

Utilization of Demolition Waste for Treatment of Acid Mine Drainage and Immobilization of Heavy Metals Released from Copper Flotation Tailings

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Keywords Copper flotation tailings, Demolition waste, Acid mine drainage, Urban renewal	Abstract: In this study, the treatment of acid mine drainage (AMD) and immobilization of heavy metals released from CFT (copper flotation tailings) were investigated using strongly alkaline demolition waste (DW), collected from the urban renewal areas. Shake flask batch tests were conducted to assess the influence of time and different ratios of CFT/DW on the acidity and heavy metal release characteristics of the drainage water. During the tests, samples were collected from filtered leachates at regular intervals to monitor pH, SO4 ²⁻ and release of heavy metals. The results indicated that the pH increased from 2.21 (t=0) to 10.37 after 120 minutes of shaking in an orbital shaker. The highest SO4 ²⁻ release ([SO4 ²⁻]=4558 mg L ⁻¹ , t=0) was measured at 1:3 dose of CFT:DW application with 257 mg L ⁻¹ (pH=10.37) at the end of 120 minutes. The addition of DW almost completely reduced the release of heavy metals from CFT due to its high alkaline content. As a result, it was suggested that DW could effectively be used for the treatment of AMD and
	result, it was suggested that DW could effectively be used for the treatment of AMD and immobilization of heavy metals released from CFT.

Bakır Flotasyon Atıklarından Ağır Metal Salınımının Önlenmesi ve Asit Maden Drenajının İyileştirilmesi için Yıkıntı Atıklarının Kullanımı

Anahtar Kelimeler Bakır flotasyon atıkları, Yıkıntı atıkları, Asit maden drenajı, Kentsel dönüşüm Öz: Bu çalışmada, kentsel dönüşüm alanlarından toplanan güçlü alkali özelliğe sahip yıkıntı atıkları (DW) kullanılarak asit maden drenajının (AMD) iyileştirilmesi ve bakır flotasyon atıklarından (CFT) salınan ağır metallerin önlenmesi araştırılmıştır. Drenaj suyunun asitliği ve ağır metal salınımı üzerinde zamanın ve farklı CFT/DW oranlarının etkisini değerlendirmek için çalkalama testleri yapılmıştır. Testler sırasında pH, SO₄²⁻ ve ağır metal salınımını izlemek için filtrelenmiş sızıntı sularından düzenli aralıklarla numuneler toplanmıştır. Sonuçlar, çalkalayıcıda 120 dakika çalkalandıktan sonra pH'ın 2.21'den (t=0) 10.37'ye yükseldiğini göstermiştir. En yüksek SO₄²⁻ salınımı ([SO₄²⁻]=4558 mg L⁻¹, t=0) 120 dakika sonunda 1:3 doz CFT:DW uygulamasında 257 mg L⁻¹ (pH=10.37) olarak ölçülmüştür. DW ilavesi yüksek alkali içeriği nedeniyle CFT'den ağır metal salınımın neredeyse tamamen azaltmıştır. Sonuç olarak, DW'nin AMD iyileştirmesinde ve CFT'den salınan ağır metallerin önlenmesinde etkin bir şekilde kullanılabileceği önerilmiştir.

1. INTRODUCTION

Acid mine drainage (AMD) is an environmental concern due to its adverse effects on the surrounding ecosystem. When sulfide minerals such as pyrite are exposed to oxidation in a humid environment, they can release protons (H^+ ions) to the aqueous environment (1). Thus, the water acquires an acidic character with a concomitant decrease in pH and increased release of most heavy metals [1-3]:

 $4\text{FeS}_{2}+15\text{O}_{2}+14\text{H}_{2}\text{O} \rightarrow 4\text{Fe}(\text{OH})_{3\downarrow}+8\text{SO}_{4}^{2-}+16\text{H}^{+}$ (1)

The resulting acidic waters with high metal content are defined as AMD. Once AMD starts, it is very hard to stop and very expensive to control. Therefore, special precautions must be taken to prevent AMD formation [4,5]. Prevention techniques should be designed at the source to reduce or eliminate the drainage of the acidic water. Prevention at the source, i.e., prevention at the acid formation stage, can be possible by increasing the neutralization capacity of the minerals, such as CFT, by applying alkaline material having neutralization potential, such as DW [6,7].

The control and management of solid wastes are one of the biggest problems. Today, although the rates vary depending on the country and the city, construction and demolition wastes have a large share in solid waste [8,9]. More than 90% of waste is generated by demolition, while new construction accounts for less than 10%. In 2015, waste from construction sites accounted for 23.1 million tons and waste from demolition sites accounted for 358.7 million tons globally [10]. The transportation and storage costs of DW are quite high. In addition, very large natural areas are occupied for the storage of these wastes, which may cause the degradation of soil in these areas with the loss of its fertility and hence, agricultural land [11,12]. Therefore, the utilization of DW is of great environmental and economic importance.

