

Prediction of acid mine drainage generation potential of various lithologies using static tests: Etili coal mine (NW Turkey) as a case study

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Abstract The Etili neighborhood in Can County (northwestern Turkey) has large reserves of coal and has been the site of many small- to medium-scale mining operations since the 1980s. Some of these have ceased working while others continue to operate. Once activities cease, the mining facilities and fields are usually abandoned without rehabilitation. The most significant environmental problem is acid mine drainage (AMD). This study was carried out to determine the acid generation potential of various lithological units in the Etili coal mine using static test methods. Seventeen samples were selected from areas with high acidic water concentrations: from different alteration zones belonging to volcanic rocks, from sedimentary rocks, and from coals and mine wastes. Static tests (paste pH, standard acid–base accounting, and net acid generation tests) were performed on these samples. The consistency of the static test results showed that oxidation of sulfide minerals, especially pyrite—which is widely found not only in the alteration zones of volcanic rocks but also in the coals and mine wastes—is the main factor controlling the generation of AMD in this mine. Lack of carbonate minerals in the region also increases the occurrence of AMD.

Keywords Acid generation prediction · Acid mine drainage · Etili coal mine · Static tests

Introduction

Acid mine drainage (AMD) from waste rock, tailings, and other mine components is the most important environmental problem in the mining industry (Lawrence and Scheske 1997). AMD is often characterized by high concentrations of metals and sulfates in solution and by generally low pH values (Mendez-Ortiz et al. 2007). These acidic solutions can potentially contaminate water resources, as well as soils (Skousen 1995; Morin and Hutt 1997; Mendez-Ortiz et al. 2007). AMD has a long history, dating back thousands of years to Phoenician times when the Iberian Pyrite Belt in Spain, from where the Rio Tinto (“Red River”) flows, was first exploited (Miller 1998; Usher and Vermeulen 2006). In the past, little attention was paid to acid generation, leading to long-term environmental problems associated with water discharges from old mine excavations and from piles of pyrite-bearing waste rocks (Hughes et al. 2007). Currently, handling and long-term storage of pyrite-bearing waste rocks are important issues at mine sites, and details of waste rock stack construction are an integral part of mine design, even before mines are opened (Sengupta 1993; Tran et al. 2003; Williams et al. 2003; Hughes et al. 2007). There are over 200,000 AMD sites within the USA (Hochella et al. 1999); more

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than 5000 km of water courses are polluted by contaminated drainage from abandoned mines in Europe (Younger et al. 2002; DiLoreto et al. 2016). The most expensive aspects of some mining operations involve long-term water management and treatment because of their acidic pH and/or unacceptably high aqueous metal concentrations (Morin and Hutt 2006). The time period for such management and treatment can span decades to centuries (Morin and Hutt 1997, 2001, 2006). Given that mineralogy and other factors affecting AMD formation are highly variable from site to site, predicting the potential for AMD can be exceedingly challenging and costly (U.S. EPA 1994; Yeheyis et al. 2009).

Static tests used to evaluate the acid generation potential of rocks and mine wastes are critical to determine the environmental impact of a mine site and to choose a waste management method to minimize that impact (Ritcey 1989; Morin and Hutt 1997; Bussiere et al. 2004; Benzaazoua et al. 2004; Yeheyis et al. 2009). Details of these tests are documented (Sobek et al. 1978; Smith et al. 1992; Morin and Hutt 1997; White et al. 1999; Mitchell 2000; Jambor 2003; Mills 2007; Lottermoser 2010). Static tests are short-term, relatively low-cost tests developed to provide an estimate of the capacity of mine waste to produce acid and to neutralize acid (Lapakko 2002). A large number of these tests can be performed to characterize the various mine rock units and from this to select samples for kinetic testing. To develop waste management plans that will confidently allow permitting to proceed and the acceptance of closure measures, proponents and operators must characterize the various wastes and other components from current or proposed mining activities to predict future performance of the materials when disposed of under ambient conditions (Lawrence and Scheske 1997). In dealing with environmental aspects of the potential exploitation of mineral and coal deposits, such as the ongoing disposal of wastes at operating mines, preparation for mine closures, and the remediation of abandoned sites, it is essential to predict the geochemical behavior of the wastes on their exposure to weathering (Jambor et al. 2002).

The Etili neighborhood in Can County (north-western Turkey) has large reserves of coal and has been the site of many small- to medium-scale

mining operations since the 1980s. Some of these have now ceased working while others continue to operate. Once activities have ceased, the mining facilities and fields are usually abandoned without rehabilitation. As a consequence of human intervention in the natural structures and topography of the earth's surface, large holes and deterioration have developed in these areas. Artificial lakes can occur as a result of both surface discharge and underground leakage into abandoned open pits; over time, these lakes become acidic (pH <4) and accumulate high metal concentrations (e.g., Al and Fe). The geological characteristics of the region and the limnological and hydrogeochemical characteristics of these acid mine lakes have been studied in detail (Gunduz and Baba 2008; Gurdal 2011; Gurdal and Bozcu 2011; Okumusoglu and Gunduz 2013; Sanliyuksel Yucel and Baba 2013; Sanliyuksel Yucel et al. 2014, 2016), but the acid generation potential of different lithological units has not yet been studied. In this work, static test methods were employed to investigate the Etili coal mine for acid generation potential of the various lithological units and mine wastes. The site provides an excellent comparison with other problematic coal mining sites.

Site description

The Etili open pit coal mine is located in Can County, approximately 40 km from Canakkale, Turkey (Fig. 1a). The lithological units can be classified into two groups: Oligocene-aged Can volcanic rocks (Ercan et al. 1995) and Early-Middle Miocene-aged Can formation (Hezarfen, Feasibility report of Çan-Çanakkale coal deposits, unpublished) (Fig. 1b). Can volcanic rocks (andesite, andesitic tuff, agglomerate, and trachyandesite lava), which comprise the basement lithology of the study area, have undergone different types of hydrothermal alteration, including silicic and argillic alterations. Can formation sedimentary rocks were deposited by calc-alkaline volcanic activity (Siyako et al. 1989). The Can formation contains heterogeneous lithologies, such as conglomerate, sandstone, claystone, coal, organic claystone, and tuff. Low-calorific-value coal is extracted by several private mining companies for local heating purposes.

