Original Scientific Paper

CHEMICAL AND RADIOLOGICAL PROFILE OF THE COAL ASH LANDFILL IN KAŠTEL GOMILICA*

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Received in May 2005
Accepted in February 2006

The objective of this study was to determine the chemical, radiological and leaching properties of slag and ash produced by a thermoelectric unit of a former factory Adriavinil and deposited in the area of Kaštel Gomilica near Split, Croatia. A total of 33 samples were analysed. The bioavailable fraction of the slag and ash was estimated using different leaching tests. The waste material was characterized by a high activity of naturally occurring radionuclides ^{238}U, ^{235}U and ^{226}Ra and by elevated concentrations of heavy metals. The concentrations of most heavy metals were three to four times as high as in the common soil. Uranium slag and ash concentration was almost 40 times higher than in control soil. More than 37 % of the total U could be removed from the slag and ash with the sea water.

KEY WORDS: *heavy metals, leaching, radionuclide, slag and ash*

There are various of sources of waste materials with technologically enhanced concentrations of naturally occurring radioactive materials, especially those with high activities of ^{226}Ra, ^{228}U and ^{232}Th. These include oil and gas industry (1-3), non-uranium mining (copper mining, niobium industry, phosphate and gold industry) (4-6), coal mining (7, 8), and energy production (9-11).

The purpose of this paper was to determine the chemical and radiological profile of the slag and ash produced by a coal burning thermoelectric unit of the former chemical factory Adriavinil. The factory was situated in a small Croatian town of Kaštel Sućurac, near Split, on the northeast coast of the central Adriatic (Figure 1). Since 1949, coal had been used to fuel a power unit. It was transported from several coal mines such as Raša in Istria (anthracite), Drinovci and Širitovci near Šibenik (brown coal and lignite) and the broader area of Livanjsko polje in Bosnia and Herzegovina (brown coal and lignite). All coal was the source of elevated uranium concentrations and its natural radionuclides. Table 1 shows the activities of ^{232}Th, ^{226}Ra and ^{238}U together with ^{238}U/^{226}Ra activity ratio of the coal cores (brown coal and lignite) of the Tušnica region in Livanjsko polje (Bosnia and Herzegovina) sampled in 1993. The content of ^{232}Th, ^{226}Ra and ^{238}U in slag and ash increased several times during combustion, which also depended on the inorganic portion of the coal. Slag and ash were deposited in the vicinity of the factory until 1973 and then moved to a more appropriate location a few hundred meters away, at the edge of the town of Kaštel Gomilica, where it was covered with a protective plastic foil and a layer of clay and humus. The area was then fenced in and grassed over. With time the area between the factory and the old depot had gradually been covered with waste materials, which contained slag and ash. At the turn between the 1980s and 1990s, slag and ash were dumped directly into the sea shallows in the south-western section of the facility.

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*Preliminary report presented at the 6th Symposium of the Croatian Radiation Protection Association with international participation, Stubičke Toplice, Croatia, 18-20 April 2005

