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Research Article

CHARACTERIZATION AND EVALUTION OF REMOVAL CONDITIONS OF LEAD-ZINC-COPPER FLOTATION PLANT WASTE

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Keywords
Characterization, Iron, Inorganic acids, Mining Waste, Sulfur.

Abstract
A large amount of mining waste occures due to the intensive mining activities in our country. These wastes may contain different pollutants depending on the type of mining facilities. The lead-zinc-copper flotation waste was investigated in this study contains significantly calcium, iron, magnesium and sulfur elements. The presence of high concentrations of these elements has an environmental problem. For this reason, in this article, firstly, the mine waste was obtained from the Gümüştaş Mining Corporation, the Lead-Zinc-Copper Enhancement Plant. Then, characterization and iron and sulphate removal experiments were investigated under different inorganic acids. Experimental studies showed that about 30% iron and 85% sulphate removal yields were obtained with 4 M HCl concentration.

KURŞUN-ÇİNKO-BAKIR İYİLEŞTİRME TESİSİ ATIĞININ KARAKTERİZASYON VE GİDERİM ŞARTLARININ DEĞERLENDİRİLMESİ

Anahtar Kelimeler
Demir, İnorganik asitler, Maden atığı, Karakterizasyon, Sülfür.

Öz
Ülkemizde yoğun madencilik çalışmaları neticesinde, fazla miktarda madencilik atığı ortaya çıkmaktadır. Bu atıklar, işletilen maden işletmesinin türüne göre farklı kirleticiler içerebilmektedir. Bu çalışma kapsamında araştırılmış olan kurşun-çinko-bakır flotasyon atığı önemli ölçüde kalsiyum, demir, magnezyum ve sülfür elementlerini içermektedir. Bu elementlerin yüksek konsantrasyonda bulunması çevresel açıdan tehdit oluşturmaktadır. Bu sebeple, bu makalede, Gümüştaş Madencilik İşletmesi, Kurşun-Çinko-Bakır İyileştirme Tesisi'nden temin edilen maden atığının karakterizasyonu neticesinde farklı inorganik asitlerle demir ve sülfat elementlerinin giderimleri değerlendirilmiştir. Deneysel çalışmalar neticesinde 4 M HCl konsantrasyonunda yaklaşık olarak %30 demir, %85 sülfat giderimi elde edilmiştir.

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1. Introduction

Tailings are defined as the processing wastes from a mill, washery or concentrate that removed the economic metals, minerals, mineral fuels or coal from the mined resource. Tailings are generally deposited in the tailing dam or pond which has been constructed using mining waste or other earth material available on or near the mine site (Kachhap, 2009). Although the mining industry has played an important role in economic development over the world, mining activities remain one of the biggest contributors to environment pollution worldwide (Fosso-Kankeu et al., 2015). The forests, land and waters are polluted as a result of mining activities wastes and waste mine drainages (Ok et al., 2011; Park et al., 2015). Mining

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industry wastes can lead to immense environmental damage if not stabilized and sufficiently remediated (Fosso-Kankeu et al., 2015; Forjan et al., 2014). These wastes need to be recovered effectively due to the metal/heavy metal or hazardous metals contents. Different metal pollutants originated from different mining facilities causes extensive contamination of shallow groundwater systems and soil water zones. For example, coal mine drainage contains elevated concentration of sulphates, iron, manganese, aluminum, other toxic, radioactive ions and total dissolved solids (Komnitsas et al., 2001). Also, heavy metal ions such as cobalt, copper, nickel, chromium and zinc were determined in the waste streams from mining operations, tanneries, electronics, electroplating and petrochemical industries and textile mill product (Chuah et al., 2005; Kazemipour et al., 2008). Cd is a non-essential and non-beneficial and found as highly toxic to plants and animals (Kefala et al., 1999). Pb also has environmental threats even at extremely low concentrations and cause brain damage for children (Ahmedna et al., 2004; Kazemipour et al., 2008). When the ferric iron discharged to surface water, it hydrolyzes to produce hydrated iron oxides and additional acidity and it contributes making the corrosive and unable to support various forms of aquatic life. Also sulfide minerals such as pyrite (FeS_2), arsenopyrite (FeAsS), chalcopyrite ($CuFeS_2$) can produce acidic solutions upon oxidation (Komnitsas et al., 2001).