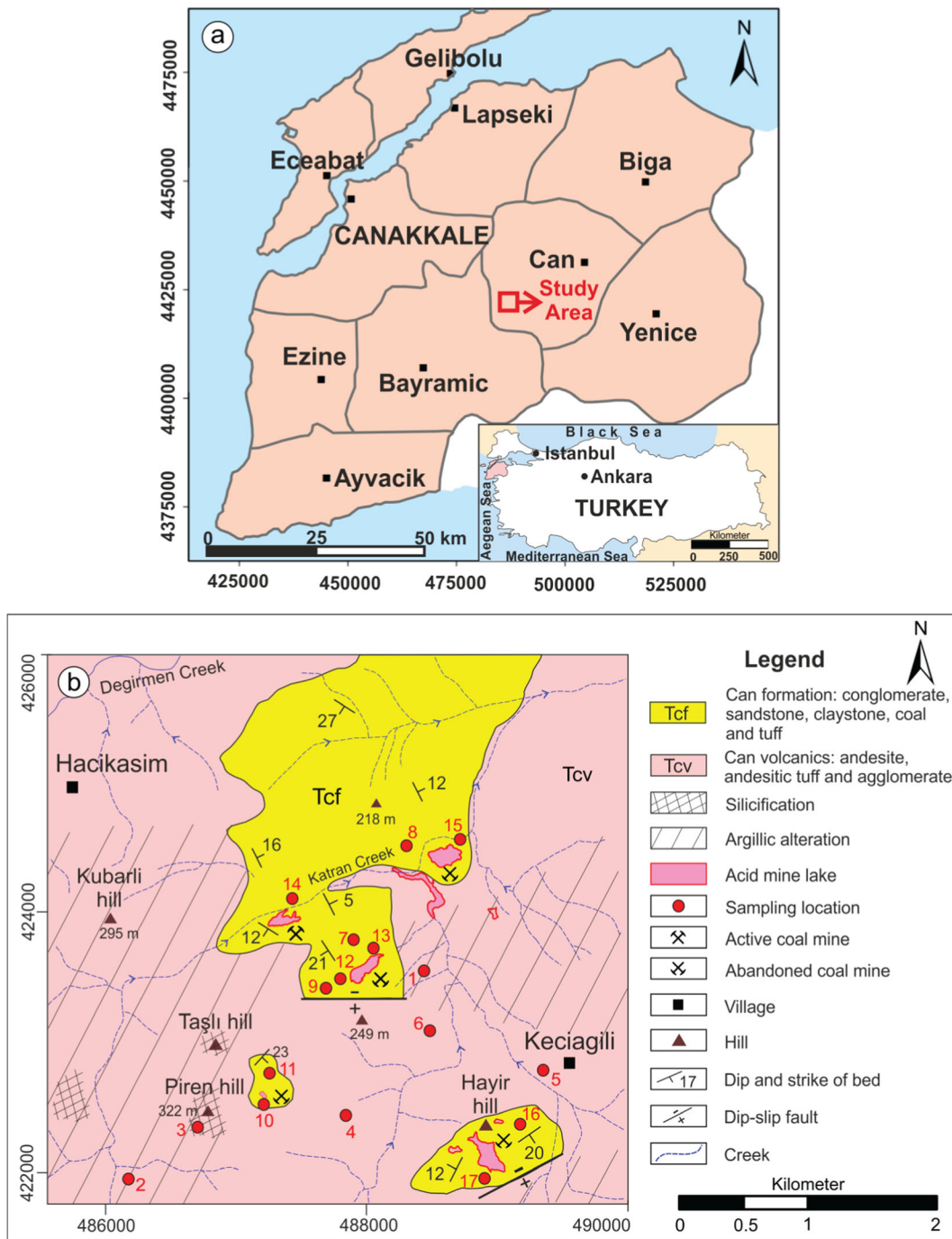


Fig. 1 a Location map and b geological map of the study area (Sanliyuksel Yucel et al. 2016) and sampling locations

The Etili coal mine contained nine acid mine lakes (AMLs) (Fig. 2), which had a total area of 11.42 ha in November 2014, according to unmanned air vehicle data (Yucel and Turan 2016). One lake was emptied because of reopening mining activities and the

remaining eight AMLs covered 9.82 ha in October 2015 (Yucel and Turan 2016). The pH and electrical conductivity values of the AMLs ranged from 3.01 to 4.24 and from 2380 to 5690 $\mu\text{S cm}^{-1}$, respectively (Sanliyuksel Yucel and Yucel 2016). The maximum Fe



Fig. 2 Panoramic snapshot from AMLs and waste heaps from Etili coal mine

and Al concentrations of the lakes were 338 to 357 mg L⁻¹, respectively (Sanliyüksel Yucel et al. 2014). The lakes are classified as class IV with respect to their pH, SO₄, Al, Fe, Mn, Ni, and Zn concentrations, in accordance with the Water Pollution Control Regulations in Turkey (SKKY 2004); in other words, they are considered to be highly polluted (Sanliyüksel Yucel and Yucel 2016).

The AMLs are situated upstream of the Kocacay River watershed of Can County, which is rich in water resources. Local drinking and irrigation water resources are supplied from an alluvium aquifer that outcrops along the Kocacay River (Okumusoglu and Gunduz 2013; Sanliyüksel Yucel et al. 2016). There are many small creeks, such as the Katran Creek, connected to the Kocacay River, where the water has a neutral pH. Because the lakes drain into Katran Creek, the pH of the creek is decreasing. Sanliyüksel Yucel and Baba (2013) stated that the creeks around Etili coal mine ranged from pH 2.9 to 5.8. They also have a reddish-brown color because of the high Fe concentration

(161 mg L⁻¹); Al, Mn, and Ni concentrations of creek water have reached 212, 40, and 0.8 mg L⁻¹, respectively (Sanliyüksel Yucel and Yucel 2016). Local media has widely reported on the death of aquatic life as a result of seepage and discharge of the lake waters into surface waters in the Etili region. This site represents an important risk to drinking water resources via water flow paths from this low-pH, high-metal concentration source.

Materials and methods

Field studies and sampling

The spatial distribution of the sampling locations is given in Fig. 1b. Based on field observations and previous studies, 17 representative rock samples were collected from exposed lithological units, particularly mining faces and rock outcrops, and mine waste samples were obtained from coal mine waste dumps to assess their acid generation potential. Approximately 5 kg of

each sample was collected in plastic bags. The sampling includes specifications for chain of custody procedures and documentation consistent with quality assurance and quality control (QA/QC) procedures.

Experimental methods

The experimental procedures for the static tests differed with respect to the aging period, sample grain size, and type and amount of acid used. Replicate samples, standards, certified reference materials, and blanks routinely submitted to ensure and confirm that the analytical results are of acceptable quality in all experiments. Details of the experimental procedures are provided below.

