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# Leaching behavior of selected trace elements in coal fly ash samples from Yenikoy coal-fired power plants

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#### ABSTRACT

Short-term leaching test results of alkaline fly ash from Yenikoy coal-fired power plant were reported in this paper. ASTM D-3987-85 and TCLP-1311 test methods were applied to determine leaching behavior of selected elements namely, Fe, Ca, Cu, Co, Cd, Mn, Ni, Pb Zn, and Cr (VI) at different particle size fractions and test conditions. Chemical, mineralogical and morphological characterizations of ash samples were also performed using chemical, XRD and SEM-EDS analysis methods. The results showed that CaO dominates in the Yenikoy fly ash sample which directly affects the mobility of trace elements by determining the pH of the leaching medium. Higher mobility values of Cd, Co Cu, Pb, Ni and Zn elements were observed for TCLP-1311 procedure. The concentration of these elements in leachates showed a tendency to increase by decreasing particle size. Overall results suggested that the amount of the selected elements in the final leachates of both tests was lower than the limit values of landfill regulations except Cr (VI).

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## 1. Introduction

Coal has always been an important energy source for Turkey. As of today, the lignite based power plants have a production capacity of 20% of the total energy production [1,2]. Turkish lignites are typically low-grade coals which have very low calorific value and high mineral matter content [3,4]. It has been reported in various studies that extreme, high ash yields can reach that range from 23% to 64% [5,6], which produce huge quantities of coal fly ash to be disposed. According to the latest numbers, total amount of ash in ponds and landfills have already reached around 100 billion tons in Turkey [7,8]. Coal contains significant amount of various trace metals. Most of these trace elements are emitted largely in the fly ash or in association with the surface species of fly ash particles during coal combustion [9-12]. It has been reported that trace element concentrations in fly ash are sometimes 4-10 times higher than their original concentrations in coal samples. The reuse of coal fly ash for different purposes has spurred interest in understanding its environmental impact with focus on metal release [9,13]. Under favoring conditions, a considerable amount of these trace elements can easily be leached out from the surface of fly ash particles by the interaction with water in ponds or landfills [14,15]. There are large numbers of papers on prediction of leaching behavior of metals from coal fly ash. The researchers reported that the particle size, initial concentration of trace elements, pH of solution, leaching time, solid-liquid ratio and other

properties of the leaching environment are important factors affecting the mobility of the trace elements in aqueous environments [16–20].

Short-term leaching tests are capable of giving critical information in considerably shorter interaction periods and simple means to compare the dissolution behavior of various components [4,21]. Toxicity Characteristic Leaching Procedure (TCLP-1311) and Standard Test Method for Shake Extraction of Solid Waste with Water (ASTM D-3987-85) are commonly used methods for this purpose. These tests are considered as static methods of analysis, and can both be applied for the determination of short-term water solubility characteristics of fly ash samples [22–24].

This study focuses on short-term leaching behavior of Fe, Ca, Cu, Co, Cd, Mn, Ni, Pb Zn, and Cr (VI) from Yenikoy fly ash using TCLP-1311 and ASTM-3987-85 tests. The mobility of these elements was investigated on different size fractions of the ash samples. The concentrations in the final leachates were compared with the related limit values of landfill regulations.

#### 2. Materials and methods

#### 2.1. Fly ash sample

Yenikoy power plant uses 4.8 million tons of low quality lignite and generates 1.2 million tons of ash per year. The samples were collected, in a dry state, from the electrostatic precipitators and stored in airtight plastic containers.

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Particle size distribution of the Yenikoy fly ash was determined using Malvern Mastersize-S laser diffraction particle size analyzer. ELE brand Rigdens Flowmeter EL38-0500 was employed to determine the surface area of Yenikoy fly ash using the procedure described in ASTM C 204-11 [25].

