



Characterization of Acid Mine Drainage in Tailings and Ore Stock Areas of Coal Mine Areas: Kinetic Test Moisture Cell Method

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Research Article

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Abstract

In addition to evaluating the economic inputs of mining, it is important to examine the symptoms and concerns of the results of these activities. Appropriate legal regulations in the operation of mining facilities are implemented in an integrated manner with mining activities and growth effects in order to prevent negative growth effects on future generations. The most important environmental problems specific to mining are the inability to adequately examine the characterization of wastes and the failure to accurately determine whether these wastes can be obtained over the years. Especially during the opening, operation and separation of coal mines, which are rich in sulfur minerals, the reactions of these minerals are exposed to temperature interruptions and the formation of Acid Mine Drainage (AMD) poses a danger to natural life. In order to estimate the AMD formation potential of ore and waste, static and kinetic tests are applied to determine the mineralogical and chemical composition of the field. Among these tests, the most appropriate test in terms of representing natural conditions is the kinetic test method. Within the scope of the study, field conditions were simulated in columns prepared according to ASTM D5744-18 standard, physical and chemical analyzes of the resulting leachate were performed and the results were evaluated. According to the data obtained, no acid formation was observed during the 20-week test period, and metal emissions were characterized at a low rate. It has been determined that the clay structures in the geology of the sampled area are effective on the leakage rate.

Keywords: Coal mine site, acid mine drainage, kinetic test, ore, pasha, mine waste

Kömür Maden Sahalarının Atık ve Maden Stok Alanlarında Asit Maden Drenajının Karakterizasyonu: Kinetik Test Nem Hücresi Yöntemi

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Öz

Madencilik faaliyetlerinin ekonomik girdilerinin değerlendirilmesinin yanı sıra bu faaliyetler sonucunda ortaya çıkabilecek çevresel kaygıların da incelenmesi önemlidir. Gelecek nesiller üzerinde olumsuz çevresel etkilerin önlenmesi için madencilik tesislerinin işletilmesine ilişkin yasal düzenlemelerin madencilik faaliyetleri ve çevresel etkilerle bütünleşik bir şekilde uygulanması gerekmektedir. Madencilğe özgü en önemli çevre sorunları, maden atıklarının karakterizasyonunun yeterince incelenmemesi ve bu atıkların yıllar içinde çevreye verebileceği kirliliğin tam olarak tespit edilememesidir. Özellikle kükürt mineralleri açısından zengin olan kömür madenlerinin açılması, işletilmesi ve kapatılması

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sırasında atmosferik işlemlere maruz kalan bu minerallerin reaksiyonları sonucu ortaya çıkan Asit Maden Drenajı (AMD) oluşumu doğal yaşamı tehdit etmektedir. Cevher ve atığın AMD oluşum potansiyelini tahmin etmek amacıyla sahanın mineralojik ve kimyasal bileşimini belirlemek amacıyla statik ve kinetik testler uygulanmaktadır. Bu testler arasında doğa koşullarını temsil etmesi açısından en uygun test kinetik test yöntemidir. Çalışma kapsamında ASTM D5744-18 standardına göre hazırlanan kolonlarda saha koşulları simüle edilmiş, ortaya çıkan sızıntı suyunun fiziksel ve kimyasal analizleri yapılarak sonuçlar değerlendirilmiştir. Elde edilen verilere göre 20 Haftalık test süresinde asit oluşumu gözlemlenmemiş, metal salınımları ise düşük oranda karakterize edilmiştir. Örnekleme yapılan bölgenin jeolojisinde bulunan kil yapılarının sızıntı oranında etkili olduğu tespit edilmiştir.

Anahtar Kelimeler: Kömür madeni sahası, asit madeni drenajı, kinetik test, cevher, atık, maden atığı

Introduction

In addition to evaluating the economic inputs of mining activities (grade, price, mine life, etc.), it is also very important to examine the environmental concerns that may arise as a result of these activities from an environmental perspective. In order to prevent negative environmental impacts on future generations, legal regulations regarding the operation of mining facilities must be implemented in an integrated manner with mining activities and environmental impacts. For this, businesses need to create appropriate environmental policies. The mining sector is one of the leading sectors of our country and one of the most basic sectors that constitutes an important input to the economy. Since mines must be operated at their location or in existing suitable facilities located nearby, it is necessary to keep mining activities separate from other industrial activities. While it is possible to establish any industrial facility in an organized industrial zone or in places close to raw materials, there is no such possibility when it comes to opening mines. Mines are operated in the location where the ore is located, with open or closed mining (gallery) methods [1]. The most important environmental problems specific to mining are the insufficient examination of the physical and chemical properties of mine wastes and the inability to accurately detect the pollution that these wastes may cause to the environment over the years. Acid Mine Drainage threatens natural life as a result of the degradation of these minerals, which are exposed to atmospheric processes during the opening, operation and closure of coal mines, which are especially rich in sulfur minerals. From AMD, which is defined as one of the biggest environmental problems of the mining industry; This situation shows that in developed countries such as the USA and Canada, the financial costs of the world's leading mining companies to eliminate the problems caused by AMD vary between 2-5 billion dollars for a total of 12500 hectares of acidic fine-grained residue and 750 million tons of acidic bedrock [2]. It provides a very good example in terms of perceiving the dimensions of the problem. The formation of acidic drainage by sulphide minerals may occur under geogenic (hydrolysis and oxidation of sulphide ores in the natural environment) conditions, or it may occur when the ore or sulphurous wastes come into contact with the air and moisture (water) environment as a result of intensive mining activities (especially open pit mines). Terminologically, acid production under natural conditions

