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Elemental characterization of lignite from Afşin-Elbistan in Turkey by k_0-NAA

Marie Kubešová[1] · **Esra Orucoglu**[2] · **Sevilay Haciyakupoglu**[3] · **Sema Erenturk**[3] · **Ivana Krausová**[1] · **Jan Kučera**[1]

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Abstract Twelve samples of lignite from several places and depths of the Kışlaköy open cast mine in the south eastern Turkey were characterized by k_0-NAA with emphasis on the potentially hazardous elements, such as As, Cd, Co, Cr, Mn, Ni, S, Sb, U. In total 39 elements were determined at least in some of the samples. The results showed a low quality and elemental inhomogeneity of the lignite material, which is used in the nearby thermal power plant. Mean values of element contents were also compared with older data from the same locality and to the world average lignite composition.

Keywords Lignite · Afşin · Elbistan · Neutron activation analysis · k_0 Standardization

Introduction

Use of lignite with a low calorific value, high ash, sulfur, and moisture contents presents potential environmental problems, such as air and water pollution as a large amount of fine particles is formed during mining and cleaning processes, because of the lignite softness. These fine particles cannot be easily handled, stored and transported [1]. The combustion of lignite is known to generate emissions of potentially hazardous trace elements, which may cause a wide range of health problems to organisms, including man, in regions with a high level of immisions as a result of both short- and long-range transport of gases and air particulates from lignite-fired power plants [2]. Environmental and health impacts of trace elements are generally related to the concentration, toxicity, and mode of occurrence of the elements in lignite [3]. Depending on the occurrence of elements, different health and environmental impacts will ensue. The organically bond trace elements tend to be vaporized upon combustion, with subsequent adsorption on the fine fly ash particles. The inorganically bond elements are generally less volatile, and tend to be retained in the bottom ash and/or in the coarser fly ash particles [4–7]. On the other hand, the major environmental concern of the elements in lignite combustion is that the elements concentrate on particulates of various sizes and may reach toxic levels for organisms, especially if bioaccumulation and biomagnification processes take place. For instance, the elevated levels of the elements in lakes, rivers or any other natural resources create a potential hazard for aquatic species [8–13]. The hazard can be reduced by taking some precautions like improved ash-handling practices or the use of geotechnical barriers around waste piles and/or dumping sites [14].

Elemental analysis is also important from the economic perspective [15–17] as it helps to understand the inorganic geochemistry of lignite and/or to evaluate a possible recovery of some valuable elements from its ash.

The Afşin-Elbistan lignite deposit is located in the southeast of Turkey in Kahramanmaraş Province within Afşin and Elbistan districts (Fig. 1). Around 95,000 people live there in the close proximity of the energetic complex, which consists of two lignite-fired power plants Afşin-Elbistan A (brought online in 1984–1987) and Afşin-Elbistan B (2006) with total power of 2795 MW. Although the

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✉ Marie Kubešová
mariekubes@centrum.cz

[1] Nuclear Physics Institute ASCR, 250 68 Řež, Czech Republic

[2] Faculty of Mines, Istanbul Technical University, 34469 Maslak, Istanbul, Turkey

[3] Energy Institute, Istanbul Technical University, 34469 Maslak, Istanbul, Turkey