The collected samples were dried at 105 °C for 24 h and ground using an agate mill to a particle size less than 100  $\mu m$  for mineralogical and chemical analysis. The crystalline minerals in the sample were identified using RIGAKU-Dmax-2200 PC equipment (Cu–K $\alpha$  radiation, 32 kV, 22 mA). Philips XL-30S FEG type scanning electron microscopy (SEM) system was used to examine morphology of the particles. The chemical composition of the ash samples were determined by Perkin Elmer AAS-2280 atomic absorption spectrometer. Concentration of total dissolved Cr (VI) was determined using the calorimetric method developed by USGS [26].

#### 2.2. Batch tests

Yenikoy fly ash sample was classified into different particle size fractions using appropriate sieves prior to use in the tests. These mono size samples were dried at 105 °C for 48 h and cooled down to room temperature in a desiccator to remove hygroscopic moisture. Two different toxicity test procedures were employed in the study. Each test procedure requires the application of special experimental conditions.

- TCLP-1311 test is applied to evaluate potential leachability of the toxic substances from the fly ash samples [22,24,27]. The tests were performed using 20:1 liquid-to-solid ratio, 40 g of fly ash from every size fractions was taken into acid-cleaned 1000 ml volume polyurethane bottles and 800 ml of extraction fluid (acetic acid solution) was added as described in US EPA protocol. The bottles were then subjected to horizontal shaking at 30 rpm in an oscillating shaker for  $18\pm2$  h and  $22\pm3$  °C.
- The water leaching test described in ASTM D 3987-85 is useful to evaluate the leaching potential of a material being exposed to normal precipitation. This method uses the liquid-to-solid ratio of 20:1. 40 g of fly ash samples was added to 800 ml of deionized water into acid-cleaned 1000 ml polyurethane bottles and agitated continuously at a rate of 29 rpm for 18 h at room temperature (25 °C).

After agitation on the shaker, the mixture was allowed to settle for 5 min for both methods, and then the aqueous phase separated by decantation. The pH of the leachates was determined immediately after collection. The leachates were then filtered through a 0.45 mm filter paper by vacuum filtration system, and acidified with nitric acid to pH 2. The sampling bottles were preserved of volume change and evaporation [23]. Element concentrations in the leachates were determined by using Perkin Elmer 2280 model atomic absorption spectrometer and Cr (VI) was measured at 540 nm with an UV-visible spectrophotometer.

#### 3. Result and discussion

## 3.1. Physical and chemical characterization of fly ash sample

Particle size is one of the important factors influencing the reaction kinetics in leaching of fly ash. The fine particles, particularly the very fine particles in coal ash play an important role in this process due to their large active surface area [28]. It may also show an effect upon chemical composition such as trace element distribution within the ash [29,30].

As depicted in Fig. 1, about 80% of the Yenikoy fly ash is under 45  $\mu$ m particle sizes. Volumetric mean particle size and 10% passing size values of the material were found as 28.41  $\mu$ m and 1.86  $\mu$ m respectively. Results of the physical characterization studies were

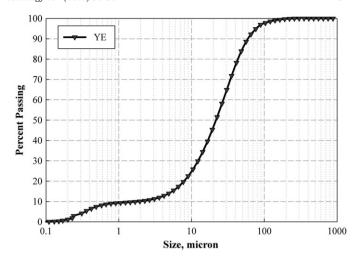


Fig. 1. Size distribution plots of Yenikoy fly ash.

found to be in agreement with the results of the study conducted by Bayat in 1999 [31].

Specific gravity as a function of porosity and surface area of the particles also influence the interactions between fly ash and aqueous solution. As seen from Table 1, specific gravity and surface area values of the sample were found as  $2.86 \text{ g/cm}^3$  and  $0.175 \text{ m}^2/\text{g}$ .

Natural pH value of fly ash is an important property which has a significant effect on mobility of trace elements in aqueous environment. Fly ash-water slurry was prepared by mixing 5 g of fly ash with 100 ml of deionized water for the determination of the pH value of the sample. The pH of slurry had been recorded at 1 h intervals for a period of 24 h using WTW brand pH/Cond. 340i portable water meter and final pH was found as 13.2. The basic character of ash leachate is generally attributed to the free calcium, soluble metal salts, oxides, hydroxides, and carbonates [4,32].