is Acid Rock Drainage (ARD); Acidic drainages caused by anthropogenic factors are called Acid Mine Drainage (AMD). As expected, the AMD formation process occurs faster than the ARD formation process. The major mechanisms of both ARD and AMD formation include hydrolysis and oxidation of the mineral pyrite (FeS_2), which is a very common mineral in nature [1]. Acid Mine Drainage greatly affects the receiving environment by changing the ambient pH and dissolved concentration of different chemical species [3]. Minerals contained in AMD can also precipitate at the bottom of receiving water bodies such as streams and rivers and affect benthic organisms [4]. In AMD, hazardous substances such as arsenic (As), chromium (Cr), iron (Fe), aluminum (Al), copper (Cu), zinc (Zn), lead (Pb), molybdenum (Mo) and nickel (Ni) are present. The presence of toxic chemical species is of primary concern [5]. Exposure to these pollutants can cause ecotoxicological, carcinogenic, mutagenic and teratogenic effects [6]. In addition to negatively impacting aquatic ecosystems, AMD also affects the quality of natural water bodies for human consumption or irrigation [7]. Therefore, preventing or effectively improving acid formation can protect human health and the environment. Remediation of acid typically relies on active (frequently driven by the input of chemicals, energy, and equipment) or passive (based on oxidation or reduction) technologies. However, these technologies have variable effectiveness in removing contaminants and also produce sludge and/or brine that can cause secondary pollution if not properly managed [8]. AMD usually contains high concentrations of Fe and sulfate (SO_4^{2-}) as well as rare earth elements and yttrium [9]. Determining the acid-producing or acid-neutralizing status of rock layers before failure [10] helps develop overburden handling and placement plans. Kinetic tests such as moisture cell sand leaching columns are important because they examine the rate of acid-producing and neutralization reactions [11]. This information from kinetic testing can complement the information provided by Acid-Base Accounting and assist regulators in permitting decisions. For example, the rate of sulfide oxidation and release of contaminants can occur rapidly before neutralizing materials have time to react. Therefore, early pulses of contaminants can be released for a short period of time when the reactions stabilize. [12] Detection of AMD formation potential plays a major role in planning mining activities and preventing their environmental impacts. According to the 14th paragraph of the 11th article of the Mining Wastes Regulation dated 15.07.2015 and numbered 29417 of the Ministry of Environment and Urbanization, "Although the provisions of this article are not applied for waste storage areas, stability measures are taken in the storage areas. In addition, sulfur-containing and acid rock drainage Potential wastes are stored in a way that prevents contact with air and water, by buffering them with wastes with neutralization capacity or by using the necessary collection techniques. With appropriate slope and shoulder systems, the leachate is treated and rehabilitated after storage. "Effects of these areas on surface/surface and groundwater are monitored with water samples taken from observation points and observation wells." Detecting AMD formation in the field and determining the measures to be taken to minimize its impact on the receiving environment. In order to estimate the AMD formation potential of ore and waste, static and kinetic tests are applied to determine the mineralogical

and chemical composition of the field. Among these tests, the most appropriate test in terms of representing natural conditions is the kinetic test method. In this context, field conditions will be simulated in columns prepared according to the ASTM D5744-18 "Standard Test Method for the Separation of Solids in the Laboratory Using a Humidity Cell" standard and physical and chemical analyzes of the resulting leachate will be performed. In addition, XRD analyzes were performed before moisture cell tests to determine the mineralogical structure of the field. This study aims to determine the potential for AMD formation in the ore and waste areas of the high-sulfur coal mine operating in Çan District of Çanakkale Province. Previous studies were examined in selecting the region where the research would be conducted. The fact that the geology of the region is suitable for AMD formation and that kinetic test studies have not been conducted before reveals the importance of this study. The kinetic tests subject to the study are used to simulate field conditions and evaluate the physical and chemical properties of the leachates they create, determine their potential to form acid mine drainage (AMD) and determine the precautions that can be taken. Thanks to this work, pollution that may arise from mining companies operating or will continue to operate in the region will be prevented. To achieve this goal;

- Appropriate samples were taken from the waste areas of the Open Pit Mine operated in Çan District of Çanakkale Province.
- That were formed as a result of excavations for coal production and were exposed to decomposition processes under natural conditions, were fragmented and especially open to atmospheric processes, were determined.
- Mechanisms were prepared to be used in kinetic tests, taking into account the field of mineralogy.
- During kinetic column experiments, pH and electrical conductivity (Ec) measurements taken over time and changes in elements and compounds determined by chemical analyzes were examined, and the relationship between AMD formation and these changes was investigated in kinetic column experiments.

Examining AMD formation potentials in ore and waste areas is very important in terms of protecting the environment from negative effects. For this reason, similar studies are carried out in sulfur-containing mining areas in our country and around the world, and it is aimed to minimize these effects by detecting the negative effects in advance. The data obtained as a result of the work to be carried out in the field within the scope of the project is of great importance in terms of preventing environmental pollution that may occur in the region. This will contribute to the preservation of the ecological balance of the region, especially by protecting the water resources from which the surrounding settlements provide drinking water and preventing the pollution of soil, air, surface and groundwater.