In Turkey, the main problem in the urban renewal process is the limited availability of storage facilities where the waste will be safely disposed of after demolition to control its harmful effects on the environment. It is necessary to investigate the potential use of DW as raw material in industrial applications, which will contribute to environmental sustainability and the protection of natural resources. When alkaline materials, such as DW, are added to an acidic environment, they not only increase the pH of the environment but also allow heavy metals in the solution to precipitate and thus, largely eliminate AMDdriven problems leading to severe environmental problems [1,13,14]. Post-mining activities are among the most important conditions for sustainable development, and developed countries successfully perform these activities. It is possible to prevent surface and underground water pollution by eliminating the potential of mining wastes to produce acid by using alkaline materials such as DW. This study investigated the effectiveness of DW utilization for AMD treatment and the precipitation of heavy metals released from CFT.

2. MATERIAL AND METHOD

2.1. Materials

CFT, a waste product of the flotation process used to enrich copper sulfide ores, was obtained from Murgul Copper Mine in Artvin, Turkey. DW was collected from the buildings in Yakutiye district in Erzurum (Turkey), demolished due to the urban renewal within the scope of the Cultural Road Project, which is being carried out with the support of Erzurum Metropolitan Municipality. CFT particles were smaller than 75 μ m in diameter, while DW sample was ground down to 75 μ m. Pure CaCO₃ powder (pH=9; CaCO₃=100%wt) was also used as a reference material (Merck-102066) to compare the effectiveness of DW on the CFT neutralization process.

2.2. Experimental Procedure

Static and kinetic tests were carried out to determine the acid-producing potential (APP) of CFT and the neutralization potential of DW. The acid-base accounting method [15] was used in the static tests to determine APP based on the total sulfide content of CFT (APP = 31.25 x 42(%S)) [16]. DW was utilized as neutralization material to treat CFT at a dose of 1.31 units per unit CFT. This dose was determined using the following stoichiometry of FeS₂ and CaCO₃ (2), which predicts the amount of lime required to neutralize all of the potential acids based on the percentage of sulfides present in CFT. FeS₂+2CaCO₃+3.75O₂+1.5H₂O \rightarrow Fe(OH)₃+2SO₄²+2Ca² +2CO₂ (2)

Kinetic tests are performed to evaluate the acid generation potential and leachate quality of mine waste and process tailings [16]. Shake flask batch tests, one of the kinetic test methods, were conducted to evaluate the effect of mixing ratio of CFT to DW and shaking time on the evolution of pH, the release of SO_4^{2-} and heavy metals into the leachate.

After the required amounts of CFT and DW samples, and distilled water were placed in a flask at the tested dosage of DW, the flask was placed onto an orbital shaker (WiseCube, WIS-20R). Test conditions for shake flask batch tests are given in Table 1. The solid-liquid ratio was set as 2 L kg⁻¹ [16]. Using pure CaCO₃ instead of DW, a control test without a neutralizing additive, e.g., DW or CaCO₃, was also conducted to compare the effect of DW on the CFT neutralization process. Under normal conditions, while it is required to use 1312.5 kg CaCO₃ to neutralize 1 ton of waste pyrite containing 42% S, 66 g CaCO₃ is required to neutralize 50 g waste pyrite. However, since DW contains CaCO₃ by 30.44%, 217 g DW was used instead of 66 g DW to neutralize 50 g CFT at 1:1 dose of CFT:DW application. Based on the purity of DW, it is assumed that DW cannot completely react with CFT and therefore, different doses of DW were also used in the shake flask batch tests (Table 1).

Table 1. Test conditions for shake flask batch tests

	CFT	DW	Distilled water
Series	(g)		(mL)
CFT (control)	50	-	100
CFT+pure CaCO ₃	50	66	232
CFT+DW (1:1)	50	217	534
CFT+DW (1:0.5)	50	109	318
CFT+DW (1:1.5)	50	326	752
CFT+DW (1:2)	50	434	968
CFT+DW (1:2.5)	50	543	1186
CFT+DW (1:3)	50	651	1042

Samples prepared were shaken in flasks under continuous stirring at 180 rpm $(23 \pm 2 \text{ °C})$ for a total of 120 minutes. During the leaching period, pH measurements were

performed every 15 minutes. Samples were taken with a syringe every 60 minutes, passed through filter paper. Filtered samples were stored at 4°C for SO₄²⁻ and heavy metals analyses.