Chemical analysis of the fly ash sample was performed to determine the composition of material and results are given in Table 2. CaO was found as the major component in the ash sample which was mostly originated from calcite in feed coal. As it was stated by Koukouzas et al., total and free CaO are mostly generated through the thermal disruption of CaCO<sub>3</sub> [33]. As seen from Table 2, the sample contains significant amount of silicon, aluminum and iron elements due to the presence of clay minerals in feed coal. Relatively

**Table 1**Some physical properties of Yenikoy fly ash.

D [4,3] D(v, 0.1)		Specific gravity (g/cm <sup>3</sup> )	Specific surface area (m <sup>2</sup> /g)	pН
28.41	1.86	2.86	0.175	13.2

**Table 2** Concentration of major and trace elements in Yenikoy fly ash [34,35].

Elements (as oxides)	(%)	Trace elements	(mg/kg)
CaO	38.24	Zn	77.84
SiO <sub>2</sub>	20.79	Cd	14.90
$Al_2O_3$	17.26	Со	77.12
$Fe_2O_3$	7.17	Ni	73.69
MgO	4.66	Cu	72.08
SO <sub>3</sub>	12.8	Pb	149.81
Na <sub>2</sub> O	2.19	Mn	262.49
K <sub>2</sub> O	1.33	Cr	220.13
BaO	0.13		



Fig. 2. XRD analysis results of the ash samples (Q = quartz (SiO<sub>2</sub>), A = anhydrite (CaSO<sub>4</sub>), L = lime (CaO), H = hematite (Fe<sub>2</sub>O<sub>3</sub>)).

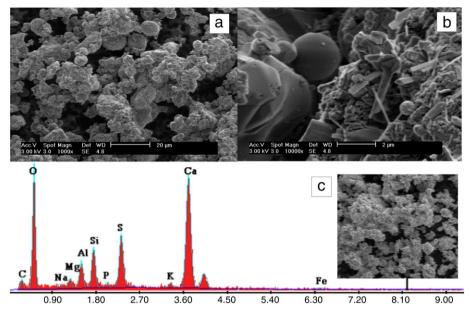


Fig. 3. (a, b) SEM image of general view of Yenikoy fly ash, (b) SEM image of a surface detail, and (c) SEM image and X-ray energy dispersed spectrum in the field.

high concentrations of Pb (149.81 mg/kg), Cr (220.13 mg/kg) and Ni (73.69 mg/kg) were also detected in the fly ash.

## 3.2. Mineralogical characterization of Yenikoy fly ash sample

X-ray diffraction analysis was performed to determine the mineralogical properties of the fly ashes. The analyses were carried out using RIGAKU-Dmax-2200 PC equipment (Cu–K $\alpha$  radiation, 32 kV, 22 mA) and the results are shown in Fig. 2.

As can be seen from Fig. 2, Yenikoy ash consists of quartz, anhydrite, lime (free and total) and hematite minerals. CaO and CaSO $_4$  minerals were detected as the major source of calcium in the sample. Quartz was observed as the dominating silicon compound, and iron was identified as in the form of Fe $_2$ O $_3$  mineral. Zhao et al pointed out that calcium oxide is one of the characteristic phases in the combustion products of a high calcium coal [36]. As mentioned by Vamvuka and Kakaras, anhydrite

was generally derived from fixing sulfur dioxide with lime and/or dehydration of gypsum in coal [37].

Morphological property of the particles affects the mobility of the trace elements in fly ashes. The particles having relatively high density and nonporous outer surface may resist leaching of heavy metals [4,20]. That's why, SEM analysis was applied to find out morphological properties of the particles using a Philips XL-30S FEG type instrument at different magnifications and results are given in Fig. 3.

As shown in Fig. 3a and b, the sample contains individual particles and agglomerates of particulates in spherical or irregular shape as it was reported by Bayat [31]. Those particles are considered to be generated by the fusion of smaller fragments and incomplete melting process. A number of individual particles were observed in the range of 1 and 10  $\mu m$  and many of them with a diameters less than 2  $\mu m$  (Fig. 3a and b). As can be seen from Fig. 3c, high amount of Ca, Si, Al was detected in fly ash.