Paste pH and electrical conductivity

The paste pH test is used to determine the acidic nature of rock and waste samples and provides an indication of immediate pH characteristics on mixing a crushed sample with water (Weber et al. 2006a). Lapakko (2002) emphasized that paste pH is a common and simple field test used to assess the presence of acid-soluble salts in mine wastes. Electrical conductivity (EC) provides an indirect measure of the quantity of water-soluble salts in the sample. A pH value below 4 generally indicates that the sample is acid generating and an EC value of greater than $2000 \mu\text{S cm}^{-1}$ indicates a high level of total dissolved solids in the waste leachates (Lottermoser 2010).

Paste pH tests were performed to obtain preliminary information on the immediate reactivity of minerals present in each sample using the Sobek procedure (Sobek et al. 1978). Before testing, the samples were ground to a particle size of less than 1 mm (18 mesh). The samples (20 g each) were placed in 50-mL beakers. Ultrapure water of the same mass as the sample (20 mL, 1:1 solid/solution ratio) was added with the aid of a digital burette and mixed with a glass rod for 10 s to make a paste. The paste pH was then measured by inserting a pH electrode into the paste. The first measurement was made at 10 min, with measurements repeated after 20 min, 1 h, 24 h, 48 h, 1 week, 2 weeks, and 3 weeks. The reaction was allowed to continue after each measurement by providing an oxygen environment: the solution was mixed for 10 s and pH and EC were measured with a Multi 340i instrument

(Wissenschaftlich-Technische Werkstätten, Weinheim). The experiments were concluded at the end of the third week.

Standard acid–base accounting

Standard acid–base accounting (ABA) is the most commonly used static test to predict AMD from mine wastes (Yeheyis et al. 2009). The ABA test evaluates the balance between maximum acid-generating processes (oxidation of sulfide minerals) and acid-neutralizing processes (dissolution of alkaline carbonates, displacement of exchangeable bases, and weathering of silicates) (Sobek et al. 1978; Ritcey 1989; Skousen et al. 2002; Yeheyis et al. 2009; Banerjee 2014). The maximum acid-producing potential (MAP) of the sample is calculated on the basis of its total sulfur (S) content: for each 1 % S contained in the material, 31.25 metric tons (t) of calcium carbonate are required for neutralizing the sulfuric acid produced from 1000 t of the material (Sobek et al. 1978; Skousen et al. 2000). The MAP value (in kg CaCO_3/t) was calculated according to the formula proposed by Sobek et al. (1978): $\text{MAP} = \text{S (wt\%)} \times 31.25$. This calculation assumes that all S is contained in pyrite, which is a reasonable approximation (Sobek et al. 1978; Weber et al. 2004, 2006a, b; Hughes et al. 2007).

The neutralization potential (NP) test measures the amount of acid that the sample can neutralize (Lottermoser 2010). This is determined by analyzing the acid consumption of a sample prepared with a particle size less than $250 \mu\text{m}$ (60 mesh). Accurately weighed 0.5-g sample was prepared for a fizz test then a few drops of 25 % HCl were dropped onto it. The density and size of the bubbles formed by the reaction that occurred when the acid solution contacted the sample were observed, and the appropriate volume and molarity of the acid to be used in the test were determined from Table 1. Accurately weighed 2-g samples were placed in a 250-mL conical flask, and the volume and molarity of acid identified from Table 1 were added. The flask was then heated in a water bath at a fixed temperature of $85 \text{ }^\circ\text{C}$ and shaken at 5-min intervals. The flask was left in the water bath for at least 2 h or until the reaction was completed (until no further bubbles or gas were released). Pure water was then added to make up the volume

Table 1 Description of fizz rating and amounts and strengths of HCl (Sobek et al. 1978)

Fizz rating	Description	Amount of acid (mL)	Strength of acid (M)
None	No reaction	20	0.1
Slight	Minimal reaction; a few bubbles per second to many fine bubbles	40	0.1
Moderate	Active bubbling with only a small amount of splashing	40	0.5
Strong	Very active bubbling that includes substantial splashing	80	0.5

to 125 mL and the solution left in the water bath for another minute. The flask was then firmly closed and left to cool to ambient temperature (28–30 °C). Standardized NaOH solution of the same concentration as the HCl was added to the cooled solution and back-titrated to pH 7.0 to determine the amount of acid consumed by the sample.

Acid consumed in the digestion of the neutralizing species was used to calculate NP according to (Mendez-Ortiz et al. 2007):

$$NP = \frac{50 \times [(a_M \times v_A) - (b_M \times v_B)]}{s_W},$$

where NP is the neutralization potential (kg CaCO₃ t⁻¹), a_M is the molarity of acid (M), b_M is the molarity of base (M), v_A is the volume of acid (mL), v_B is the volume of the base (mL), and s_W is the mass of the sample (g).

Net acid generation test

The net acid generation (NAG) test is based on the principle that hydrogen peroxide accelerates the oxidation of iron sulfide minerals (Miller et al. 1990). The acid produced dissolves neutralizing minerals present, and the net result of the acid production and neutralization can be measured directly (Brough et al. 2013). This test does not require S determinations and is therefore more readily conducted in a field laboratory than other static tests (Lapakko 2002).

Samples with particle sizes less than 75 μm (200 mesh) were prepared. The pH of the H₂O₂ must be between 4 and 7; if the pH is below 4, it is recommended that dilute NaOH be added until a value between pH 4 and 6 is reached (Mills 2007). The pH of the H₂O₂ solution was measured and recorded as 3.25. With the aid of a digital burette, 5 mL of 0.025 M NaOH was added and the pH value raised to 4.65. The accurately weighed 2.5-g pulverized sample was placed in 500-mL

conical flask, 250 mL of 15 % H₂O₂ was added, and watch glass was placed on the top of the flask. The solution was left under a fume hood for at least 2 h while reactions continued (until no further bubbles or gas were released) and then placed in a 70 °C water bath. The heating procedure served to remove excess peroxide and to reveal the current neutralizing ability of the sample (Stewart et al. 2006). The solution was left in the bath for at least 2 h or until the reactions were complete, then removed and left to cool. When the solution reached ambient temperature, pH value of the solution (NAGpH) was measured and recorded before titration with standardized NaOH. Miller et al. (1997) stated that if the solution pH exceeded 2.5, 0.1 M NaOH should be employed; otherwise, 0.5 M NaOH should be chosen. Pure water was added to bring final volume up to 250 mL and the solution was back-titrated to pH 4.5 and then pH 7 to determine the net acidity produced by the acid-generating and acid-neutralizing reactions occurring in the sample (NAG), as outlined in Lawrence (1990) and Miller (2000) (Akabzaa et al. 2007):

$$NAG = \frac{49 \times v_B \times b_M}{s_W}$$

where NAG is the net acid generation (kg H₂SO₄ t⁻¹), v_B is the volume of the base (mL), b_M is the molarity of base (M), and s_W is the mass of sample (g).

