

## Material characterizations and copper, zinc, and lead removing capacity of Çayırhan fly ash

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### Abstract

This research focuses on the removal of toxic heavy metals in wastewater using coal fly ash (CFA) from Çayırhan thermal power plant, which is among the biggest coal producers in Turkey. The chemical and physical properties of fly ash were determined. The adsorption, desorption, and precipitation experiments were done for different purposes. The effects of metal concentration, agitation time, particle size, ash amount, and pH in the metal removal were examined. The results indicated that the main mechanism for metal removal was precipitation due to the alkaline characteristics of fly ash. A very small percentage of adsorbed metal was released during the desorption test. Statistical analysis of the experiments was carried out with the selected parameters at 4 levels. Statistically speaking, for all metals (Cu, Zn, Pb) and all initial ppm levels (10, 50, 100 ppm), initially 50 ppm zinc solution showed the best metal removal capacity.

**Key Words:** Coal fly ash, heavy metals, adsorption, precipitation, statistical analysis

### 1. Introduction

Industrial waste management is essential for sustainable ecological balance. Fly ash is the by-product of the coal combustion process for energy generation, and is recognized as an environmental pollutant (Bayat, 1997). Since the production of fly ash is an environmental problem, applications for the utilization of fly ash have been undertaken worldwide over the years. One of the very important utilization areas of fly ash is the adsorption of metallic ions at the fluid/solid interface and this has been studied for several years (Rio and Delebarre, 2003; Dermatas and Meng, 2003; Hong et al., 2009, Lu et al., 2009; Özdemir and Yapar, 2009). Moreover, fly ash produced by coal combustion is considered in numerous studies aimed at valorization. Different applications (cements, roads, and backfill) already allow a recycling of an important part of fly ash produced (Weshe, 1991; Tütünlü, 2000; Sarkar et al., 2004). The use of fly ash for metallic ions' removal from aqueous solution is the current concern. Studies have shown that fly ash might be beneficial for the removal of heavy metal ions in wastewaters (Querol et al., 1999; Dermatas and Meng, 2003; Rio and Delebarre, 2003; Tuzcu, 2005; Tuzcu and Atalay, 2006; Zheng et al., 2008).

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In the present study, the chemical and physical properties of Çayırhan thermal power plant's fly ash were determined. The adsorption, desorption, and precipitation experiments were done to quantify the effects of different parameters in the light of the chemical and physical properties of the fly ash. The effects of metal concentration, agitation time, particle size, ash amount, and pH on metal removal were examined.

## 2. Experimental

### 2.1. Materials

#### 2.1.1. Chemical analysis of Çayırhan fly ash

The chemical composition of the coal fly ash sample was determined using an X-ray fluorescence spectrometer. Table 1 shows the results of the chemical analysis of the test sample. More than half of the fly ash's composition is occupied by  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Fe}_2\text{O}_3$ .

**Table 1.** Chemical analysis of the sample (XRF).

$\text{Na}_2\text{O}$	3.30%	$\text{Al}_2\text{O}_3$	11.20%
$\text{K}_2\text{O}$	1.60%	$\text{SiO}_2$	50.30%
$\text{SO}_3$	5.25%	$\text{TiO}_2$	0.50%
$\text{MgO}$	4.90%	$\text{P}_2\text{O}_5$	0.40%
$\text{CaO}$	14.50%	$\text{Fe}_2\text{O}_3$	8.05%

#### 2.1.2. Physical analysis of Çayırhan fly ash

- Specific gravity determination

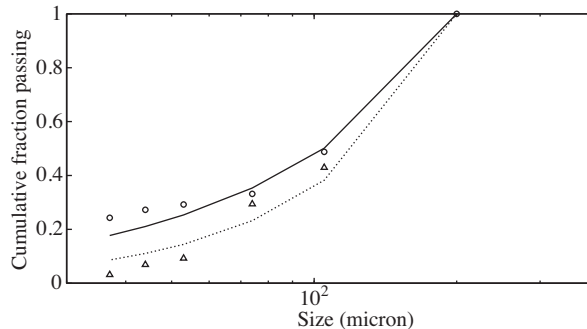
The specific gravity of fly ash was determined by a water and air pycnometer. The average specific gravity of CFA was found to be 1.97 (Table 3).

- Size analysis

##### *By screening*

The screen analyses of the CFA both were done by dry and wet screening methods. The  $\sqrt{2}$  Tyler mesh series were used. Dry and wet screen results are given in Figure 1. The Gates-Gaudin-Schuhmann distribution model  $((x/k)^m)$  was fitted to the experimental data with the distribution parameters given in Figure 1.

