Effect of particle size distribution and acid treated coal bottom ash on TSS and COD removal from textile effluent using fixed bed column

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Abstract. Most wastewater treatment plant (WWTP) of textile industries in Bandung area have not operated optimally due to several reasons, such as high processing load and high operational cost. On the other hand, bottom ash which is a major pollutant generated from coal-fired boiler has good potential as adsorbent. In this paper bottom ash obtained from a textile industry in Bandung has been used to remove the organic contaminant from textile effluent in order to reduce the burden to WWTP. The ash was treated using acid solution prior to adsorption and filtration of waste, in order to avoid leaching of its metal content by wastewater and to enhance its adsorption capacity. The best contact time on COD removal and the adsorption capacity were obtained in batch processes. The effect of bed particle size to pressure drop, the influence of contact time, bed particle size on TSS filtration and COD removal were also studied using a fixed bed column. Acid treatment of ash was carried out using 0.25 M HCl solution. One gram of bottom ash was leached by 20 mL of acid solution to give 1.402 ppm of chromium effluent (below the threshold limit of 5 ppm). The highest adsorption of acid-treated bottom ash on Rhodamine B 233 was achieved on 2 h duration of adsorption, with adsorption capacity of 15.87 mg Rhodamine/g ash. Filtration of wastewater on fixed bed of finer grain (1.0625 mm) as well as coarser grain (1.200 mm) provided 55 % TSS reduction, 30% volumetric rate reduction, and 95% COD reduction in less than one hour.

1. Introduction

Textile industry is one of the most important sectors in Bandung and its surrounding areas. There are around 350 textile industries by 2016. The textile industries generally have difficulties in meeting wastewater discharge limits, particularly with regard to dissolved solids, pH, BOD, COD, heavy metals, and color of effluent. On the other hand, a large amount of bottom and fly ashes that are produced during combustion of coal in power plant requires large dumping sites for the safe disposal, due to the relatively low level of ash utilization rate.

Adsorption, a displacement phenomenon on a surface, is strongly influenced by particle size distribution and adsorbent porosity. Most of textile industries in West Java use chain grate boiler which operates at a relatively low combustion temperature that produces a fairly large amount of bottom ash. The bottom ash has a relatively high carbon content, estimated around 30%. Due to the heating process, there is an increase in carbon porosity. Therefore, it is potential to be used as an effective adsorbent. To reduce the load of textile wastewater treatment plant (WWTP), an initial treatment of waste can be conducted using coal bottom ash. The leaching studies of coal bottom ash indicate that the heavy metals...
in the bottom ash and fly ash are bound to different fractions with different strengths. From the environmental and utilization perspectives, heavy metals in ashes posed different levels of environmental contamination risk.

Therefore, the bottom ash needs to be extracted prior to utilization as an adsorbent in order to give the environmentally responsible release of wastewater. The heavy metals content of the effluent resulted from acid treatment should meet the threshold limit. Besides that, ash treatment with acid is necessary to improve the adsorption performance.

The effective size and uniformity of grain to characteristics of granular media treatment systems [1]. Many researchers had investigated the utilization of coal ash as a low-cost adsorbent and filter medium for uptaking of organic compound from waste effluents, including adsorption of reactive dyes [2], utilization of NaOH-activated coal bottom ash as Cd^{2+} ion adsorbent [3], utilization of bottom ash to adsorb heavy metals [4], organic pollutants[5]and dyes [6,7], utilization of coal ash as an adsorbent of organic matter in brackish water [8], and the utilization of bottom ash as a filter medium for textile wastewater treatment. Treatment of artificial textile dyes waste (methylene blue) were adsorbed using fly ash [9]. The use of coal ash as an adsorbent was chosen due to its high silica and alumina content. Coal ash is a low-cost adsorbent compared to other adsorbents, but the ash contains some heavy metal elements as trace elements such as Fe, Mn, Zn, Cu, Cr, Ni, Mg, Pb, Li, Co, Hg, Cd and As which are potentially extracted when contacted with acidic solutions. Therefore, coal ash is classified as a hazardous waste [10]. Different elements have different leaching behaviors due to differences in elemental properties and pH of the solution as well as leaching time [11]. The fly ash treated with acic solution increased the adsorption surface area by 60% [12].