Geochemical analysis

Mine waste and sedimentary and volcanic rocks were analyzed for total S and major element content at ACME Laboratories (Canada). Major and trace element concentrations were determined by inductively coupled-plasma mass spectrometry (SCIEX-ELAN 9000; PerkinElmer). Total S was determined via infrared adsorption using a LECO C–S analyzer. Mineralogical analyses of coal, claystone, mine waste, and volcanic rock samples were performed by X-ray diffraction

(XRD) using a Philips PW 1830 instrument at the General Directorate of Mineral Research and Exploration Analysis Laboratories in Ankara, Turkey. Scanning electron microscopy (SEM) (Philips FEI XL30-SFEG) coupled with energy-dispersive X-ray spectrometry (EDX) was performed at the Center for Material Science at the Izmir Institute of Technology, Turkey.

Results and discussion

Mineralogical and geochemical characterization of rock samples and mine wastes

The geology of the study area and its immediate vicinity has created a suitable environment for numerous mineral deposits. Coal and kaolin are two economically important resources extracted from the region. According to geochemical data, the SiO₂ content of the andesite rocks ranged from 55.9 to 57.2 %, with Al₂O₃ of 17.6–18.2 % and Fe₂O₃ of 6.9–8.0 % (Table 2). XRD analyses of the argillic altered andesite samples indicated that these included kaolinite, pyrite, and illite/mica-, feldspar-, and smectite-group minerals (Table 3). Yigit (2009) concluded that alteration zones of Can volcanic rocks contained a large volume of disseminated and fracture-fill pyrite. Argillic-altered andesite rock, which includes cubic pyrite crystals smaller than 1 μm, was observed by SEM (Sanliyüksel Yucel et al. 2016).

Oxides of the major elements in Can coals are dominated by SiO₂ (24.8–30.1 %), Al₂O₃ (10.4–14.8 %),

Fe₂O₃ (5.5–6.3 %), and others, including MgO, CaO, Na₂O, TiO₂, K₂O, P₂O₅, MnO, and Cr₂O₃, each of which comprise less than 1.0 %. The minerals present in the coal samples comprised quartz, kaolinite, amorphous matter, pyrite, gypsum, cristobalite, and smectite-, zeolite-, and feldspar-group minerals. The total inorganic mineral content of the coal was 19 %, comprising 5.0 % gypsum, 4.8 % pyrite, 4.5 % kaolinite, 3.7 % quartz, and 0.9 % smectite-group clay minerals (Tuncali et al. 2002). Pyrite is observed in framboidal and cubic crystal forms and common mineral in Can formation coal. Lustrous pyrite was observed in macroscopic quantities (Fig. 3a). Well-developed euhedral pyrite crystals are shown by SEM (Fig. 3b), containing 65.3 % S and 34.7 % Fe, as quantified by EDX analysis (Fig. 3c). Total S content of the coal ranged from 3.97 to 6.04 wt%; organic S was between 1.6 and 3.0 %, pyritic S comprised between 0.65 and 1.10 wt%, and sulfate S was between 0.85 and 1.54 wt% (Gurdal 2011). The presence of the high S content is attributed to the peat environment and regional volcanic activity, as well as to the alkaline depositional environment that has intensive sulfide mineralization (Gurdal and Bozcu 2011).

The major oxides in claystone, located in the same depositional environment with coal, are dominated by SiO₂ (48.0–52.3 %), Al₂O₃ (15.2–18.2 %), and Fe₂O₃ (3.0–4.1 %). XRD analyses of the claystone indicated that this composed of kaolinite group minerals, opal-CT, quartz, pyrite, feldspar-group minerals, and gypsum. Sulfur layers associated with iron oxide in Can formation can be easily distinguished in the field indicated by the

Table 2 Major element concentrations of rocks and mine wastes (%)

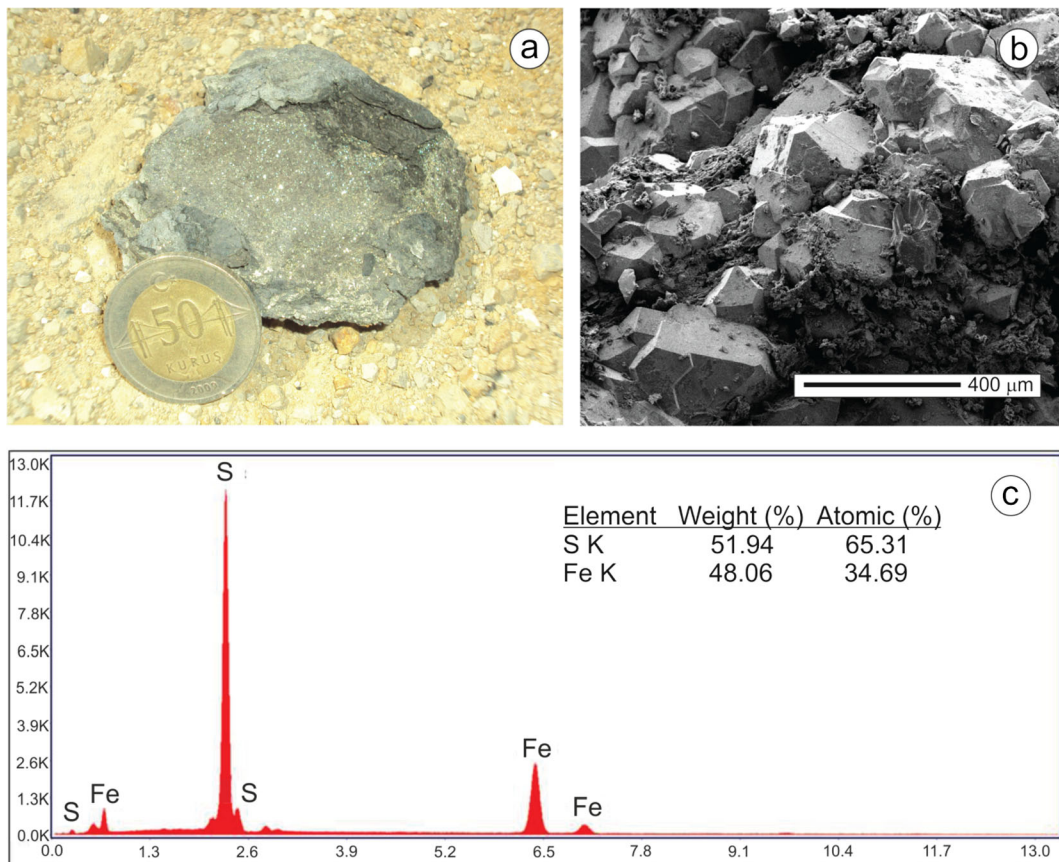
Sample name	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	MnO	Cr ₂ O ₃	LOI	Sum
Andesite	55.87	17.56	6.89	1.08	3.06	2.85	3.77	0.75	0.09	0.03	0.002	7.5	99.45
	57.23	18.19	8.04	0.56	0.92	1.01	4.31	0.65	0.10	0.08	0.003	8.7	99.79
Coal	24.78	10.4	6.25	0.18	0.89	0.25	0.44	0.12	0.10	0.01	0.004	55.9	99.32
	30.12	14.75	5.46	0.25	1.16	0.29	0.33	0.36	0.05	0.01	0.005	47.1	99.88
Claystone	52.26	18.22	3.01	0.81	3.47	0.51	1.85	1.01	0.29	0.001	0.003	18	99.43
	48.01	15.21	4.08	0.75	4.83	0.41	1.29	0.94	0.47	0.002	0.004	23.8	99.79
Mine waste	44.61	14.13	6.57	2.09	0.22	1.14	0.93	0.64	0.14	0.002	0.004	28.9	99.37
	46.50	13.9	4.85	1.44	0.41	1.27	1.39	0.17	0.08	0.001	0.004	29.2	99.21