The dashed line in the figure represents for dry screening where  $k = 206$  and  $m = 1.427$ . The solid line stands for the wet screening where  $k = 210$  and  $m = 0.997$ .



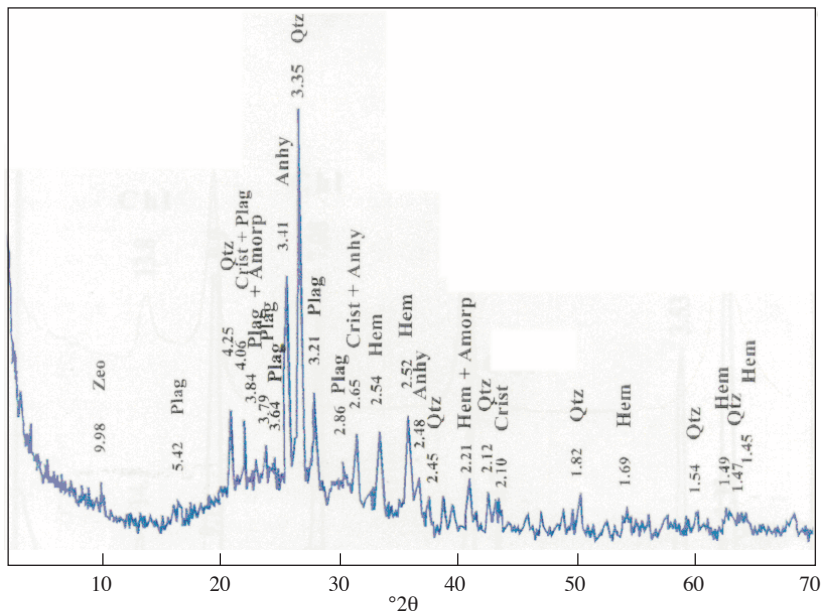
**Figure 1.** Dry and wet screening of CFA.

### Laser size analysis

The laser size analysis was done using Mastersizer 2000 Version 5.1. The  $d_{50}$  value obtained was about  $100 \mu\text{m}$  in screening, whereas  $124.663 \mu\text{m}$  was found from the laser size analysis. Surface and volume mean diameters was  $14.165 \mu\text{m}$  and  $159.199 \mu\text{m}$ , respectively.

- X-Ray diffractometry (XRD) analysis

The peaks of diffraction were recorded and plotted against a horizontal scale with  $2\theta$ . The XRD results of the CFA sample showed that quartz, plagioclase, anhydrite, hematite, cristobalite, and zeolite are the main constituents. The XRD pattern of the CFA is shown in Figure 2.



**Figure 2.** X-ray diffraction pattern of fly ash.

- Scanning Electron Microscopy (SEM)

Morphological properties of coal fly ash were determined using scanning electron microscopy (SEM-JEOL JSM 6400). It was recorded that the fly ash particles mainly contained small, glass-like particles of spherical and irregular shape. Generally, besides some amorphous and crystalline parts, non-opaque cenosphere (hollow) and plerosphere (filled with micro spheres) dominate the morphology in finer size fractions. The crystals present in ash particles may be acicular, elongated, or irregularly shaped. In general, the larger particles show a greater irregularity compared to smaller ones. Opaque amorphous particles predominantly might have arisen from coal components that were not combusted completely. The complete opaque spheres are mostly magnetite or other iron oxides, alone or in combination with silicates. The non-opaque particles are mostly silicates derived from clays and siltstones associated with the coal. Results of the XRD and XRF analyses show that more than half of the fly ash composition is occupied by silica, which is non-opaque spherical particles. The complete opaque spheres are mostly aluminum and iron oxide compounds. The constituent of the fly ash assembly is shown in Figure 3a-d. The elemental analysis of the plerosphere type of particles is shown in Table 2.