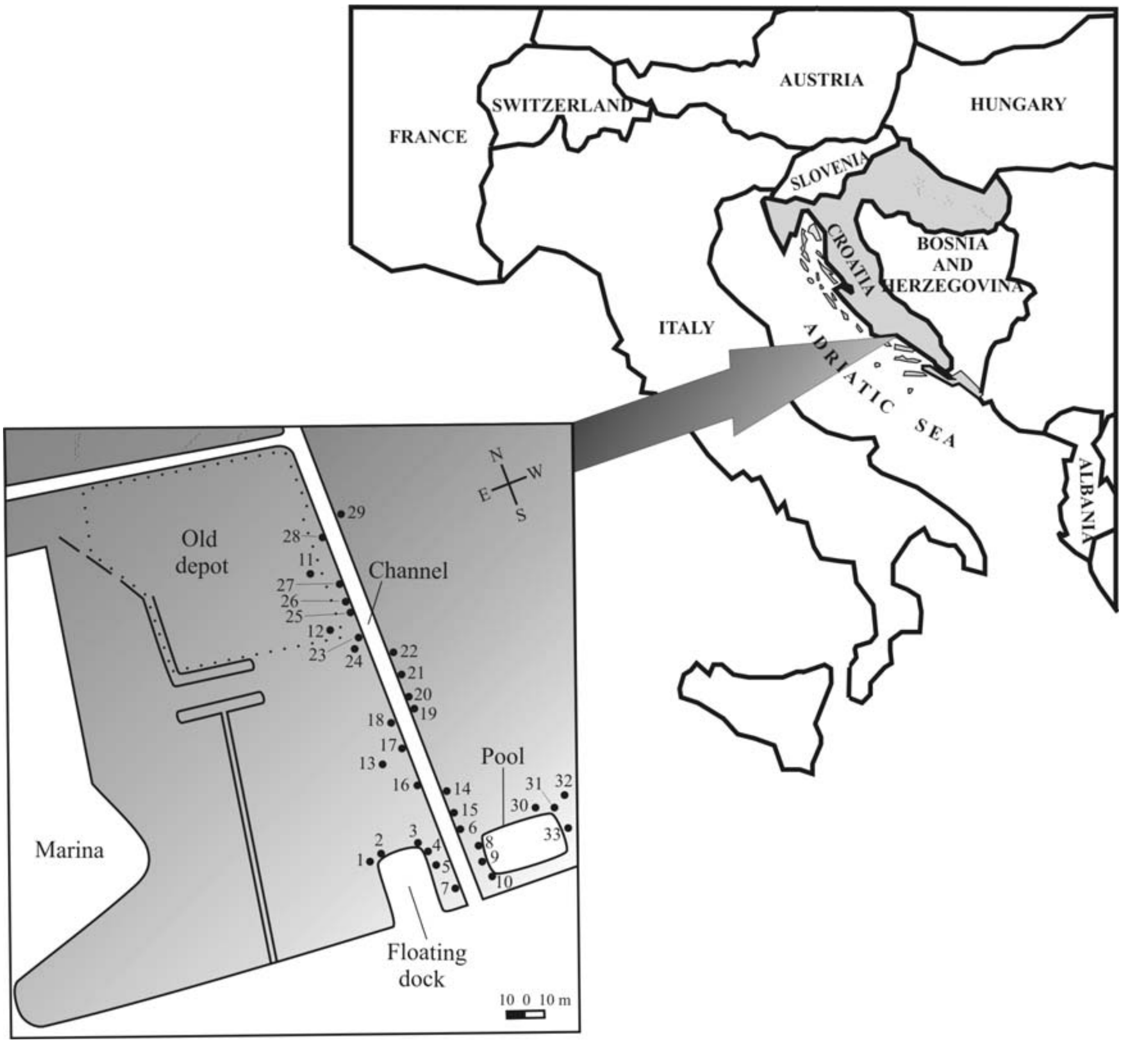

Figure 1 *Location of the depot of the fly and bottom ash mixture and sampling points*

A floating dock in the southern section of the factory is mostly buried in the deposited material and is in direct contact with the sea. There is also a channel running through the depot in the north-south direction and a pool to the southwest which are directly exposed to the deposited waste materials. Furthermore, a marina which is under construction reaches the western section of the old depot and the new section of the depot. Last but not least the nearest settlement is only 20 m away.

A total of 33 samples of slag and ash were collected and analysed in the spring of 2004 in order to obtain preliminary data on the present state of the new section of the depot.

MATERIALS AND METHODS

One sample of about 1 kg of slag and ash was taken from each location (33 altogether), put in a plastic bag and transported to the laboratory. The material was dried at 105 °C and the fraction <0.5 mm was disaggregated in an agate mortar to determine the composition and activities of selected radio-nuclides and perform XRD analysis and leaching tests.

The concentrations of Ca, Ti, V, Cr, Mn, Fe, Cu, Zn, Rb, Sr, Y, Zr, Pb and U in bulk samples were determined using the energy-dispersive X-ray fluorescence (EDXRF) spectrometer (12). Samples were irradiated by X-rays generated from a ^{109}Cd annular source. The incident angle was 49.76 °. The X-ray radiation profile of the sample was made using a Si (Li) detector (Canberra), cooled with the liquid nitrogen. The detector size was 30 mm^2, Si thickness 3 mm, Be window 25 μm, FWHM for 5.9 KeV ^{55}Fe 165 eV. The emerging angle was 74.05 ° and the distance was 1.5 cm. The spectra were collected by a Genie – 2000 software (Canberra). The collecting time was 10,000 s for thin targets and 7,000 s for thick targets (solid slag and ash samples). Spectral data were analysed using the WinAxil software (Canberra). The calibration file for the thin targets (water samples) was created on the basis of measurements of standard solutions (Merck) prepared and analysed in the same way as samples. Elemental concentrations in the thick targets were calculated using the the WinFund package for fundamental parameter approach with ECH (coal fly ash) as the standard reference material.

