General characteristics of rare earth and radioactive elements in Dong Pao deposit, Lai Chau, Vietnam

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ABSTRACT

One of the important rare earth deposits is the Dong Pao localized in Lai Chau province, West-North of Vietnam. Generally, the deposit is composed of two parts, the lower-and the upper. The lower part is composed of dolomite, limestone and intrusive rocks, while the upper part of the profile is represented by a weathered zone containing soil and fragments of mixed barite-fluorite ores. The concentrations of natural radionuclides, chemical compositions of ores, including rare earth elements (REE) in solid samples, were determined by gamma spectrometer equipped with a HPGe detector, laser ablation inductively coupled plasma mass spectrometry (LA ICPMS) and activation method, respectively. In the samples taken from the ore bodies within the weathered zone the REE concentration is about 10 wt.% and both ^{238}U and ^{232}Th amount to 0.01 wt.%, while in the samples from the hard part of ore the REE and ^{238}U as well as ^{232}Th contents amount only to 0.3 wt.% and 0.001 wt.%, respectively. So the enrichment of the REE and natural radioactive elements in the deposit is a consequence of the weathering processes. The water samples were taken from the streams, natural tap and thermal water intakes localized in the studied deposit and surrounding region. The ^{238}U, ^{234}U, ^{228}Ra, ^{226}Ra concentrations in the water samples were prepared by the adequate radiochemical procedures and measured using an alpha spectrometer coupled with silicon semiconductor detector and α/β liquid scintillation counter. In the stream water, the concentrations of both ^{226}Ra and ^{228}Ra vary from 100 to above 300 mBq/L, while in the natural tap and thermal waters they amount to tens mBq/L. The concentration of ^{238}U, ^{234}U in the thermal water is 80 and 110 mBq/L respectively, while in the surface water concentrations of uranium isotopes are below 30 mBq/L.

Keywords: REE, natural radionuclides, weathering zone, enrichment, surface and thermal waters, Dong Pao deposit, Lai Chau Vietnam.

1. Introduction

The rare earth elements (REE) play an increasingly important role in the world economy. REE are implicated in many technologies associated with energy, electronic, nanomaterials, hybrid car components and others. Due to the production of spare parts of hybrid

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cars, wind turbines, battery alloys, magnets, aerospace, the future demand for REE is expected to increase to above 25 times in comparison with the current needs (Hoatson et al. 2011, Curtis 2011, Damascena et al., 2015). What is more in some countries the REE are even treated as economically and politically strategic commodities (Damascena et al., 2015).

Vietnam is a country located in the Indochina Peninsula with the marine coastline extending over more than 1500 km making beach sands a resource of great REE potential (Hou 2005). Apart from that there are two very rich REE deposits in Vietnam, i.e. Nam Xe and Dong Pao. Both are located in Lai Chau province, Northwest Vietnam (Kušnir 2010; Pham Ngoc Can et al. 2011). These deposits were discovered in the middle of the XX Century and are continuously investigated (Fromaget 1941; Fromaget & Saurin 1952; Dovzikov et al., 1965; Le Thac Xinh et al., 1988; Tran Trong Hoa et al., 2010; Pham Ngoc Can et al., 2011), but up to date some problems e.g. 3-4D models still require further examination.

In the scope of the Bilateral Collaborative Project between Hanoi University of Mining and Geology (HUMG) and AGH University of Science and Technology (AGH UST) Kraków, Poland No. 01/2012/HD-HTQTSP, on November 2015 the AGH UST delegation visited Dong Pao REE deposit and performed a geological survey to collect ore samples from both the upper weathered zone and the lower - drill core hard rocks from limestone and dolomite sections, and water samples from streams, natural tap waters and thermal spring in the deposit area and its surrounding. The aim of this work is to determine the chemical, rare earth and radioactive element concentrations in the solid samples. The obtained results together with the archival data serve as background to qualify the characteristic parameters of the REE deposit in question.

Water samples were collected and analyzed to characterize the impact of the ores on the local environment, which are responsible for transportation of radioactive elements.

