



# Characterization and comparison of leaching behaviors of fly ash samples from three different power plants in Turkey



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## ABSTRACT

This study reports the characterization and the leaching behaviors of three fly ashes taken from different low grade-lignite-fired stations. Fly ashes were characterized in terms of their physical, chemical, mineralogical and morphological properties. Leaching behaviors of these ashes were studied by means of two standard leaching tests, namely TCLP and ASTM D3987. In this context, both the eluates and solid residues of these tests were investigated. Some selected major, minor and trace element (Ag, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Sb, Se, Sr, Ti, Zn) concentrations were analyzed in the leaching eluates. Calcium was a major cation having the highest extraction ratio in all samples regardless of the leaching test used. On the other hand, in ASTM tests, the extraction ratios are higher than 0.1% for selected elements (except Mg, Zn) whereas in TCLP tests, they are higher than 1% for the majority of selected elements. Solid residues were also analyzed to determine the mineralogical and morphological transformations during the leaching process. While lime and anhydrite were consumed in Yatagan fly ash (YFA) and Soma fly ash (SFA), secondary phases such as ettringite and/or calcium silicate hydrates precipitated in their solid residues; however, similar transformations could not be observed in Seyitomer fly ash (SoFA). Therefore, YFA and especially SFA can be accepted as inert wastes due to the ettringite formation. Since ettringite stabilizes several toxic elements such as As, Cr, Se and Sb by incorporating them to its structure.

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## 1. Introduction

Lignite coal is an important source of power generation in Turkey. It is estimated that there will be a boost in the consumption of coal hereafter to satisfy the growth in the demand for electricity [1,2]. As of today, the thermal power plants supply more than 25% of the total electric energy demand of Turkey [3]. Every year, million tons of coal have been burned in thermal power plants and massive volumes of fly ash and other combustion by-product materials are generated in Turkey. As far as today, almost 100 billion tons of ash have already filled up in power plant ponds and landfills [4,5]. Turkish coals, which are burned in thermal power plants, have low calorific value due to containing variable amounts of incombustible inorganic component and humidity [6,7]. Inorganic content of coal consists primarily of calcite, dolomite, quartz, kaolinite/chlorite, illite/muscovite, smectite, gypsum, pyrite, plagioclase, K-feldspar and small amounts of other minerals based on the mineralogical and chemical composition of [8]. During combustion of coal, these minerals transform into new forms with variety of properties. Additionally, trace element concentrations increase due to the ignition of carbon as carbon dioxide [8,9]. As a result, fly ash contains significant amount of hazardous leachable elements such as As, B, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Se, Sr, Ti and Zn, as well as a great deal of

major element compounds as oxides, hydroxides and sulfates of iron, aluminum and calcium [10,11]. Some studies also indicate that heavy metals favorably concentrate on the particle surfaces, making them more easily to be extracted from fly ash by the contact with aqueous solution in ponds and landfill areas [7,9,11–13]. In addition, there are a great number of studies [9–12,14–19] on the leaching behavior of different elements from fly ash. Dissolution behavior of the elements in leaching environments mainly relates to the particle size, surface area, leaching solution and fly ash pH, initial concentrations of the elements, leaching period, solid/liquid ratio, morphological structure and the other features of the leach medium. If not properly disposed of, these elements can cause a risk into the environment that consists of soil, groundwater and surface water [2,9,10,20,21]. Dissolution of the elements from fly ashes is a slow operation in which the solid–liquid equilibrium cannot be reached even after long contact time [7,11,17]. However, the rate of the process may be enhanced by stirring or shaking which increases the possibility and the efficiency of interactions between particles and leach solution. Therefore, by means of the shorter contact time, preliminary information can be obtained about the long-lasting leaching process [7,14]. Toxicity Characteristic Leaching Procedure (USEPA TCLP 1311, 1992) [22] and Standard Test Method for Shake Extraction of Solid Waste with Water (ASTM D-3987-85, 2004) [23] are generally applied tests to various wastes for this aim. Both tests can also be carried out for the determination of short-period leaching behaviors of fly ash samples in water and weakly acidic

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conditions such as acid rain [14]. However, in this process, not only leach medium properties but also fly ash properties are important. When the fly ash especially alkaline type contacts with the aqueous solutions, calcium aluminate sulfate hydroxide hydrate phases, mostly ettringite,  $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$ , are formed as hydrolysis products. During the ettringite formation, oxyanions (As, B, Cr, Mo, Sb, Se, V, etc.) and metallic ions substitute for  $\text{SO}_4^{2-}$  and  $\text{Ca}^{2+}$ , respectively [17,24,25]. These substitutions decrease with the ion concentrations of the leaching solution. However, similar formations are not observed for acidic fly ash.

The overall objectives of this study are to present the characteristics of three fly ashes taken from different thermal power stations in Turkey and to compare their leaching behaviors. For this reason, physical, chemical, mineralogical and morphological investigations, and two standard leaching tests were performed on the samples. The leaching ratios of Ag, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Sb, Se, Sr, Pb, Ti and Zn in fly ash samples were determined. In addition, their solid leach residues were investigated as mineralogical and morphological to reveal probable phase transformations, which affect largely the leached element concentrations, in the course of leaching process.