The objectives of this paper were to study the effect of coal bottom ash particle size distribution on the extent of total suspended solid (TSS) removal in a fixed bed column and to investigate the effect of ash initial treatment on the COD removal of artificial wastewater using batch process and a fixed bed column.

2. Materials and methods
The bottom ash utilized as adsorbent and filter medium in this study originated from a textile industry in Cimahi, Province of West Java, Indonesia. The TSS content of artificial wastewater was prepared by mixing fine particles of bentonite with tap water. The COD content was prepared by dissolving Rhodamine B 233 in tap water.

2.1. Filtration for TSS removal
The filtration was conducted by means of an experimental seepage column (100 cm height and 5 cm inside diameter, made of Plexiglass), completed with pressure drop indicators, which were installed at different heights in the filter. The initial ash treatments were grinding and sizing to reduce the size from about 5 cm to 14- 20 mesh (1.40 – 0.85 mm). The ground ash then washed using aquadest at 80°C for 4 h with stirring, followed by filtration and drying at 100°C. Ash washing was conducted to reduce the water-soluble metal oxide content as well as to clean ash pores from the impurities. The artificial wastewater with TSS content of 460 ppm was fed from a water tank to the upper constant head controller, flow throughout the column and then discharged via the lower constant head controller (valve). The flow rate and hydraulic head along the filtration path were observed manually every 30-60 min. The effect of particle size on pressure drop, effluent flow rate reduction and TSS removal was investigated for eight hours of operation.

2.2. Adsorption studies
2.2.1. Adsorbent preparation. Trace element analysis on coal bottom ash showed that the chromium content a hazardous metal was quite high (110 ppm). Therefore, it was necessary to leach the chromium content to prevent the environmental effects. The leaching was performed by mixing the ash in 0.25 M HCl solution (with acid to ash ratio = 20: 1), with stirring for 4 h, followed by filtration and drying at 105°C for 12 h in an oven [13]. Acid treatment on ash was also intended to open the pores of ash which
will increase adsorption capacity. The best contact time of COD removal was obtained for initial concentration of 750 ppm.

2.2.2. Isotherm adsorption. In order to identify the appropriate adsorption isotherm of COD, a series of batch adsorption experiments of artificial wastewater were performed using the best contact time obtained from the former experiment. Langmuir equilibrium isotherms were analyzed according to the experiment data and the adsorption capacity of the ash was estimated.

2.2.3. Column test. Fixed bed experiments were performed with constant bed height and best particle sizes obtained from the filtration experiments. As is known, adsorption is a transient (time dependence) process, the amount of material adsorbed within a bed depends both on position and time. As fluid enters the bed, it comes in contact with the upper layer of absorbent, then solute filling up the available sites. The adsorbent near the entrance is saturated and the fluid penetrates farther into the bed before all solute is removed. Thus, the active region shifts down through the bed as time goes on. The breakpoint was obtained at the effluent's TSS of 200 ppm. Analysis of TSS was conducted by means of SNI 06-6989.3-2004. Analysis of COD was conducted by means of SNI 6989.73-2009 method. Chromium concentration in the effluent was measured with AAS GBC Avanta Ver. 2.02.

3. Results and discussion

3.1. Filtration

The experiment to study deep bed filtration was based on a visual observation of pressure drop, measuring the flow rate and TSS of the effluent.

![Figure 1. Progress of the filter bed resistance in time.](image)

From Figure 1, it is found that over the operation time the pressure drops of both filter beds getting bigger with time. The bed filter with grain diameter of 1.200 mm gave lower pressure drop and longer period of time than that of 1.0625 mm. The bed with finer grain showed a very rapid increase in head loss. The pressure dropped along the column height fit smoothly with curve, indicated that clogging occurred evenly in the whole bed.

The filtration was intended as a preliminary treatment to WWTP, which effluent will be further treated. Therefore, the effluent of filtration was considered to be satisfactory with TSS content of 200 ppm. The effect of contact time on velocity of effluent and TSS level were presented on Figure 2a and 2b, respectively.

