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Use of fly ash, phosphogypsum and red mud as a liner material for the disposal of hazardous zinc leach residue waste

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ABSTRACT

Increasing amounts of residues and waste materials coming from industrial activities in different processes have become an increasingly urgent problem for the future. The release of large quantities of heavy metals into the environment has resulted in a number of environmental problems. The present study investigated the safe disposal of the zinc leach residue waste using industrial residues such as fly ash, phosphogypsum and red mud. In the study, leachability of heavy metals from the zinc leach residue has been evaluated by mine water leaching procedure (MWLP) and toxicity characteristic leaching procedure (TCLP). Zinc removal from leachate was studied using fly ash, phosphogypsum and red mud. The adsorption capacities and adsorption efficiencies were determined. The adsorption rate data was analyzed according to the pseudo-second-order kinetic, Elovich kinetic and intra-particle diffusion kinetic models. The pseudo-second-order kinetic was the best fit kinetic model for the experimental data. The results show that addition of fly ash, phosphogypsum and red mud to the zinc leach residue drastically reduces the heavy metal content in the leachate and could be used as liner materials.

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1. Introduction

Mining and industrial activities have caused extensive heavy metal contamination by introducing heavy metals directly into the surrounding environment [1]. In many developed countries regulations require all hazardous waste disposal facilities to be lined with suitable impermeable barriers to protect against contamination [2]. A landfill cover is a multilayer system that serves to reduce the emission of landfill gas into the atmosphere and the infiltration of water into the waste. The only legal requirement is that the amount of water that percolates through the cover must not exceed 5 L (m^2 year)$^{-1}$ for hazardous waste landfills and 50 L (m^2 year)$^{-1}$ for non-hazardous waste landfills, which corresponds to a hydraulic conductivity of about 10^{-10} and 10^{-9} ms^{-1}, respectively. The landfill cover is usually built using a combination of natural mineral materials (e.g., gravel, sand, till, bentonite and clay) and synthetic materials (e.g., geomembranes) [3,4]. Compacted clay liners are widely used as landfill liners to isolate hazardous and other waste materials from surrounding environments, and to prevent the heavy metals commonly found in landfill leachates from migrating into groundwater [1]. However, the application of such materials may become extremely expensive because of the lack of suitable clay materials at the disposal site or because of high costs of synthetic liners. In such instances, fly ash or other industrial waste materials, either alone or in combination with synthetics and other liners, if effective, would have the potential to provide to the industries that have to dispose waste in secure landfills [2].

Fly ash (FA), a solid by-product from coal combustion in electric power plants, is composed of metallic oxides, silicates and other particulate matter [5]. During the past 50 years, the use of coal to the generate electricity has increased substantially, as has the generation of fly ashes. Ash from coal combustion or the incineration wastes is currently, however, more often discarded, for instance in lagoons, settling ponds or landfills. The worldwide production of coal ash for instance is estimated to exceed 550×10^6 tonnes year^{-1}. The disposal of fly ash is becoming more expensive each year due to large land needs for its disposal [6–8]. The best way to solve the disposal problem fly ash is to decrease the quantity for disposal with the utilization of fly ash in the industry. Fly ash has been increasingly utilized in construction application, such as fills, concrete, pavements, wastewater treatment, landfill barrier material, grouts and others [2,9,10].

Phosphogypsum (PG) is a by-product resulting from the phosphoric acid process for manufacturing fertilizers. PG consists primarily of $CaSO_4 \cdot H_2O$ and contains some impurities such as P_2O_5, F^- and organic substances. The quantity of PG produced is very large: for every tonne of phosphoric acid made, about 3 tonnes of phosphogypsum are yielded. It is estimated that more than 22 million tonnes of anhydrous P_2O_5 are produced annually worldwide, generating in excess of 110 million tonnes of gypsum by-product. Approximately 3 million tonnes of PG are generated each year

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in Turkey. This product is discarded in landfills, rivers and ponds [11–14]. Relatively little of this by-product is currently utilized by the cement and gypsum industries as a set retarded for cement and for making gypsum plaster and bricks. PG has also been reused with some success in stabilized road bases, stabilization of base course, wastewater treatment, building constructions and unbound road bases [12,13,15–18].

