



The mobility of heavy metals in mine tailings associated with polymetallic ore mining activities in the Eastern Carpathians, Romania

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Abstract

The present study was conducted on a series of 20 samples collected from the topstratum of seven mine tailings mounds and one mound of processed ore. The material studied is linked to the mineralization of polymetallic sulphide ore in the Tulgheș metamorphic unit (Eastern Carpathians, Romania). The aim of the study was to quantify the mobile fraction from each sample, for As, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sn and Zn, as well as to investigate its correlation to the pH and Eh conditions within the topstratum. The initial pH and Eh, as well as the initial content, were measured for each sample; the latter was established using XRF. The leaching procedure was performed by means of acetic acid extraction – 0.1M for 1g of sample, while the concentrations of the elements within the extracts were measured using ICP–OES. The results indicate that As and Sn were the least mobile elements, while the highest mobility was found to have been that of Mn and Co. The highly acid environments increased the mobility of Fe and Cr, and decreased that of Pb. The mobility of Zn, Cu and Co was found to be correlated, as they formed a distinct group.

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Introduction

Acid mine drainage usually occurs when sulphide-rich material becomes exposed to oxygen and water, especially where iron sulphides such as pyrite are involved, and, although the process occurs naturally, mining activities

intensify its impact by increasing the amount of exposed sulphides (Akcil and Koldas, 2006).

Taking into account that pH values usually have a significant contribution when it comes to the increase in mobility of certain heavy metals and that tailings containing environmentally hazardous material are often deposited in the

immediate vicinity of important rivers, such as Moldova, Bistrița or Olt, the study of the conditions under which certain elements increase their mobile fraction becomes of great importance in the assessment of the polluting potential of these abandoned materials.

Several scientists and researchers (Stumbea, 2010; Ionce, 2010; Nagy-Korodi

et al., 2011 a.o.) have also conducted studies on the pollution occurring in soils adjacent to abandoned mining areas, or on the way in which heavy metals are distributed in relation to the physico-chemical environment and the mineralogy of the top-stratum of tailings mounds (Balaban et al., 2011a,b).

