the continuous growth of the population has had a positive impact on the textile industry's development.

However, the fast development of the textile industry generates wastewater from the textile production process, causing significant adverse impacts. The wastewater generated from the textile production process has a relatively high color, COD, and heavy metals and is a by-product of the dyeing and rinsing process (Hadiwidodo et al., 2009). The treated textile wastewater from the waste water treatment plant (WWTP) outlet still has a relatively high color content because the dyes used in the textile industry are generally made of azo compounds. This compound is very stable, difficult to degrade, and harmful to humans and the environment if discharged directly into the environment (Kustomo and Santosa, 2019).

On the other hand, the textile industry production process requires steam produced from coal-fired boilers. It is projected that the steam requirement for this industry had reached 4 million tons in 2018 (BPPT, 2018). Burning coal produces residues in the form of bottom ash and fly ash. The Government Regulation of the Republic of Indonesia No. 101 of 2012 has stated that these two wastes are hazardous and toxic. In the most recent regulation (The Government Regulation no, 22 of 2021), fly ash and bottom ash produced from low-temperature combustion facilities are still classified as hazardous wastes. The waste generated from coal combustion at low temperatures contains chemical compounds such as arsenic, lead, mercury, and chromium harmful to the environment and public health; thus, further management and control are necessary (Febriana et al., 2021). Several alternatives can be used to address the problem of fly-and-bottom ash waste treatment. This includes utilizing the fly- and bottom ash as one of the raw materials for cement. However, bottom ash is not adequately suitable if used as a cement raw material since it has a high residual carbon content of more than 6% by mass (Wardani, 2012). One of the opportunities in the utilization of bottom ash as an adsorbent. Bottom ash contains SiO2 and Al2O3, which are thought to be similar to zeolite (Bertolini, 2013). The coal residues can be used as adsorbents to remove COD (Cahyono, 2012), and Sururi et al. (2016) have found that zeolite can be used as an adsorbent to remove organic compounds in water. Moreover, the high carbon content in the bottom ash makes it similar to carbon characteristics even the bottom ash has undergone a process at high temperatures (1200˚C - 1400˚C), which causes bottom ash to have a large number of pores, thus enabling them to be used directly as an adsorbent (Azizzah and Lisha, 2019). Burton et al. (2013) have proposed the adsorption method to reduce the color and COD concentration in wastewater.

Efforts to utilize bottom ash require an activation process because the presence of metal impurities such as Fe, Mg, Ti, and K can inhibit the adsorption process. The activation could also expand the surface and clean the bottom ash pores (Telaumbanua, 2017). Activation can be done physically through a heating process to the water content in the pores can be evaporated, and the surface area is increased (Fatimah, 2019). Meanwhile, chemical activation can be done by adding an acid (hydrochloric acid) or base at a specific concentration to change the bottom ash structure to improve the absorption capacity of the adsorbent.

Previous studies on the use of bottom ash as an adsorbent have reported that the efficiency of coal ash without activation was 97.1%, with a particle size of 100 mesh, a maximum height of 11 cm, and a minimum flow rate of 15 mL/min for COD removal (Mouhri et, al, 2021). Kuntari (2017) also found that the efficiency of bottom ash which was activated physically through the drying process at a temperature of 500°C for 15 minutes in color adsorption, was 91.2% at pH 1.5 with a contact time of 80 minutes. In addition, Arifatunnisa et al. (2022) reported that the color adsorption process achieved 30.26% using bottom ash which was physically activated through 1-hour incineration at a temperature of 100°C. This removal efficiency for color in textile wastewater occurred at pH 2, 180 minutes contact time. For bottom ash which was activated by hydrochloric acid 0.25 M and dried at 105°C temperature for 12 hours, was able to remove COD in Rhodamine B 233 artificial solution up to 95% at 2 hours contact time (Wahyuni et al., 2018). Commonly, the activation process was conducted through a drying process at 500 - 1000 OC in 15 - 60 minutes; this process uses large and expensive electrical energy. These conditions hinder the application of this technique. This study investigated the characteristic of bottom ash without and with activation by hydrochloric acid, including the effect of pH, stirring speed, and contact time on the adsorption process. The result of this study is expected to be one of the solutions and references for reducing the bottom ash wastes by utilizing the waste as an adsorbent for removing color and COD in textile wastewater. Moreover, using natural energy such as sunlight for the adsorbent drying process will make the adsorbent preparation easier and cheaper. Hence, it will increase the opportunities for their application in the field.