Materials and Methods

Acid mine drainage; It is defined as the phenomenon in which iron minerals such as pyrite, pyrrhotite and marcasite are exposed to oxidation in a humid environment in a mining area, giving protons (H⁺

ions) to the aqueous environment as a result of the reactions and the solution turning into solution. Acidic mineral waters with corrosive and solvent character generally contain up to 50 mg/l Cu, up to 1000 mg/l Fe, and up to 12 mg Pb, as well as high sulphate (800 - 1800 mg/l) content. /l. and may contain up to 1700 mg/l of Zn metals. In addition, AMDs formed as a result of the leaching process contain cyanide, thiosulfate, etc. It is also known to contain [13]. Although pyrite (FeS_2) and arsenopyrite (FeAsS) are mainly responsible for AMD formation, iron sulfides (Fe_xS_x), pentlandite ($(\text{Fe, Ni})_9\text{S}_8$), chalcopyrite (CuFeS_2), villamaninite (Cu_2S), covellite (CuS), molybdenite Weathering of other sulfide-rich or sulfide-containing minerals such as (MoS_2), sphalerite ($(\text{Fe, Zn})\text{S}$), millerite (NiS) and Galena (PbS) contributes to the formation of AMD [14]. These minerals are typically organic-rich (reducing) and are found in sediments (e.g. coal beds) [9]. These minerals are typically encountered in organic-rich (reductive) sediments (e.g., coal deposits) or in rocks altered by sulfur-rich hydrothermal fluids, such as volcanogenic sulfide ore, which produce metals such as copper, gold, and zinc. When exposed to the atmosphere, typically through mining activities (e.g., surface or deep mines, tailings piles, and tailings), they oxidize and form AMD; Iron sulfide minerals (typically pyrite) are the main minerals behind the formation of AMD and others. Sulfur minerals contribute only to a limited extent [15].

Table 1. Chemical decomposition processes effective in the formation of acid mine drainage

Chemical Degradation Process Stages	Explanation
$\text{FeS}_2 + 7/2\text{O}_2 + \text{H}_2\text{O} \rightarrow \text{Fe}^{2+} + \text{SO}_4^{2-} + 2\text{H}^+$	Oxidation of Pyrite
$\text{Silicates} + \text{H}^+ \rightarrow \text{SiO}_2 + \text{Al}^{3+} + \text{Mg}^{2+} + \text{Ca}^{2+} + \text{Mn}^{2+}$	Dissolution of Clays with Acid
$\text{CaCO}_3 + \text{H}^+ \rightarrow \text{Ca}^{2+} + \text{H}_2\text{O} + \text{CO}_2$	Neutralizing Acid with Calcite
$\text{Fe}^{2+} + 1/4\text{O}_2 + \text{H}^+ \rightarrow \text{Fe}^{3+} + 1/2\text{H}_2\text{O}$	Ferro Iron Oxidation
$\text{Fe}^{3+} + 3\text{OH}^- \rightarrow \text{Fe}(\text{OH})_3$	Ferric Iron Hydrolysis “Yellow Layer Formation”
$\text{Al}^{3+} + 3\text{OH}^- \rightarrow \text{Al}(\text{OH})_3$	Aluminum Hydrolysis
$\text{Fe}^{2+} + 1/4\text{O}_2 + 5/2\text{H}_2\text{O} \rightarrow \text{Fe}(\text{OH})_3 + 2\text{H}^+$	Iron Oxidation and Hydrolysis Reactions
$\text{H}^+ + \text{HCO}_3^- \rightarrow \text{H}_2\text{O} + \text{CO}_2$	Acid Neutralization of Bicarbonate Ion
$\text{H}^+ + \text{OH}^- \rightarrow \text{H}_2\text{O}$	Acid Neutralization with Hydroxyl Ion



Figure 1. Abandoned coal mine site in çanakkale çan region with amd formation observed. turning to red color after oxidation with atmospheric weather conditions.

The type of sulfur minerals found in mine wastes, humidity, oxygen concentration, presence of bacteria, properties and amounts of alkaline minerals are the main factors affecting the formation of AMD. On the other hand, the distribution of sulfide and alkaline ores in the waste pile, the surface area of the sulfide ores and therefore the grain size and oxidation rate are important parameters affecting the formation of AMD. The potential of mine tailings to produce acids and release pollutants depends on several factors [16]. The main factors affecting the formation of AMD include sulfur minerals (Table 2), water, oxygen, the presence of bacteria that catalyze oxidation, and the heat generated as a result of the reaction. Secondary factors are; While it refers to tertiary factors such as neutralization of the acid produced, it consists of dissolved metal ions and their effects that change the character of the waste through acid production. Tertiary factors; Physical properties of materials, storage of acid-producing and neutralizing materials, and hydrological regime near the waste. The view of the AMD pond in the abandoned coal mine area, which turned red after oxidation due to atmospheric weather conditions, is given in Figure 1. Forms Fe^{2+} and SO_4 and H^+ as shown in the reaction in Table 1. This process is key to AMD formation. Mineral composition, environmental microbial activity, and external conditions such as oxygen and water influence pyrite oxidation and H^+ release, determines the speed. In an environment where there is sufficient oxygen and necessary microorganisms ($\text{pH} > 3.5$), Fe^{2+} released in the reaction, depending on the reaction, it can be oxidized to Fe^{3+} . Since the pH is 2.3–3.5 $\cdot \text{Fe}^{3+}$ will precipitate $\text{Fe}(\text{OH})_3$ and release H^+ as shown in the reaction. As the H^+ released increases, the pH will gradually decrease. When $\text{pH} < 2$, $\text{Fe}(\text{OH})_3$ will hydrolyze again, allowing Fe^{3+} to return to solution and

promoting the oxidation of FeS_2 (When $\text{pH} < 3$, the oxidation rate of FeS_2 by Fe^{3+} is about 10– In the reaction [17] from O_2) as shown It is 100 times faster (Figure 2).