2. Geological setting

The Dong Pao Rare Earth deposit is situated in the Northwest Vietnam, within the geographic coordinates of 103°32'37''-103°33'46''E and 22°18'84''- 22°19'13''N, occupying an area of about 120 km^2 (Le Khanh Phon et al., 2015). From the tectonic point of view this deposit belongs to the Son La-Lai Chau zone bounded in the east by the Red River fault and in the west by the Ma River suture zone (Figure 1). Within the Ma River suture zone there are a few intrusive units such as the Fan Si Pan Massif, Posen batholith and Muong Hum granite. The Fan Si Pan Massif is composed of granites and metagranites separated by narrow intercalations of Neoproterozoic metasediments, mainly mica schists and marbles. In this massif, the youngest igneous unit is the Yensun granite of Paleogene age (Żelaźniewicz et al., 2013). The Precambrian Posen batholith is characterized by the variability in composition of mafic veins. This unit contains also migmatic patches and gneisses (Żelaźniewicz et al., 2013). The Muong Hum unit is characterized by the fine-grained alkali granite and syenite. This suit is poor in Sr, P, Ti, Ba, Ca but rich in Rb, Zr, Hf including REE up to 780 ppm. Based on geochemical characteristics, Żelaźniewicz and co-workers (2013) suggested that the Muong Hum granite was associated with either continental rifts or mantle plumes.

In the investigated deposit there is the Dong Pao syenite, this formation occupies an area of 8.9 km^2 trending NW-SE with length up to 5.5 km. The alkali syenite, porphyritic syenite are the main rocks of the Dong Pao granite, in which the orthoclase (63-92%), plagioclase (5-15%), quartz (2-20%), biotite, (2-6%) and pyroxene (0-8%) are the major minerals, and zircon, apatite and leucite are the minor ones.
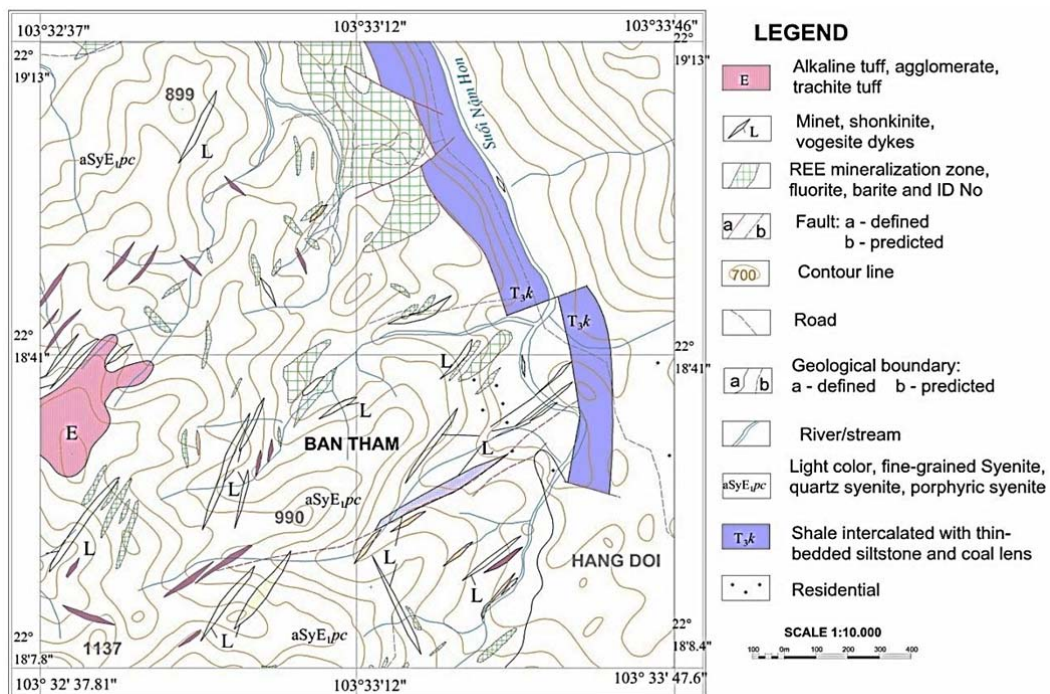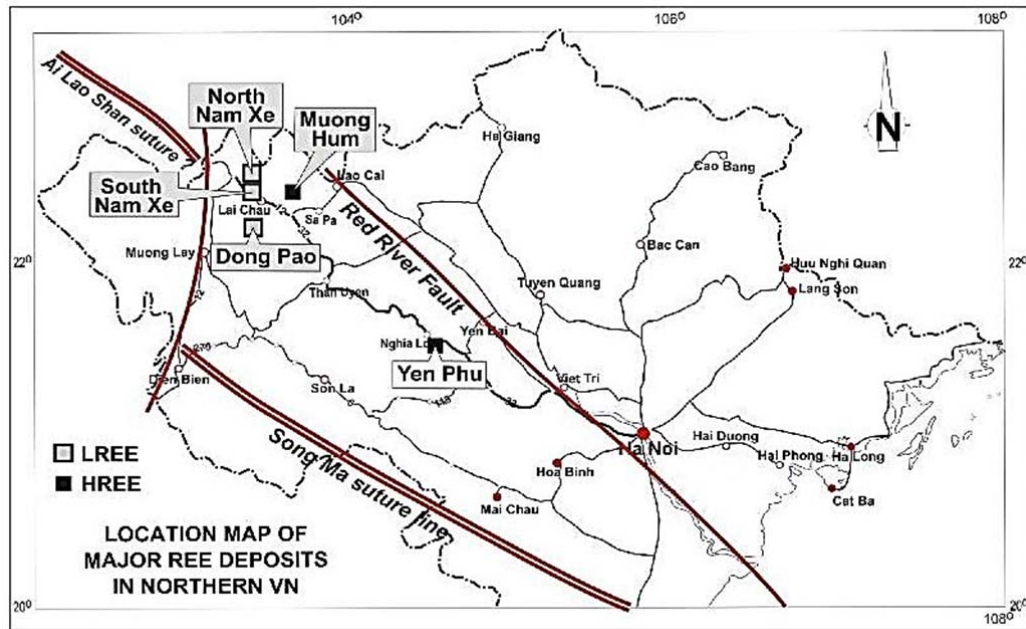
Figure 1. Geological sketch of Dong Pao Rare Earth Deposit on the background of the tectonic sketch of the Indo-china block (part of the North-West Vietnam)