Red mud is a waste material formed during the production of alumina when the bauxite ore is subjected to caustic leaching. For every tonne of alumina produced, 0.5–2 tonnes of red mud are produced. Red mud, due to its high aluminum, iron and calcium content has been suggested as a cheap adsorbent for the removal of heavy metals, dyes, phosphate, nitrate, fluoride and arsenic [19–23]. The other alternative uses of red mud are the production of bricks and other ceramic products as a partial substitute for clay [24,25].

Zinc is the most important non-ferrous metal after copper and aluminum. In the world, zinc is generally produced from primary sulfide ores or concentrates [26]. The common processes to produce zinc are pyrometallurgical or hydrometallurgical, depending on the type of ore used as a charge. Zinc carbonate has been used as primary source for zinc production at Çinkur plant in Turkey where Waelz processing dilute-H_2SO_4 leaching-electrowinning route was utilized. It has been estimated that more then 100 tonnes of zinc leach residue are generated daily in Çinkur plant. Pyrometallurgical and hydrometallurgical wastes of zinc production industry poses major environmental problems and considered hazardous and toxic due to the presence of heavy metals like Zn, Pb, Cd, Mn and Co. Due to the high metal content and its environmental significance these residues were immobilized with suitable methods such as adsorption, stabilization–solidification and vitrification [27–30]. Heavy metals are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders. Zinc is one of the most important pollutants for surface and ground water. Because of its acute toxicity and non-biodegradability, zinc containing liquid and solid wastes are accepted as hazardous wastes [31–33].

The present study investigated the safe disposal of the zinc leach residue using industrial waste materials. Also, this study aims at investigating the sorption behavior of zinc metal on fly ash, phosphogypsum and red mud. The kinetics of adsorption of zinc on fly ash, phosphogypsum and red mud were studied using different models.

2. Materials and methods

2.1. Materials

2.1.1. Zinc leach residue

The zinc leach residue sample used in this study was obtained from the Çinkur Zinc–Lead Metal Plant of Kayseri, Turkey. The chemical composition of the sample is presented in Table 1. As seen in the table, sample contains 22.49% SiO_2, 22.40% PbO, 13.20 ZnO, 11.03 Fe_2O_3, 6.76% CaO and 6.16% Al_2O_3 as main components. According to XRD analysis, the zinc leach residue includes detectable amounts of anglesite ($PbSO_4$), gypsum ($CaSO_4{\cdot}2H_2O$), and zinc sulfate hydrate ($ZnSO_4{\cdot}2H_2O$). The mineralogical composition of zinc leach residue is found in Table 2.

2.1.2. Fly ash

The fly ash sample used for this study was obtained from Soma Thermal Power Plant in Turkey. The chemical composition of the fly ash was evaluated by using X-ray Fluorescence techniques and the results are presented in Table 1. The total amount of SiO_2, Al_2O_3, Fe_2O_3 and CaO content is about 90%. The mineralogical composition of this material was found by X-ray diffraction method, and the results are given in Table 2.

Table 1
Results of chemical analysis of the zinc leach residue, fly ash, phosphogypsum and red mud.

Oxides	Materials (%) weight			
	Zinc leach residue	Fly ash	Phosphogypsum	Red mud
SiO_2	22.49	55.17	3.87	15.64
Al_2O_3	6.16	24.56	0.20	20.10
Fe_2O_3	11.03	6.80	0.32	36.24
CaO	6.73	2.65	33.5	2.68
MgO	0.48	2.75	0.42	–
Na_2O	–	0.28	0.13	9.99
K_2O	0.82	0.84	–	–
TiO_2	0.27	1.12	–	4.76
P_2O_5	–	–	0.48	–
MnO	0.78	0.42	–	–
ZnO	13.20	–	–	–
PbO	21.40	–	–	–
CuO	0.10	–	–	–
BaO	0.46	–	–	–
Cr_2O_3	0.08	–	–	–
SO_3	15.39	0.89	42.86	0.06
CO_2	–	–	–	2.93
F	–	–	1.12	–
Loss of ignition	0.61	4.52	17.10	8.39

2.1.3. Phosphogypsum

Phosphogypsum was obtained from TÜGSAŞ Fertiliser Plant in Samsun, Turkey. The chemical composition of the phosphogypsum was evaluated by using X-ray Fluorescence techniques and the results are presented in Table 1. The sample was washed with distilled water to remove any non-adhesive impurities and small particles and then dried at 70 °C for 24 h to remove moisture. The mineralogical composition of phosphogypsum is found in Table 2.