Table 2. Some important metal sulfides, of which pyrite and marcasite are the dominant acid producers [18].

Metal Sulphide	Chemical Formula
Pyrite	FeS_2
Marcasite	FeS_2
Pyrrhotite	Fe_{1-x}S
Chalcocite	Cu_2S
Chalcocite	Whoa
Chalcopyrite	CuFeS_2
Molybdenite	MoS_2
Millerite	NiS
Galena	PbS
Sphalerite	ZnS
Arsenopyrite	FeAsS

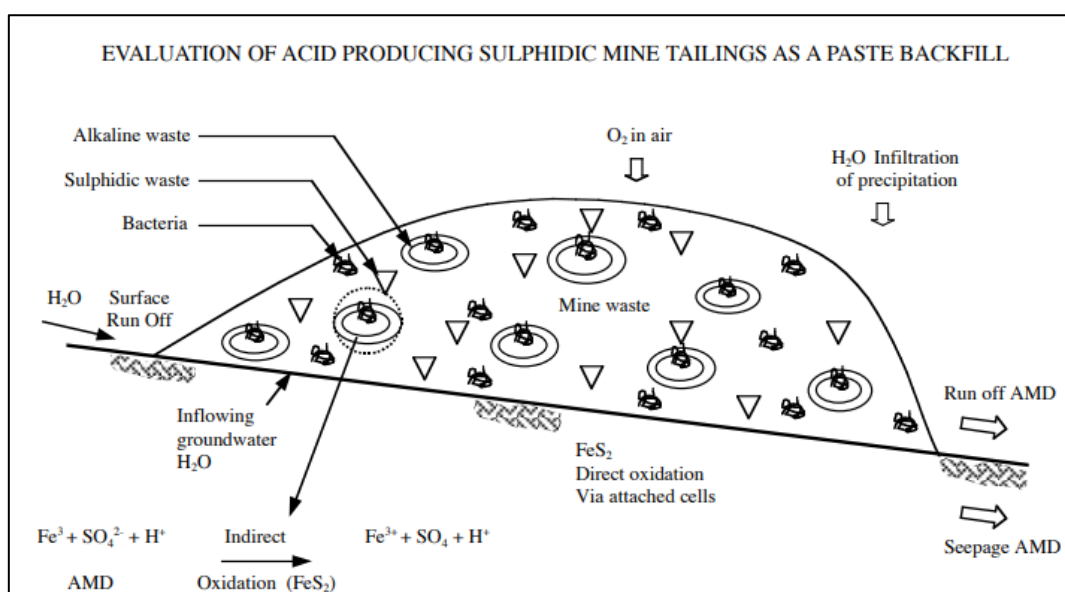


Figure 2. Bacterial oxidation of pyrite and acid formation via direct and indirect paths [19]

Geology and Stratigraphy of the Study Area

The basement rock unit observed in the study area and its surroundings is the Cretaceous -Paleocene aged complex, according to Okay et al. [20] it is named as Çetmi ophiolite mélange. Çetmi ophiolite mélange, which crops out in the west of Karabiga, consists mostly of spilite, various types of limestone blocks and less of shale, sandstone, serpentinite and radiolarite. Spilites are dark green, blackish green colored spilites generally do not show foliation and consist of albite, pinkish Ti-Augite and chlorite. Only eclogites located around tectonic eclogite blocks show a distinct foliation and a greenschist facies mineral assemblage characterized by actinolite and epidote [20]. This unit is unconformably covered by

the Early-Middle Miocene aged Doyran Volcanics and the continental Çan Formation, which was deposited at the same time as this volcanism [21]. The youngest geological unit in the region is Quaternary alluviums. In the study area where TKİ Çan Lignites Enterprise is located, there are Çan volcanics [22], which have reached the surface by cutting older units on the Biga Peninsula, and Çan formation [23] and Kulfa formation [24], which consist of clastic and visual sediments containing lignite. The average coal thickness in the Çan Formation, where coal production takes place, is approximately 17 meters.

Findings and Conclusions

Previous Studies Performed in the Field

Turkish Coal Enterprises (TKİ), Çan Lignite Enterprise Directorate (ÇLİ) Çanakkale province, Çan district, with the aim of determining the acid production potential of the waste areas of the open pit enterprise with license number IR: 3378, by Kütahya Dumlupınar University Faculty of Engineering, Mining Engineering Department in 2020. A study was conducted and as a result of the sulfur analysis and static tests of the samples taken, the acid production potential (APP) of the field was found to be 19.99 kg CaCO₃ t⁻¹ on average and the neutralization potential (NP) was 80.55 kg CaCO₃ t⁻¹ on average. In addition to minerals with neutralizing properties such as clay, montmorillonite, kaolinite, andesite, muscovite and calcite, the presence of minerals with acid-producing properties such as pyrite has also been determined. The following findings were determined as a result of static tests carried out by Dumlupınar University for the purpose of determining AUP in the mining area under investigation.