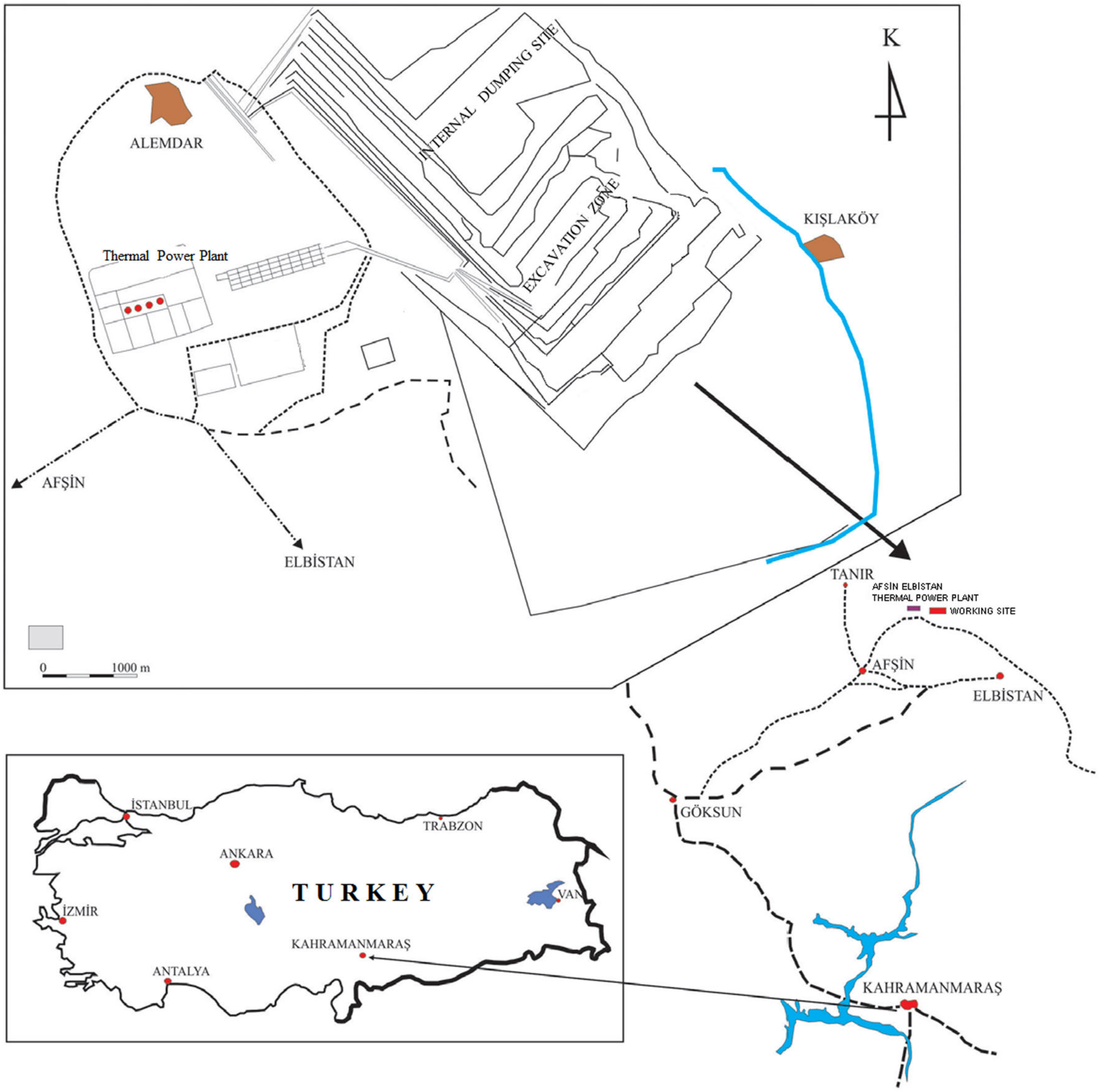

Fig. 1 Location of the Kıslaköy open cast mine and the nearby thermal power plant

quality of the deposit is low with an average calorific value of 1070 kcal/kg, ash content of 17 %, and moisture content of 55 %, the deposit is one of the most important sources of electricity generation in the country [18–20].

Geologically, it is a multi-layer deposit interlayered with clay and calcareous gyttja (sapropel clay), which is a formation of sapropelic, black or brown mud with organic matter containing many gastropod shells [21]. The lignite, formed in the Pliocene is directly beneath the gyttja, having a thickness of 25–45 m. The three different cross sections of the Kışlaköy sector have been described by Yorukoglu [18]. The thickness of the lignite increases from east to west and north to south. Faults are observed in the south part of the Kışlaköy sector, while the gyttja is a dominant unit in the overburden, the lignite band includes transitional layers of coal and gyttja. Several clay layers are interbedded with the gyttja and lignite [22]. The intercalations of lignite with clay or gyttja formations strictly affect the mineral content of the coals in the area [15, 18, 21, 23].

This small-scale study was conducted as a preliminary environmental survey on the pollution from a lignite mining area and the possible relationships between the pollution source and its environment. The goal of this study was to determine the concentrations of major, minor and trace elements in lignite samples in order to identify the possible sources of environmental contamination.