2.1.4. Red mud

The red mud used in this study was obtained from Seydişehir Aluminium Plant, Konya, Turkey. The chemical composition of this waste material product is presented in Table 1. The mineralogical composition of red mud is found in Table 2.

2.2. Experimental procedure

2.2.1. Leaching tests

Leaching occurs when a leachant contacts a waste and carries away contaminants from the waste. A leachant can contact the waste either by flowing around the waste or by flowing through the waste or in a combination of both. In an actual landfill situation, the relative importance of these two ways of contact is dependent on the permeability of the solidified waste and its surrounding materials. Several leaching procedures have been developed to simulate the leaching processes of hazardous wastes in landfills or natural environments in order to evaluate the possibility of human health hazard threats of tested wastes [29,34]. For this purpose, batch leaching experiments, MWLP and TCLP test methods were used to evaluate the leaching and pollution potentials of pollutants in the waste.

2.2.1.1. Mine water leaching procedure (MWLP). The aim of this method is to predict the leachability of heavy metals and trace ele-

Table 2
Mineralogical analysis of the materials used.

Materials	Mineralogy
Zinc leach residue	Anglesite, gypsum, zinc sulfate hydrate
Fly ash	Mullite, quartz, hematite, anhydrite, free lime, amorf and glassy phase
Phosphogypsum	Apatite, quartz
Red mud	Hematite, boehmite, quartz

ments from zinc leach residue and other industrial solids when in contact with acidic, metal bearing groundwater, such as acid mine drainage. The method, developed at the West Virginia Water research Institute (WVWRI), involves the serial addition of acid mine water to an ash sample until all alkalinity has been exhausted by the acid water [6]. A 100 g sample placed in a 2 L of acid mine water was added. If mine water was not available, a 0.001 M H_2SO_4 solution was used. The bottle was agitated for 18 h at 30 rpm. At the end of the extraction, the leachates were filtered, and the sample was rinsed back into the container using 2 L of fresh leachant solution. The procedure was repeated until the pH of the leachate reaches that of the unreacted mine water (~3.5).

2.2.1.2. Toxicity characteristic leaching procedure (TCLP). The toxicity characteristic leaching procedure (TCLP), as given in EPA's SW846, is designed to determine the mobility of both organic and inorganic analyses present in liquid, solid, and multiphasic wastes. In order to determine pollution potentials of the zinc leach residue, the toxicity characteristic leaching procedure was applied. The TCLP method first requires the determination of the appropriate extraction fluid to be used (extraction fluid 1 or 2). Acidic wastes are extracted with buffered acetic acid (pH 4.93 ± 0.05). Alkaline wastes, on the other hand, are extracted with unbuffered acetic acid (pH 2.88 ± 0.05) [6,35,36]. For this purpose, 25 g of samples were placed separately in a plastic bottle together with 500 ml of leach solution, sodium acetate/acetic acid buffer solution. The mixtures were then agitated at 30 rpm and 23 °C for 18 h. The zinc metal concentration in the filtrate was determined using AAS (Atomic Absorption Spectrophotometry, UNICAM 929 Model).

2.2.1.3. Batch leaching experiments (BLE). Batch leaching experiments were used to evaluate the leaching and pollution potentials of pollutants in the waste material. Effects of the pH, contact time and liquid/solid ratio on leaching behaviors of the pollutants in the zinc leach residue were investigated in the batch leaching experiments. Batch leaching experiments applied to zinc leach residue were carried out at a constant contact time and liquid/solid (L/S) rate depending on pH [26,30]. At the end of each experiment, the mixtures were filtered and then the final pH of the leachates was measured by a pH meter. The leachates were maintained highly acidic by adding nitric acid (1 ml of HNO_3 solution) to prevent the metal ion precipitation and stored at 4 °C for metal analysis. The experiment was performed in duplicate and mean values were taken into account.

2.3. Preparation of mixtures

All materials were dried in an oven at approximately 103 °C before grinding. Then, mixtures of fly ash (FA), phosphogypsum (PG) and red mud (RM) were blended to prepare composite liner. The mixture design of the composite materials was based on dry weight percentages (10%, 300 g for each three components) of total zinc leach residue (3 kg of zinc leach residue, ZR). These percentages were used for all laboratory tests.