2. Methodology

2.1 Sample Preparation

The wastewater sample was filtered using Whatman 42 filter paper to separate the solution from the suspended solids. There were two treatments for the used bottom ash: with and without activation. Inactivated bottom ash (BA) was obtained by grounding and filtering the bottom ash using a 100-mesh sieve to ensure uniform particle size. At the same time, acid-activated bottom ash (BHCl) was generated by soaking the bottom ash in 2% hydrochloric acid for 24 hours, followed by drying at room temperature utilizing sunlight (sun drying) for ± three days to reduce the water content. This was conducted after the bottom ash was grounded and filtered using a 100-mesh sieve.

2.2 Analysis of Bottom Ash Characteristics

The measurement of the bottom ash in the sample was conducted to identify heavy metal content and metal oxides such as AI2O3, SiO2, Fe2O3, NaO, P2O5, MnO, CaO, and MgO, K2O, and TiO2. This was conducted by Na2CO3 and HF smelting to determine the elements of Al, Fe, Na, Mn, Ca, Mg, K, Na, and heavy metals, which were then analyzed using the Atomic Absorption Spectrophotometer (Shimadzu AA 7000). Meanwhile, Ti and P elements were measured using UV-Vis Spectrophotometry (Shimadzu UV-1700), and SiO2 was measured using the gravimetric method (Saputra, 2015). The proportion of each metal oxide was calculated following this equation:

$$\%\ \text{Metal Oxide} = \frac{\text{Sample volume x dilution x measured concentration x f}}{\text{sample weight x }10^{6}} \text{x}100 \ldots\ldots (1)$$

2.3 pH Point of Zero Charge (pH_{pzc}) Determination

The pHpzc measurement was performed by mixing the wastewater solution and 150 mg of adsorbent at pH 2, 4, 6, 8, 10, and 12. The stirring was conducted for 1 hour. After 48 hours, the final pH was measured, and the data was plotted on a straight-line curve of the initial pH against the final pH (Vijayakumaran, 2009). The value of pHpzc determines the adsorption process dynamics (Ramdhani et al., 2018). The observed pH > pHpzc refers to the surface of the adsorbent being negatively charged, pH < pHpzc indicates the surface of the adsorbent is positively charged, and if pH = pHpzc, then it is neutrally charged (Ikhlaq et al., 2012).

2.4 Color and Chemical Oxygen Demand (COD) Adsorption

The variations in this study include contact time, stirring speed, and pH. The adsorption measurements were carried out on each 5 grams adsorbent added to 150 ml of filtered wastewater at pH of 6, 8, and 10 variations. The pH adjustment was carried out by adding 2% hydrochloric acid and 2% Sodium hydroxide with stirring speeds of 30 and 60 rpm at contact time variations of 30, 60, and 90 minutes. Samples for color measurement were measured according to the Standard Methods for the Examination of Water and Wastewater 2120 C, Spectrophotometric Method using UV-Vis Spectrophotometry (Shimidzu UV mini-1240) with a maximum wavelength of 410 nm. Meanwhile, the COD sample was measured based on the Standard Methods for examining water and wastewater 5220C closed reflux with a titrimetric method.

3. Result and Discussion

3.1 Bottom Ash Characteristics

Based on the measurement results of metal oxides in the bottom ash laboratory used in this study, the characteristics can be seen in Table 1 below:

Table 1 Bottom ash composition (in %)

Compound	*Bottom Ash*	
	Unactivated	**HCl-Activated**
SiO_2	43.77	41.84
TiO_2	1.22	0.57
Al_2O_3	12.38	9.06
Fe_2O_3	8.93	7.69
MnO	0.06	0.05
CaO	0.46	0.31
MgO	1.48	1.20
NaO	1.11	1.34
K_2O	1.34	3.46
P_2O_5	0.49	0.40
Cu	0.0059	0.0061
Pb	0.0021	0.0018
Zn	0.0029	0.0011
Ni	0.0045	0.0038
Cr	0.0020	0.0023
Co	0.0032	0.0025
Mn	0.0186	0.0135