Experimental

Samples of lignite were taken from Kışlaköy open cast mine. Representative samples of at least 1 kg were taken from three different locations of each bench starting from third bench through sixth bench. Boulder like samples were collected randomly at the beginning, middle and end of each bench where bench faces were excavated by bucket wheel excavator previously. Totally 12 samples were collected from four benches of the mine. The sampling locations are shown in Fig. 2. The collected raw samples were crushed with hammer into medium fragments and then sampled by systematic grid sampling for homogenization [24]. They were not cleaned before crushing.

A mass down to 100 g was used for grinding in the IKA-mill down to mm scale. Approximately 25 g of each lignite material was provided to the Nuclear Physics Institute ASCR in Řež, Czech Republic. Here, the samples were milled again in an agate planetary ball mill to ensure finer particles of the lignite powder and consequent homogeneity. "As received" moisture content was determined for all lignite samples and coal SRMs by drying approximately 1 g of each material at 105 °C for 5 h in an oven.

Five types of NIST SRMs were used to ensure quality control and quality assurance during analysis: 1547 Peach Leaves, 2711 Montana Soil, 1633b Constituent Elements in Coal Fly Ash, 1635 Trace Elements in Coal (subbituminous), and 1632B Trace Elements in Coal (Bituminous) [25]. Samples were weighed (Table 1) and packed for irradiations into acid-cleaned disc-shaped polyethylene (PE) capsules with a 25-mm diameter.

Short irradiation (1 min.) of the samples, Si standard (21.66 mg) and Au+Mn+Rb [26] monitor sets was carried out in channel H1 of the LVR-15 reactor equipped with a fast pneumatic transfer system. Gamma-ray spectra of samples and monitors were measured using a coaxial HPGe detector (PGT, relative efficiency 20.3 %, resolution FWHM 1.75 keV at 1332.5 keV, peak-to-Compton (P/C) ratio 49.8:1). The decay times and counting geometries were chosen according to activities and half-lives of the radionuclides present to achieve determination of as many elements as possible with a low uncertainty. Due to the fact that Si standard and samples were measured in different counting geometries coincidence correction factors and detection efficiencies had to be recalculated for all the geometries of interest for the 1273.4 keV line of ^{29}Al formed by the ^{29}Si(n, p)^{29}Al nuclear reaction with fast neutrons.

For long irradiation, the samples, a Ni standard (to be able to use the ^{58}Ni(n, p)^{58}Co reaction with fast neutrons), and Au+Mo+Rb monitor sets [27] were formed into a column as indicated in Table 1, and hermetically sealed in an Al irradiation container. All PE capsules were wrapped into a thin Al foil to ensure good heat transfer from the samples through the Al container walls during irradiation. The Al container was irradiated in the LVR-15 reactor in channel H8 located at the outer perimeter of the LVR-15 reactor active core, in a Be reflector block, with the whole reactor spectrum for 3 h. The irradiations were carried out within the CANAM infrastructure (MEYS project No. LM2011019).