2.3. Analytical Procedure

The mineralogical compositions of CFT and DW samples were determined by X-Ray diffraction (XRD) (RIGAKU, D/Max-2000). SO₄²⁻ concentration was analyzed as specified in method 375.4 of EPA [17]. The pH was measured using an Orion 420A + pH/mV/ORP device. The CaCO₃ content was determined using a calcimeter [18]. The total sulfide content of CFT was evaluated by using the methods given in the Turkish Standards Institution [19]. Heavy metal concentration was determined using inductively coupled plasma mass spectrometry (ICP-OES; PerkinElmer Sciex Elan). The scanning electron microscopy (SEM, FEI Quanta 400 MK2) coupled with Energy Dispersive X-ray Spectrometry (EDS, EDAX Genesis XM4 Imaging detector) was used for morphological analysis and identification and quantification of elements after 1:3 dose (max) of CFT:DW application.

3. RESULTS AND DISCUSSION

3.1. Characteristics of CFT and DW

The pH value and total sulfide content of CFT obtained from Murgul Copper Mine were measured as 2.21 and 42% respectively. The CaCO₃ content of DW obtained from urban renewal areas in Erzurum was determined as 30.44% with a pH of 11.07.



Figure 1. The XRD pattern of a) CFT and b) DW

The XRD analysis results indicate that the major minerals in the CFT sample are pyrite (FeS₂) and quartz (SiO₂); and the major minerals in DW are dolomite (CaMg(CO₃)₂), anorthite (CaAl₂Si₂O₈) and quartz (SiO₂), respectively (Figure 1).

3.2. Experimental Results

To determine the neutralization potential of DW applied on CFT at different doses, the changes of the leachate pH values over time were investigated (Figure 2). At the end of 120 minutes of shaking, pH remained stable around 2.21 in the control group. Significant increases were observed in the pH values of CFT leachate treated with pure CaCO₃ and different doses of DW at the first 15 minutes. No significant changes were observed in the following periods. In the pure CaCO₃ applied sample, the pH value of the leachate, which was 2.21 (t=0), increased to 6.60 after 15 minutes of shaking. The greatest pH change occurred at 1:3 dose of CFT:DW application. It was noted that the pH value increased from 2.21 to 10.09 after 15 minutes and 10.37 after 120 minutes of shaking. Johnson and Halberg [20] and Zinc and Griffith [21] stated in their studies that acidity was neutralized by increasing the pH of the environment with alkaline materials like lime or limestone.



Figure 2. The effect of time on the leachate pH values at different application doses of DW on CFT

After separating the filtered leachate, the solids remaining on the filter paper were dried and examined under SEM-EDS. SEM images and the EDS pattern of the leaching residue obtained after 1:3 dose (max) of CFT:DW application show that the main elements are Si, Ca, Al and Mg due to neutralization of CFT with DW applications (Figure 3).

No significant changes in SO42- concentration of the leachates were observed in the control group depending on the pH value, while serious decreases were observed in the SO₄²⁻ value in pure CaCO₃ and DW applications after 60 minutes, and not much change was observed at the end of 120 minutes. The SO42- value was measured as 982 mg L⁻¹ (pH=7.03) and 970 mg L⁻¹ (pH=7.23) with pure CaCO₃ application at the end of the 60 and 120 minutes, respectively. In DW application, the lowest SO₄²⁻ value was observed at the 1:3 dose of CFT:DW. At this dose application, the SO42- value, which was 4558 mg L-1 at the beginning (t=0, pH=2.21), was observed as 274 mg L⁻¹ (pH=10.14) and 257 mg L⁻¹ (pH=10.37) after 60 minutes and 120 minutes, respectively (Table 2). DW application affected the pH value and SO42- concentration in the leachate. As the application doses of DW increased, the pH of the leachate increased and SO42- concentration decreased due to the neutralization capacity of DW. Since SO_4^{2-} ion precipitated in the form of CaSO₄ and its concentration decreased in the environment. Madzivire et al. [22] and Name and Sheridan [23] stated in their studies

that the SO_4^{2-} concentration decreased depending on the increase in pH.





Figure 3. SEM images and EDS pattern of leaching residue obtained after 1:3 dose (max) of CFT:DW application

Ct.a	Time	11	SO4 ²⁻
Series	(min)	рп	(mg L ⁻¹)
Start	0	2.21	4558
CET (control)	60	2.25	4417
CI-I (control)	120	2.06	4550
	60	7.03	982
CF1+Pule CaCO ₃	120	7.23	970
CET DW(1.1)	60	9.31	852
CF1+DW (1:1)	120	9.56	841
$CET \mid DW(1,0.5)$	60	8.66	757
CF1+DW(1:0.3)	120	8.91	748
$CET \mid DW(1, 1, 5)$	60	9.65	665
CI-1+D w (1.1.3)	120	9.86	652
CET + DW (1:2)	60	9.81	443
$CI^{+}I^{+}DW(1.2)$	120	10.07	426
$CET \mid DW(1,2.5)$	60	9.97	378
CF1+DW(1:2.5)	120	10.24	365
$CET \mid DW(1,2)$	60	10.14	274
$CI^{-1}+DW(1.5)$	120	10.37	257