Following the EDXRF analysis, all the remaining material was mixed and homogenised, and one gram of the mixture was extracted either by 1 mol L^{-1} NH$_4$Oac, pH 7 for one hour or by 2x10^{-2} mol L^{-1} of citric acid, ascorbic acid, EDTA or oxalic acid for eight hours (13). Waste material was also extracted with sea water (solid/liquid ratio=1:10) for 24. At the end of the contact time, the solid part was separated from the leachate by filtration through white ribbon filter paper. The extracts were diluted to 100 mL with double distilled water, divided in two sub-samples, and adjusted to pH values 3 and 11 by the addition of concentrated HNO$_3$/NH$_4$OH. After the pH adjustment, samples were preconcentrated by 1 mL of freshly prepared 1 % (w/v) solution of ammonium-pyroloidinedithiocarbamate (APDC). After 20 min of complexation, the suspension was filtered through a Millipore filter paper (0.45 μm) and irradiated for 10,000 s.

Composite sample of slag and ash was also prepared for the XRD analysis. Powdered samples were examined at room temperature using a Philips PW 1830 X-ray diffractometer with Cu tube (current = 40 mA; voltage = 40 kV), graphite crystal monocromator, and proportional counter. Diffraction data were processed by Philips X'Pert software. The relative intensities of X-ray diffraction lines were compared with literature values (12).

Waste materials were placed in the counting container of 125 cm^3, sealed and stored for at least 4 weeks to allow ingrowth of gaseous ^{222}Rn. At the end of the ingrowth period, gamma-spectrometry was performed on a HPGe semiconductor detector with a Canberra 8192 channel analyzer. Spectra were recorded for 80,000 seconds and analyzed using the Genie 2K Canberra software. Activities of ^{40}K were calculated from the 1460.75 keV-peak, of ^{226}Ra from the 609.3 keV-peak of its ^{214}Bi progeny, and of ^{228}Ra from 911.1 keV-peak of its ^{228}Ac progeny. The activities of ^{235}U were calculated from 186 keV-peak after subtraction of the overlapping ^{226}Ra contribution and were used for the calculation of ^{238}U activity, assuming the natural ^{235}U/^{238}U activity ratio of 0.04603 (12, 14, 15).

RESULTS AND DISCUSSION

Radiological profile

Figure 2 shows the activities of ^{40}K, ^{232}Th, ^{226}Ra and ^{238}U in collected samples of fly ash. Thes activities seem to depend on coal used for energy production in the power unit. In most ash samples, ^{238}U activity was up to 30 % lower than that of ^{226}Ra, while in the coal samples (Table 1) their average activities were similar. This could be explained by the fact that most of the samples were taken either from the surface of the deposit with a damaged protective cover or from the channel exposed to the rainwater circulation. Since uranium was more susceptible to leaching from ash with rainwater than ^{226}Ra, the resulting ratio ^{238}U/^{226}Ra shifted towards radium. Alkaline solutions promote the dissolution of the glassy components of

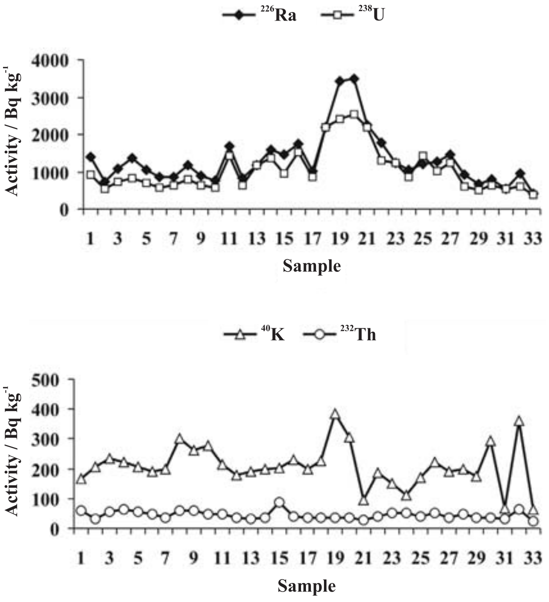

Figure 2 *Activities of ^{40}K, ^{232}Th, ^{226}Ra and ^{238}U measured in slag and ash from the Kaštel Gomilica, Croatia*