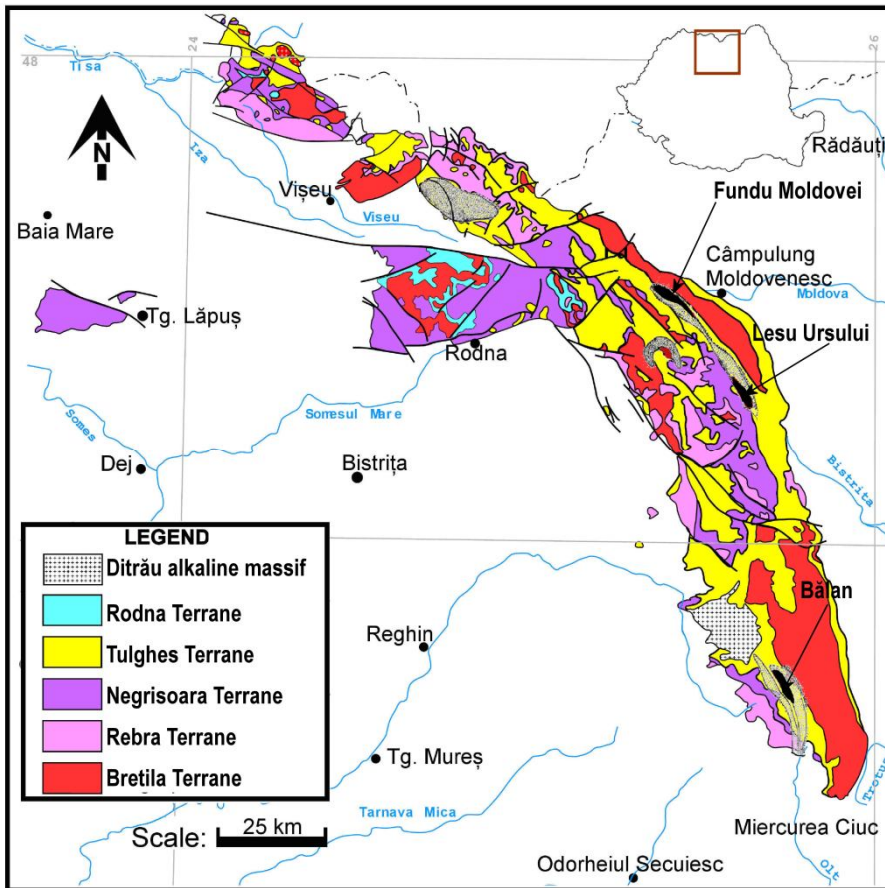


Fig. 1 Location of the three perimeters studied – Fundu Moldovei, Lesu Ursului and Bălan (modified from Balintoni, 2010).

Geographical and geological setting

1. Location and topography

In order to analyze element mobility in various environments and under various con-

ditions, the present study was aimed at some of the most important polymetallic sulphide ore mining districts in the Eastern Carpathians, namely those of Fundu Moldovei, Leșu Ursului and Bălan (Fig. 1).

The village of Fundu Moldovei lies in the western part of Suceava County, on the banks of the Moldova river, while the Leșu Ursului mining district is located further south, between the villages of Crucea (to the north) and Holda (to the south), on the left bank of the Bistrița river. The Bălan mining sector is located even further south, in the northeastern part of Harghita County, at the base of the Hășmașu Mare mountains and on the banks of the Olt river.

2. Geological setting

The materials from the three mining perimeters studied are all related to the Tulgheș metamorphic unit from the Crystalline-Mesozoic Zone of the Eastern Carpathians. Săndulescu et al. (1981) divided the area into the Infracuvonian, Subbucovinian and Bucovinian nappes. The last two have a basement consisting of the following Variscan nappes: Rodna, Pietrosu Bistriței, Putna, and Rarău. According to a model published by Balintoni et al. (1983), which was internation-

ally accepted and promoted by Krätner (1988), the Variscan nappes consist of the following metamorphic units: Bretila, Tulgheș (which corresponds to the Putna nappe), Negrișoara, and Rebra.

Afterwards, Liégeois et al. (1996) and Krätner (1996) started to gradually introduce the notion of *terrane* in relation to the Romanian Carpathians, describing five pre-alpine structures, as follows: the Carpien terrane, the Negrișoara terrane, the Tulgheș terrane (which represents the Tulgheș group), the Rodna terrane, and the Bistrița terrane.

Later, Balintoni (1997) simplified this model by describing the Bretila, Tulgheș, Negrișoara, Rebra and Rodna lithogroups and referring to the first four as tectono-stratigraphical terranes. Pană et al. (2002) attributed a peri-Gondwanan origin to these terranes.

The lithology of the Tulgheș lithogroup varies and it has been described by Balintoni (1997) as being dominated especially by white and black quartzites and quartzeous-feldspathic rocks.

Table 1 The samples and their locations

No. of samples	Name given to the sample series	Name of deposit	Type of deposit	Mining district
2	DNST	Dealul Negru	Ex-draining pool	Fundu Moldovei
2	P1ST	Prașca I	Mine tailings	Fundu Moldovei
2	P2ST	Prașca II	Mine tailings	Fundu Moldovei
4	ISST	Isipoaia	Mine tailings	Leșu Ursului
3	PUST	Pârâul Ursului – Puțul 7	Mine tailings	Leșu Ursului
2	PCST	Pârâul Câinelui	Mine tailings	Leșu Ursului
4	FCST	Fagul Cetății – Iazul 4	Ex-draining pool	Bălan
1	IOPP	Processed ore mound	Processed ore	Bălan

The Tulgheș metamorphic unit is characterized by low-degree metamorphism, and the polymetallic mineralization is associated to the Tg₃ formation, which is of a rhyolitic metasedimentary origin.