Table 2. SO4²⁻ concentration in the leachates

Depending on their neutralization capacity, the effects of different application doses of DW on heavy metal release from CFT were investigated. Concentrations of the heavy metals in the leachate at the end of 60 and 120 minutes during the shake flask batch tests and standard limits allowed by WHO [24] are given in Table 3. No significant changes in Cd, Cr, Cu, Fe and Zn concentrations were observed in the control group in the leachate after 120 minutes of shaking with initial values of 3.45 mg L⁻¹, 2.99 mg L⁻¹, 264 mg L⁻¹, 479 mg L⁻¹ and 20.14 mg L⁻¹ (t=0), respectively. At the beginning of the tests, the heavy metal concentrations in the leachates were high while they decreased with the addition of pure CaCO3 and DW application. In pure CaCO3 application and all dose applications of DW; Cd, Cr, Cu, Fe and Zn values decreased below the limit values of 0.003 mg L⁻¹, 0.05 mg L^{-1} , 2 mg L^{-1} , 0.3 mg L^{-1} and 3 mg L^{-1} , respectively at the

end of 60 minutes. The results indicated that DW reduced the Cd, Cr, Cu, Fe and Zn concentrations in the leachate, due to the alkaline content of DW, which neutralized the acidity, reducing the leachability and solubility of metals under alkaline conditions achieved in the tests [25]. Szymanski and Janowska [26] and Tenodi et al. [27] stated that the decrease in pH might cause an elevated level of heavy metals in the leachate and negatively affect the surrounding ecosystems. Feng et al. [28] and Rose [29] neutralized acid by using DW in similar studies and showed that no iron was found in the leachate due to the precipitation. Cui et al. [30] and Rodriguez-Jorda et al. [31] explained that the release of heavy metals could be prevented by using alkaline materials such as DW.

Table 3. Heavy metal concentrations in the leachate and limit values						
Series	Time	Cd	Cr	Cu	Fe	Zn
	(min)	(mg I ⁻¹)				

			÷-			
	(min)	$(mg L^{-1})$				
Start	0	3.45	2.99	264	479	20.14
CFT	60	3.12	3.02	257	496	22.11
(control)	120	4.33	3.01	265	481	21.18
CFT+Pure	60	< 0.005	< 0.005	0.033	< 0.005	0.269
CaCO ₃	120	< 0.005	< 0.005	0.034	< 0.005	0.092
CFT+DW	60	< 0.005	< 0.005	0.671	< 0.005	< 0.005
(1:1)	120	< 0.005	< 0.005	0.556	< 0.005	< 0.005
CFT+DW	60	< 0.005	< 0.005	0.744	< 0.005	< 0.005
(1:0.5)	120	< 0.005	< 0.005	0.871	< 0.005	< 0.005
CFT+DW	60	< 0.005	< 0.005	0.478	< 0.005	< 0.005
(1:1.5)	120	< 0.005	< 0.005	0.308	< 0.005	< 0.005
CFT+DW	60	< 0.005	< 0.005	0.114	< 0.005	< 0.005
(1:2)	120	< 0.005	< 0.005	0.161	< 0.005	< 0.005
CFT+DW	60	< 0.005	< 0.005	0.174	< 0.005	< 0.005
(1:2.5)	120	< 0.005	< 0.005	0.103	< 0.005	< 0.005
CFT+DW	60	< 0.005	< 0.005	0.076	< 0.005	< 0.005
(1:3)	120	< 0.005	< 0.005	0.068	< 0.005	< 0.005
Limit values [24]		0.003	0.05	2	0.3	3

4. CONCLUSIONS

This study investigated the utilization of DW for the treatment of AMD and immobilization of heavy metals released from copper flotation tailings. The results showed that as a result of DW applications at different doses on the CFT neutralization process, the greatest pH change was observed at 1:3 dose of CFT:DW application. The pH value increased from 2.21 to 10.37 at the end of 120 minutes. Depending on the increase in pH, SO₄²⁻ concentration decreased from 4558 (t=0, pH=2.21) to 257 mg L⁻¹ (pH:10.37) and Cd, Cr, Cu, Fe and Zn concentrations were almost completely reduced in the leachate at 1:3 dose of CFT:DW application after 120 minutes of leaching. Considering the environmental quality and health, neutralizing acidity and removing heavy metals released from CFT is of paramount importance, and it could be achieved using DW generated during demolition activities.

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