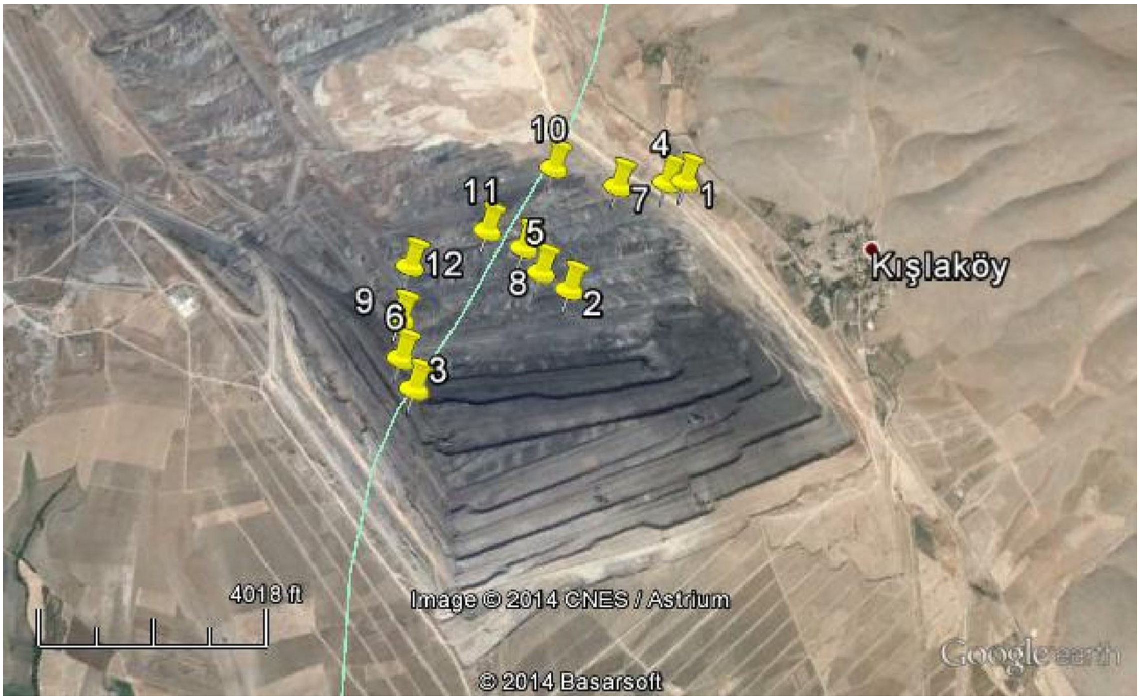

Fig. 2 Sampling locations of the material used for analysis

Table 1 Position of samples in irradiation tube and their masses

Position	Sample	Mass (mg)
1	Au+Mo+Rb-1	Monitor
2	SRM NIST-1547	108.94
3	Blank	195.39
4	Lignite-1	107.27
5	Lignite 2	111.14
6	Lignite-3	114.64
7	SRM NIST-1632B	107.01
8	Lignite-4	108.62
9	Lignite-5	114.08
10	Lignite-6	111.40
11	SRM NIST-1635	102.91
12	Au+Mo+Rb-2	Monitor
13	Ni	0.20058
14	Lignite-7	116.02
15	Lignite-8	115.85
16	Lignite-9	114.16
17	SRM NIST-1633B	56.70
18	Lignite-10	111.14
19	Lignite-11	110.34
20	Lignite-12	114.89
21	SRM NIST-2711	54.26
22	Au+Mo+Rb-3	Monitor

Prior to activity measurements of the irradiated samples and monitor sets, the cover Al foil was removed and the surface of the PE capsules was cleaned by washing with dilute nitric acid and deionized water. Gamma-ray spectra of samples and monitors were measured using a coaxial HPGe detector (CANBERRA, relative efficiency 77.8 %, resolution FWHM 1.87 keV at 1332.5 keV, and peak-to Compton ratio of 82.5:1). Both CANBERRA and PGT detectors were connected to a CANBERRA Genie 2000 γ-spectrometer through a chain of linear electronics, which contained a loss-free counting module (LFC CANBERRA 599, dual mode) to correct for pile-up effect and dynamic changes of dead time.

Two counts were performed for each sample after decay times of 5 days (counting time of 90 min., measurement geometry 10 cm for coal, 12 cm for SRMs NIST 1633B, 2711, 1635, 1633B, and 1 cm for blank and SRM NIST 1547) and 5 weeks (counting time of 6 h, measurement geometry 1 cm for all samples).

Measurement of the Au+Mo+Rb monitor sets was carried out in the geometry of 10 cm after a decay time of 4 days.

CANBERRA Genie 2000 software was used to control measurements and to evaluate the spectra obtained. Results of k_0-NAA were calculated using the Kayzero for Windows program [28]. In this program, the values of thermal and epithermal self-shielding factors $G_{th} = G_e = 1$ were used (as calculated in Kayzero for Windows program) [28]. The tolerance for peak identification was set to 1.0 keV (one sided).

Neutron flux parameters were determined using the bare triple-monitor method [29, 30] as follows (value ± standard deviation): channel H1 $\alpha = 0.025 \pm 0.001$, $f = 26.6 \pm 0.3$, $F_c = 343{,}000 \pm 6000$, channel H8 $\alpha = 0.018 \pm 0.005$, $f = 51.9 \pm 2.9$, $F_c = 233{,}000 \pm 11{,}000$.