A sedimentary sequence in the Dong Pao area is composed of Devonian siltstone-mudstone formation, Permian limestones, and bauxites, and iron-bearing formations covered conformably by Early and Middle Triassic fine grained grey limestone. These rocks are overlain by the Late Triassic conglomerates and sandstones and in some places with Cretaceous red beds (Tran Van Tri, 2011; Żelaźniewicz et al., 2013; Faure et al., 2014; Halpin et al., 2015). According to Nguyen Tien Du et al., 2011, there are 17 ore bodies in the studied area. The thickness of the ore bodies varies from tens to above 500 m and length of separate ore bodies vary from 300 to 1000 m. Subcrops of the ore bodies were sampled. The weathered zone is composed of altered fragments of barite-fluorite ores cemented with REE carbonates mixed with clay and Fe- Mn hydroxides. In the area, there are many streams flowing along the region relief, the main streams being Nam Hon and Nam Nu. The Nam Hon in the West flows along the trend of rock formations, and in the North, there is the Nam Nu stream crossing near perpendicularly the barite and fluorite bodies in the region. At some places, there are karstic caves, especially there where carbonate formations occur. In the area, the water is recharged from the surface reservoirs and is used by people for daily life and agriculture.

3. Sampling and analytical methods

3.1. Sampling

After the Conference on the Earth Sciences held at HUMG on November 2015, the AGH-UST group made a geological reconnaissance and collected some ore samples P1, P2 in the weathered upper surface soil (Figure 2) and fragments of drill cores KF-133, LK-122 (Figure 3) following the information by geologists working in place. The samples were collected and analyzed to compare results with the existing data of the ore bodies numbered as follows: F4, F7, F9, F10, F14, F16, F17.

The water samples were collected from the Nam Hon (F4), Nam Nu streams (P1), from the thermal spring located 200 m Southeast from the deposit (TM) and from the artificial concrete container built at the geological office (F9). The tap water in the container is recharged from rain water flowing from the mountain and lead by the bamboo pipes and finally reaches to the cement container; in the paper, this water is defined as natural tap water. All the collected samples were shipped and analyzed at AGH-UST laboratories.