Table 3 XRD results of rocks and mine wastes

Sample name	Explanations
Andesite	Kaolinite, quartz, illite/mica-group minerals, feldspar-group minerals, pyrite Kaolinite, smectite-group minerals, pyrite, feldspar-group minerals, illite/mica-group minerals
Coal	Quartz, opal-CT, gypsum, pyrite, kaolinite, mica/illite, smectite-group minerals, feldspar-group minerals Kaolinite, amorphous matter, quartz, pyrite, gypsum, zeolite-group minerals
Claystone	Kaolinite-group minerals, opal-CT, quartz, pyrite, feldspar-group minerals, gypsum Kaolinite-group minerals, cristobalite, quartz, opal-CT, gypsum, pyrite
Mine waste	Quartz, cristobalite, amorphous matter, kaolinite-group minerals, alunite, feldspar-group minerals, illite/mica-group minerals, chlorite-group minerals, pyrite, gypsum Kaolinite-group minerals, illite/mica-group minerals, opal-CT, gypsum, quartz chlorite-group minerals, feldspar-group minerals, pyrite, jarosite, goethite

yellow color (Fig. 4a). Jarosite exhibits small ($<1 \mu\text{m}$) and sub-rounded crystals in SEM images (Fig. 4b). EDX analysis of the mineralized S layer shown in Fig. 4c indicated that Fe, S, K, and O are the main components.

The major oxides in mine waste were dominated by SiO_2 , Al_2O_3 , and Fe_2O_3 , in the ranges of 44.6–46.5, 13.9–14.1, and 4.9–6.5 %, respectively. The mine wastes generally contained quartz, cristobalite, pyrite, opal-CT, gypsum, and illite/mica-, kaolinite-, chlorite-,

**Fig. 3** a Field view, b SEM image, and c EDX spectra of cubic pyrite crystals in coal

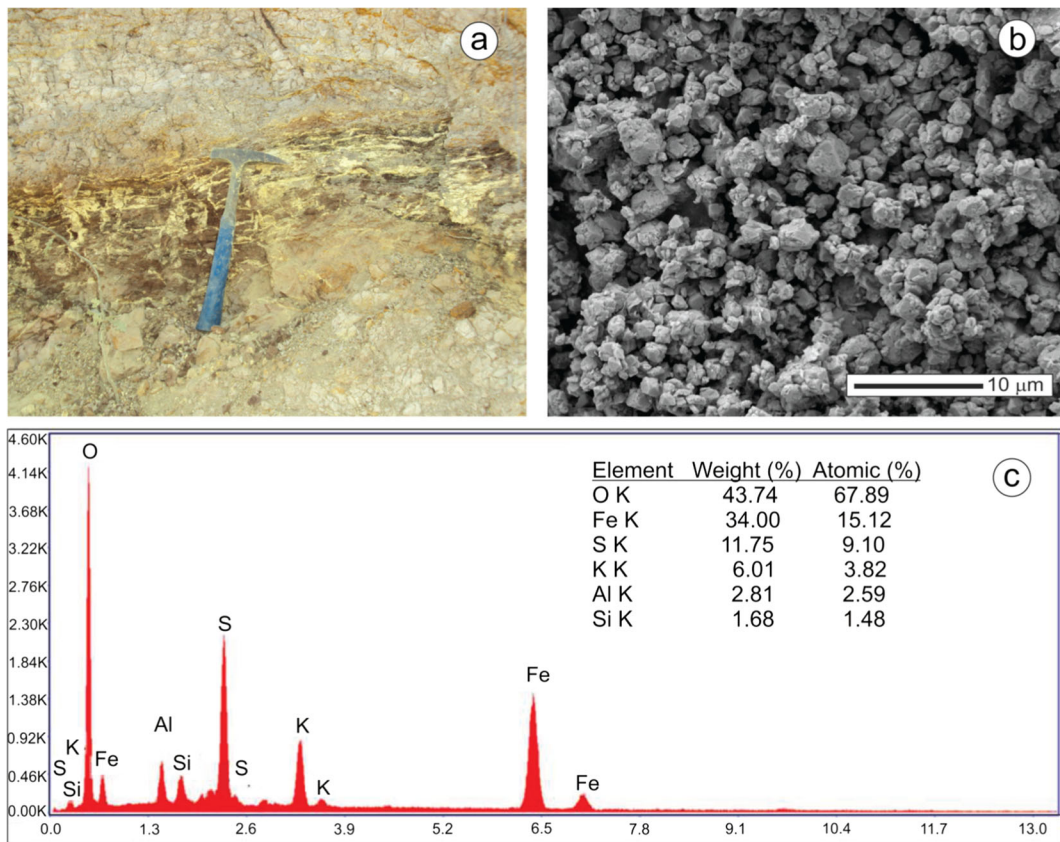


Fig. 4 a Sulfur layer associated with iron oxide in Can formation. b SEM image of jarosite. c EDX spectra of jarosite containing aluminum silicate

and feldspar-group minerals, in addition to jarosite and alunite. The mine wastes contained 1.69 % total S, including 0.35 % pyritic S, 0.71 % sulfate S, and 0.63 % organic S. The total area of mine wastes around Etili coal mine was calculated approximately 1 km² using satellite images in 2013 (Sanliyüksel Yucel and Yucel 2016). The mine waste consists of sand to silt-clay in particle size (29 to 76 % passing a 75-µm sieve). Because of mining activities, shrinkage of the grain size and increasing of the surface area of this geological unit have the potential to accelerate the acid generation process by facilitating the oxidation of sulfide minerals.