**Table 2.** Elemental analysis of glass-like particle.

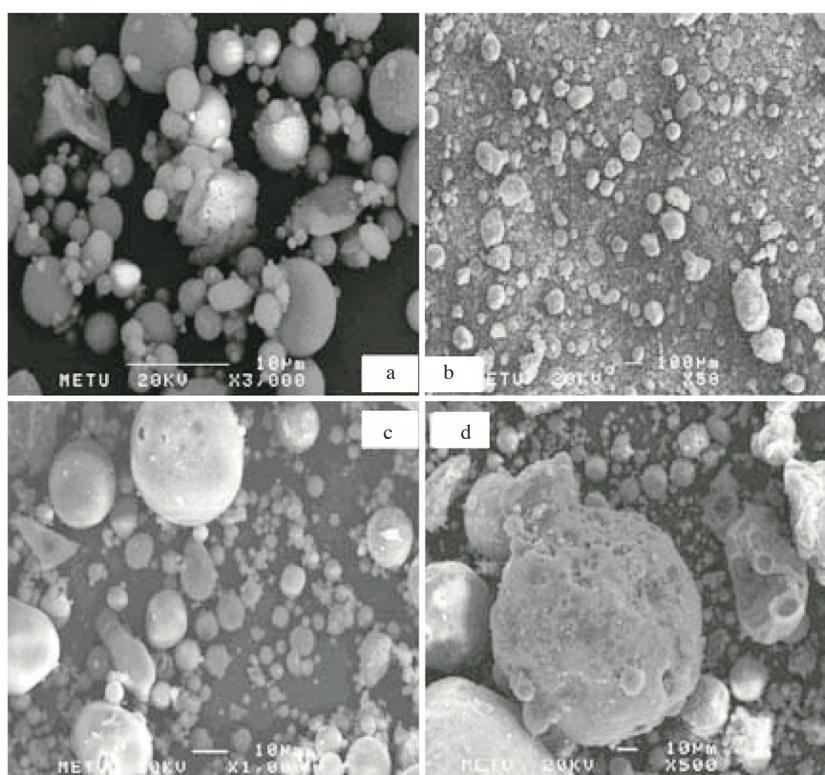
Element	Weight content %	Atomic content %	Compound content %	Formula
O	48.34	62.76	0	
Na	3.46	3.13	4.67	Na <sub>2</sub> O
Al	7.12	5.48	13.45	Al <sub>2</sub> O <sub>3</sub>
Si	33.75	24.96	72.2	SiO <sub>2</sub>
K	3.17	1.68	3.81	K <sub>2</sub> O
Ca	2.94	1.52	4.11	CaO
Fe	1.23	0.46	1.76	Fe <sub>2</sub> O <sub>3</sub>

- Porosity measurement

The pore size distribution of particles smaller than 100,000 Å (10 μm) was able to be measured due to the powder composition of the fly ash (Table 3). The macro pore values could not be examined. The total porosity value was 15.05% by volume.

- Specific surface area measurement (BET)

The specific surface areas of different sizes of coal fly ash were determined by single point BET nitrogen adsorption (Micromeritics Flowsorb II 2300). The measurements were done for 3 size fractions: +300 μm,



**Figure 3.** a) General view of CFA particles, b) General view of CFA particles, c) Spherical, glass-like particles, d) Non-opaque, spongy, hollow spherical particles.

-300+212  $\mu\text{m}$  and -75+53  $\mu\text{m}$ , respectively. It is commonly known that the specific surface area increases as the particle size decreases (Table 3), but this was not the case for the CFA sample. The largest specific surface area was found in the +300  $\mu\text{m}$  size fraction. The reason for this would be the lack of porosity in smaller size fractions.

- Effect of time and fly ash amount on the pH of distilled water

This test was done to quantify the alkaline property of the fly ash. Different amounts of fly ash were added and mixed with double distilled water. The effect of agitation time and fly ash amount on the double distilled water show the alkaline property of the fly ash. The increase in agitation time (treatment of distilled water with fly ash only) results in an increase in the pH of the distilled water (Table 3).

**Table 3.** Physical properties of fly ash.

Property	Value			
Specific gravity	1.97			
$d_{(50)}$ (screening)	100 ( $\mu\text{m}$ )			
$d_{(50)}$ (laser size analysis)	125 ( $\mu\text{m}$ )			
Surface mean diameter	14.165 ( $\mu\text{m}$ )			
Volume mean diameter	159.199 ( $\mu\text{m}$ )			
Porosity (macro)	15.05 (%)			
Specific surface area at 300 $\mu\text{m}$	2.0979 ( $\text{m}^2/\text{g}$ )			
Specific surface area at 252 $\mu\text{m}$	0.5467 ( $\text{m}^2/\text{g}$ )			
Specific surface area at 63 $\mu\text{m}$	0.3879 ( $\text{m}^2/\text{g}$ )			
Natural pH values of $\text{Cu}^{++}$ solution				
10 ppm	6.0			
50 ppm	5.7			
100 ppm	5.5			
Natural pH values of $\text{Zn}^{++}$ solution				
10 ppm	6.3			
50 ppm	6.0			
100 ppm	5.9			
Natural pH values of $\text{Pb}^{++}$ solution				
10 ppm	6.4			
50 ppm	5.7			
100 ppm	5.6			
Effect of time and fly ash amount on the pH of distilled water				
Time (min)	Fly ash amount			
	(g)			
	1	2	4	8
	pH			
0	6.6	6.6	6.6	6.6
5	11	11	11.2	11.5
10	11	11	11.4	11.5
30	11	11	11.5	11.6
60	11	11	11.5	11.6

A review of physical properties of Çayırhan fly ash is given in Table 3.