Table 1 shows that the main three compounds in the inactivated bottom ash were 43.77% $SiO2$, 12.38% $Al2O3$, and 8.93% $Fe2O3$, consistent with Yunita (2017) that found the composition of bottom ash included silica dioxide ($SiO2$), aluminum oxide ($Al2O3$), iron oxide ($Fe2O3$). Bertolini (2013) also reported that the primary compound of bottom ash were silica, alumina, and iron oxides. Meanwhile, the composition of $K2O$, CaO, $TiO2$, and MgO was below 5%. In addition, bottom ash contains a heavy element that can interfere with the adsorption process. As seen in Table 1, Mn was the highest heavy metal compound. Meanwhile, Cr was the lowest. It is hoped that heavy metal elements will not influence the adsorption process.

Meanwhile, in hydrochloric acid-activated bottom ash, the levels of $SiO2$, $Al2O3$, and $Fe2O3$ decreased by 1.93%, 3.32%, and 1.23%. The metal oxides and other heavy metals also decreased compared to BA conditions. This condition indicates that the chemical activation process using an acid activator at low concentrations effectively dissolves $Al2O3$ and $SiO2$. This was because hydrochloric acid is a water-binding agent that is more effective for dissolving aluminum, iron, magnesium, and calcium silicate and bounding the adsorbent, enabling cleaner and opened pores (Dewi, 2019). The decrease in aluminum content in the acid activation process, according to Ozka and Ulku (2005), occurred due to the dealumination process so that the acid can dissolve $Al2O3$ even at low concentrations.

3.2 pH_{pzc} Determination

pHpzc is a condition where the surface of the adsorbent has a neutral charge so that the pH of the adsorbent suspension does not contribute to the acid or base functional groups of the adsorbent

(Ramadhani et al., 2018; Wardiyati, 2010). Figure 1 shows that the pHpzc in each type of adsorbent occurred at pH 8.

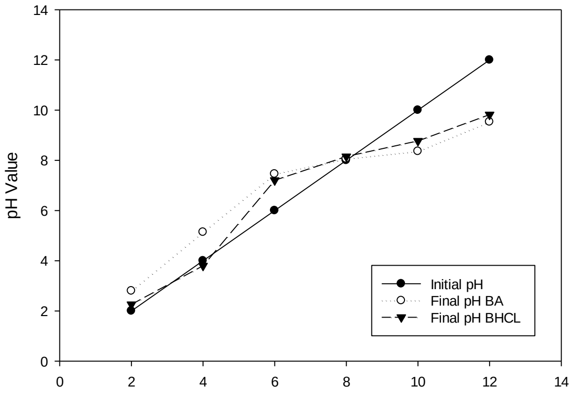

Figure 1. Determination of pH_{pzc}

Previous studies have found that the effect of pH on the adsorption process was: (i) if the pH of the system were lower than the pHpzc of the adsorbent, the bottom ash surface would be positively charged; (ii) if the pH was above pHpzc then the bottom ash surface will be negatively charged; and (iii) if the pH of the system was equal to the pHpzc, the bottom ash surface has no charge (Ramdhani et al., 2018; Sururi et al., 2016; Kuntari, 2017). The measurement of pHpzc is essential in the adsorption process. Every surface creates a charge (positive/negative). It is necessary to know the value where the charge is proportional to the pH of the solution surrounding the particles (AL-DEG, 2000).

3.3 Adsorption Process

Color in textile industrial wastewater is one of the main parameters for successful treatment. Most of the colors used in the textile industry are synthetic dyes that are difficult to decompose, resulting in a high concentration of COD. The processing of textile waste to reduce the concentration of color and COD is very complex because of its varying chemical structure (Sayan, 2006). The adsorption process using bottom ash is expected to reduce the concentration of color and COD of textile industry waste. The pH parameter is the degree of acidity that can express the level of acidity or alkalinity in a solution (Zilius, 2017). The pH of the solution can affect the adsorption process because pH can enhance and inhibit the adsorption process. Determination of the optimum pH is to review how the effect of pH on the activation process when the pH of the solution is below pHpzc (pH 6) and above pHpzc (pH 8). The adsorption measurements on color and COD parameters can be seen in Figure 2 at a stirring speed of 60 rpm.