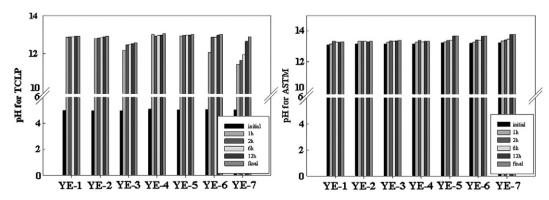


Fig. 4. (a) Variations in the pH value of the solutions during the TCLP tests, and (b) variations in the pH value of the solutions during the ASTM tests.

**Table 3a**The concentration of heavy metals in Yenikoy TCLP leachates (mg/kg).

Samples	YE-1 (+212 μm)	YE-2 (212–150 μm)	YE-3 (150–106 μm)	YE-4 (106–75 μm)	YE-5 (75-53 μm)	YE-6 (53–38 μm)	YE-7 (-38 μm)	YE (as is)	Waste acceptance criteria, mg/kg		
									Inert	Nonhazardous	Hazardous
Fe	4.2	3.2	4	2.8	4	4.6	3.4	3.6			
Ca	67,040	55,360	58,400	48,320	78,720	85,120	74,880	72,320			
Zn	2	1.6	1.8	1.6	2	2	2	1.8	4	50	200
Cd	2.8	2.2	2.4	2	2.8	2.8	3	2.8	0.04	1	5
Co	3.2	2.8	2.2	2	2	2.6	2.4	2.4			
Ni	4	3.6	4.8	4	3.4	3.4	5.6	4.6	0.4	10	40
Cu	2.8	2.6	2.6	2.6	3.2	3.2	3.4	3.4	2	50	100
Pb	11	10	11	10.4	14.2	15	14.4	14.0	0.5	10	50
Mn	1.8	1.6	1.6	1.4	1.6	1.6	1.8	1.8			
Cr 6	2.01	2.54	3.56	3.65	4.012	10.28	13.4	9.6	0.5	10	70

#### 3.3. Results of the toxicity test procedures

In this part of the study, two standard leaching tests (TCLP 1311 and ASTM3987-85) were used to determine the mobility of trace elements in Yenikoy fly ash. TCLP 1311 test simulates the solubility of different elements when fly ash exposed to a weak acid rain water environment. ASTM 3987-85 test characterizes water leaching behavior of fly ash at low concentrations of solid material.

Fly ash samples were classified into different particle size fractions, YE-1 (+212  $\mu m)$ , YE-2 (212–150  $\mu m)$ , YE-3 (150–106  $\mu m)$ , YE-4 (106–75  $\mu m)$ , YE-5 (75–53  $\mu m)$ , YE-6 (53–38 $\mu m)$ , YE-7 (–38  $\mu m)$ , YE (as is) and subjected to leaching tests to determine the fractionation and mobility of trace elements in aqueous environments.

As it was stated by Koukouzas [33], solubility of some trace elements may significantly change depending on the pH of the solution. For this reason, pH variations should be monitored during toxicity test procedures, and the final pH values should be taken into consideration in the evaluation of the test results. The high concentrations of calcium may cause secondary precipitation reactions and limit the solubility of some metals [38,39].

Variations in the pH values were closely monitored and recorded using the same conditions with toxicity test procedures prior to the tests. It is important to note that the pH of the solutions was adjusted to 5 to provide the initial condition for TCLP test method. However, the solution pH was measured over 12 after 1 h (Fig. 4).

For all fractions of fly ash samples, initial pH values of leachates were observed in the range of 12 to 13 after water addition step of ASTM test methods. As seen from Fig. 4a and b, the final pH values of the leachates are varying between 12 and 13.5 for different fractions of fly ash sample. At the end of the TCLP test, slightly lower pH values were measured compared to ASTM method.