In Turkey, the discharge standards of wastewater originating from the mining industry were published in the Official Gazette in 2004 (Official Gazette, 2004). According to this regulation, necessary standard concentrations to be provided for the mining wastewater are given for chemical oxygen demand (COD), suspended solid matter (SSM), Pb, total cyanide, Fe, Zn, S, Hg, Cd, Cu and Cr elements. However, for waste sludges that accepted to the I., II., III. class landfill site the limit concentrations for antimony, arsenic, barium, cadmium, chromium, lead, mercury, molybdenum, nickel, selenium, sulphate and zinc are given in the Regulation on the Landfill of Mining Wastes (Official Gazette, 2010b). The mining activity companies must meet the standards that cause no adverse effects on humans or receiving environment before giving the waste sludge to facilities. Generally, the remediation of the mine wastes requires pH adjustment, physical oxidation or reduction, neutralization and heavy metal removal. The traditional methods of removing heavy metals include precipitation and co-precipitation of dissolved metals, foam flotation, flocculation, sedimentation, ultra-filtration, evaporation, bio-sedimentation, adsorption and ion exchange (Ghirişan et al., 2007).

One of the ways to effectively mobilize specific metals from mine tailings can be listed as hydrometallurgical processes, lime-based chemical precipitation and ion exchange. The main hydrometallurgical processes focus on the leaching. Leaching occurs when an aqueous solution containing a waste and lixiviates (acids or bases) flows through the wastes, dissolving solid minerals from their host rocks. The researchers must choose ideal lixiviates according to their economical, globally applicable and easily recyclable features. However, the treatment processes of mining wastes or acid mine wastewater vary according to the mining sectors, the concentration of dissolved metal ions present in the wastewater (Jafaripour et al., 2015).

Gaber et al. (2011) found that citric acid is a promising extractant for the removal of heavy metals from sewage sludge. Their study showed that acid leaching at pH 2.43 with citric acid seemed to be highly effective in extracting Cu, Zn, mostly after 1 day of extraction time, for Cr, Ni for 5 days leaching time, for Pb removal for 10 days, respectively. Yoshizaki and Tomida (2000) found that phosphoric acid of 8% concentration with hydrogen peroxide showed good removal rates of heavy metals comparable by 1 N hydrochloric acid from sewage sludge. They found that copper was easily removed from the sludge in the presence of hydrogen peroxide. Removal of zinc, lead, cadmium with hydrochloric acid was found around 50%. In the case of using hydrochloric acid containing hydrogen peroxide, over 80% of zinc, lead, etc. was removed from the sludge (Yoshizaki and Tomida, 2000).

Nariyan et al. (2017) obtained the mine water from Pyhäsalmi mine and they used the electrocoagulation treatment for the removal of copper, silicon, manganese, aluminum, iron, zinc and sulfate. They used the aluminum and iron electrodes in a batch system. They searched the effect of current density, time and various electrodes on pollutants removals. Their study showed that about 100% Al, 100% Cu, 90% Si, 100% Zn, 30% Mn and 30% SO_4^{2-} were obtained with iron electrode at 70 mA/cm^2 current density for 120 min reaction time and about 80% Fe, 100% Cu, 100% Si, 40% Zn, 20% Mn and 40% SO_4^{2-} with aluminum electrode at the same experimental conditions.

Nariyan et al. (2018) also studied of precipitation with lime plus electrocoagulation for sulfate removal from Pyhäsalmi mine water. Sulfate concentration was decreased from 13,000 mg/L to 1600 mg/L with calcium oxide (lime). Besides, sulfate reduction was achieved from 13,000 mg/L to 250 mg/L with electrocoagulation and calcium oxide pre-treatment.

Kaur et al. (2018) studied with both Bayer precipitates and thermally activated Bayer precipitates to remove Al, Cu, Fe, Zn and Ni in AMD samples and achieved satisfactorily results for both Bayer precipitates. They obtained manganese proved to be a challenge as removal appeared to be inhibited regardless of the alkaline material employed. For all tested samples, they found that thermally activated Bayer precipitates were found to be more efficient overall than Bayer precipitates.

Summary of some studies about recovery of metals from mining wastes are given in Table 1.