Results and discussions

Agreement of the mean element contents found in SRMs with certified values was tested with E_n number defined as [31] using the same methodology of evaluation as described elsewhere [32–34].

Discrepant results ($E_n > 1$) for NIST SRMs were obtained only for a few elements for which the following explanations may be considered. For NIST SRM 1547—Cr, Yb, for NIST SRM 2711—Eu, Ge, La, Sm, for NIST SRM 1633B—Nd, Sb, for NIST SRM 1632B—Br, for NIST SRM 1635 Hf. In all cases uncertainties of these noncertified values may be larger than those presumed in this work (10 %, $k = 2$), in NIST SRM 1547 we found repeatedly a higher Na content than the certified value as in our previous works [26, 27, 33, 34] and in SRM 1635 a lower value of Cr, which is probably caused by insufficiently resolved spectral interference between peaks 320.1 keV of ^{51}Cr and 319.4 keV of ^{147}Nd.

Concentrations of three elements were not evaluated: mercury due to the possible volatility losses during irradiation, gold due to its inhomogeneous distribution in the analytical blank at very low concentrations, and tungsten in coal due to the possible contamination with this element due to the milling using the IKA-Mill at Istanbul Technical University.

The element contents determined in the individual lignite samples collected in different relative depths with respect to the first sampling point at bench three are given in Tables 2 and 3. In these tables, values for elements that were below detection limits in all lignite samples are not shown. The maximum detection limits for these elements were as follows: silver <0.4 mg/kg, cadmium <3.1 mg/kg, copper <60 mg/kg, erbium <45 mg/kg, gadolinium <4 mg/kg, germanium <0.17 %, holmium <1.4 mg/kg, indium <0.07 mg/kg, iridium <10 mg/kg, molybdenum <110 mg/kg, niobium <900 mg/kg, osmium <0.18 mg/kg, palladium <42 mg/kg, rhenium <19 mg/kg, ruthenium <0.4 mg/kg, tin <70 mg/kg, tellurium <80 mg/kg, thulium <5 mg/kg, yttrium <0.9 %, zirconium <0.1 %,

Table 2 Major elements and moisture content

Lignite sample	1	2	3	4	5	6	7	8	9	10	11	12	Range	Mean value[a]	Values from [15]	Values from [35]
Relative depth (m)	0	−13	−22	−21	−31	−46	−52	−67	−70	−74	−85	−91	0–91	–	–	–
Moisture (%)[b]	12.7	2.3	15.9	11.3	13.0	6.1	10.3	12.6	13.1	10.5	10.6	11.8	2.3–15.9	10.9	12.7	8.9
Al, % (7 %)[c]	2.04	0.41	2.55	1.14	0.85	0.478	4.52	1.07	0.71	3.39	3.41	2.6	0.41–4.52	*1.59*	–	1.6
Ca, % (7 %)[c]	3.94	26.5	3.31	5.28	3.87	25.1	2.42	3.92	3.4	2.62	2.34	2.56	2.34–26.5	7.11	4.3	11.1
Fe, % (7 %)[c]	1.2	0.283	1.55	0,82	0.377	0.341	2.64	1.02	0.371	2.23	3.77	2.43	0.283–3.77	1.42	–	0.9
K, % (7 %)[c]	0.188	0.0541	0.254	0.114	0.074	0.0545	0.429	0.074	0.063	0.311	0.318	0.129	0.0541–0.429	*0.12*	–	0.2
Mg, % (7 %)[c]	0.39	0.305	0.44	0.307	0386	0.332	0.77	0.497	0.415	0.75	0.61	0.46	0.305–0.77	0.472	0.4	0.2
Na, mg/kg (7 %)[c]	696	232	517	317	263	266	636	331	295	114	105	450	105–696	352	–	1000
S, % (20 %)[c]	3.8	<4,4	2.8	4.6	3.1	<2.9	<2.2	3.7	4.2	<1.9	4.5	4.4	<1.9–4.6	3.75	3.71	3.2
Si, % (30 %)[c]	<8	<4	<8	<6	<2.3	<2.7	14.5	<3.6	<3	<6	12.9	<8	<2.3–14.5	NE	–	–

NE not evaluated

[a] Arithmetic mean in normal letters, median in italics

[b] Moisture determined from as received samples

[c] Element, unit (relative uncertainty, $k = 2$), based on dry mass

praseodymium <5 mg/kg, lutetium <0.2 mg/kg, and platinum <150 mg/kg.