Based on Figure 2a and 2b, the bed with grain size of 1.200 mm was considered more satisfactory than the bed with grain size of 1.0625 mm, which reduced TSS value from 430 to 200 ppm and 100 ppm during one hour and two hours of contact time (one hour service run), with 30% effluent velocity reduction (from 36.67 to 24.42m³/m².day). This filtration belongs to the medium speed (quick sand filter: 100-475 m³/m².day; slow sand filter: 4 - 8 m³/m².day).
3.2. Adsorption

3.2.1. Adsorbent treatment and contact time. Acid treatment of 1.200 mm bottom ash using 0.25M HCl solution with ratio of 1 g ash to 20 mL of acid leached the highest heavy metal with chromium by 74.52%, which gave effluent concentration of 1.4016 ppm (below the threshold limit of 5 ppm [14]).

![Figure 2. Effect of time on (a) velocity and (b) TSS of effluent.](image)

![Figure 3. COD removal in batch adsorption.](image)
The determination of best contact time was performed by means of batch adsorption for COD removal. The result is presented in Figure 3. Based on Figure 3, the best contact time was two hours. The figure also showed that ash treatment using solution of 0.25 M HCl enhanced the COD removal from wastewater, almost 100% higher than that of water-washed ash of which 68 and 34%, respectively.

The enhancement of treated ash adsorption was in line with the work of Bada and Potgieter-Vermaak [12] who found that the fly ash treated with acid solution has increased the adsorption surface area by 60% (due to increasing pore volume). The appearance of the liquid underwent adsorption for longer time than two hours was cloudy, indicated that for contact time longer than two hours, the adsorbent was eroded resulting in the adsorbate release from the adsorbent.

3.2.2. Isotherm adsorption. The adsorption equilibrium data plot in the form of an isotherm adsorption is presented in Figure 4.

![Figure 4. Isotherm adsorption of COD removal.](image)

The shape of isotherm adsorption curve was categorized as favorable, which permitted higher solid loadings at lower solution concentrations. The mathematical interpretation of the adsorption isotherms was studied using the Langmuir model. For the Langmuir model linearization gives:

$$
\frac{C_e}{q_e} = \frac{1}{KQ_0} + \frac{C_e}{Q_0}
$$

$q_e =$ Concentration of COD in mg/g of ash; $C_o =$ Initial concentration of COD in solution in ppm; $C_e =$ Final concentration of COD in solution in mg/L.

The plot of $C_e/q_e$ versus $C_e$ is presented on Figure 5, which gives a straight line with intercept of $1/(KQ_0)$ and slope of $1/Q_0$.

![Figure 5. Langmuir isotherm adsorption.](image)
Figure 5 shows that the isotherm adsorption was linear with $R^2$ value of 0.924, indicating the applicability of Langmuir model. The adsorption capacity was found from the slope of the curve to be 15.873 mg/g ash.

3.2.3. Breakthrough curve and evaluation of continuous adsorption. Evaluation of adsorption performance in the continuous process was conducted by observing the emerging of unabsorbed solute in the effluent (breakthrough curve). The plot is presented on Figure 6. Artificial wastewater with COD of 750 ppm and TSS value of 430 ppm was prepared as feed mixture in order to examine the effect of TSS content on the adsorption.

![Breakthrough curve of adsorption](image)

Figure 6. Breakthrough curve of adsorption.

Figure 6 shows that in the bed with grain diameter of 1.200 mm the unabsorbed solute emerged in the effluent at four hours service time, and increased with time. As the time went on the COD of effluent increased, but the process did not continue for more than 8 h because the effluent flow rate kept decreasing. Therefore, the breakthrough curve was not completed yet. However the bed with grain diameter of 1.06 mm, the effluent remained clean until 8 h of operation.

4. Conclusion

The results showed that the bed column with coarse grain provided better TSS reduction than finer grain. TSS removal was achieved at relatively short service run with the medium speed filtration. The COD removal with acid-treated coal bottom ash increased by 100%. In column with coarse grain, the unabsorbed solute started to emerge in the effluent at 4 h of service time and increased with time. Whilst the bed with fine grain resulted clean effluent until 8 h of operation.

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