## 2.2. Methods

### 2.2.1. Adsorption experiments

The adsorption tests were carried out to see the metal removal capacity of CFA. Three analytical grade metal salts were used as heavy metal source: copper chloride ( $\text{CuCl}_2$ ), zinc chloride ( $\text{ZnCl}_2$ ), and lead chloride ( $\text{PbCl}_2$ ). From these metal salts, synthetic solutions were prepared. The studied ppm levels of the solutions were 10 ppm, 50 ppm, and 100 ppm for each of the metals. The initial pH values of the solutions are given in Table 4. The experiments were carried out in 200 mL beakers with 100 mL metal solutions, and varying fly ash amounts and time values. The prepared solutions were treated with 1 g, 2 g, 4 g, and 8 g of fly ash, that is, solid/liquid (fly ash/metal solution) ratios were 1%, 2%, 4%, and 8%, respectively, for different treatment durations (5 min, 10 min, 30 min, 60 min). The treatment processes were carried out using a magnetic stirrer at room temperature for those particular durations. At the end of the treatment period, the final concentrations of filtrated solutions were measured using an ATOMSPEK H1580 Analytical Instrument. It should be stated that, instead of giving enough time for the chemical equilibrium, 60-min treatment was carried out in the experiments.

**Table 4.** Initial (natural) pH studied for adsorption experiments.

Concentration (ppm)	pH		
	$\text{Cu}^{++}$	$\text{Zn}^{++}$	$\text{Pb}^{++}$
10	6	6.3	6.4
50	5.7	6	5.7
100	5.5	5.9	5.6

### 2.2.2. Precipitation experiments

It is known that particular metals have different stabilities at different pH values. Hence, it was aimed to observe the dramatic change (decrease) in the total dissolved metal concentrations by precipitation by changing (increasing) the pH of the solutions. These tests were carried out in the absence of fly ash. The natural pH values of the solutions were changed to evaluate the metal precipitation at different pH values. The natural pH values of the solutions were changed by addition of NaOH. The different pH values studied are shown in Table 5. The final concentrations of copper zinc and lead chloride solutions were measured after treatment.

**Table 5.** Different pH values studied for precipitation experiments.

$\text{Cu}^{++}$			pH values					
			$\text{Zn}^{++}$			$\text{Pb}^{++}$		
10 ppm	50 ppm	100 ppm	10 ppm	50 ppm	100 ppm	10 ppm	50 ppm	100 ppm
6	5.7	5.5	6.3	6	5.9	6.4	5.7	5.6
8.2	6.9	5.8	8.4	7.3	8	9.9	9.8	9.1
9.8	10.4	6.2	9.5	9	10.6	10.3	10.3	10.1
10.7	11.3	9.9	9.9	11.4	11.6	10.8	11	10.8
11.7	11.7	11.9	11.5	11.8	11.8	11.2	11.7	11.2

### 2.2.3. Desorption experiments

The desorption (metal release) stage of the experimental series was related to the metal retention capacity of the fly ash used in the adsorption experiments described above. Desorption experiments were carried out only at the level of 100 ppm, which was naturally the most acidic pH level of the studied ppm levels in adsorption experiments. The experiments were carried out in 2 stages: first was the metal removal from 100 ppm (Cu, Zn, Pb) metal containing solutions with 2 g of fly ash. In the second stage 2 g of fly ash (used in adsorption) was treated, that is, agitated for 10 h and subjected to metal analysis periodically at 2, 6, and 10 h, in 100 mL of double distilled water.

### 2.2.4. Metal removal test on a mixture solution

A final test was carried out on combined solutions of lead, copper, and zinc metal ions. The combined solution was prepared containing 100 ppm of each metal. In these tests, efforts were made to understand the effective mechanism for metal removal from solution, i.e. the precipitation or adsorption. In order to elucidate the mechanism of metal removal with fly ash 2 different procedures were followed:

- i. The alkaline property of fly ash, which is the reason for precipitation, was tested in the absence of fly ash. The water mixed with CFA was used as the precipitator for the metals. In order to do this, fly ash treated water was prepared: 80 g of fly ash was added to 1 L of distilled water and agitated for predetermined time intervals for 5, 10, and 15 min. After that, the water was filtrated from CFA. The appropriate amounts of metal salts were added to the CFA treated (alkaline property) water to obtain 100 ppm concentration for each metal. The metal ions added in alkaline solution were left to stand for 15 min. The metal concentrations were measured after the process.
- ii. To understand the role of adsorption in metal removal, 8 g of fly ash added to combined solutions of 100 ppm lead, zinc, and copper. The solution was mixed for 5, 10, and 15 min using a magnetic stirrer. After filtration a chemical determination of lead, zinc, and copper ions was performed on the filtrate.