Table 1. Literature reviews about recovery of metals from mine tailings

Researchers	Mine type	Methods and Exp. Conditions	Recovery rate (%)
Fosso-Kankeu et al., 2015	Gold mine tailings	(Acidic) 0.05g/ml S/L 200 rpm, 55 °C 240 min 1 M H_2SO_4	100% Zn 10%Fe 20%Ni
Falagan et al., 2017	Copper mines	(Biological) 45 °C pH 1.8-1.0 Aerobic/anaerobic Acidithiobacilus caldus Sulfobacillus thermosulfido oxidans	100% Zn 100% Cu
Rosa et al., 2017	Tungsten mine tailings	(Acidic) L/S = 10 current intensity:800 mA 360 min 0.8 mol/L citric acid	68%As 24%Fe 65%Mn
Ye et al., 2017	Lead zinc mine tailings	(bioleaching) 10% pulp density 50 days	0.82% Pb 97% Zn 71% Fe
Liu et al., 2007	Lead and zinc mine	(bioleaching) 1%(w/v) solid conc. 13 days	98% Zn 96% Cu 43% Pb
Lambert et al., 2014	Copper mining residues	(electrochemical) current intensity: 2.0 A 100 min	97% Cu
Luo et al., 2017	Alunite tailings	(basic) <90 °C 60 min 300-1100 rpm >13.5 mol/L KOH	~100% alunite

Electrochemical methods can be evaluated technologically in the recovery of metals from mining waste. In this regard, Wang et al. (2003) studied arsenic removal by re-precipitation with iron hydroxide by electrochemical pH adjustment. Another researchers investigated the heavy metal removals (Cu, Zn, Ni, Mn) by monopolar iron electrodes and electrocoagulation method. However, the selective metal removal was not considered in these methods (Al Aji et al., 2012). Sarı (2005) examined the removal of Zn, Cd, Pb, Cr, Ni, Cu by bioleaching method in a waste slurry obtained from a metal industry which is coated with copper, nickel and chrome by electrolysis method. He found that the bioleaching methods may require longer periods, but also it can be used as an effective method because it has a higher metal dissolution rate and is a more environmentally friendly and economical process.

In this study, the waste sludge characterization and removal efficiencies of iron and sulphate elements were evaluated. The waste sludge was obtained from Gümüştaş Mining Corporation Lead-Zinc-Copper Flotation (Enrichment) Plant.

2. Materials and Methods

2.1. Waste Sludge Characterization and Analytical Methods

A waste sludge sample was obtained from Gümüştaş Mining Corporation Lead-Zinc-Copper Flotation (Enrichment) Plant, in Gümüşhane Province, Turkey. The sample was dried at 60 ° C for 24 hours (FN 500, Nuve). All analyzes were done with waste dry sample. The metal contents of the sample were analyzed with S-METAXHB1 and METAXHB2 methods with ICP-OES (Agilent 5100 SVDV, USA) device in the ALS laboratority in Czech Republic. XRD (Bruker D8 Advance Twin-Twin) analysis of the sample was analyzed using CuKα x-rays at the YETEM Laboratory in Suleyman Demirel University, (SDU).The total solids (2540B method) and total volatile solids (2540G method) tests of the sample were carried out according to the Standard Method in the Water and Wastewater Laboratory of the SDU Environmental Engineering Department (APHA, 2005). The pH value of the dry sample was measured by the pH meter (Hanna HI 221) after dilution of the sample with purified water by 1/5 taking reference of Kilic et al. 2011. The sulfate concentration (Sulfaver 4 Method, Method: 8051), iron concentration (Ferrover Method, Method: 8008) were determined with Hach DR 5000 spectrophotometer.

2.2. Removal Experiments

Removal experiments were conducted with hydrochloric acid (HCl), nitric acid (HNO_3) and phosphoric acid (H_3PO_4). 0.2 g/L dried waste sludge and 50 mL, 1 M acid solution was added to mixed batch reactor. Then the samples were mixed (150 rpm) in a temperature-controlled orbital shaker (Gallenkamp) at 20 °C. Time intervals were selected as 0, 10, 20, 30, 60, 90, 120, 180, 240 min. After removal experiments, the samples were centrifuged at 6,000 rpm for 10 min (Shimadzu Centrifuge, Japan) to precipitate the waste sludge. The supernatant was diluted by 1:10 ratio using nitric acid solution (pH~1.5-2) to avoid the precipitation of metals and then samples were kept in refrigerator at 4 °C until analysis.

3. Results and discussions

3.1 Waste Sludge Characterization

The solids content of the waste sludge sample is given in Table 2. According to the results given in Table 2, mining waste sludge has been found to have a total solids content of 178 g/L.

Table 2. Solid contents of mining waste sludge

Parameters	Concentrations (g/L)
TSM	178.86±19.05
TVS	174.50±13.37

XRD result of the mining waste sludge is given in Fig. 1.