The polymetallic sulphides hosted within the Tg₃ formation are represented either by pyrite-chalcopyrite disseminations (such as the Bălan, Leșu Ursului and Fundu Moldovei

perimeters) or by galena-sphalerite-chalcopyrite (such as the Burloaia and Leșu Ursului perimeters) (Krätner et al., 1983).

Samples and analytical methods

1. Sampling

The present study was conducted on 20 samples collected from various deposits related to polymetallic sulphide ore mining in

the Eastern Carpathians, as shown in Table 1. The chosen deposits originate from the Fundu Moldovei (Fig. 2), Leșu Ursului (Fig. 3) and Bălan (Fig. 4) mining districts. The sampling

operation was performed with a manual-drill soil sampler, using methods described in the literature (Borlan and Răuță, 1981; Clichici and Stoici, 1986; Florea et al., 1986).

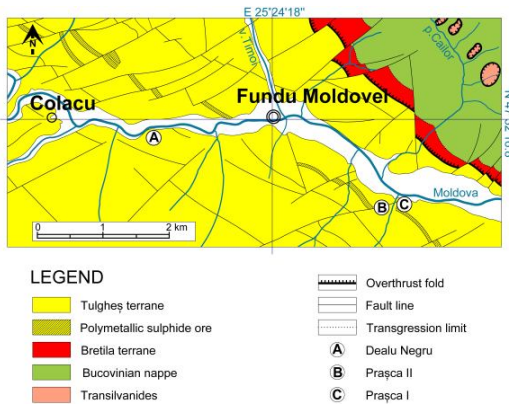


Fig. 2 Location of the deposits studied within the Fundu Moldovei mining district (modified from Petrescu et al., 1975).

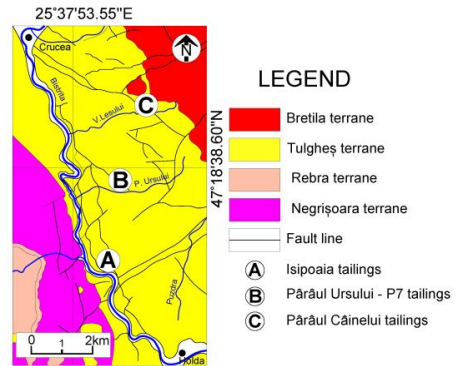


Fig. 3 Location of the deposits studied within the Leșu Ursului mining district (modified from Krätner and Bindea, 2002).

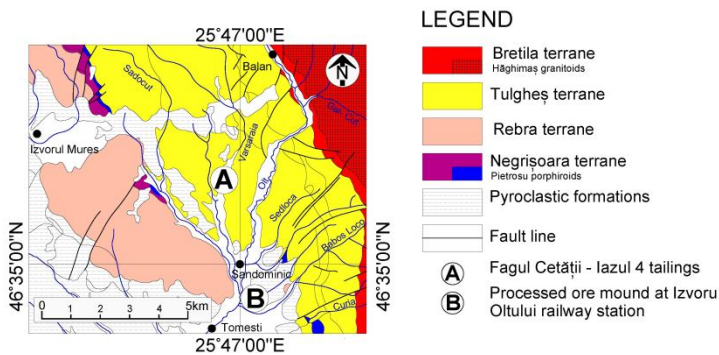


Fig. 4 Location of the deposits studied within the Bălan mining district (modified from Krätner and Bindea, 2002).

2. Sample preparation

The samples were dried at room temperature for 3 days, and then in the electric oven, at 40–50°C, for 8 hours. The coarse fractions ($\phi > 2.00$ mm) and traces of vegetation were subsequently removed from the dried samples.