Table 1 *Activities of ^{232}Th, ^{226}Ra and ^{238}U (in Bq kg^{-1}) measured in coal cores of the Tušnica region in the Livanjsko polje (Bosnia and Herzegovina), sampled in 1993 and the ratio ^{238}U/^{226}Ra in collected samples*

Coal core	depth / m	^{232}Th	^{226}Ra	^{238}U	^{238}U/^{226}Ra
A	18.2-23.2	28.9±1.8	410.2±2.8	398.7±11.8	0.972
	23.5-29.4	22.7±1.8	330.4±2.6	424.1±11.2	1.284
B	21.2-26.2	6.5±1.7	584.5±3.5	696.9±15.3	1.192
	26.2-31.0	16.1±1.7	606.8±3.4	651.1±14.6	1.073
C	0.0-0.8	7.2±0.8	83.2±0.8	204.6±3.6	2.459
	1.0-1.5	7.4±1.7	147.3±2.0	182.9±8.0	1.242
D	0.0-1.0	5.0±1.7	177.1±2.0	178.9±8.3	1.010
	1.1-2.0	7.8±1.6	116.2±1.7	210.3±7.4	1.810
E	0.0-1.0	17.3±1.8	681.4±3.7	641.5±15.6	0.941
	1.0-2.0	25.9±1.8	464.7±3.0	450.7±12.6	0.970
	2.8-3.5	9.2±1.7	453.0±3.1	336.0±12.8	0.712
	3.7-4.5	10.2±1.7	482.7±3.1	285.7±12.5	0.592
	4.6-5.0	7.0±1.7	532.2±3.3	442.5±13.6	0.831

fly ash and increase uranium solubility through the formation of uranium-carbonate species. On the other hand, leachates of fly ash are rich in dissolved sulfate which minimises the solubility of radium through the formation of its highly insoluble sulfates.

The Shapiro-Wilks' W test showed normal distribution only for ^{40}K while the activities of other three radionuclides significantly varied. This is expected since the radionuclides originated from the coal supplied from five different sources. ^{226}Ra and ^{238}U activities were up to fifty times higher than their average activities characteristic for the surrounding soils of the Middle and Upper Eocene flysch. ^{40}K and ^{232}Th showed no elevation compared to soil activities.

Results of mineralogical analysis

The predominant minerals present in the fly and bottom ash mixture are calcite, quartz, monteponite (CdO) and bassanite ($CaSO_4 \times 0.5H_2O$).

Results of chemical and statistical analysis

Basic statistic parameters for the concentrations of the elements Ca, Ti, V, Cr, Mn, Fe, Co, Cu, Zn, Rb, Sr, Y, Zr, Pb and U measured in slag and ash are given in Table 2. Since the flysch soil from the island of Krk is well profiled (16, 17) and has almost the same origin as the soil on which the investigated material was deposited, it served as reference material. This is why Table 2 also includes the mean concentrations of elements measured in reference soil and the ratio between mean concentrations of elements in slag and ash and mean concentrations in reference soil.

The respective means of U, Ni, V, Sr, Cu, Pb and Y were approximately 36, 4.3, 3.7, 3.2, 1.8, 1.6 and 1.5 times higher in slag and ash than in reference soil samples (Table 2). Zn, Fe, Cr, Ca and As had similar concentrations, while the concentrations of Mn, Rb, Zr and Ti were 5.5 to 1.7 times lower in slag and ash than in reference soil.

Basic statistic parameters and Shapiro-Wilks' W test showed a significant deviation from the normal distribution of Zn, Fe, Cr, Ca, Sr, As and U. Only zinc exhibited bimodal distribution while the cause of deviation for the other six elements were the outliers. Following the subtraction of outliers from the dataset and the logarithmic transformation of the values, the data were subjected to factor analysis. Four significant factors were extracted that explained 73.5 % of the total variability. On the basis of factor loadings, three groups of elements were identified (Table 3). This was in agreement with the results obtained by correlation among elements. The first group was represented by Cu, Fe, Co, Ti, Y, Rb and Zr; the second by Cr, V, U, Pb and Ni, and the third by Mn, Ca and Sr. Arsenic did not correlate either positively or negatively with any other element of interest, while Zn showed only a weak correlation with Pb. Uranium showed a significant positive correlation with elements associated with oxides and sulfides (Cr, V, Pb, Ni), thus confirming the prevalently terrestrial origin of coal deposits. Positive correlation between U and Sr suggests that coal deposits were formed in carbonate soil.