- In the pH measurements made for a short time (3 days) in the samples taken from the field, it was determined that the pH values varied between 6.67 - 9.79, therefore there was no acid production in the short term.
- The average sulphide-sulfur value of 95 different samples taken from different coordinates of the waste area was determined as 0.64%
- The acid production potentials of the samples were determined with a theoretical approach (ABA standard method) according to sulfur values, and the average acid production potential of the waste area was calculated as 19.99% kg CaCO₃/t.
- The average neutralization potentials of the samples taken from the license areas were determined as 80.55 kg CaCO₃/t.

In this study, the acid production potential and neutralization potential of the tailings area were determined by static tests that can be answered in a short time based on prediction. In the final evaluation of such fields; it is recommended to use leaching-based kinetic tests in which the field is simulated by monitoring the oxidation and degradation of samples subjected to water and oxygen over a longer period of time.

Sampling

Sampling procedures: 2 waste samples containing high sulfur were taken on 03.08.2021, taking into account the Static Test results. Approximately 50 kg samples were collected from the Research Pit dug at a depth of 8 meters with a diameter of 9 inches using the Rok -Bit Blow Hole Drilling Machine (9 inch diameter, 8 meter rod length), CEN_TR 15310-3_2006 Characterization. Bulk Sample Reduction from Waste Subsampling Standard was reduced according to Method 2 and taken as 20 kg. Sampling procedures and sample reduction processes carried out in the field are given in Figure 3. The samples were transferred to a 40 micron thick, 35x70 sized plastic nylon bag, a legibly written note describing the sample was placed inside the bag, and the mouth of the bag was closed with waterproof, strong adhesive tape. Explanatory information was noted on both sides of the bag with an indelible pen and the prepared sample bags were transported to the Laboratory. The physical properties of the samples are given in Table 3.

Table 3. Information about the samples

Simple Code	Sample Quantity	Colour	Homogeneity	Particle Type	Gas Output	Heat Production	Reaction Occurrence
CN-7	20 kg	Dark brown	Homogeneous	various type	NO	NO	NO
CN-25	20 kg	Dark brown	Homogeneous	Monotype	NO	NO	NO



Figure 3 Sampling processes view

Preparation of Field Samples for Laboratory Experiments

The samples collected as a result of field studies were made ready for analysis using appropriate techniques (Homogenization, Drying, Particle Size Reduction, Preparation of Subsamples) according to the TS EN 15002 "Waste Characterization - Preparation of test sample pieces from laboratory samples" standard. The samples were dried under atmospheric conditions (at room temperature) for 3

days to remove moisture. After the drying process, all samples were passed through a 6.3 mm (0.25 inch) sieve, and samples with a grain size larger than 15.2 cm were passed through crushers and gradually refined. For this purpose, it was first passed through the crusher set at 1.92 cm, then through the crusher set at 0.95 cm, and finally through the crusher set at 0.64 cm. After each crushing process, the samples were passed through a 6.3 mm sieve and the next stage was started. Since crushing processes cause the release of acid-producing and acid-consuming minerals, they may cause the characteristic features of the samples to deteriorate. This effect was taken into account in the analysis processes. Then the samples;

- Using a Sample Divider with a 2.54 cm slit, the sample was split into 8 1 kg test samples and stored in a ziplock bag. One of the test samples was loaded into the moisture cell for kinetic testing.
- One of the separated test samples was taken and crushed through a 1.7 mm (10 mesh) sieve to pass 95%, and then the sample was divided into 2 with a 6.35 mm slotted sample separator.
- These samples were ground to pass through a 150 μm (100 mesh) sieve and 250 grams of sample was taken for XRD analysis.

Minerological Tests of Samples

In order to determine the mineralogical characteristics of the geological units that create waste in the wastewater field and to detect the minerals that will create acidity through neutralization, PANalytical was conducted in Çanakkale. Analyzes were made with Empyrean brand XRD (X-Ray Diffraction) device. Onsekiz Mart University ÇOBİLTÜM - Science and Technology Application and Research Center. The mineralogy test results of the samples are given in Table 4, and the distribution according to mineral groups is given in Figure 4.

Table 4. Minerological analysis results

Mineral	Formula	Group	CN-7	CN-25
Pyrite	Fe_2S_3	Oxide Group	3.9%	3.5%
Cristobalite	SiO_2	Silicate Group	24.4%	27.8%
Montmorite	$2\text{Al}_2\text{O}_3 \cdot 8\text{SiO}_2 \cdot 2\text{H}_2\text{O} \cdot \text{nH}_2\text{O}$	Silicate Group	28.1%	29.7%
Quartz Low	SiO_2	Silicate Group	-	9.6%
İlitis	$\text{K}_{0.65}\text{Al}_{2.0}(\text{Al}_{0.65}\text{Si}_{3.35}\text{O}_{10})(\text{OH})$	Silicate Group	-	19.0%
Zeolite	$(\text{M}^+, \text{M}^{+2})\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 9\text{SiO}_2 \cdot \text{nH}_2\text{O}$	Silicate Group	1.0%	0.8%
Albite High	$\text{NaAlSi}_3\text{O}_8$	Silicate Group	42.5%	-
Plaster	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	Sulfate Group	-	1.6%
Aragonite	$(\text{Ca}, \text{Sr}, \text{Pb}, \text{Zn})\text{CO}_3$	Carbonate Group	-	8.0%