## 2. Materials and methods

### 2.1. Materials

Seyitomer, Yatagan and Soma B (1–4) power plants, located in Western Turkey, are the pulverized coal fired thermal power plants, which are operated in subcritical conditions. They consume low quality lignites supplied from the nearest Miocene aged coal reserves, Seyitomer lignite field (upper and lower seams) [26], Mugla lignite field (Eskihisar, Bagyaka and Tinaz Basins) [2] and Soma lignite field (lower seam k1 at the southern Soma Basin) [27] respectively. The mineralogy of the fired coals in these power plants was studied earlier by Karayigit et al. [26] in detailed. According to their findings, feed coals are composed mainly of clay minerals (smectite, illite and kaolinite/chlorite), quartz, feldspar, calcite, pyrite and gypsum. Moreover, Seyitomer lignites contain remarkable amount of siderite [26].

Studied samples were taken from the discharge of the electrostatic precipitators of these power plants in dry state. Then the samples were homogenized, divided and stored in airtight bags.

### 2.2. Characterization methods

Dried representative fly ash samples were ground less than 100  $\mu\text{m}$  for mineralogical and chemical investigations. The mineralogical investigations were realized with Rigaku Miniflex 2 X-ray diffraction (XRD) equipment (Cu K $\alpha$  radiation, 30 kV, 15 mA). Major and minor element (apart from sulfur) analyses were carried out using Analytic Jena NovaA 300 atomic absorption spectrometer (AAS). The samples were prepared using lithium tetra borate ( $\text{Li}_2\text{B}_4\text{O}_7$ ) fusion method [28] for this aim. Sulfur content was determined by gravimetric method. The trace element analyses were performed by Varian 710-ES inductively coupled plasma–optic emission spectroscopy (ICP–OES) following the total metal digestion process.

The specific densities and the particle size analyses of the samples were performed by Micromeritics AccuPyc II 1340 gas pycnometer and Horiba LA–950 laser scattering particle size distribution analyzer respectively. Specific surface areas were determined by Quantachrome NovaWin2.

Initial and equilibrium pH values and conductivities of the samples were measured by Hanna HI 4521 pH/conductivity bench top meter. Fly ash–water slurries were prepared according to the TCLP [22,29]. Five grams of the each sample was transferred to a 500 ml beaker and added a 96.5 ml of distilled water, enclosed with a watch glass, and stirred strongly using a magnetic bar for 5 min. Then the initial pH

and conductivities were measured. The same procedure was repeated at intervals of 1 h for a period of 24 h.

Morphological investigations and semi quantitative elemental analyses were carried out using a Jeol JXA 733 microprobe equipped with secondary electron image unit (SEM) and energy dispersive spectrometer (EDS).

### 2.3. Leaching tests

There are various standard test methods introduced by several institutes and agencies for investigation of leaching behavior of the materials. In this study, two different leaching tests, namely TCLP and ASTM D3987 methods, were applied to the samples.

TCLP test which was used by a number of researchers [1,14,29–38] was carried out to the samples to determine the potential leachability of some elements in acidic conditions. In addition, ASTM D3987 test was applied to find out the leach potential of the samples being exposed to normal environmental conditions. In these tests, solid-to-liquid (S:L) ratio is generally applied between 1:3 and 1:10. However, Zandi and Russel [38] stated that S:L ratio (1:10) was enough for Ca, Cr, K, Mo and Sr, while S:L ratio (1:20) was more appropriate for As, B, Ba, Mg, Na, Ni, Se, V and Zn. Therefore, in this study, S:L ratio was applied as 1:20 for both tests and 50 g of sample from each fly ash was added into 2-liter polyurethane bottles. After that, for TCLP tests 1 l of appropriate leach solution (acetic acid or acetic acid–NaOH buffer solution) was added into the bottles while for ASTM D3987 tests 1 l distilled water was added. Then, the bottles were horizontally shaken at 30 rpm in a shaker for 18 h and  $20 \pm 3$  °C. After shaking, the suspensions were settled for 5 min and then the liquid phases were decanted. The pH of the pregnant liquid phases (eluates) was measured subsequently. Then the eluates were filtered by vacuum filtration using glass fiber filter with a 0.45  $\mu\text{m}$  filter holder and were acidified with nitric acid to a pH lower than 2. They were kept in refrigerator in order to avoid the volume change and evaporation. Element concentrations (Ag, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Sb, Se, Sr, Pb, Ti, Zn) in eluates were determined by ICP–OES. Both leaching tests were studied by the parallel series and the measurement of leached element concentrations was also performed duplicate and the results were given as the mean of duplicates. The solid leach residues were also investigated in terms of the mineralogical and morphological transformations by means of XRD and SEM respectively.

## 3. Result and discussions

### 3.1. Physical and chemical characterization of fly ashes

The leaching behavior of the fly ashes depends on the physical and chemical properties of the ashes, such as particle size, surface area, bulk and specific densities, pH, LOI and conductivity. Some properties of the samples are shown in Table 1. SoFA has the highest mean size and specific surface area while SFA has the lowest. On the contrary, SoFA has the lowest bulk density and specific gravity whereas SFA has the highest one. In addition, SoFA contains higher LOI value than the others owing to the high amount of unburned coal.

pH is an important parameter on leaching process on which there is also a general agreement in the literature [1,2,7,9–12,14,19,20,30,31,33,

**Table 1**  
Some physical and chemical properties of fly ashes.

	Mean size ( $\mu\text{m}$ )	Bulk density ( $\text{g}/\text{cm}^3$ )	Specific gravity	Specific surface area ( $\text{m}^2/\text{g}$ )	Initial pH	Final pH TCLP	Final pH ASTM	LOI
SoFA	161	0.58	2.10	18.1	10.34	5.68	11.29	4.27
YFA	131	0.89	2.38	3.3	12.33	5.45	12.64	1.10
SFA	96	1.12	2.68	3.3	12.77	12.44	12.78	1.54

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