It may be seen from Tables 1 and 2 that the composition of individual layers is different. The similarity and/or dissimilarity of the elemental composition becomes clearly visible from results of cluster analysis (Fig. 3), in which we used complete linkage, Euklidean distance [35] and element contents below detection limits were taken as if present at this level (the substitution of missing values by a mean would greatly distort the data set, especially in cases of a large fraction of missing values). Two distinct clusters are obvious in Fig. 3. In the left cluster, there are samples No. 2 and No. 6, which contain more than 25 % of calcium suggesting that the corresponding layers are rich in calcareous compounds, according to [36] mostly calcite. In this cluster, a low content of terrigenous elements in samples No. 4, 5, 8, and 9 seems to indicate that these samples probably originate from gyttja (e.g., calcareous sapropel) layers. In the right cluster, the samples No. 1, 3, 7, 10, 11, and 12 can be considered as “real lignite” deposited between dirt beds. Two samples of this group (No. 7 and 11) contain much more silicon than the other samples, suggesting a significant enrichment of the corresponding layers with quartz. For some elements, there is a trend of decreasing contents with the depth (Na, Se, Cs, Cr); the opposite trend can be seen for the elements (Fe, Sb, Co, Ba, As, U). In an older work [35], lignite samples from this mine were collected in a pre-boiler mill of the Afşin -Elbistan power plant and analyzed by ICP-AES and ICP-MS. This type of sample collection can provide a representative sample with the “average” composition of lignite from the mine. To be able to compare our results with the above “average” composition, we evaluated mean values of element contents in our twelve samples from different depths of the mine. In cases of a normal distribution of the data sets, as ascertained by Kolmogorov–Smirnov and Shapiro–Wilk tests [35], arithmetic means were calculated, in the opposite cases, and in cases where there was a large fraction of values below detection limits, medians were calculated as the mean values (Tables 2 and 3). In general, our mean compares well with the mean reported in [36], except for a lower content of Na, and higher contents of Cs and Ni. There is also a fairly good agreement between our mean values and those evaluated from lignite borehole samples of the Kışlaköy open cast mine analyzed by ICP-MS [15]. A comparison of our mean values with the world average (arithmetic mean) of lignite elemental composition [37] shows that the element contents of the Kışlaköy lignite do not exceed the world average, except for higher contents of the elements Br, Cr, U, and V. From the comparison of our mean values with the data obtained for the samples from the pre-boiler mill of the