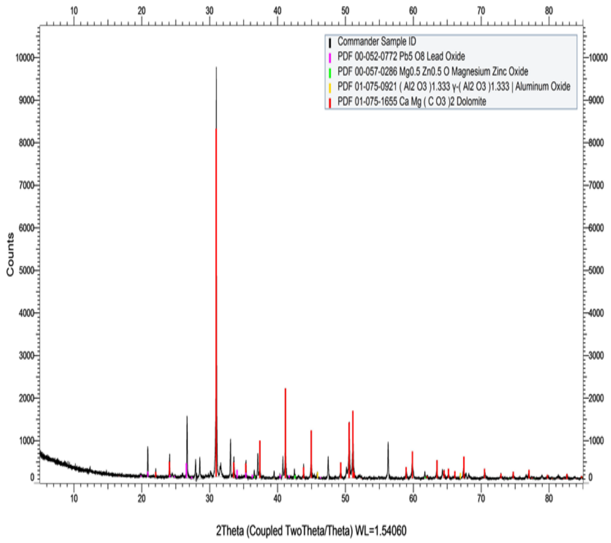


Figure 1. XRD analysis results of mining waste sludge

According to the XRD results shown in Fig. 1, it has been found that the waste sludge to be characterized contains approximately 85% of dolomite ($CaMg(CO_3)_2$), 9% of lead oxide, 3% of aluminum oxide and 2% of magnesium zinc oxide. These results are consistent with the high concentration of elements detected by the ICP device.

The elemental analysis results of mining waste sludge are given in Table 3.

Table 3. Metal contents of mining waste sludge

Elements	Conc. (mg/kg dry matter)	Elements	Conc. (mg/kg dry matter)
Aluminum	1,040	Manganese	20,600
Antimony	44.2	Mercury	<1
Arsenic	754	Molybdenum	3.54
Barium	39.3	Nickel	14.7
Bismuth	<5	Phosphorus	54.4
Boron	<5	Potassium	271
Cadmium	18	Selenium	<10
Calcium	170,000	Silicon	<250
Chromium	7.96	Silver	20.2
Cobalt	3.97	Sodium	261
Copper	621	Strontium	55.3
Iron	86,300	Sulfur	95,200
Lead	3,560	Thellerium	<5
Lithium	<5	Thallium	<2.5
Magnesium	91,500	Tin	<5
Titanium	20.7	Vanadium	13.6
Zinc	4,260	Zirconium	<25

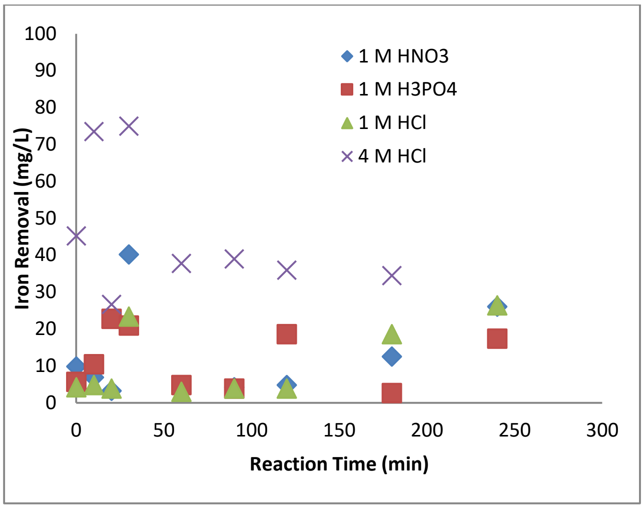
According to the results obtained in Table 3, the concentrations of calcium, iron, lead, magnesium, manganese, sulfur and zinc were found to be high. According to the analysis results, 170,000 mg/kg calcium, 86,300 mg/kg iron, 3,560 mg/kg lead, 91,500 mg/kg magnesium, 20,600 mg/kg manganese, 95,200 mg/kg sulfur and 4,260 mg/kg zinc concentration were found. Our characterization results were consistent with other studies about lead-zinc mine tailings (Liu et al., 2007; Ye et al., 2017).