Zandi et al. and Izquierdo et al. suggested that the surface of a fly ash particle is only microns in thickness and can contain leachable heavy metals due to condensation on to the surface. According to previous studies, some elements such as Ca, Cr and Mg are reported to the surface of the fly ash particles, while Al, Si, K, Pb and many

other trace elements are distributed throughout the particle [40,41]. Most of the cationic elements including Mg, Cu, Mn and Ni, show lower leaching rates due to their chemical behavior in solution below pH 10. Strong alkaline conditions over pH>10 may induce their release due to desorption/dissolution reactions. The leachability of heavy metals such as Zn and Cd is minimal around pH 7. The solubility of these elements tends to increase at pH levels above and below 7. Solubility of Cr increases with increasing pH for pH levels over 7 and decreases in strong basic conditions (pH>11) [38–40].

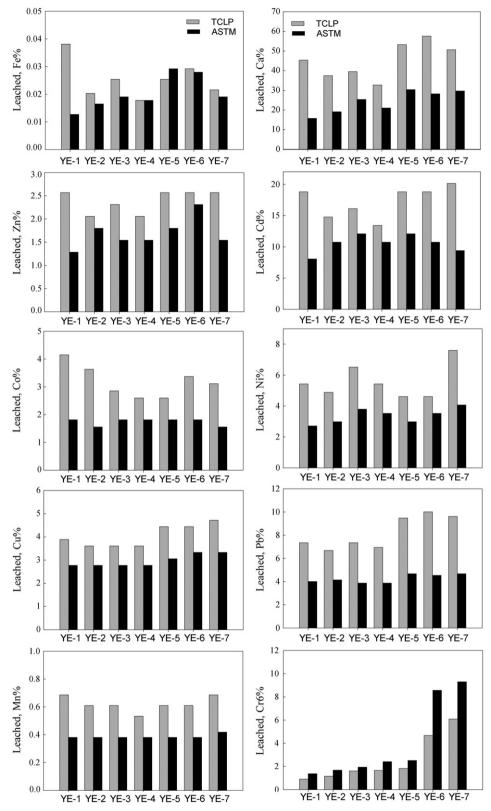
It can be said that very similar results with the previous studies about Yenikoy fly ash were obtained for ASTM method. However, different trace element concentrations were detected in the leachates of TCLP tests compared to the same studies [17,33]. This is most probably due to the difference between the presented final pH values in this paper and the final pH values reported by the researchers [17,33], which are 13 and 5 respectively.

The results of the leaching tests were in Tables 3a and 3b along with the limit values of waste acceptance for land filling stated in the Annex 2 of the 2003/33/CE Council Decision (based on 1999/31/EC Directive) [9]. As seen from Tables 3a, 3b, for all the fractions, most of trace elements gave higher leaching rates for TCLP 1311 compared to ASTM method due to its slightly harsher conditions. It is also possible to say that higher trace element concentrations (Cd, Pb, Cu) were observed in the leachates of finer particle size fractions for TCLP 1311 method [42]. Higher Cr (VI) concentrations were detected in the leachates of the finest particle size fraction for both test methods. It is worth pointing out that concentration of Cd $^{+2}$ , Ni $^{+2}$ , Pb $^{+2}$  and Cr (VI) showed higher values than inert limit for waste acceptance criteria for TCLP 1311 method.

The relationship between the ash particle size and percentage of trace elements in leachates is given in Fig. 5. It becomes evident from Fig. 5 that the highest releases for the studied fly ash samples correspond to Ca. Ca concentration in the leachates were found to be higher concentration in the particle size fraction of finer than 75  $\mu$ m (YE-5, YE-6, YE-7, Fig. 5). As seen from Fig. 5, the percentages of other selected elements in solution are very low compared to Ca

The concentration of heavy metals in Yenikoy ASTM leachates (mg/kg).