Table 2 *Basic statistic parameters for elemental concentrations measured in slag and ash from the Kaštel Gomilica, Croatia. Mean values of the measured elements in the Krk soil (Mean$_{soil}$) together with the ratio between mean elemental concentrations in slag and ash and mean elemental concentrations in the Krk soil (Mean$_{slag\ and\ ash}$/Mean$_{soil}$). Mean elemental concentration; SE-standard error of mean; M-median*

Element	Mean	SE	M	Minimum	Maximum	SD	Skewness	Kurtosis	Mean$_{soil}$	Mean$_{slag\ and\ ash}$/ Mean$_{soil}$
Zn/mg kg^{-1}	150.5	9.9	143.0	56.0	307.0	57.0	1.2	1.4	155.9	1.0
Cu/mg kg^{-1}	40.3	1.8	42.0	19.0	64.0	10.2	0.2	-0.3	22	1.8
Ni/mg kg^{-1}	149.0	7.4	140.0	48.0	240.0	42.3	-0.1	0.4	34.3	4.3
Co/mg kg^{-1}	11.7	0.6	11.7	4.9	22.8	3.2	1.2	3.7	12.1	1.0
Fe/%	3.775	0.199	3.805	1.260	7.810	1.141	1.3	4.4	4.401	0.9
Mn/mg kg^{-1}	307.1	20.9	320.0	80.0	670.0	119.8	0.8	1.5	1686.2	0.2
Cr/mg kg^{-1}	136.2	9.5	120.0	69.6	250.0	54.5	0.7	-0.5	153.4	0.9
V/mg kg^{-1}	329.9	19.1	320.0	146.6	630.0	109.4	0.7	0.4	90.1	3.7
Ti/%	0.30	0.01	0.30	0.10	0.47	0.08	-0.5	0.9	0.51	0.6
Ca/%	9.77	0.60	9.60	4.30	17.90	3.45	0.8	0.5	12.8	0.8
Y/mg kg^{-1}	73.4	4.2	70.0	28.0	133.0	24.4	0.3	0.2	50.1	1.5
Zr/mg kg^{-1}	184.2	9.5	182.0	65.0	281.0	54.6	0.1	-0.6	380.6	0.5
Sr/mg kg^{-1}	434.6	29.3	399.0	233.0	898.0	168.4	1.0	0.5	134	3.2
Rb/mg kg^{-1}	43.8	3.0	44.0	6.5	89.0	17.3	0.4	1.6	143.5	0.3
As/mg kg^{-1}	21.4	1.2	19.0	14.0	42.0	7.0	1.5	1.9	27.1	0.8
U/mg kg^{-1}	93.8	9.1	76.0	34.0	227.0	52.0	1.3	1.0	2.6	36.1
Pb/mg kg^{-1}	42.8	2.1	42.0	24.6	74.0	12.3	0.5	-0.2	26.6	1.6

Table 3 *Factor loadings (equamax rotated) matrix associated to the principal component analysis of elemental concentrations measured in slag and ash mixture from the Kaštel Gomilica, Croatia. (n=33; marked loadings are > 0.70)*

Variable	Factor 1	Factor 2	Factor 3
Zn	-0.01	0.56	-0.41
Cu	0.76*	0.39	0.24
Ni	0.47	0.71*	-0.15
Co	0.87*	0.12	0.12
Fe	0.86*	0.11	0.10
Mn	0.28	-0.13	0.83*
Cr	0.08	0.76*	0.44
V	0.07	0.85*	0.20
Ti	0.92*	-0.03	-0.18
Ca	-0.24	0.23	0.88*
Y	0.78*	-0.14	0.15
Zr	0.78*	-0.30	-0.30
Sr	-0.16	0.59	0.72*
Rb	0.75	0.04	-0.42
As	0.37	-0.41	0.10
U	-0.05*	0.84*	0.17
Pb	-0.03	0.84*	-0.14
Expl.Var	5.24	4.39	2.87
Prp.Totl	0.31	0.26	0.17

Leaching Tests

The deposited slag and ash were in direct contact with the sea water. It is generally believed that among different soil phases (exchangeable/adsorbed, carbonates, Fe/Mn oxides, organic matter, and residual), the most important chemical form is the exchangeable/adsorbed phase (12,13). It represents weak binding sites that could be easily mobilised by rain/sea water. Accordingly, data on heavy metal concentrations in the exchangeable fraction are more valuable than the total metal content determined in an environmental sample. The leaching properties of the sea/rain water can largely be modified by various naturally occurring organic complexing agents (13). In this study, different organic acids were applied in order to simulate leaching conditions that naturally occur in the environment.