3.2. Removal experiments

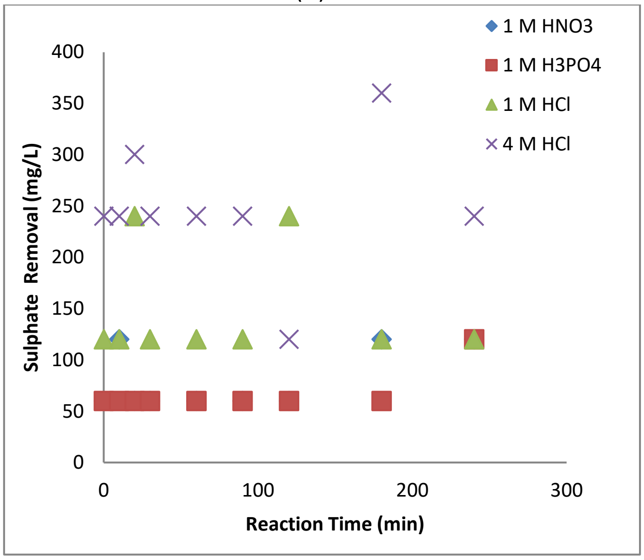
Removal experiments were conducted with inorganic acids such as phosphoric acid (H_3PO_4), hydrochloric acid (HCl) and sulphuric acid (H_2SO_4). Figure 2 shows the removal of iron (A) and sulphate (B) concentration with different kinds of acids.

Fig. 2A shows that effect of acid type wasn't changed the removal of iron. Tested all type of acids such as H_3PO_4, HCl and H_2SO_4 showed similar effects and iron removal efficiencies were obtained as 1-10% with 1 M concentrations, after 300 min reaction time. While the acid concentration is increased to 4 M, iron removal yield was increased to only %30. This results were similar with other studies (Meer and Nazir, 2017; Sayilgan and Kurklu, 2018).

Fig. 2B shows that the sulphate removal efficiencies with tested inorganic acids. About 50-400 mg/L sulphate concentrations were obtained in acidic solutions. These concentrations correspond to 20-85% sulphate removal. Sulphate removals were also increased with higher acid concentrations. High concentrations of sulphate and calcium contents may interference the measurement of some elements and removal efficiencies. Due to these reasons, it is better to wash mining waste sludge with distilled water before the removal experiments.



(A)



(B)

Figure 2. Iron removal (A) and sulphate removal (B) concentrations of mining waste

3.3. Assessment of waste in terms of regulation

Table 4 shows the limit values of regulation on the use of Soil for Domestic and Urban Treatment Sludge and tested waste sludge characterization.

Table 4. Metal contents of mining waste sludge and regulation limit values

Heavy metal (Total)	Limit values (mg kg^{-1} dry matter) Regulation on the Use of Soil for Domestic and Urban Treatment Sludge (27661)	Mining waste sludge metal contents (mg kg^{-1} dry matter)
Lead	750	3,560
Cadmium	10	18
Chromium	1,000	7.96
Cupper	1,000	621
Nickel	300	14.7
Zinc	2,500	4,260
Mercury	10	<1

Table 4 shows that considering the maximum allowable heavy metal contents in the stabilized treatment sludge that can be used in the soil given in Annex-1B within the scope of the Regulation on the Use of Soil for Domestic and Urban Treatment Sludge, published in the Official Gazette dated August 3, 2010, numbered 27661, the concentration of lead, cadmium and zinc elements in the tested waste sludge was found above the limit concentrations (Official Gazette, 2010a). According to these results, firstly lead, cadmium and zinc elements have to be removed from waste sludge. Then, considering the same regulation, after removal metals, the waste sludges have to be stabilized and could be used in soil.

It is aimed to carry out studies about the recovery of elements over the limit values stated in the regulation after the characterization stage and detected elements in high concentration even though they are not already included in the regulation, considering the possibility that this waste is completely mixed with water. The limit values in the relevant regulations must be met in order for the sludge from the mining activities to be directly deposited into the landfill. However, it may be regarded as a stabilizing sludge which can be used in the soil after reduced below the limits specified in the relevant regulations for hazardous and/or metal/heavy metals from mining waste sludge. Lead, cadmium and zinc concentrations were found to be higher than the limit values within the scope of the related regulations in Turkey.

4. Conclusions

The mining waste sludge's metal/heavy metal contents have to be reduced the limit values in the relevant regulations. After the removal and stabilization steps these wastes could be directly deposited into the landfill. In this study, the waste sludge that has been characterized does not meet the limit values for elements such as lead, cadmium and zinc within the scope of the relevant regulations. However, there are other pollutants with high concentrations such as calcium, iron, magnesium and sulphate even if not in the regulation. For this reason, the removal of sulphate and iron elements was evaluated in this study. About 30% iron and 85% sulphate removal were obtained with tested inorganic acids. As a result, our research on techno-economic solutions in which all elements of high concentration can be appropriately removed are in progress.

Conflict of Interest

No conflict of interest was declared by the authors.

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