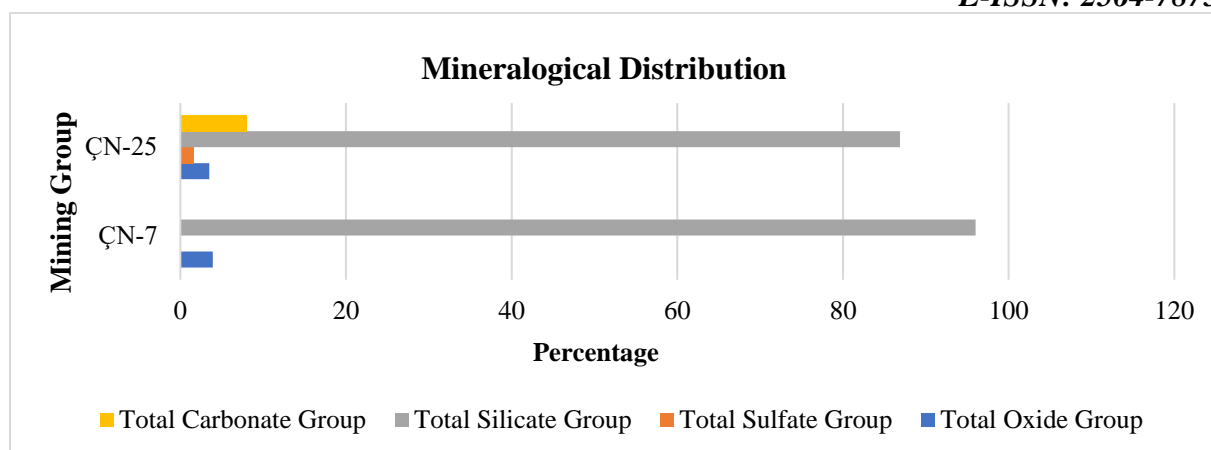


Figure 4. Mineralogical distribution graph

Kinetic Tests (Moisture Cell Test)

Kinetic tests are the next step up from static tests and are essentially simulation tests. They are performed to reduce uncertainties in static tests, verify the data obtained, identify decisive reactions, detect acid production rate and determine drainage water quality. In this technique, the field is simulated by monitoring the oxidation and degradation of samples over time as they are exposed to water and oxygen. Kinetic tests known in the literature are the "Moisture Cell" test, the "Column" test, the "Soxhlet" test, "Lkstahton" Test, "British Columbia These are tests such as the "Research " test. Considering that acid production in waste areas will develop over time, kinetic tests should be used when evaluating new and old mine areas and waste areas. Method for 2 samples with Acid Producing Potential to test AMD formation in the waste area. for Laboratory Kinetic Humidity Cell Test procedures were carried out in accordance with the " Weathering of Solid Materials Using a Humidity Cell" standard . pH, Acidity, Conductivity, Alkalinity, Acidity, Anion-Cation and Metal were determined by planning the moisture cell method in the wastewater collected weekly, every fifteen days or monthly. Parameters were analyzed. The samples to be loaded into the cells were 100% sieved or broken, and since their dimensions were 6.3 mm, cells with an inner diameter of 10.2 cm and a height of 20.3 cm were used. ASTM D5788-18 Standard Option A method was used for weekly cycles. According to this method, after the first leakage sample was taken from the cells, three days of dry air (relative humidity less than 10%) and three days of water-saturated air (relative humidity around 95%) were applied to the cells weekly. humidity). Approximately 2 L/min of dry air and moist air were supplied to the cells during all weekly cycles (Figure 5).



Figure 5. Kinetic test setup view

Grain Size Distributions in the Moisture Cell

The particle size distribution of the samples loaded into the moisture cell is given in Figure 6.