The granulometric fraction with a diameter of less than 2 mm was then ground, first in an agate grinder, then in a “Fritsch” planetary mill, for 60 minutes, at a speed of 200 rpm (Clichici and Stoici, 1986), until a granulometric diameter of less than 0.01 mm was achieved for XRF, pH, Eh and leaching analyses.

3. pH and Eh determination

The pH was determined using the potentiometric method, in twice-distilled water: 10 g of sample for 25 mL of twice-distilled water, with a contact time of 30 minutes, at room temperature. For the pH determinations, a "pH-Meter Basic 20+" was used along with a couple of electrodes: calomel (used as reference) and a pH-measuring electrode.

The redox potential was determined directly, using the process of suspension in twice-distilled water: 10 g sample for 25 mL of twice-distilled water and a contact period of 45 minutes, at room temperature. For the Eh determinations, a "pH-100" potentiometer, along with a couple of electrodes: platinum and calomel (for reference) (Florea et al., 1986; Bloom, 2000), were used.

4. Heavy metal contents

The chemical contents for major and trace elements were determined by means of X-Ray Fluorescence. Powdered samples were analyzed using a PANalytical Axios and a PW2400 spectrometer. The samples were prepared through mixing with a flux material and melting into glass beads. The beads were analyzed by means of wavelength dispersive X-ray fluorescence spectrometry.

In order to determine loss on ignition, (LOI), 1000 mg of sample material were heated at 1030°C for 10 min. After being mixed with

5.0 g of lithium metaborate and 25 mg of lithium bromide, the residue was fused at 1200°C for 20 min. The calibrations were validated through analyses of reference materials. "Monitor" samples, as well as 130 certified reference materials (CRM), were used for the correction procedures. The analyses were carried out at the Federal Institute for Geosciences and Natural Resources (BGR) in Hannover, Germany.

5. Leaching analysis

The leaching analysis was carried out at the Federal Institute for Geosciences and Natural Resources (BGR) in Hannover, Germany, through acetic acid extraction: 1g of sample material was extracted overnight using 40 mL 0.1 M of acetic acid (with a pH of 2.88). The extract was then separated from the residue through centrifugation, at a speed of 3000 rpm.

Afterwards, the liquid was decanted and element concentrations were measured by means of ICP-OES.

Results and discussions

As shown in Table 2, the pH and Eh values for the studied samples generally revealed a highly acid environment, with the exception of the 3 samples collected from the Pârâul Căinelui tailings, which showed a rather reducing and neutral-to-slight-alkaline environment.

Table 2 The pH and redox potential for the studied samples

Sample	pH	Eh [V]	Sample	pH	Eh [V]
DNST5	3.03	0.389	PUST10	2.89	0.492
DNST13	2.61	0.476	PUST14	7.22	0.324
P1ST1	3.90	0.392	PCST13	7.96	0.229
P1ST3	5.12	0.280	PCST18	8.08	0.246
P2ST2	3.44	0.412	PCST19	7.23	0.244
P2ST3	4.60	0.340	FCST17	3.78	0.397
ISST2	2.36	0.517	FCST26	2.94	0.439
ISST5	6.43	0.330	FCST30	4.02	0.368
ISST7	2.86	0.560	FCST39	6.36	0.334
ISST13	2.29	0.570	IOPP4	2.29	0.431