Prototype landfill systems were constructed to determine zinc removal from in landfill. Each system was made using an open-ended plastic tank with approximately 6 L (15 cm × 15 cm × 25 cm). The different waste composite liner materials were settled on floor of the landfill systems. Then 3 kg of zinc leach residue was placed on the liner materials. Leachate samples were prepared by adding distilled water. The leachate derived from the landfill systems was filtered and acidified with concentrated nitric acid to pH < 2. The zinc metal concentration in the filtrate was determined using AAS.

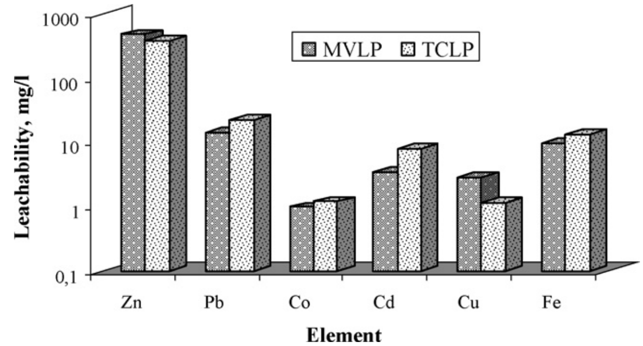

Fig. 1. Leachability of major elements in zinc leach residue.

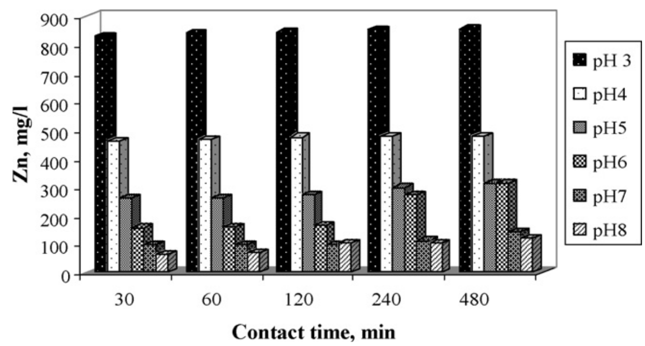

Fig. 2. The changes of Zn concentration released from zinc leach residue depending on pH (L/S:20).

3. Results and discussion

3.1. Leaching behavior of zinc leach residue

The zinc leach residue contains different heavy metals such as Zn, Pb, Co, Cu, Fe and Cr. The MWLP and TCLP test results and allowable maximum concentrations of these heavy metals are presented in Fig. 1. The test results show that most of the heavy metal releases exceed Turkish and EPA standards.

Waste characterization tests are static experiments, performed on short duration, useful for determining the intrinsic properties of the waste with respect to one or several controlled parameters. For example; concentration of the contaminants released can change depending on pH, contact time and amount of rains or water contacted [37–39]. In this study, effects of pH, contact time and liquid/solid ratio on the dissolution of metal constituents in the zinc leach residue were investigated. The obtained results are presented in Figs. 2 and 3. The concentrations of the zinc leachate increased with the increasing contact time and the decreasing pH. The results

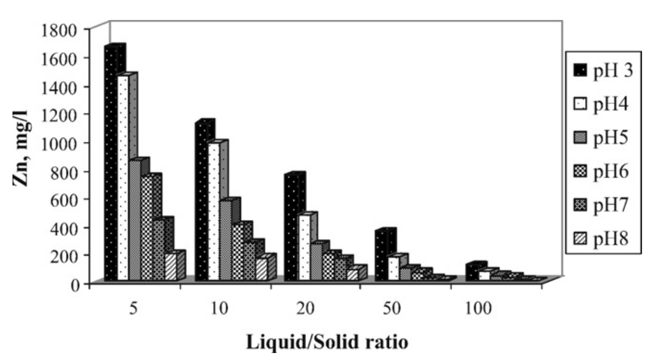

Fig. 3. The changes of Zn concentration released from zinc leach residue with liquid/solid ratio depending on pH (contact time: 60 min).