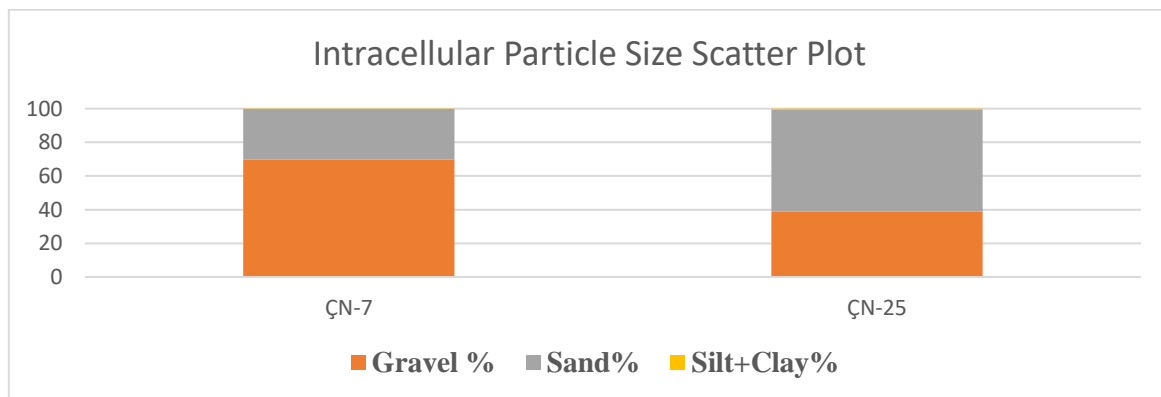


Figure 6. Intra-cellular particle distribution plot

Fixing the First Leak

The first water leaching is flood leaching. Week 0 leaching started with the first addition of water. This determines the initial characteristics of the leak. Moisture cell testing consists of a 7-day cycle. The sample was always filtered on the same day of the week (Monday). 1 L of water was added using a measuring tape and this amount could be 500 ml depending on the type of sample. During the water addition process, air inlets and water inlets were closed and water was added from the sides of the cell walls. After the filtration process was completed, the upper lids were closed and the filtration process was left overnight. When the filtration process was completed, the weights of each cell and collection containers were weighed and recorded. The pH and conductivity values of the collected leachate were measured, the collected sample volume was recorded and analyzed as Week 0 Sample. Then, to complete the weekly cycle, the air inlets of the cells were opened again and the operations were started.

Next Weekly Leachate

On the 7th day of the first weekly cycle, the second water was added and filtered and called the 1st week sample. The same procedures were carried out in subsequent weekly cycles.

Leachate Analysis

Priority pH, Dissolved Oxygen and Conductivity parameters of the leachate collected from moisture cells were measured and recorded. Then, filtration was performed using a filter with a pore diameter of 0.45 µm, and the solids remaining in the filter were taken back into the moisture cell. Leachate pH, alkalinity, acidity and conductivity analyzes is 20 weeks. These analyzes should be performed at weeks 0, 1, 2, 4, 6, 8, 10, 12, 14, 16, 18 and 20 of the test. There is no need to analyze weekly samples for chemical characterization; Instead, analysis at minimum 0, 1, 2, 4, 6, 8, 12, 16 and 20 weeks is sufficient. Analyzes were carried out for 20 weeks and the results were interpreted.

Discussion and Conclusion

The obtained measurement and analysis results were interpreted with reference to the PD CEN TR 16363-2012 “Characterization of wastes, kinetic testing for evaluation of acid production potential of sulfuric wastes originating from mining industries” standard.

Evaluation of Minerological Analyzes

While Oxide and Sulphate Group minerals, which may have acidic properties, were found in small amounts in the samples, Silicate and Carbonate Group minerals with Neutralization Potential were predominantly detected. When minerals with neutralization potential were examined, it was determined that silicates, which have lower neutralization capacity than carbonates, were predominantly present. In the mineralogical description of the samples, gypsum and pyrite minerals were determined as the main sulfur sources. Gypsum sulphate sulfur; Pyrite disulfide contains sulfur. Gypsum is known to have moderate solubility in water (4). Pyrite represents the presence of sulfur that has not yet undergone oxidation. Plaster does not have an increasing or decreasing effect on acidity (6). When pyrite undergoes hydrolysis and oxidation, it releases protons into water and increases the acidity of the environment.

Leachate Analysis

The results of the leachate analyzes performed to determine the quality of the drainage water of the waste samples loaded into the moisture cells are given in Table 5 and Table 6, and the following findings were obtained according to the analysis results.

Table 5. Physical analysis results

pH												
Example	0.	one.	2.	4.	6.	8.	10.	12.	14.	16.	18.	20.
CN-7	7.14	7.31	7.60	7.23	7.50	7.75	7.47	7.33	7.17	7.50	7,10	6.92
CN-25	7.11	7.51	7.54	7.34	7.45	7.78	7.50	7.40	7.45	7.70	7.27	7.27
CONDUCTIVITY $\mu\text{S/cm}$												
CN-7	3460	1260	956	709	516	1662	665	636	542	662	713	747
CN-25	7730	2770	2044	1610	1475	1159	933	783	539	538	556	584
ALKALINITY mg (CaCO₃)/L												
CN-7	48.2	46.8	52.6	38.0	41.6	64.0	29.6	26.6	36.0	37.4	54.8	58.2
CN-25	109.4	112.2	103.2	93.6	92.8	89.6	83.6	98.8	81.0	66.0	65.0	91.2