Figure 2. The samples collected from the weathered (upper) zone

Figure 3. The core samples

3.2. Analytical methods

To determine the chemical and REE composition, the solid samples were sent to Bureau Veritas Mineral Laboratories in Vancouver Canada, which possesses the Certificate of Analysis No KRA15000229.1. At the Veritas Mineral Laboratories, the chemical composition of the solid samples is analyzed by laser ablation with induced plasma coupled with the mass spectrometer (LA ICP MS), the REE are analyzed using instrumental neutron activation method (INAA). The LA ICP MS consists in the generation of the fine particles by a laser beam focused on the sample surface, the process known as Laser Ablation. Then the ablated particles are transported to the secondary excitation source of the ICP-MS instrument for digestion and ionization. The excited ions in the plasma torch are subsequently introduced to a mass spectrometer detector for elemental analysis. The LA ICP MS instrument uses the laser wavelength of 193 nm with 14 J/cm^2 of the power energy density for ablation, in ICP MS the power of 1350W for RF generation, the 14 L/min and 0.9 L/min argon gas for plasma torch and auxiliary respectively (Liu et al., 2008). The ICP-MS is calibrated using the NIST standard samples of well known chemical composition. The background of the instrumental neutron activation method can be briefly described as follows: an analyzed sample is subjected to a thermal neutron flux and radioactive nuclides are produced. As these radioactive nuclides decay, they emit gamma rays of characteristic energies for each nuclide. Comparison of the intensity of these gamma rays with those emitted by a standard, one can estimate quantitatively the concentrations of the various nuclides.

The sample for gamma measurement was ground until the grains were less than 2 mm in diameter, then it was dried in an oven at 120°C for 24 hours to ensure that moisture was completely removed, then weighted accurately and packed in the aluminum cylindrical Marinelli beaker of 720 ml capacity and sealed to prevent escape of radon. The sealed samples were left for at least 22 days to reach secular equilibrium between the ^{222}Rn and ^{226}Ra. The activity concentrations of ^{40}K, ^{226}Ra and ^{232}Th were determined using the gamma-ray spectrometer coupled with HPGe detector of the relative efficiency of 42% and resolution of 1.9 keV for 1332 keV line and calibrated using the IAEA reference materials RGU, RGTH, RGK as standard samples. The

gamma lines 609.3 keV (46.1%), 1120.3 keV (15.0%) and 1764.5 keV (15.9%) from [214]Bi were used to determine the activity concentration of [226]Ra, while that of [232]Th were determined from the gamma lines of 911.2 keV (29.0%) from [228]Ac and 583.0 keV (30.9%) and 2614.4 keV (35.8%) from [208]Tl. For [40]K, its activity concentration was determined from its 1461 keV gamma line Jodłowski and Kalita (2010).

The chemical composition of the water samples was analyzed using an ICP-AES PerkinElmer Optima 7300 DV spectrometer. The principal of the ICP-AES is similar to the LA ICP MS, but in the ICP-AES the laser ablation is not needed and instead of the atomic mass measurements, the wavelength of the atomic spectral lines is measured. The condition parameters of ICP of both instruments ICP-AES and LA ICP MS are similar. The ICP-AES is calibrated with a multi-element standard solution of the Merck® company.

The uranium isotopes in the water samples were precipitated together with the manganese oxide, then the obtained sample was dissolved in HCl 9M and transmit through the Dowex resin exchange column. The uranium ions were rinsed from the resin column by adding the HCl 0.1M and again precipitated using the neodymium chloride. The precipitate was placed onto the plastic filter of 0.1 μm porosity and measured using an alpha spectrometer coupled with silicon PIPs detector. To control the chemical yield and determine the [238]U and [234]U concentrations, the trace quality amount of [232]U solution was added into the studied water sample at the beginning of the chemical procedure (Nguyen et al., 2010).

The radium isotopes were precipitated from the water sample together with barium as the sulfate compound, then the obtained precipitate was cleared up from the other messing isotopes by dissolution it in the EDTA solution. The radium was again precipitated though decreasing pH sample to 4.5 by adding the acetic acid. The precipitate was washed using distilled water and centrifugal machine, then placed into the special glass vial of 22 mL and mixed with the gel scintillation cocktail of 12 ml. The sample was measured using the Wallac 1414 α/β Liquid Scintillation counter (Nguyen et al., 1998). The quality of the used methods was tested by comparing many measurements done by several international organizations.