Table 3 Minor and trace elements content

Lignite sample	1	2	3	4	5	6	7	8	9	10	11	12	Range	Mean[a]	Values from [15]	Values from [35]	World Average [X]
Relative depth (m)	0	−13	−22	−21	−31	−46	−52	−67	−70	−74	−85	−91	0–91	–	–	–	–
As, mg/kg (7 %)[b]	15.1	1.64	17.7	13.6	8.8	2.50	17.2	10.2	9.7	13.7	23.6	36.3	1.64–36.3	14.7	13	10	33.37
Ba, mg/kg (16 %)[b]	111	97	125	93	53	77	152	140	72	183	219	170	53–219	124	163	162	249.91
Br, mg/kg (7 %)[b]	21.2	7.7	27.9	40.9	34.0	11.5	23.2	24.4	30.9	14.6	24.1	25.3	7.7–40.9	23.8	–	–	5.35
Ce, mg/kg (7 %)[b]	25.1	4.77	29.1	9.1	8.2	5.24	26.6	8.4	7.7	20.8	28.6	13	4.77–29.1	*11.0*	23	14	25.20
Cl, mg/kg (20 %)[b]	53	50	46	76	61	40	41	66	59	57	59	75	40–76	57	–	–	99.39
Co, mg/kg (7 %)[b]	4.75	1.28	4.87	3.04	1.60	1.24	17.3	2.32	1.73	11.2	14.1	8.5	1.24–17.3	*3.9*	12	3.9	32.01
Cr, mg/kg (7 %)[b]	270	17.5	465	213	180	20.3	247	122	124	132	139	88	17.5–465	168	–	146	54.51
Cs, mg/kg (7 %)[b]	15.0	3.85	2.45	1.66	1.93	0.99	6.05	1.12	0.98	2.88	3.00	1.91	0.98–15	*2.20*	1.7	1.2	1.63
Dy. mg/kg (12 %)[b]	1.70	<0.6	2.56	0.67	0.50	0.39	2.04	0.60	0.56	1.64	2.01	1.05	<0.6 –2.56	1.25	1.9	1.0	1.94
Eu, mg/kg (7 %)[b]	0.427	0.072	0.599	0.187	0.128	0.095	0.500	0.150	0.125	0.404	0.57	0.263	0.072–0.599	0.293	0.5	0.3	0.39
Ga, mg/kg (14 %)[b]	5.9	1.21	7.8	3.70	2.77	1.38	13.4	4.1	2.55	9.1	12.8	8.8	1.21–13.4	6.1	6.5	4.5	5.22
Hf, mg/kg (10 %)[b]	1.43	0.258	1.46	0.62	0.45	0.267	2.36	0.598	0.465	1.55	1.72	1.09	0.258–2.36	1.02	–	–	3.11
I, mg/kg (19 %)[b]	6.4	3.4	5.1	8.9	6.3	3.39	3.4	7.0	5.8	5.3	5.4	4.8	3.39–8.9	5.4	–	–	–
La, mg/kg (7 %)[b]	12.8	2.00	15.1	4.45	3.13	2.48	13.5	3.89	2.79	10.8	15.4	6.3	2.00–15.4	*5.4*	11	7.4	10.48
Mn, mg/kg (7 %)[b]	94	60.7	137	57.4	12.3	45.1	95	68.8	27.4	128	115	96	12.3–137	78.1	98	78	72.90
Nd. mg/kg (12 %)[b]	12.1	2.1	15.2	4.6	3.6	2.9	13.2	3.8	3.2	10.5	13.6	6.7	2.1–15.2	*5.7*	11	6.4	11.58
Ni, mg/kg (20 %)[b]	33	<20	113	30	25	<9	147	46	24	89	144	124	<9–147	*39.5*	–	16	54.17
Rb, mg/kg (13 %)[b]	11.6	4.0	15.6	6.2	3.8	3.3	27.9	4.4	3.5	22.7	20.6	9.4	3.3–27.9	*7.8*	13	10	32.64
Sb, mg/kg (9 %)[b]	0.384	0.100	0.50	0.529	0.337	0.088	0.87	0.440	0.351	0.61	1.21	1.85	0.088–1.85	*0.47*	–	–	0.80
Sc, mg/kg (7 %)[b]	4.91	0.92	6.55	2.13	1.81	1.07	10.3	2.30	1.84	7.1	7.2	4.6	0.92–10.3	4.2	5.5	5.0	2.80
Se, mg/kg (9 %)[b]	9.9	0.47	12.4	7.1	6.6	0.70	8.1	6.16	3.31	3.55	3.62	4.98	0.47–12.4	5.6	–	–	3.86
Sm, mg/kg (7 %)[b]	<0.13	0.229	1.91	0.417	0.253	0.308	1.49	0.74	0.300	1.16	1.34	0.90	<0.13–1.91	0.82	2.0	1.3	–
Sr, mg/kg (16 %)[b]	85	245	69	116	122	232	112	150	142	149	149	135	69–245	142	130	162	206.82
Ta, mg/kg (9 %)[b]	0.292	0.064	0.376	0.154	0.104	0.075	0.597	0.149	0.092	0.355	0.484	0.260	0.064–0.597	0.250	0.4	1.4	16.38
Tb, mg/kg (8 %)[b]	0.284	0.045	0.399	0.101	0.083	0.062	0.318	0.102	0.083	0.257	0.317	0.170	0.045–0.399	0.185	0.3	0.2	2.01
Th, mg/kg (7 %)[b]	3.69	0.562	3.60	1.44	1.27	0.664	5.57	1.39	0.97	4.11	3.98	3.48	0.562–5.57	2.56	3.4	1.5	3.30
Ti, mg/kg (10 %)[b]	1250	300	1610	690	514	322	2680	700	437	1590	2020	1250	300–2680	1110	1672	1000	–
U, mg/kg (7 %)[b]	19.3	2.37	20.9	14.7	14.9	4.45	21	19.1	10.1	15.3	22.8	30.9	2.37–30.9	16.3	25	12	6.06
V, mg/kg (9 %)[b]	230	20.0	320	197	158	37.2	231	197	148	265	273	257	20–320	194	–	161	37.28
Yb, mg/kg (10 %)[b]	1.09	0.174	1.58	0.37	0.286	0.216	1.26	0.345	0.275	1.03	1.30	0.69	0.174–1.58	*0.53*	0.9	0.6	0.78
Zn, mg/kg (8 %)[b]	21.9	6.3	43.0	24.2	10.0	5.53	64.9	8.6	10.7	44.5	44.3	19.7	5.53–64.9	25.3	29	27	75.54