Table 3 Major elements contents (mg/kg) of the studied samples

Sample	As	Co	Cr	Cu	Fe	Mn	Ni	Pb	Sn	Zn
DNST5	159	10	5	481	88060	225	3	414	28	125
DNST13	188	<4	6	843	89319	232	<3	2748	26	281
P1ST1	20	<3	<4	77	38120	248	<2	64	<13	91
P1ST3	22	<3	<4	73	40778	294	<2	122	<13	99
P2ST2	35	<3	<4	213	40988	302	<2	341	<13	145
P2ST3	14	21	57	100	38260	550	27	22	13	93
ISST2	594	11	62	674	105547	186	9	1927	62	242
ISST5	157	28	80	263	46933	759	55	119	13	570
ISST7	435	7	73	742	95405	294	18	660	17	513
ISST13	585	14	93	818	160243	728	41	1404	62	524
PUST10	370	13	94	722	97223	720	28	821	29	349
PUST14	137	18	67	323	46933	643	28	618	17	372
PCST13	9	15	86	44	37700	565	31	27	13	100
PCST18	20	10	97	30	49661	449	37	69	13	81
PCST19	25	30	136	75	50990	1673	86	42	13	152
FCST17	164	45	41	1698	103518	767	15	537	36	592
FCST26	294	27	61	705	115759	829	17	1122	43	345
FCST30	173	65	29	1311	104148	751	17	332	31	482
FCST39	135	68	67	2485	121424	1324	30	535	40	938
IOPP4	997	320	9	4608	318528	108	48	2508	83	278

* The contents in bold are below the detection limit of the device

Table 4 Major elements contents (mg/kg) of the extracts

Sample	As	Co	Cr	Cu	Fe	Mn	Ni	Pb	Sn	Zn
DNST5	<0.02	0.024	<0.02	0.531	23.3	0.65	0.22	<0.05	<0.01	0.244
DNST13	<0.02	0.021	<0.02	3.110	14.0	0.52	<0.05	1.38	<0.01	0.842
P1ST1	<0.02	<0.02	<0.02	0.410	6.0	0.15	<0.05	<0.05	<0.01	0.010
P1ST3	<0.02	<0.02	<0.02	0.233	6.0	0.53	<0.05	<0.05	<0.01	0.138
P2ST2	<0.02	<0.02	<0.02	2.110	10.1	1.15	<0.05	<0.05	<0.01	2.020
P2ST3	<0.02	0.620	<0.02	2.070	13.8	6.92	0.06	<0.05	<0.01	0.623
ISST2	<0.02	0.189	<0.02	6.950	27.6	2.46	0.12	3.30	0.016	5.2
ISST5	<0.02	1.070	<0.02	5.350	15.3	14.4	0.98	0.53	<0.01	19.9
ISST7	<0.02	0.235	<0.02	7.640	39.0	5.56	0.26	<0.05	<0.01	20.1
ISST13	<0.02	0.244	0.388	12.000	187	11.0	0.44	<0.05	<0.01	14.0
PUST10	<0.02	0.292	<0.02	2.930	9.2	103.0	0.13	0.14	<0.01	2.29
PUST14	<0.02	0.443	<0.02	2.520	11.5	11.0	0.22	8.90	<0.01	4.90
PCST13	<0.02	0.366	0.036	0.294	147	12.8	0.15	<0.05	0.010	0.590
PCST18	<0.02	0.010	<0.02	0.113	20.7	8.90	0.08	<0.05	<0.01	0.243
PCST19	<0.02	0.350	<0.02	0.408	30.5	22.0	0.32	<0.05	<0.01	0.735
FCST17	<0.02	0.293	<0.02	33.000	31.7	8.50	0.07	2.50	<0.01	10.20
FCST26	<0.02	0.675	<0.02	2.990	6.4	23.2	0.11	<0.05	<0.01	0.960
FCST30	<0.02	0.642	<0.02	8.000	27.6	27.7	0.10	1.60	<0.01	7.960
FCST39	<0.02	0.705	0.023	26.600	59.5	31.3	0.17	2.20	<0.01	12.5
IOPP4	<0.02	0.737	0.033	12.700	410	1.05	0.13	<0.05	0.044	5.50