4. Results and discussions

Table 1 summarizes the measured oxides of the main metals and some trace elements for the collected solid samples. Table 2 presents the ranges and average concentrations of some oxides of the selected ore bodies reported by Nguyen Tien Du et al., 2011. Comparing the data in both Table 1 and Table 2, we can see that the data presented by this work are contained in the ranges of the data given by Nguyen Tien Du et al., 2011, but all the values of the oxides analyzed by us are comparable with the minimum values in the ranges of the adequate oxides. The calcium, silicon, and iron are principal components of the carbonate rocks. Based on the data of this work, the analyzed oxides in the borehole cores and weathered zone (soil) in the forms of vertical bars are presented in the Figure 4a, 4b, and 4c. The Ca, Mg, Fe and alkali metals (Na, K, Cs) are very vulnerable to weathering processes resulting their concentrations in soil are far lower than that in hard rock by several times, while Mn, Ti, Sc, Co, and Be are very resistant and enriched to several folds in the weathered zone. According to Rösler and Lange (1972), the weathering degree can be appreciated using the weathering index defined by the formula:

$$V_i = \frac{CaO + MgO + Na_2O + K_2O + H_2O}{SiO_2 + Al_2O_3 + CaO + MgO + Na_2O + K_2O} \quad (1)$$

The oxides in formula (1) are expressed in mole units, and the weathering index for studied samples is equal to 0.187, 0.076, 0.731 and 0.700 for P1, P2, LK122 and KF-133 respectively.

Table 1. Analyzed concentrations of the metal oxides and trace elements in the collected samples

sample	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	MnO %	Cr_2O_3 %	Ni ppm	Sc ppm	Ba ppm	Be ppm	Co ppm	Cs ppm
P1	6.76	0.16	0.98	0.01	14.58	0.01	0.01	0.04	0.23	0.71	0.012	20	4	50000	2	2.2	0.1
P2	8.23	0.23	1.94	0.01	6.27	0.01	0.01	0.07	0.39	1.13	0.011	20	3	50000	2	4.5	0.1
KF-133	1.66	0.81	2.75	0.31	49.41	0.01	0.32	0.03	0.01	0.17	0.002	20	1	50000	1	1	0.7
LK-122	2.37	0.07	1.99	0.16	51.52	0.01	0.02	0.02	0.85	0.16	0.002	20	1	16778	1	1	0.1

Table 2. Ranges and average values of concentrations of the metal oxides (%) in the ore bodies (Nguyen Tien Du et al., 2011)

| Ore body	SiO_2	Al_2O_3	Fe_2O_3	P_2O_5	CaO	TiO_2	PbO	ZnO
F4	5.34-52.8	7.08-70.30	1.22-12.01	0.11-1.12	0.06-50.83	0.04-1.01	0.08-0.97	0.02-0.21
	(22.77)	(29.56)	(4.42)	(0.38)	(6.86)	(0.33)	(0.34)	(0.06)
F7	7.08-70.30	0.35-22.64	0.98-20.64	0.02-1.78	0.00-41.43	0.04-1.14	0.04-0.90	0.02-0.34
	(29.56)	(8.30)	(4.82)	(0.13)	(6.39)	(0.37)	(0.24)	(0.10)
F9	2.02-58.53	0.35-27.79	1.08-20.94	0.02-1.30	0.04-41.39	0.02-2.11	0.06-0.82	0.01-0.26
	(15.70)	(5.48)	(3.04)	(0.12)	(5.50)	(0.22)	(0.29)	(0.06)
F10	1.92-62.22	0.40-39.27	1.56-12.91	0.02-0.63	0.04-53.47	0.05-1.12	0.04-1.39	0.02-0.35
	(24.84)	(8.41)	(5.79)	(0.12)	(4.48)	(0.50)	(0.37)	(0.11)
F14	5.88-42.62	1.56-12.96	1.19-30.96	0.04-4.02	0.11-43.60	0.05-0.81	0.12-1.15	0.03-0.45
	(24.00)	(6.09)	(12.65)	(1.34)	(7.65)	(0,33)	(0.36)	(0.15)
F16	1.29-44.91	0.33-21.19	3.45-22.13	0.20-5.16	0.00-32.47	0.05-1.10	0.09-1.22	0.05-0.66
	(17.19)	(5.94)	(10.95)	(0.92)	(2.60)	(0.49)	(0.46)	(0.26)
F17	20.95-59.06	6.75-15.65	4.66-16.34	0.37-1.77	0.00-21.15	0.27-0.58	0.14-1.30	0.04-0.17
	(46.01)	(12.93)	(9.24)	(0.70)	(1.33)	(0.40)	(0.33)	(0.07)