### 2.2.5. Statistical analysis of the selected parameters

For the experimental design and statistical analysis of the experiments, it is meaningful to include all parameters (parameters due to main effects and interaction effects) in a regression equation. In this study, main effects were time, fly ash amount, and pH of the solution, and so logically it is necessary to include all these parameters and their interaction effects in our final regression line. Considering the direct effect of fly ash amount on the pH of the solutions, it is realistic to include only time, fly ash amount, and their interaction effect in the statistical model. For this reason 4 selected levels of time (5, 10, 30, and 60 min) and fly ash amounts (1, 2, 4, and 8 g) were analyzed in the statistical software Minitab 15.

## 3. Results and discussion

### 3.1. Effect of time and fly ash amount on metal removal

In general, at all 3 concentrations levels (10, 50, and 100 ppm) of metals (copper, zinc, and lead), there was a decrease in the total metal ion concentration with time. However, the noticeable or sharp decrease in metal ions occurs within the first 7.5 min. After that, there was a gradual decrease in metal ions. Copper metals show

better removal characteristics than the other metals. At the same ppm levels, i.e. 10 ppm copper and 10 ppm zinc and lead, 50 ppm copper 50 ppm zinc and lead, and 100 ppm copper and 100 ppm zinc and lead, copper concentration were well decreased compared to the other 2 metals (zinc and lead). It is shown in Figures 4-6 that time has positive effects on the removal of metal ions for all levels of fly ash addition. The metal removal occurred at all concentration levels in the first 10 min usually; after that there was no significant change in the metal concentrations.

Fly ash amount also has a positive effect on the removal of metallic ions from synthetic wastewater solutions. The initial metal concentrations of synthetic wastewater decrease with the increase in CFA for all metals.

### 3.2. Effect of particle size on metal removal

Although experiences dictate that there was no significant effect of particle size of CFA on removal of heavy metal ions from synthetic wastewaters, the better removal of heavy metals occurred in the biggest size fraction +300 micrometer for all metals. However, studies (Heechan et al., 2005; Grigorios et al., 2010) have shown that the screened larger size fraction shows better adsorption capacity than the as-received size fly ash.

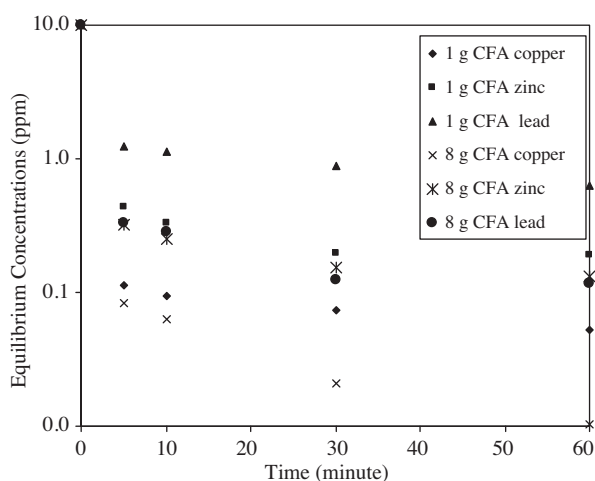


Figure 4. Elemental analysis of glass-like particles.

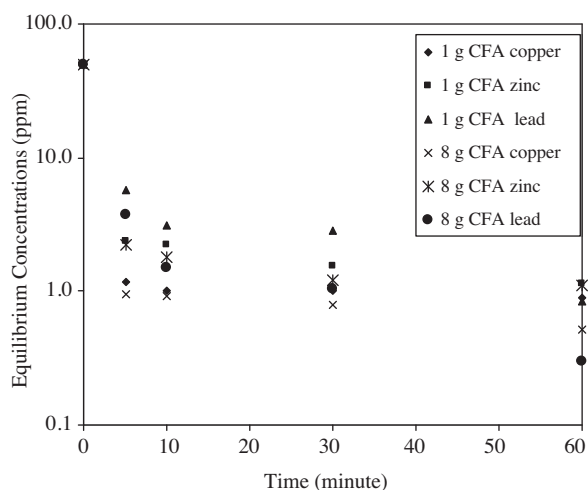


Figure 5. Specific volume vs. pore size histogram.