Hutt and Morin (1999) stated that if the total S content of a rock is greater than 0.3 %, it oxidizes rapidly if exposed to water and oxygen and may produce AMD. In this study area, the total S content of the rock samples and mine waste ranged from 0.08 to 6 %. Altered volcanic rocks, coal, and mine wastes ($\Sigma S > 0.3 \%$) therefore have a strong capacity

for AMD generation. All investigated lithologies contained pyrite minerals; the mine wastes also contained highly soluble metal sulfates, such as jarosite and alunite minerals, which provide further acidity. Secondary minerals may control the composition of AMD. Gypsum probably occurs as a consequence of secondary oxidation of sulfides. Gypsum, iron hydroxides, and other secondary minerals that have slowly precipitated on the surface of the pyrite can form a coating that may impede further reaction with water and oxygen (Villeneuve et al. 2003).

Carbonate minerals are the largest source of neutralization potential in extractive waste (Kaartinen and Wahlström 2009). Calcite is the most important neutralizing agent because of its common occurrence in a wide range of geological environments and its rapid reaction rate (Lottermoser 2010). Paktunc (1999) stated that a waste material to be considered as having no AMD potential, it must contain at least

12 times as much calcite as pyrite. Carbonate minerals were, however, not determined by geochemical analysis in these samples: only silicate minerals, such as chlorite, feldspar, kaolinite, mica, quartz, zeolite, and opal-CT, were identified. It is known from field observations, and has been quantified in laboratory studies, that most silicates and aluminosilicates react slowly relative to the rates of sulfide oxidation and carbonate dissolution (Jambor et al. 2002).

Acid generation prediction using static tests

Paste pH and electrical conductivity

The paste pH is indicative of the present drainage of pH of a sample and acidic drainage will only result when the rate of acid generation exceeds the rate of acid neutralization (Price 2009). The paste pH of the mine waste samples ranged from 2.3 to 3.8 (Table 4), and this value is similar to pH value

of creeks around Etili coal mine reported by previous studies. The lowest paste pH value of 2.1 was measured in coal. The paste pH value of volcanic rock samples ranged from 3.1 to 8.3. Unaltered andesite (sample 5) had a pH value of 8.3, indicating no risk for acid generation potential. Paste pH values of selected samples (argillic-altered andesite, coal, and claystone) were less than the critical level of pH 4.0, implying that considerable dissolution occurred within the first 10 min (Table 5). Initial low pH can be related to oxidation that occurs on the surface of the material. A slight increase in pH with time indicated the dissolution of neutralizing minerals. The presence of late-dissolving silicate minerals may be indicated. EC value of the solutions increased with time. The highest EC value ($6045 \mu\text{S cm}^{-1}$) was measured in coal at the end of the third week (Fig. 5 and Table 5), indicating that the highest ion transfer to water occurred from coal. The pH

Table 4 Summary of the static test results

Sample Name	Lithology	Alteration	Paste pH	EC	Total sulfur	MAP	NP	NNP	NPR	NAGpH	NAG ($\text{kg H}_2\text{SO}_4 \text{ t}^{-1}$)		AMD classification
				$\mu\text{S cm}^{-1}$	wt%	kg $\text{CaCO}_3 \text{ t}^{-1}$					pH = 4.5	pH = 7	
1	Andesitic tuff	Argillic	3.1	1285	1.70	53.12	2.05	-51.07	0.03	2.91	13.48	32.57	PAF
2	Andesite		3.9	1070	1.16	36.25	2.47	-33.77	0.06	3.56	6.40	29.41	PAF
3	Andesite	Silicic	5.5	417	0.22	6.87	4.32	-2.55	0.62	4.02	4.91	24.95	PAF
4	Andesite	Unaltered	6.5	264	0.11	3.43	60.87	57.43	17.70	4.85	-	16.11	NAF
5			8.3	122	0.08	2.50	67.87	65.37	27.15	5.60	-	8.58	NAF
6			6.9	135	0.17	5.31	81.12	75.81	15.27	4.62	-	11.62	NAF
7	Claystone	-	3.0	1640	1.03	32.18	0.65	-31.53	0.02	2.75	15.38	38.41	PAF
8			3.9	958	0.42	13.12	1.35	-11.77	0.10	3.40	5.29	27.53	PAF
9	Coal		2.4	2890	4.02	125.62	0.32	-125.3	0.002	2.13	25.81	45.57	PAF
10			2.1	3510	5.98	186.87	0.22	-186.65	0.001	1.72	27.36	50.23	PAF
11	Mine waste		3.2	1295	0.71	22.18	2.10	-20.08	0.09	2.72	19.28	36.08	PAF
12			3.8	960	0.65	20.31	0.97	-19.33	0.04	3.12	8.23	27.00	PAF
13			2.3	3135	2.74	85.62	2.02	-83.6	0.02	2.68	23.22	44.59	PAF
14			2.7	2260	1.38	43.12	3.07	-40.05	0.07	2.99	13.07	34.12	PAF
15			2.6	2450	1.07	33.43	2.07	-31.362	0.06	3.18	9.76	30.71	PAF
16			2.5	2710	1.69	52.81	1.37	-51.43	0.02	3.01	10.64	29.18	PAF
17			2.7	2325	1.27	39.68	1.82	-37.86	0.04	3.10	8.31	28.94	PAF
Minimum			2.1	122	0.08	2.50	0.22	-186.65	0.001	1.72	4.91	8.58	
Median			3.1	1295	1.07	33.43	2.05	-31.53	0.06	3.10	11.85	29.41	
Maximum			8.3	3510	5.98	186.87	81.12	75.81	27.15	5.60	27.36	50.23	

PAF potentially acid forming, NAF non-acid forming

Table 5 Paste pH and EC values of some selected samples

Time (h)	Paste pH			EC ($\mu\text{S cm}^{-1}$)		
	Coal	Clay	Andesite (argillic altered)	Coal	Clay	Andesite (argillic altered)
0.16	2.2	3.9	3.2	52	1060	1092
0.33	2.0	3.9	3.1	140	1100	1190
1	2.0	3.8	3.1	221	1107	1477
24	1.7	3.7	3.1	2070	1118	1682
48	1.7	3.8	3.0	3755	1182	1711
168	1.8	3.7	3.0	5085	1610	2472
336	1.9	3.7	3.0	5995	1837	2635
504	1.9	3.7	3.0	6045	1901	2872

and EC values of selected samples suggested the potential for high acid generation under appropriate conditions.