Table 4 shows that only negligible amounts of V, Cr, Pb, Zn, Cu and Ni were released from slag and ash either by an exchangeable agent (ammonium acetate, pH 7) or by the sea water (pH 7). Organic acids were more efficient in heavy metal removal than ammonium acetate. The high affinity of Cu, Pb and Zn for organic complexes was the reason for their higher extraction in organic acids compared to the other measured elements. The percentage of elements found in the extract obtained by organic acids was less than 10 %, which suggests that they strongly bind to the mineral particles of ash under the testing conditions. Uranium, on the other hand, was easily removed from the ash. A significant amount of total uranium was extracted from slag and ash by all leaching agents save for ammonium acetate which removed only 1.6 % of the total U. The removal efficiency varied from 17.2 % for EDTA to over 50 % for oxalic acid. Almost 40 % of total uranium was removed from slag and ash by seawater. It is therefore likely that seawater greatly influences the distribution, dilution, and uptake of uranium from slag and ash into the living systems.

Table 4 *Percentages of elements extracted from slag and ash by different extraction agents*

Element	Percentage of element released from the ash					
	Sea water	NH_4Ac	Ascorbic acid	EDTA	Citric acid	Oxalic acid
Pb	0.4	2.5	2.0	3.1	6.7	5.4
U	37.2	1.6	26.4	17.2	43.2	51.2
Zn	0.5	1.7	1.7	3.2	8.5	9.8
Cu	0.3	1.6	1.2	7.5	6.4	3.8
Ni	0.1	0.2	0.3	0.3	2.2	1.6
Co	0.3	2.0	4.6	0.1	9.8	8.2
Cr	0.2	0.8	0.8	1.0	1.7	1.9
V	0.6	1.7	0.8	0.1	1.2	1.8

We used canonical correlation analysis to test a possible correlation between heavy metals and radionuclides in the ash. All measured elements represented the left set of variables while four radionuclides represented the right set of variables. The two tested sets showed a perfect positive correlation, as the canonical R was 1, χ^2=377.4 and p=0.000000. All three canonical roots were significant. Each was interpreted on the basis of canonical weights and canonical factor loadings for each set. It was found that mostly five variables from the left set (V, Cr, the total U, Pb, Sr) and the two ones from the right set (^{226}Ra, ^{238}U) contributed to the correlation.

CONCLUSION

The investigated slag and ash showed a significant variability in chemical composition and in the activities of selected radionuclides due to a different origin of coal used in the former factory Adriavinil. The mean concentrations of elements in the ash were 1.5 to about 36 times higher than reference soil concentrations. The greatest differences were found for U, Ni, V and Sr. The activities of ^{226}Ra and ^{238}U in slag and ash were about 50 times higher than in reference soil. The extractable portion of heavy metals under all tested conditions was less than 10 %, save for total uranium which showed a high leaching potential either by seawater or weak organic acids. As our results are preliminary they require further toxicological testing.

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Sažetak

KARAKTERIZACIJA ŠLJAKE I PEPELA ODLOŽENOG U KAŠTEL GOMILICI

U radu su kemijski i radiološki okarakterizirani uzorci šljake i pepela. Otpadni materijal je nastao radom termoelektričnog postrojenja bivše tvornice "Adriavinil", a odložen je u Kaštel Gomilici u Hrvatskoj. Ukupno su analizirana 33 uzorka. Biodostupnost frakcija šljake i pepela određena je različitim testovima za izluživanje. U otpadnome materijalu određena je velika aktivnost radionuklida iz prirodnog niza, ^{238}U, ^{235}U i ^{226}Ra, i povišene koncentracije teških metala. Koncentracije većine teških metala 3 do 4 puta su veće nego u kontrolnim uzorcima, dok je koncentracija urana veća 40 puta. Utvrđeno je da se više od 37 % ukupnog urana iz uzorka može ukloniti izluživanjem u morskoj vodi.

KLJUČNE RIJEČI: *šljaka i pepeo, teški metali, radionuklidi, izluživanje*

REQUESTS FOR REPRINTS:

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