Table 6. Chemical analysis results

SULFATE mg										
Example	0.	one.	2.	4.	6.	8.	12.	16.	20.	
CN-7	1197.0	394.2	304.6	87.5	176.8	661.1	226.5	283.0	285.5	
CN-25	1596.0	906.3	716.6	639.3	640.8	467.7	297.4	200.2	194.7	
CADMINIUM (Cd) mg										
CN-7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
CN-25	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
ZINC (Zn) mg										
CN-7	0.016	<LOQ	<LOQ	0.017	<LOQ	<LOQ	<LOQ	0.014	0.006	
CN-25	0.015	<LOQ	<LOQ	0.015	<LOQ	<LOQ	<LOQ	0.014	<LOQ	
IRON (Fe) mg										
CN-7	0.246	<LOQ	<LOQ	0.016	0.076	<LOQ	<LOQ	0.081	0.188	
CN-25	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	
MAGNESIUM (Mg) mg										
CN-7	12.66	4.62	3.85	3.97	2.48	6.30	2.04	4.57	4.54	
CN-25	39.37	10,18	9.55	9.62	10.87	9.40	6.87	5.90	5.26	
SODIUM (Na) mg										
CN-7	417.56	142.25	124.96	70.63	56.37	165.43	90.79	88.90	85.82	
CN-25	823.39	317.18	224.70	107.63	98.37	67.31	41.19	27.66	22.11	
Potassium (K) mg										
CN-7	14.08	4.02	3.02	2.77	2.17	4.41	2.05	2.25	2.51	
CN-25	28.62	11.06	8.86	6.22	6.86	3.01	2.46	1.36	1.09	
CALCIUM (Ca) mg										
CN-7	93.33	32.66	29.74	13.78	54.78	71.69	14.49	32.11	30.95	
CN-25	182.86	65.76	81.36	57.88	134.01	109.58	77.28	70.04	61.97	
FLUORIDE (F) mg										
CN-7	0.41	<LOQ	0.39	0.71	0.13	0.59	0.60	0.39	0.47	
CN-25	0.81	0.48	0.79	0.42	0.62	0.62	0.32	0.55	0.37	
CHLORIDE (Cl) mg										
CN-7	3.81	5.85	16.96	10.29	6.87	12.96	9.54	10.44	8.29	
CN-25	25.10	5.11	13.82	10.47	8.71	11.21	5.34	9.97	7.29	

*LOQ = Measurement Detection Limits

pH

pH values vary between 6.92 and 7.78. pH values increased until the 8th week and started to decrease between the 8th and 20th weeks. The pH value in the MP-7 code sample, which does not contain carbonate minerals with high neutralization potential in its structure, is lower than the MP-25 code sample, which contains carbonate minerals.

Alkalinity

When the alkalinity results are examined, it is seen that the results of the sample coded CN-25, which contains carbonate minerals in its structure, are higher than the results of the CN-7 sample, which consists mostly of silicates.

Sulfate

Considering the sulfate concentrations, the sample coded CN-25, which contains the sulfate group gypsum mineral in its structure, decreased from 1.596.0 mg to 194.7 mg after 20 weeks. It was observed that the CN-7 sample, which does not contain sulfate group minerals, released a maximum of 1.197.0 mg sulfate.

Conductivity

It was observed that the conductivity results of the CN-25 coded sample containing carbonate minerals were higher than the CN-7 coded sample containing predominantly silicate groups and gradually decreased in weekly analyses.

Ficklin Metal Values

Ficklin metal value was calculated in two samples by considering the sum of Cd, Co, Cu, Zn, Pb and Ni elements mobilized under low pH conditions of filtered waters. According to the relationship between Ficklin metal and pH, the waste samples were characterized by low metal release, as the Ficklin metal value was below 1000 µg/L under neutral conditions.

Leakage Rate and Hydraulic Structure of the Material

The amount of element leached per unit mass and time is called leaching rate. Hydraulic Structure describes the general character of a rock as shown in terms of grain size and shape, degree of crystallinity and arrangement of the particles that compose it. This structure has an impact not only on the "hydraulic conditions" (including air intake), but also on the weathering and sensitivity to weather conditions and the formation/release conditions of the components. The texture and hydraulic properties of the material are important factors that determine contact with water and air in the kinetic test cell. At the site scale, the (physical) heterogeneity of the waste area will lead to nonuniform flow fields (preferential flow paths) that have an impact on the contact between water, air and waste and the transport of

decomposition products. While calculating the leakage rates of the samples, it was calculated that the leakage rate of the sample coded CN-25 was higher than that of CN-7.

Evaluating Whether the Material Is Acidic

When the pH values of the samples are examined, no acid formation is observed in either sample during the 20-week period. In addition to sulfate emissions, there are also easily soluble neutralizing minerals that create alkalinity and cause the consumption of hydrogen ions produced.

Defining the Acid Consuming Reaction Rate

Identification of acid-consuming reactions can be done using Humidity cell tests in response to the rate of acid production in a system where sulfur oxidation is stopped by water saturation and the flow through a batch reactor or reactor. Since minerals that rapidly consume acid will be calcium carbonate minerals, calcium and magnesium values in the leachate will give indicators of acid-consuming minerals. Silicate minerals (especially calcium-rich plagioclase and mafic minerals) also have the potential to consume acid. When the 20-week leachate results are examined, it is seen that the rapidly acid-consuming Ca+Mg values of the CN-7 coded sample are 418.6 mg, and the CN-25 coded sample is 947.8 mg. These results support the results obtained in mineralogical tests.

Predicting When Material Will Become Acidic

Calculating the Neutralization Potential exhaustion time and Acid Potential exhaustion time can be used when assessing when the material will become acidic. It can be said that the materials will not produce acid if the neutralization potential consumption is longer than the acid potential. For this calculation, the sulfate production rates of the samples, that is, the total acidity produced, are important. Accordingly, in the example coded CN-7, it is estimated that the NP consumption period will be 5.03 years later and the AP consumption period will be 2.61 years later. In the example coded CN25, the NP consumption period will be 17.27 years and the AP consumption period will be 11.71 years later. Since AP will be consumed before NP, it is predicted that the two waste samples will not produce acid in the long term.

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Authors Contribution All authors read and approved the final manuscript.

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