[a] Arithmetic mean in normal letters, median in italics

[b] Element, Unit (average uncertainty, $k = 2$), based on dry mass

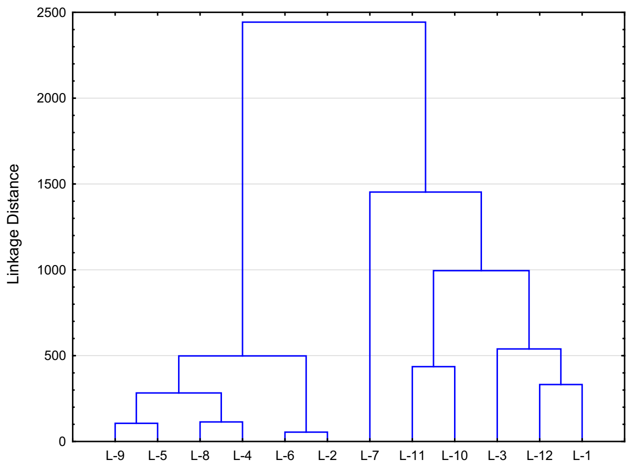

Fig. 3 Tree diagram for 12 layers of the Kışlaköy lignite mine

Afşin-Elbistan power plant [36] it is obvious that the mined material is not additionally treated or cleaned before combustion. Thus, even the non-lignite dirt bed material is fed to the boilers of the plant. In case of uncontrolled additions of such materials, the boiler fouling, corrosion, and catalyst poisoning in conversion technology may occur, because of the presence of the alkali metals, and the elements Cl, S, Zn, and Mn. On the other hand, the presence and/or controlled addition of calcareous materials, such as present in some layers of the Kışlaköy lignite mine may lead to a reduction of sulfur dioxide and trace element emissions on lignite combustion. Hydrated lime or limestone as sorbents proved to be efficient for this purpose [38].

Conclusions

Method of k_0-NAA was used for elemental analysis of 12 samples from layers of different depths of the Kışlaköy open cast mine in Afşin-Elbistan district in Turkey. Our results showed that the lignite layers in this mine are interlaid with layers of dirt beds, rich in silicaceous and calcareous compounds, or presumably consisting of gyttja (calcareous sapropel). Both lignite and "parasitic" layers contain appreciable quantities of macro- and trace elements that are of environmental concern, especially those identified by the U.S. Clean Air Act Amendments of 1990 as potentially Hazardous Air Pollutants, i.e., As, Cd, Co, Cr, Mn, Ni, Se, and radionuclides, e.g., U. Also the presence of alkali metals, Cl, S, Zn, and Mn in the materials combusted can cause severe technical and economical problems in the power plant operation.

Our results indicate the need for regular environmental pollution monitoring around the Kışlaköy mine and the Afşin-Elbistan power plants concerning both immisions from the mines, power plants, and waste piles and leakages from the waste piles. Based on results of the long-term measurements of the mine, power plant and waste pile (dumping site) discharges, environmental impact assessment could be done and/or possible measures for pollutant abatement in the power plants could be evaluated.

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