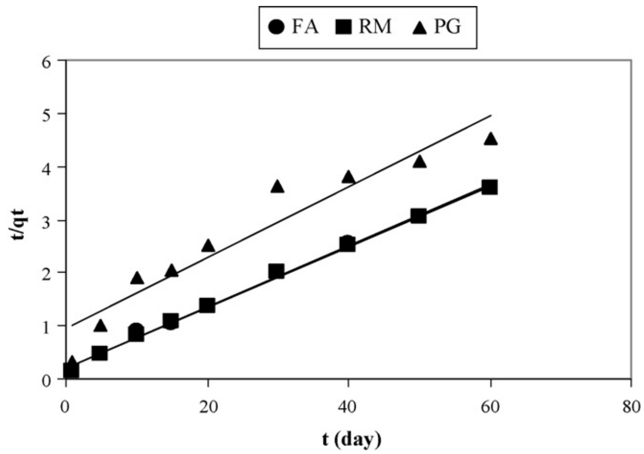

Fig. 4. Linerized pseudo-second-order kinetics plots for the adsorption of Zn^{2+}.

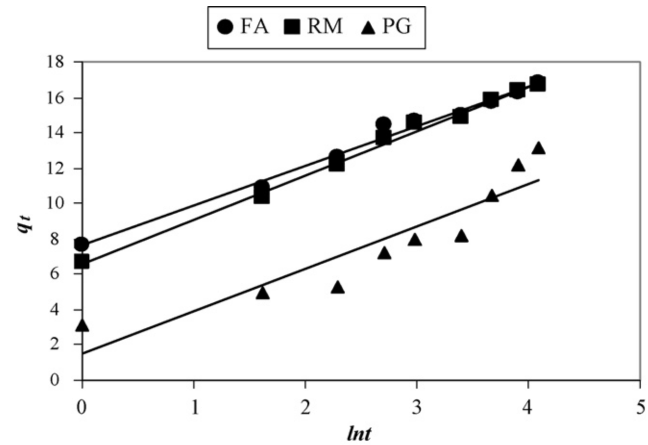

Fig. 5. Linerized Elovich kinetics plots for the adsorption of Zn^{2+}.

show that concentrations of the zinc dissolved from the sample varied with the concentration ranges of 65–856 mg/L.

In order to effect of the liquid/solid ratio on the metal leachability from the waste, the leachates having different initial pH in the range of 3–8 were contacted with the zinc leach residue which has 5–100 range of liquid/solid ratio at temperature 23 °C and contact time of 60 min. Fig. 2 indicates that the metal concentrations released into the solution decreased with increasing liquid/solid ratio.

3.2. Adsorption kinetics

Adsorption kinetic shows a large dependence on the physical and/or chemical characteristics of the adsorption material which also influences the adsorption mechanism. To describe the changes in the sorption studied ions with time, several kinetic models were tested. Three kinetics models; pseudo-second-order, Elovich and intra-particle diffusion models were used to test experimental data examine the adsorption kinetics [40–42].

3.2.1. Pseudo-second-order model

Pseudo-second-order model is given as [43–45]

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \qquad (1)$$

where q_e and q_t are the amount of the heavy metal (mg g^{-1}) adsorbed at equilibrium (mg g^{-1}), k_2 (g mg^{-1} day^{-1}) is the rate constant of the second-order kinetic equations. The pseudo-second-order kinetic for Zn^{2+} removal using fly ash, phosphogypsum and red mud is presented in Fig. 4. The second-order kinetic rate constant k_{id} and correlation coefficients are presented in Table 3. The correlation coefficients for the second-order kinetic model were higher than 0.996 indicating the applicability of this kinetic model of the adsorption process of zinc ions on fly ash and red mud.

3.2.2. Elovich model

The rate parameter for the Elovich equation is determined as [40,42]

$$q_t = \beta \ln(\alpha) + \beta \ln t \qquad (2)$$

where α (mg g^{-1} day^{-1}) and β (g mg^{-1}) are the equilibrium rate constants for Elovich model. The equation constants can be obtained from the slope and intercept of a straight-line plot of q_t against $\ln t$. The values of constants can be obtained from the slope and intercept of the plots (Fig. 5). The values of kinetic constants are presented in Table 3.