* The contents in bold are below the detection limit of the device

Table 5 The mobile fraction (%) for each heavy metal from the studied samples

Sample	As	Co	Cr	Cu	Fe	Mn	Ni	Pb	Sn	Zn
DNST5	< 0.013	0.240	< 0.400	0.110	0.026	0.289	7.333	< 0.012	< 0.036	0.195
DNST13	< 0.011	< 0.525	< 0.333	0.369	0.016	0.224	< 1.667	0.050	< 0.038	0.300
P1ST1	< 0.100	< 0.667	< 0.500	0.532	0.016	0.061	< 2.500	< 0.078	< 0.077	0.011
P1ST3	< 0.091	< 0.667	< 0.500	0.319	0.015	0.180	< 2.500	< 0.041	< 0.077	0.139
P2ST2	< 0.057	< 0.667	< 0.500	0.991	0.025	0.381	< 2.500	< 0.015	< 0.077	1.393
P2ST3	< 0.143	2.952	< 0.035	2.070	0.036	1.258	0.222	< 0.227	< 0.077	0.670
ISST2	< 0.003	1.718	< 0.032	1.031	0.026	1.323	1.333	0.171	0.026	2.149
ISST5	< 0.013	3.821	< 0.025	2.034	0.033	1.897	1.782	0.445	< 0.077	3.491
ISST7	< 0.005	3.357	< 0.027	1.030	0.041	1.889	1.444	< 0.008	< 0.059	3.918
ISST13	< 0.003	1.743	0.417	1.467	0.117	1.511	1.073	< 0.004	< 0.016	2.672
PUST10	< 0.005	2.246	< 0.021	0.406	0.009	14.300	0.464	0.017	< 0.034	0.656
PUST14	< 0.015	2.461	< 0.030	0.780	0.025	1.711	0.786	1.440	< 0.059	1.317
PCST13	< 0.222	2.440	0.042	0.668	0.390	2.264	0.484	< 0.185	0.077	0.590
PCST18	< 0.100	0.100	< 0.021	0.377	0.042	1.981	0.216	< 0.072	< 0.077	0.300
PCST19	< 0.080	1.167	< 0.015	0.544	0.060	1.315	0.372	< 0.119	< 0.077	0.484
FCST17	< 0.012	0.651	< 0.049	1.943	0.031	1.109	0.467	0.466	< 0.028	1.723
FCST26	< 0.007	2.500	< 0.033	0.424	0.006	2.800	0.647	< 0.004	< 0.023	0.278
FCST30	< 0.012	0.988	< 0.069	0.610	0.027	3.687	0.588	0.482	< 0.032	1.651
FCST39	< 0.015	1.037	0.034	1.070	0.049	2.363	0.567	0.411	< 0.025	1.333
IOPP4	< 0.002	0.230	0.367	0.276	0.129	0.968	0.271	< 0.002	0.053	1.978

*The contents in bold are below the detection limit of the device

Table 6 The correlation factors between the pH and Eh conditions and the mobile fraction for the most significant elements

	Eh	pH	Fe	Mn	Co	Cu	Ni	Zn
Eh	1.00	< -0.90	-0.34	0.24	0.18	0.07	0.13	< 0.52
pH	< -0.90	1.00	0.32	-0.15	0.05	0.01	-0.25	-0.30
Fe	-0.34	0.32	1.00	-0.14	0.04	-0.10	-0.15	-0.08
Mn	0.24	-0.15	-0.14	1.00	0.17	-0.23	-0.21	-0.17
Co	0.18	0.05	0.04	0.17	1.00	< 0.48	-0.17	< 0.42
Cu	0.07	0.01	-0.10	-0.23	< 0.48	1.00	-0.23	< 0.50
Ni	0.13	-0.25	-0.15	-0.21	-0.17	-0.23	1.00	-0.07
Zn	< 0.52	-0.30	-0.08	-0.17	< 0.42	< 0.50	-0.07	1.00

* The most significant correlation coefficients are in bold

After determining the contents of heavy metals both in the studied samples (Tab. 3) and in their corresponding extracts (Tab. 4), a percentage of mobile fraction was calculated (Tab. 5), taking into account the initial content of the sample for each of the elements, as well as a correlation matrix for the most conclusive values (Tab. 6).