### 3.3. Effect of pH on metal removal

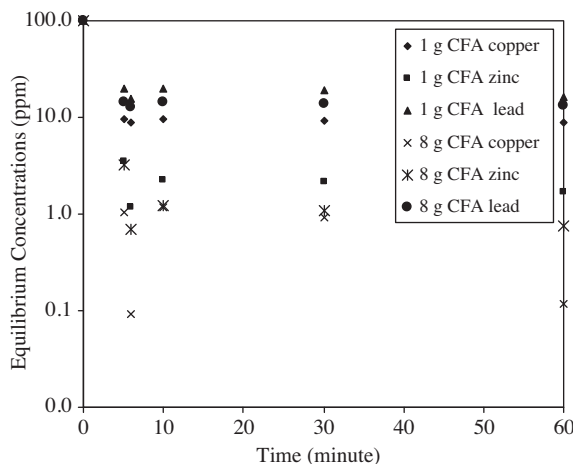
Fly ash has an alkaline property and it allows the precipitation of metallic ions. At alkaline pH levels, metal ions tend to precipitate and for this reason the effect of the adsorption mechanism was clearly seen at acidic pH values.

To understand the effect of fly ash (amount) on the pH of the solutions, natural pH levels of 10, 50, and 100 ppm Cu, Zn, and Pb solutions were treated with 1 and 8 g of fly ash for 15 min. A positive effect of the fly ash (amount) was observed. The contribution of the pH of the solutions by addition of fly ash is shown in Table 6.

In addition, precipitation tests were carried out to check the studied metals' (Cu, Zn, Pb) stability zones and to compare adsorption and precipitation characteristics. After 15 treatment with changed pH values, almost all the metal ions were removed from the solutions. The effect of pH is shown in Figures 7-9. It was understood



that, for all ppm levels and metals in general, copper shows a tendency to precipitate between pH 8 and 11, zinc between pH 9.5 and 11, and lead between pH 10 and 11. Between these pH ranges, Cu, Zn, and Pb metals were in the form of Me-OH and they resulted in a reduction in total dissolved metal ions in solutions.



**Figure 6.** Effect of time and fly ash amount on the equilibrium concentration of initial 10 ppm copper, zinc, and lead solutions.

**Table 6.** pH ranges of solutions after treatment with 1 and 8 g of fly ash.

Concentration (ppm)	measured pH values					
	copper		zinc		lead	
	Fly ash amount		Fly ash amount		Fly ash amount	
	1 (g)	8 (g)	1 (g)	8 (g)	1 (g)	8 (g)
10	10.5	11.5	10.5	11.5	10.5	11.5
50	8.5	9.5	8	9	8	9
100	6.5	7.5	6.3	7.3	6.5	7

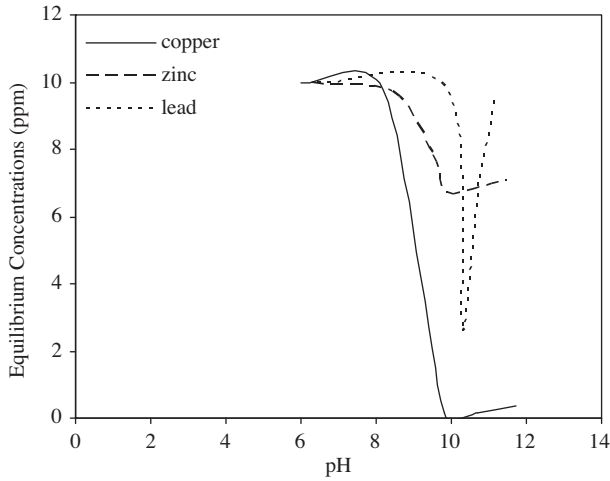
### 3.4. Results of desorption experiments

In desorption experiments, it was observed that the lead metal containing fly ash released the adsorbed metal more than the other metals. Considering all 3 metals, it can be seen that the desorption of heavy metal ions was nearly negligible at natural pH of the fly ash added distilled water. The results demonstrated that heavy metal removal involves stable adsorption and complexation of the metal cations on the reactive surfaces of fly ash. The studied lead ppm level in the desorption experiment was 100 ppm; at this high ppm level a maximum metal release of 0.225 ppm at the end of the 10-h agitation was observed, which is acceptable. The very small percentages of adsorbed metal released during the desorption tests indicated that the metal sequestered in fly ash remains stable over time. Figure 10 shows the results of metal release experiments; a very low amount of adsorbed metal was released after many hours.