To ensure that the experimental processes simulated ambient conditions, rain water was sampled during field work in December 2012. The pH value of rain water was measured as 5.9. The paste pH test was applied to mine waste using rain water and ultrapure water. When rain water was contacted with waste, the pH value was 2.5 after 10 min, increasing

to pH 2.7 in the third week (Table 6). The paste pH values using rain water and pure water were compatible (Fig. 6). The analogous EC values were also compatible at the end of the third week, reaching maximum values of 4205 and 4395 $\mu\text{S cm}^{-1}$, respectively. The different EC values are attributed to the different initial pH values of the waters; furthermore, the measurement temperatures and the samples were not completely identical.

Acid–base accounting tests

Determination of the NP that will be available under ambient conditions is crucial for the development of waste management plans to prevent and control the generation of AMD from mine wastes and other mine components during mining operations and after mine closure (Lawrence and Scheske 1997). The NP values of

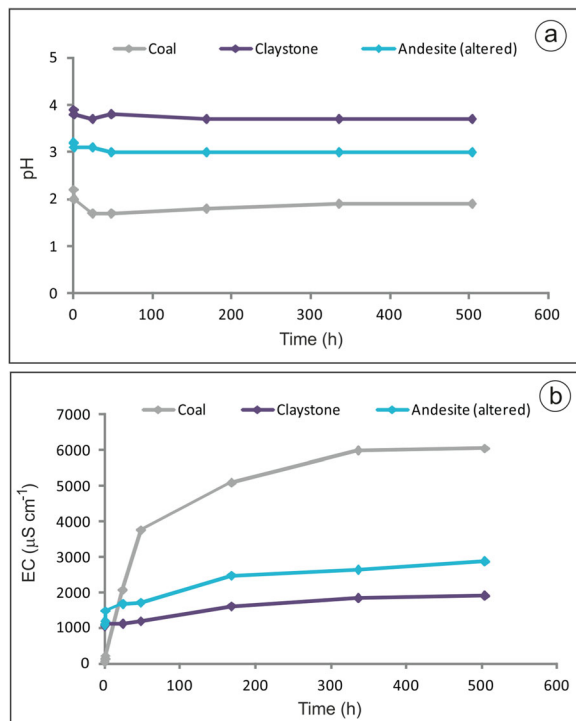


Fig. 5 Time versus **a** paste pH and **b** EC values of some selected samples

Table 6 Paste pH and EC values of mine waste

Time (h)	Paste pH		EC ($\mu\text{S cm}^{-1}$)	
	Ultra pure water	Rain water	Ultra pure water	Rain water
0.16	2.4	2.5	4200	2870
0.33	2.4	2.5	4395	2900
1	2.4	2.5	4575	3185
24	2.4	2.6	4605	3520
48	2.5	2.6	4485	3625
168	2.6	2.7	4405	4000
336	2.6	2.7	4405	4155
504	2.6	2.7	4395	4205

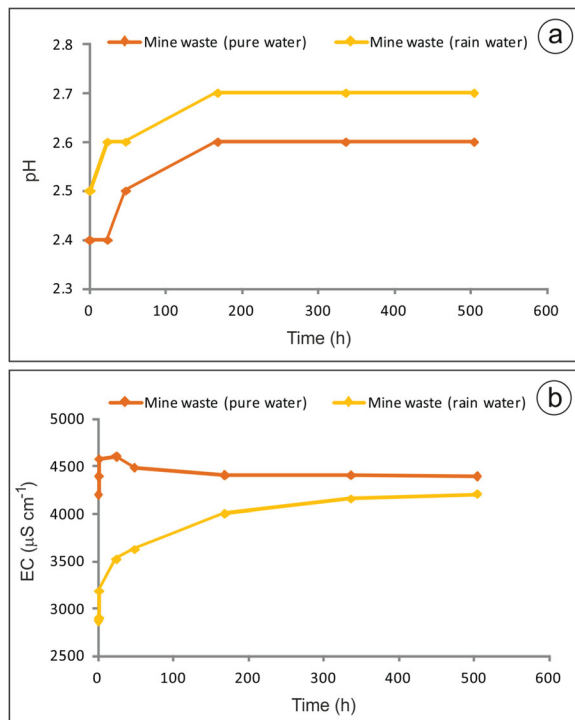


Fig. 6 Time versus **a** paste pH and **b** EC values of mine waste

various lithologies in Etili coal mine ranged from 0.22 to 81.12 with a median of $2.05 \text{ kg CaCO}_3 \text{ t}^{-1}$ (Table 4). Net neutralization potential (NNP) was calculated by subtracting the MAP from NP (Villeneuve et al. 2003). The NNP value is one of the criteria used to determine the acid production potential of a sample (Lottermoser 2010): theoretically, rocks with positive NNP values have no potential for acidification, whereas rocks with negative NNP values do. In practice, a safety factor is applied and rocks with a significantly positive NNP value are generally regarded as having no acidification potential (greater than $+20 \text{ kg CaCO}_3 \text{ t}^{-1}$); rocks with a significant negative NNP value (less than $-20 \text{ kg CaCO}_3 \text{ t}^{-1}$) are potentially acid generating. Materials with intermediate NNP values have uncertain acid generation potential ($-20 \text{ kg CaCO}_3 \text{ t}^{-1} < \text{NNP} < +20 \text{ kg CaCO}_3 \text{ t}^{-1}$) (Ferguson and Morin 1991; Miller et al. 1991; Lottermoser 2010). According to this classification, unaltered andesite (samples 4, 5, and 6) had no acidification potential, silicic altered andesite (sample 3) and claystone (sample 8) were uncertain, and all other samples were potentially acid generating.