As and Sn contents in the extract were below the detection limit of ICP-OES, which leads to the conclusion that their mobility is highly reduced under the conditions of the leaching (pH = 2.88).

As shown in Figure 5, Fe started to gradually increase its mobility at pH values lower than 2.36 (sample ISST2), maintaining a low,

relatively constant mobility at higher pH values, with the exception of sample PCST13, where, at a pH of 7.96 and a redox potential of 0.229 V, it reached its peak mobility.

Zn tended to gain mobility as the redox potential was increasing (Fig. 6) towards the strong oxidizing domain, and its peak mobility was obtained for sample ISST7, where the re-

dox potential reached 0.560 V. The mobility of Zn showed a clear correlation with the redox potential, rather than the pH, in relation to which it remained relatively independent. The same tendency was observed for Cu and for the valid measurements for Co, as the three elements formed a distinct inter-correlated group (Table 6).

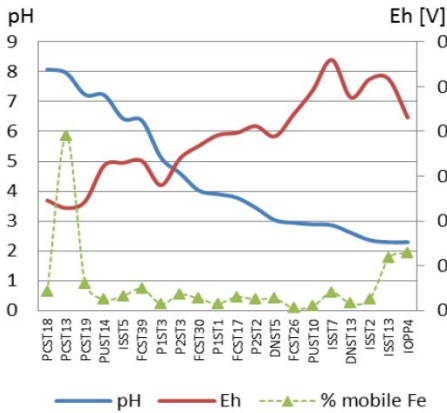


Fig. 5 The mobility of Fe vs. Eh and pH.

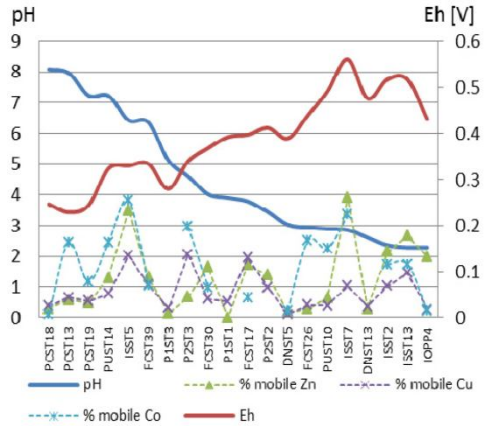


Fig. 6 The mobility of Zn, Cu and Co vs. Eh and pH.

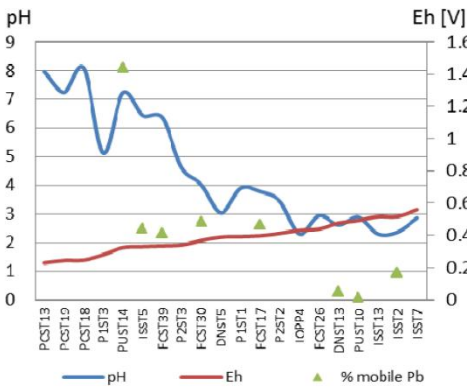


Fig. 7 The mobility of Pb vs. Eh and pH.

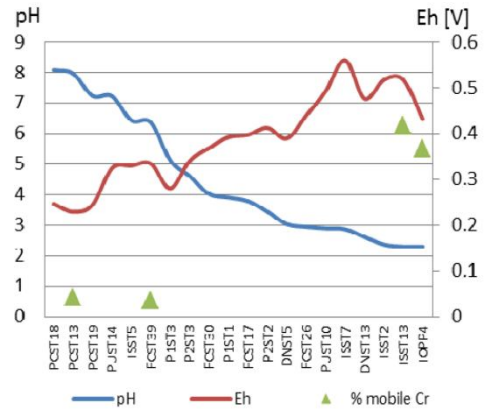


Fig. 8 The mobility of Cr vs. Eh and pH.