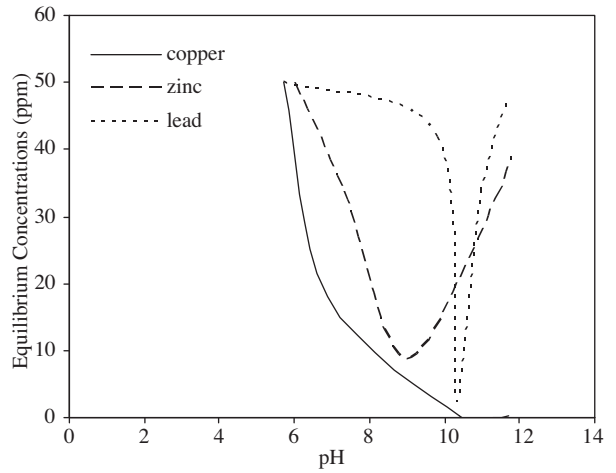
### 3.5. Results of metal removal test on a mixture solution

The results of the experimental tests for solutions containing a mixture of metals indicated that the dominant mechanism in metal removal was precipitation due to the alkaline characteristics of fly ash. The rest of the

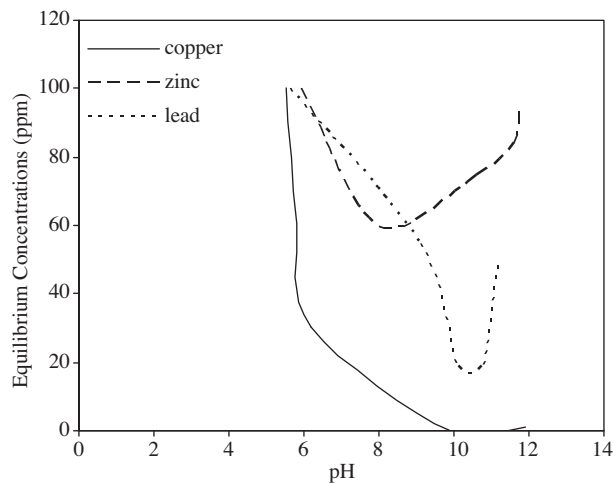
metal removal process was due to the adsorptive properties of fly ash. The pores of zeolite content and some particles give fly ash a high internal surface area and porous structure available for adsorption. The findings also indicate that fly ash has greater amenability of copper and zinc to removal compared to lead metal. The results confirmed that metal removal from the single metal solution was higher than the metal removal from the mixture of metal solutions. The equilibrium concentrations of all filtrates are plotted in Figure 11.



**Figure 7.** Effect of time and fly ash amount on the equilibrium concentration of initial 50 ppm copper, zinc, and lead solutions.



**Figure 8.** Effect of time and fly ash amount on the equilibrium concentration of initial 100 ppm copper, zinc, and lead solutions.

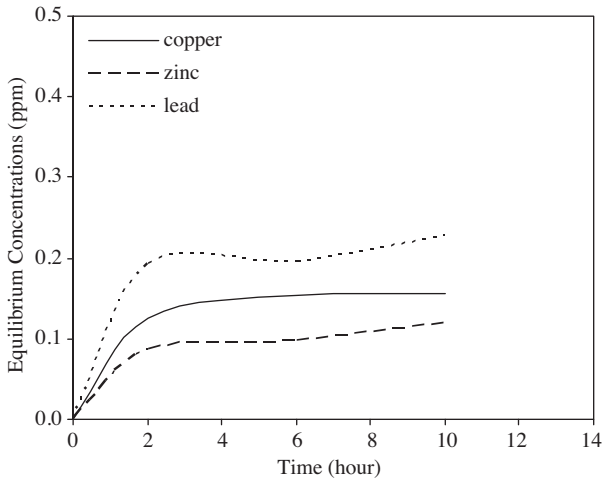


**Figure 9.** Effect of pH on the equilibrium concentration of initial 10 ppm copper, zinc, and lead solutions.

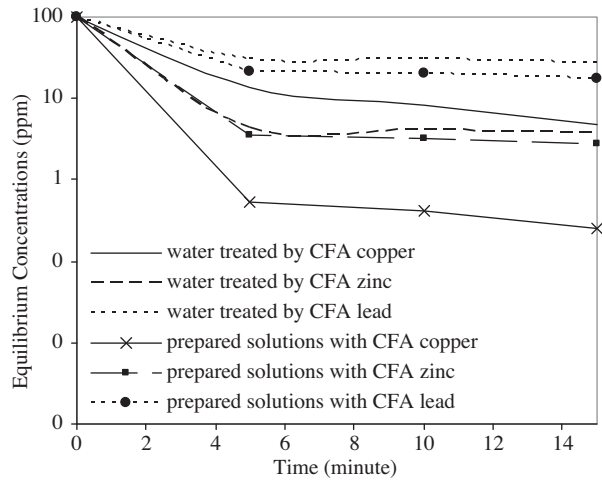
### 3.6. Statistical analysis results

The regression statistical models for all different metals and studied initial ppm values were created after carrying out the experiments. The effects of time and ash amount are the statistical parameters that were measured. The mathematical equations given in Table 7 will serve for the future needs in the absence of experimental data. If one wants to find out the final concentrate of the initial 10, 50 and 100 ppm copper, zinc, and lead solutions,

entering the desired ash amount and time to the equations will be enough. The strength of the statistical models was tested with the determination of adjusted-R<sup>2</sup> (adjusted coefficient of determination) value. Unlike generalized-R<sup>2</sup>, adjusted-R<sup>2</sup> increases with the positive contribution of the parameters (ash amount, time) to the model. However, generalized-R<sup>2</sup> would increase with the addition of any extra parameter to the equation. Therefore, observing an increase (or higher) in adjusted-R<sup>2</sup> value has a real physical meaning.