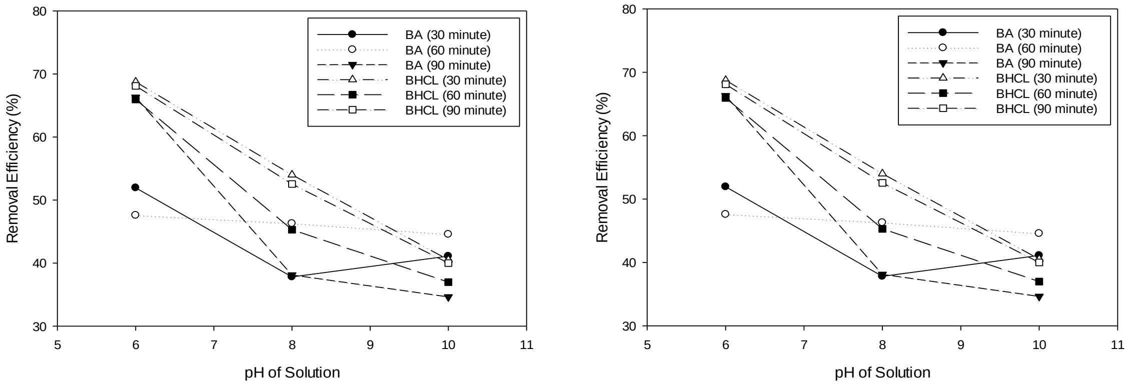

Figure 2 Removal efficiency at pH (a) color (b) variations

Figure 2 shows that the optimum conditions for removing color and COD with an inactivated adsorbent (BA) occurred at pH 6. Under these conditions, the efficiency for removing color and COD with contact times of 90 minutes and 30 minutes was 66.30% and 31.55%. This phenomenon occurs because the pH of the solution (6) is below pHpzc (8) (pH < pHpzc), the adsorbent surface becomes positive and supports anionic adsorption (Ramdhani et al., 2018). The study by Jarusiripot (2014) has reported that when the pH of the solution is less than pHpzc, the surface of the adsorbent was positively charged and thus supported the adsorption of anionic species of dye molecules. Lin and Yang (2007) found that the bottom ash surface became negatively charged as the pH increased. This condition prevented organic anionics; thus, the COD adsorption decreased at high pH.

Meanwhile, in bottom ash activated by hydrochloric acid (BHCL) at pH 6, the removal efficiency was 68.73% and 28.93%, with a contact time of 30 minutes. The removal efficiency in the BHCL condition showed that the COD parameter decreased by 2.62% compared to the BA adsorbent. For the color parameter, a 2.43% increase in the removal efficiency was observed because the pores owned by BHCl were cleaner and more open.

The effect of various stirring speeds (30, 60, and 90 minutes) on the adsorption process can be seen in Figure 3. Stirring speed is one of the factors that affect the adsorption process. This stirring allows the adsorbent to contact the waste to be absorbed. The selected stirring speed must be adequately determined because it will detach bonding between adsorbent and adsorbate if the stirring speed is too fast. Vice versa, if the stirring speed used was too slow, the collisions between the adsorbents and adsorbate will be less effective.