Based on the valid measurements obtained for Pb, one can conclude that, overall, Pb decreased its mobility with the decrease of the pH values (Fig. 7), from a peak obtained for

sample PUST14, namely a pH of 7.22 and an Eh value of 0.324 V. The opposite tendency was observed in the case of the valid measurements obtained for Cr, which leads to the conclusion

that this element increases its mobility at low pH values and redox potentials associated to the strong oxidizing domain (Fig. 8).

Mn showed a mobility that is independent from the pH and Eh of the samples (Fig. 9), with a maximum value obtained for sample PUST10, at a pH of 2.89 and a redox potential of 0.492 V. Ni displayed a similar trend, revealing its mobility to be independent of the

pH and Eh (Fig. 10). Its peak was reached in sample DNST5, with a pH of 3.03 and a redox potential of 0.389 V.

Under the given leaching conditions, the elements with the greatest mobility were Mn, Co, Zn and Ni, while the least mobile ones were As, Sn and Fe. An average degree of mobility can be attributed in this case to Cr, Pb and Cu (Table 7).

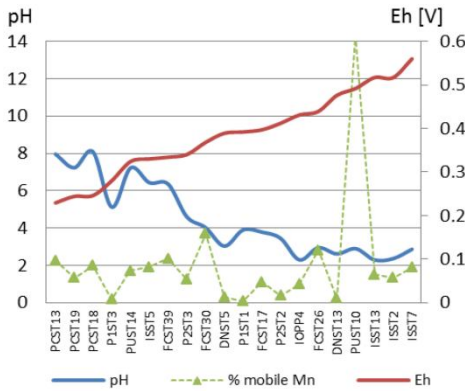


Fig. 9 The mobility of Mn vs. Eh and pH.

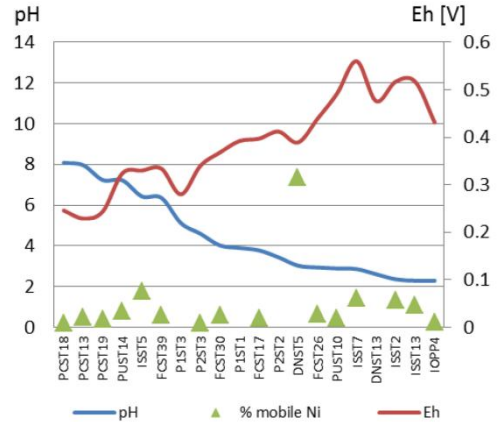


Fig. 10 The mobility of Ni vs. Eh and pH.

Table 7 The average percent of mobile fraction for each studied element

Element	Average percent of mobile fraction
As	< 0.045
Sn	< 0.052
Fe	0.055
Cr	0.215
Pb	0.435
Cu	0.852
Ni	1.128
Zn	1.262
Co	1.728
Mn	2.075

Conclusions

The pH and Eh analyses revealed physico-chemical environments varying from a slightly alkaline pH and reducing conditions, to highly acid pH and strong oxidizing conditions. The slightly alkaline conditions encountered at Pârâul Cânelui are most probably due to the talings age and can also be linked to a higher quantity of carbonates, due to the proximity of carbonatic Holdița lithozone (Al et al., 1994; Balintoni, 1997; Dold, 1999; Sobek et al., 1978; Paktunc, 1999).

Low pH values corresponded to increased mobility in the case of Fe and Cr, and decreased mobility for Pb, while Zn was found to increase its mobile fraction towards the oxidizing domain of redox potential values. The mobile fractions for Co and Cu were

slightly correlated with Zn, displaying similar tendencies.

Mn and Ni values could not be correlated with either the pH or the Eh, revealing a mobility degree which is relatively independent of these parameters.

As and Sn had the lowest mobility of all the studied elements, with average values for the mobile fraction under 0.052%, in contrast to Mn and Co, which had the highest average mobile fraction – around 2%. The rest of the elements – Fe, Cr, Pb, Cu, Ni and Zn – displayed average mobile fractions, between 0.055% and 1.26%.

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