The neutralization potential ratio (NPR), defined as (NP/MPA) (Price et al. 1997; Mendez-Ortiz et al. 2007), was used as the criterion to evaluate the capacity of a

material to generate AMD. If the NPR value is <1 , the sample is said to be acid generating, and if the NPR is between 1 and 2 ($1 < \text{NPR} < 2$), the sample is considered to be potentially acid generating. If the sample has an NPR between 2 and 4, then it is potentially acid consuming. If the NPR is greater than 4, the sample is considered to be acid consuming (Price and Errington 1998; Skousen et al. 2002). NPR values ranged between 0.001 and 27.15, and according to this classification, 14 samples were categorized as acid generating (Fig. 7). The NPR values of unaltered andesite (samples 4, 5, and 6) were higher than 4, and these samples were considered to be acid consuming.

According to the assumptions of the ABA test, acid-consuming minerals are completely reacted in nature and the rate of dissolution of acid consumers is the same as that of acid producers. MAP is calculated on the basis of the total S content. The presence of S in the mineralogical composition of a rock indicates that it may have acid generation potential, but this does not mean that that all S dissolves completely. Owing to the presence of non-acid-producing S-containing minerals (e.g., gypsum) in the study area, the calculated MAP results were higher than usual. In addition, complete dissolution during the experimental process, especially dissolution of silicate minerals, will never occur: the experimental NP values were therefore lower than the actual values.

Net acid generation test

According to experimental results of NAG test, oxidation of pyrite, pyrrhotite, arsenopyrite, and chalcopyrite

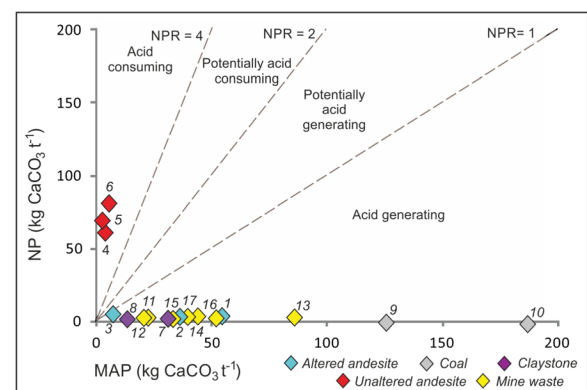


Fig. 7 NPR screening criteria

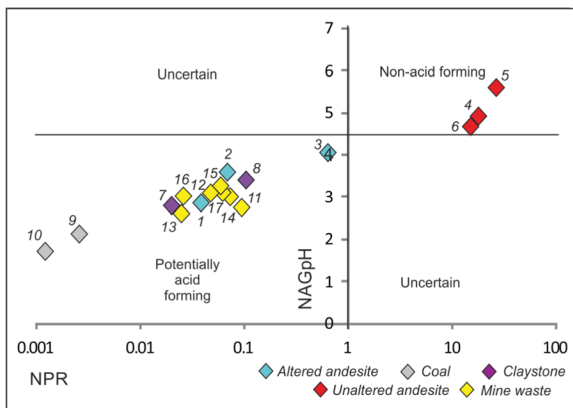


Fig. 8 NPR versus NAGpH

produces a final NAG solution with pH lower than 4.5 (Stewart et al. 2003). Pyrite is a common mineral in the alteration zones of volcanic rocks, coals, and mine wastes and NAGpH of these lithologies were measured lower than 4.5 in this study (see Table 4). Charles et al. (2015) stated that in general, a NAGpH less than 4.5 and a NAG value greater than 5 kg H₂SO₄ t⁻¹ are indicative of a potentially acid-forming material. This criterion is compatible with our results.

The samples were subsequently classified using combined ABA/NAG classification system (Fig. 8). NPR was compared with NAGpH to discriminate between non-acid-forming samples and potentially acid-forming samples. Unaltered andesite (samples 4, 5, and 6) gave non-acid-forming classifications; all other samples were potentially acid forming. NPR results compared reasonably well with NAGpH results for potentially acid-forming samples, generally confirming that these may produce acidity on exposure to oxygen and water.

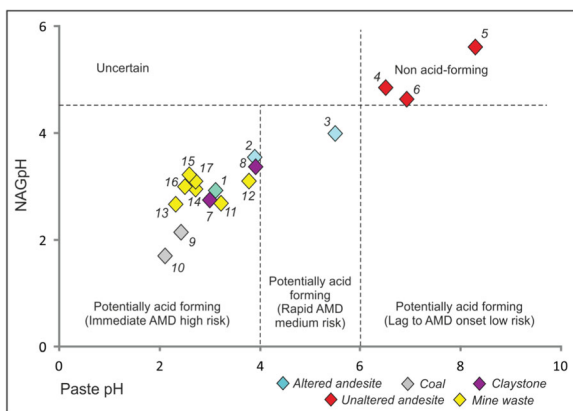


Fig. 9 Paste pH versus NAGpH

The samples of higher sulfur values lay in the lower left hand quadrant of the paste pH versus NAGpH classification (Fig. 9) and were potentially acid forming. However, unaltered rocks also lay upper right hand quadrant in the non-acid-forming region. According to this classification, unaltered andesite (samples 4, 5, and 6) was non-acid forming, while silicic altered andesite (sample 3) was in the “potentially acid forming (rapid AMD medium risk)” category; all other samples were considered to fall into the “potentially acid forming (immediate AMD high risk)” class. Both results identified unaltered volcanic rocks as non-acid generating.

Conclusions

AMD generation related to geological and anthropogenic processes is a common environmental problem in many countries, including Turkey. The abundance of pyrite in altered volcanic rocks, coal, and mine wastes was evidenced by field observations and XRD and SEM/EDX analysis. Oxidation of pyrite is the main factor controlling generation of AMD in the Etili region. Because of coal-mining activities, the sulfide minerals are liberated and are present as small particles in mine waste; these oxidize rapidly if exposed to water and oxygen. The generation of AMD is associated with mine wastes that have a major influence on the quality of drainage generated. Carbonate-group minerals were not determined by geochemical analysis. Silicate minerals are slow weathering and cannot effectively lower the acid levels alone. Static test results showed that the mine wastes and altered volcanic and sedimentary rocks, especially coals, have a strong capacity for AMD generation and are defined as potentially acid generating. Unaltered volcanic rocks are non-acid generating. Consistency between results from paste pH, ABA, and NAG tests was obtained. These tests (supported by geochemical and mineralogical analysis) are sufficient for the preliminary evaluation of AMD possibilities in any mining project in the Etili coal mine.

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