In Vietnam, there is the tropical climate, and in the region the intensive vegetable farming resulting in the surface water being rich on CO_2, ammonium and the occurrence of a lot of deep fractures in the rocks. The alkaline elements are leached from the rocks by a chemical reaction as follows:

$$RSiO_2 + CO_2 + H_2O = RCO_3 + SiO_2 + H_2O$$
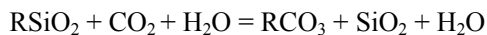
where R are Ca, Mg, Fe, Na_2, K_2, Cs_2…

The REE concentrations determined for the collected samples and the REE average concentrations of the ore bodies reported by Nguyen Tien Du et al., 2011 are summarized in Table 3 and Table 4 respectively. Both the data and ΣLREE/ΣHREE in the Nguyen Tien Du and co-workers report (2011) are comparable with that of the samples from the upper weathered zone, which can be connected with the fact that the samples presented in the Nguyen Tien Du et al., (2011)'s report were taken from the weathered zone.

The REE concentration in the weathered zone reaches to 10 wt.%, so the studied deposit can be classified to one of the richest REE deposits in the World (Hoatson et al., 2011). The patterns of the REE and radionuclides in the weathered and hard rocks are similar (Figure 5), but their contents in the weathered zone are enriched to near forty fold in comparison to that in the hard rock (Table 4). The ratio (ΣLREE/ΣHREE) in the studied samples ranged from 56 for the hard rock to 121 for weathered one. According to Hornig-Kjargaard, 1998 and Żelaźniewicz et al., 2013, the mentioned phenomena can be explained that at the beginning the rare elements were within the intrusive formations Muong Hum and/or Dong Pao syenite (cf. Figure 1), then they underwent enrichment due to thermal metamorphic processes occurring in the carbonatite formations, so the REE ores were formed as lenses or dykes of various shapes and dimensions. Finely the REE were enriched by the surface chemical weathering processes.
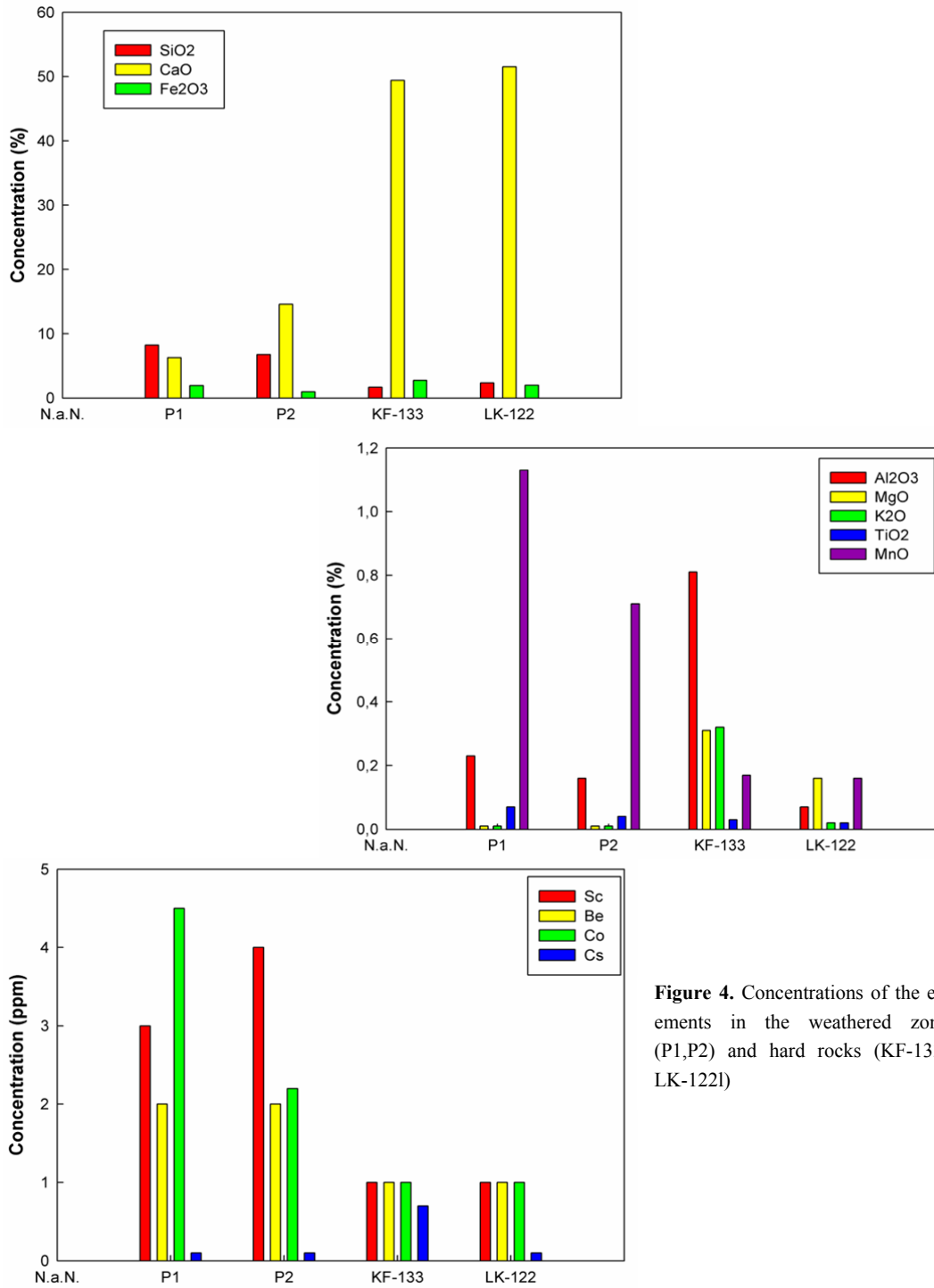
**Figure 4.** Concentrations of the elements in the weathered zone (P1,P2) and hard rocks (KF-133, LK-122l)