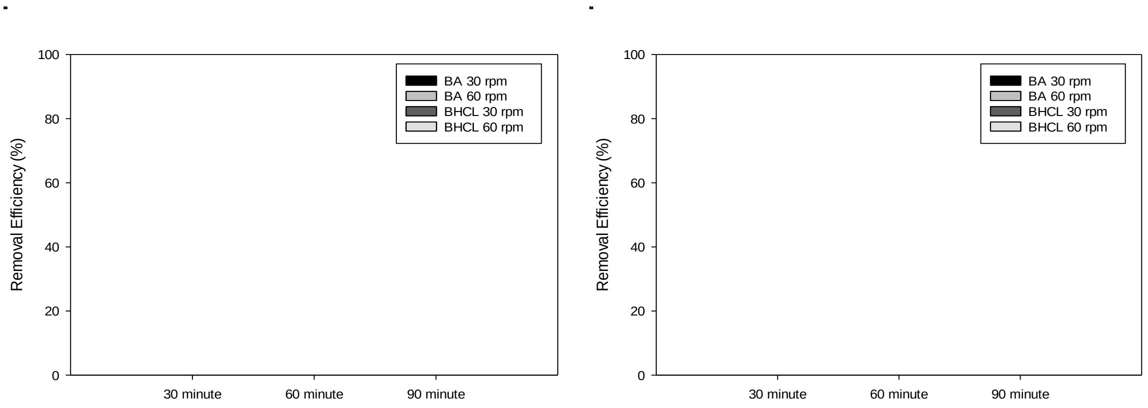

Figure 3 Removal efficiency at stirring speed of 30 and 60 rpm for color (a) and COD (b)

As seen in Figure 3, each type of adsorbent had different the best contact times for the color adsorption. A contact time of 30 minutes and 60 minutes have the best stirring speed at 30 rpm with the efficiency of 52% (BA) and 77.44% (BHCL). Meanwhile, with a contact time of 90 minutes, the best color removal occurred at a stirring speed of 60 rpm of 66% for BA and 68% for BHCL. BA's best COD adsorption process was effective at a stirring speed of 30 rpm with a removal efficiency of 38% at 30 minutes contact time, followed by 32.37% at 60 minutes and 20.18% at 90 minutes contact times. Meanwhile for BHCL, the best removal efficiency was 40.54% (at 30 minutes), 20.27% (at 60 minutes), and 17.57% (at 90 minutes). This result shows that longer contact time affects the stirring speed in the process. If the stirring speed is too fast, the attached adsorbate would be broken again. Therefore, in this study, the stirring speed of 30 rpm was more effective than the stirring speed of 60 rpm. Sutrisno (2014) has found that a lower stirring speed (40 rpm) had better adsorption efficiency than a higher stirring speed (80 rpm) with 90% at room temperature conditions. This suggests that the greater the stirring speed, the removal efficiency will decrease. The optimum contact time is the length of time required for stirring between the adsorbate and adsorbent to reach equilibrium. The optimum contact time dramatically affects the adsorption process. The effect of contact time in the adsorption process, which was determined based on measurements at pH 6, pH eight, and 10 with a stirring speed of 60 rpm, can be seen in Figure 4.

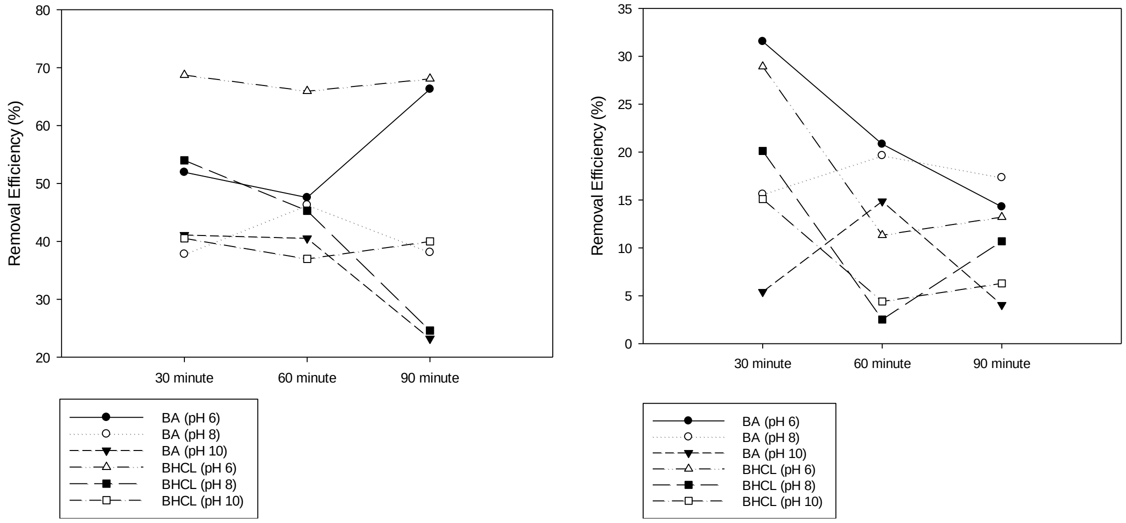

Figure 4 Removal efficiency of contact time of 30, 60, and 90 minutes for color (a) and COD (b)