3.2.3. Intra-particle diffusion model

The rate parameter for intra-particle diffusion equation is given using the following equation [43,46,47]:

$$q_t = k_{id} t^{1/2} + C \qquad (3)$$

where C is the intercept and k_{id} is the intra-particle diffusion rate constant (mg g^{-1} day$^{-1/2}$). Consistent with Eq. (3), the values of q_t correlated linearly with values of $t^{1/2}$ and the rate constant k_{dt} directly evaluated from slope of the regression line. In this model, due to the porous nature of the adsorbent, pore diffusion is expanded to be surface adsorption. Fig. 6 exhibits the intra-particle diffusion model of zinc adsorption on fly ash, phosphogypsum and red mud. As can be seen from Fig. 6, FA and RM are consistent with intra-particle diffusion but FJ is not. The intra-particle diffusion rate constants k_{id} and correlation coefficients are presented in Table 3.

The parameter values obtained from the application of kinetic models were used to predict the variation of adsorbed Zn^{2+} ion with time. The resulting curves and kinetic parameters are compared to the experimental data in Figs. 4–6 and Table 3, respectively. It can be easily seen from Table 3, relative errors of the pseudo-second-order model are less than in all other models. The correlation coefficients (R^2) of the pseudo-second-order model were high, indicating that the pseudo-second-order kinetic model best describe the adsorption of zinc ion on waste materials such as fly ash, phosphogypsum

Table 3
The kinetic constants for the removal of Zn^{2+} by fly ash, phosphogypsum and red mud.

	Pseudo-second-order			Elovich			Intra-particle diffusion		
	q_e	k_2	R^2	α	β	R^2	k_{id}	α	R^2
FA	17.361	0.016	0.9963	29.370	2.251	0.9872	1.392.10^8	1.2281	0.8860
RM	17.452	0.016	0.9975	13.735	2.506	0.9942	0.133.10^8	1.3756	0.9034
PG	14.836	0.005	0.9134	1.915	2.373	0.8435	21.620	1.4664	0.9712

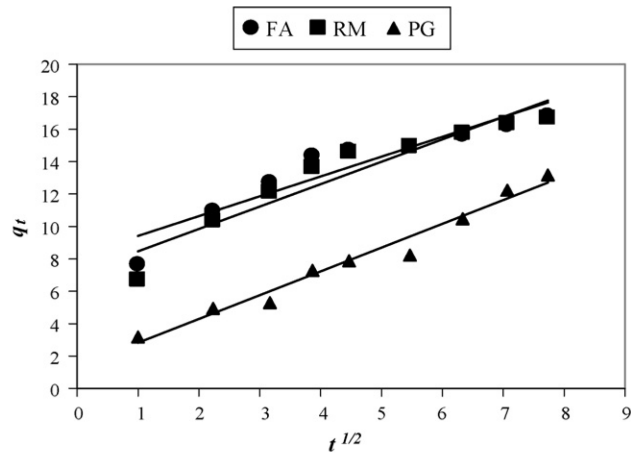

Fig. 6. Linerized intra-particle diffusion kinetics plots for the adsorption of Zn^{2+}.

and red mud. The calculated q_e values agreed very well with the experimental data in the case of pseudo-second-order kinetics.

4. Conclusions

The fly ash, phosphogypsum and red mud waste materials investigated in this study showed a potential to be used in landfill composite liner materials. The following conclusions can be drawn from this study.

- The leaching properties under different conditions and pollution potentially by MWLP and TCLP of the zinc leach residue were investigated. The test results show that zinc, lead and cadmium concentrations dissolved from the zinc leach residue by MWLP and TCLP are higher than the maximum allowable concentrations. MWLP and TCLP results indicate that concentrations of 478 and 376.5 mg/L of zinc leachate were present in the leach residue, respectively.
- The concentrations of the zinc leachate increased with the increasing contact time and the decreasing pH. The results show that concentrations of the zinc dissolved from the sample varied in the concentration ranges of 65–856 mg/L.
- The results demonstrate that addition of fly ash, phosphogypsum and red mud to the zinc leach residue drastically reduces the heavy metal content in the leachate. Fly ash and red mud performs better than phosphogypsum. Fly ash and red mud are effective adsorbents for the removal of zinc ion. The Al, Fe oxides and hydroxides of red mud and fly ash, principally the hematite, were the other active components in heavy metal adsorption. The adsorption of zinc ion on Al, Fe oxides and hydroxides occurred on positively charged surfaces through the formation of specific inner-sphere bonds.
- Three kinetic models were employed in modeling the adsorption mechanism of zinc ion. The kinetic modeling of the zinc adsorption on the waste adsorbents well followed the pseudo-second-order model with the correlation coefficients higher than 0.99 for fly ash and red mud.

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