Samples	YE-1 (+212 μm)	YE-2 (212–150 μm)	YE-3 (150–106 μm)	YE-4 (106-75 μm)	YE-5 (75-53 μm)	YE-6 (53–38 μm)	YE-7 (−38 μm)	YE (as is)	Waste acceptance criteria, mg/kg		
									Inert	Nonhazardous	Hazardous
Fe	2.0	2.6	3.0	2.8	4.6	4.4	3.0	3.2			
Ca	23,200	28,160	37,440	31,040	44,800	41,600	43,840	40,160			
Zn	1.0	1.4	1.2	1.2	1.4	1.8	1.2	1.4	4	50	200
Cd	1.2	1.6	1.8	1.6	1.8	1.6	1.4	1.4	0.04	1	5
Co	1.4	1.2	1.4	1.4	1.4	1.4	1.2	1.2			
Ni	2.0	2.2	2.8	2.6	2.2	2.6	3.0	2.6	0.4	10	40
Cu	2.0	2.0	2.0	2.0	2.2	2.4	2.4	2.4	2	50	100
Pb	6.0	6.2	5.8	5.8	7.0	6.8	7.0	6.6	0.5	10	50
Mn	1.0	1.0	1.0	1.0	1.0	1.0	1.1	1.0			
Cr (VI)	3.02	3.69	4.26	5.32	5.54	18.85	20.46	14.8	0.5	10	70



 $YE-1 \; (+212 \; \mu m), \; YE-2 \; (212-150 \; \mu m), \; YE-3 \; (150-106 \; \mu m), \; YE-4 \; (106-75 \; \mu m), \; YE-5 \; (75-53 \; \mu m), \; YE-6 \; (53-38 \; \mu m), \; YE-7 \; (-38 \; \mu m)$ 

Fig 5. Results of TCLP 1311 and ASTM 3987-85.

which indicates that these elements are either bound in the glassy phases or there are no favorable conditions for leaching.

According to particle size analysis, the Yenikoy ash contains high amount of fines which makes the material to show very similar leaching

characteristics with these fine fractions (Tables 3a and 3b). As can be seen from Fig. 6, higher element concentrations were detected in the leachates of TCLP 1311 method except for Cr (VI). The highest solubility values were observed for Ca, Pb, Cr (VI) respectively. Compared with

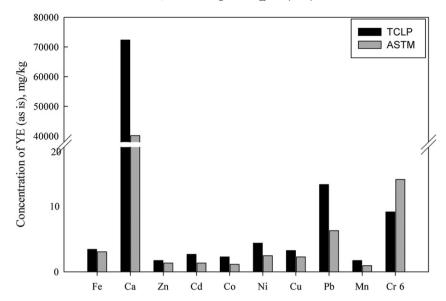


Fig. 6. The concentration of heavy metals in YE (as is) TCLP and ASTM leachates (mg/kg).

the regulatory requirements for waste acceptance, Zn yielded very low concentrations in both test methods and should therefore not be regarded as a matter of concern. The elements of Ni and Cu gave lower concentrations than the limit value of non-hazardous materials for both test methods, whereas the concentration of Cr (VI) was slightly higher for ASTM 3987-85 method. Based on the presented results, it can be concluded that releases of the selected trace elements were detected as lower than the hazardous material limit values of waste acceptance for land filling stated in the Annex 2 of the 2003/33/CE Council Decision (based on 1999/31/EC Directive).

#### 4. Conclusions

TCLP 1311 and ASTM 3987-85 toxicity test methods were applied to find out the mobility of some selected trace elements. According to the results, Ca was found as the most mobile element while Mn and Fe had the lowest mobility. The dissolution of selected trace elements was observed as higher for the particles below 38 µm size. For the leaching tests of original fly ash material, the highest solubility values were observed for Ca, Pb, and Cr (VI) respectively. The results suggested that releases of these trace elements were all lower than the allowed limits for solid wastes imposed by the EC Directive. The concentrations of Cr (VI) in some fractions exceeded the non-hazardous material limit value, that's why it should carefully be monitored. The results presented in this paper are useful and enlightening for understanding the mobility of some heavy metals from fly ash samples of Yenikoy power plants. However, long-term leaching tests are still required to obtain further information about leaching behavior of these selected trace elements for making a more adequate assessment.

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