Figure 4 shows that the adsorption of color at each pH has the best contact time. At pH 6, the best contact time for BA was 90 minutes with an efficiency of 66.30%; meanwhile, for BHCL, the highest efficiency was 54% at 30 minutes contact time. The best contact time at pH 8 was 60 minutes with an efficiency of 46% (BA) and 30 minutes contact time with 4% efficiency for BHCL. The best efficiency at pH 10 (41%) occurred at a contact time of 30 minutes for BA and 40.52% for BHCL. Meanwhile, for COD removal, the best efficiency at pH 6 was 31% for BA and 28.9% for BHCL, which occurred at 30 minutes. For BA, the best contact time was 60 minutes, with an efficiency of 19% at pH 8 and 14% at pH 10. While for the BHCL adsorbent type, the best contact time was 30 minutes, with 13% and 15% removal efficiencies at pH 8 and 10, respectively. Therefore, contact time influences the efficiency obtained that the longer the contact time, the greater the chance for the adsorbate to be diffused by the adsorbent. These results strengthened a previous study conducted by Gandhimathi (2013) that reported the best adsorption capacity occurred at a contact time of 30 minutes and 45 minutes, with the best adsorption capacity of 90.34% at pH eight and stirring speed of 150 rpm. This study also showed that the efficiency would gradually decrease once the optimum condition has been achieved. After reaching the optimum contact time, the adsorbent will experience desorption. Since the adsorbent has been saturated, the pores have been filled (Syaifie et al., 2019). Previous studies have shown that either activated or inactivated bottom ash can adsorb color and COD in wastewater from 40.6% to 97.1%. The following Table 2 summarizes previous studies' results regarding the effectiveness of bottom ash adsorption for reducing color and COD in wastewater.

Table 2 Summary of Previous Studies Results on the effectiveness of bottom ash adsorption

No	Adsorbent Type	Waste Type	Condition	Drying	Test Parameters	Variation	Effectiveness	Source
1	Bottom Ash	Textile wastewater	Bottom Ash with a particle size	drying was used with sunlight	Color	➢ BA, pH 6, 60 rpm, 90	66.30% 77.40%	This study

No	Adsorbent Type	Waste Type	Condition	Drying	Test Parameters	Variation	Effectiveness	Source
			of 100 mesh (BA) and Bottom ash with chemical activation of 2% hydrochloric acid (BHCl)	exposure for three days	COD	minute ➢ BHCl, pH 6, 30 rpm, 30 minute ➢ BA, pH 6, 60 rpm, 30 minute ➢ BHCl, pH 6, 30 rpm, 30 minute	31.55% 40.54%	
2	Bottom Ash	Palm Oil Mill Waste	Bottom ash with a particle size of 500 m and activated using hydrochloric acid	The oven was used for the drying at a temperature of 70°C for two days	Color	Contact time 18 hours, heavy dose of adoseben / addition of 10% hydrochloric acid activation , pH 6	81.15 %	Saleh, 2019
3	Coal Ash	Pulp Mill Waste	Bottom ash is not activated	Bottom ash was not dried	Color	Particle size 100 μm Contact time 60 minute Adsorbent dosage 2 g/L	88 % 80% 82%	Fu, 2019
4	Bottom Ash	Paper-making waste and coking waste	The bottom ash is sifted	Bottom ash was not dried, but during the adsorption experiment, the temperature was set at 25 ± one °C for 3 hours	COD, Color	Contact time 3 jam Effective particle size 0.074 mm	Coking waste COD 45 % Color 76% Paper-making waste COD 40,6% Color 79%	Sun, Wei-Ling, 2007