**Figure 10.** Effect of pH on the equilibrium concentration of initial 50 ppm copper, zinc, and lead solutions.



**Figure 11.** Effect of pH on the equilibrium concentration of initial 100 ppm copper, zinc, and lead solutions.

**Table 7.** Statistical analysis results.

Initial concentrations (ppm)	Regression equations	R-Sq (adj) (%)
10 (Cu)	$0.115 - 0.000940 \times \text{time} - 0.00480 \times \text{ash} - 0.000033 \text{ time} \times \text{ash}$	86
50 (Cu)	$51.13 - 0.00621 \times \text{time} - 0.0175 \times \text{ash} - 0.000297 \text{ time} \times \text{ash}$	78.3
100 (Cu)	$11.0 - 0.0954 \times \text{time} - 1.28 \times \text{ash} + 0.00696 \text{ time} \times \text{ash}$	66.9
10 (Zn)	$0.405 - 0.00435 \times \text{time} - 0.0138 \times \text{ash} + 0.000141 \text{ time} \times \text{ash}$	64.9
50 (Zn)	$2.44 - 0.0226 \times \text{time} - 0.0435 \times \text{ash} + 0.000465 \text{ time} \times \text{ash}$	89.7
100 (Zn)	$3.21 - 0.0361 \times \text{time} - 0.101 \times \text{ash} + 0.00014 \text{ time} \times \text{ash}$	59.6
10 (Pb)	$1.21 - 0.0134 \times \text{time} - 0.126 \times \text{ash} + 0.00126 \text{ time} \times \text{ash}$	70
50 (Pb)	$5.17 - 0.0738 \times \text{time} - 0.300 \times \text{ash} + 0.00329 \text{ time} \times \text{ash}$	73.8
100 (Pb)	$22.1 - 0.115 \times \text{time} - 0.885 \times \text{ash} + 0.00863 \text{ time} \times \text{ash}$	84.6

By analyzing the results based on the experimental data, copper seems to show better removal characteristics than the other metals. However, after carrying out statistical analysis for all metals (Cu, Zn, Pb) and all initial ppm levels (10, 50, 100 ppm), statistically speaking, with 2 parameters and interaction effect only (time, fly ash amount, and their interaction), the best adsorption performance was found to belong to initial 50 ppm zinc solution and the worst one to initial 100 ppm zinc solution. Regression equations and adjusted-R<sup>2</sup> values can be seen in Table 7.

#### 4. Conclusions

It can be concluded based on the chemical analysis that the tested CFA from the Çayırhan Mine shows Class C fly ash properties according to ASTM C 618 (Silicon dioxide ( $\text{SiO}_2$ ) plus aluminum oxide ( $\text{Al}_2\text{O}_3$ ) plus iron oxide ( $\text{Fe}_2\text{O}_3$ ), min, % = 50.0%). Based on the results of the XRD and XRF analyses, more than half of the fly ash composition was occupied by silica, non-opaque spherical particles, and complete opaque spheres were mostly aluminum and iron oxide compounds. The correlation of the SEM and BET results and metal removal experiments for 3 different size classes show that the coarser fraction of fly ash may show higher adsorptive properties than finer fractions due to the larger specific surface area at coarser size fractions. The larger specific surface area at coarser fractions may be explained by the fact that the coarse fractions contain significant amounts of alumina silicate compounds and porous particles contained therein.

Heavy metal removal studies were conducted to investigate the adsorption, precipitation, and desorption capacity of CFA under different conditions and the following conclusions can be drawn from this study.

- Metal adsorption is a very quick process. Although time has positive effects on the removal of heavy metals, a sharp decrease in metal concentrations occurs within the first 7.5 min.
- The removal of heavy metal ions increases with increasing pH and copper is the most sensitive metal for changing pH among the studied metal ions. At higher pH ranges a considerable amount of metal is precipitated in the form of Me-OH and results in reduction of total dissolved metal ions in solution.
- Amount of fly ash has a positive effect on removal of heavy metal ions from synthetic wastewater.
- The results demonstrated that desorption of heavy metal ions from fly ash was nearly negligible at natural pH.

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