No	Adsorbent Type	Waste Type	Condition	Drying	Test Parameters	Variation	Effectiveness	Source
5	Fly ash	Textile waste water	Fly ash was dried for 24 hours and then sieved into 4, 8, 16, 30, 50, 100, and 200 mesh sizes,	Fly ash was dried at a temperature of 105°C for 24 hours	Color (*methylene blue)*	The dose of adsorbent is 7.5 g/L, contact time is 30 minutes, stirring speed is 200 rpm, room temperature, pH 10	92%	Hasan, 2019
6	Bottom ash	Tanning wastewater	Bottom ash was dried at 80˚C for 24 hours and sieved to a size of 80-125 m	Bottom ash was dried at a temperature of 80°C for 24 hours	COD	pH 6.36, 25˚C, 80 m, particle size 100 m	97.1%	Mohri, 2021

Previous studies showed that the COD adsorption process would be effective when the wastewater was at pH 6, the temperature of 25℃, and the adsorbent condition was physically activated by drying the adsorbent at 80℃ for 24 hours and sieved adsorbent to a size of 100 m. This was conducted to clean and open the adsorbent pores to enable maximum COD adsorption by 97.1%. Meanwhile, the adsorption of color was effective (92%) if the waste conditions were at pH 10, room temperature, stirring speed of 200 rpm, contact time of 30 minutes, and the adsorbent was treated with physical activation by drying the adsorbent for 24 hours then crushed and sieved using 4, 8, 16, 30, 50, 100, and 200 mesh sieves (Hasan, 2019).

The observed efficiency obtained in this study was lower than in previous studies. For inactivated adsorbent, the COD removal of 31,55% occurred at pH 6, contact time 30 minutes, and stirring Speed 60 rpm. Meanwhile, for HCl activated adsorbent, the removal efficiency of COD was 40.54% at pH 6, contact time 30 minutes, and stirring speed 30 rpm. The removal efficiency of color was 66,30% for inactivated adsorbent at pH 6, contact time 90', and stirring speed of 60 rpm. HCl obtained an efficiency of 77.40% activated adsorbent at pH 6, contact time 60 minutes, and stirring speed 30 rpm. Compared to the results of Saleh's research (2019) that used bottom ash adsorbent, which was dried at a temperature of 70℃ for two days, the removal efficiency for color reached 81.15%. This difference was because the adsorbent used in this study was only dried using sunlight for ± three days. Thus, there was the possibility that the adsorbent pores were not completely opened. In addition, the stirring speed used was relatively lower than those used in other previous studies. However, the adsorption of color and COD parameters in textile wastewater by bottom ash activated through sunlight exposure as such in this recent study could minimize the costs for bottom ash wastes utilization and process. Therefore, this could be a promising method for textile wastewater treatment plants either in the pre-treatment or subsequent processes. A further study investigating bottom ash as an adsorbent in continuous scale reactors would be necessary.

4. Conclusion

The bottom ash residue's chemical characteristics were suitable for being used as adsorbents. The main compounds of the bottom ash from coal boiler combustion residues in the textile industry include SiO_2 of 43.77% and Al_2O_3 of 12.38%, with minor components of Fe_2O_3, CaO, MgO, NaO, K_2O, TiO_2, P_2O_5. The heavy metal elements in the bottom ash were Cu, Pb, Zn, Ni, Cr, Co, and Mn. After activation using hydrochloric acid, the content of Al_2O_3 and SiO_2 decreased by 3.32% and 1.92%, indicating a dealumination process had occurred. The best result of dye removal on the inactivated adsorbent was 66.30%, at pH 6, contact time of 30 minutes, and stirring speed of 60 rpm. While the best conditions for HCl activated adsorbent in removing color that reached 77.40% occurred at pH 6, stirring speed of 30 rpm with a contact time of 30 minutes, The best COD removal for both inactivated adsorbent (31.55%) and activated adsorbent (40.54%) occurred at pH six and contacted time of 30 minutes, but with different stirring speeds: at stirring speed of 60 rpm for inactivated adsorbent, and 30 rpm for the activated adsorbent. Both inactivated and activated adsorbents from bottom ash, which was dried by sunlight, can remove color and COD. This technique can be applied through serial processing or during the pre-treatment process in wastewater treatment plants. According to this study results, it is hoped that the bottom ash could be considered an adsorbent in wastewater treatment, although further research is needed on continuous scale reactors.

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