Table 3. Measured concentrations of the rare earth and radioactive elements (ppm) and weathering index (V_i) for the collected samples

Elements	P1	P2	KF-133	LK-122
La	39240	31100	798	823
Ce	44600	36000	1340	1240
Pr	3540	2990	122	104
Nd	9830	8390	381	326
Sm	903	783	43.6	39.0
Eu	212	183	10.1	9.7
Gd	583	494	26.8	24.8
Tb	37.6	33.1	2.6	2.2
Dy	122	112	11.0	9.0
Ho	13.3	12.2	1.4	1.3
Er	30.0	25.5	3.2	2.9
Tm	3.6	3.3	0.5	0.4
Yb	19.2	17.4	2.7	2.7
Lu	1.9	1.8	0.4	0.3
ΣREE	99100	80100	2750	2580
^{238}U	104.4	115	11,9	11,2
^{232}Th	104	83.3	5,2	8,8
^{40}K (%)	0,11	0.8	0.05	0,07
V_i	0.187	0.076	0.700	0.731

Table 4. The average concentrations of the REE (ppm) in the ore bodies (Nguyen Tien Du et al., 2011)

Element	F4	F7	F19	F10	F14	F16	F17
La	9001	18925	14932	13735	8374	9017	7982
Ce	11615	24371	19166	17686	10988	11821	10313
Pr	1094	2286	1812	1677	1023	1098	967
Nd	3186	6612	5237	4846	2954	3156	2808
Sm	327	671	543	492	315	337	289
Eu	169	334	267	246	158	169	141
Gd	159	322	358	238	150	16	141
Tb	18	36	29	27	17	18	16
Dy	48	97	79	72	47	51	43
Ho	7	15	12	11	7	8	7
Er	22	31	25	24	14	15	14
Tm	2	5	4	3	3	3	2
Yb	7	14	11	10	7	7	6
Lu	1	2	2	2	1	1	1
ΣLREE	25392	53199	41957	38682	23812	25598	22500
ΣHREE	264	522	520	387	246	119	230
ΣLREE/ ΣHREE	96	102	81	100	97	215	98
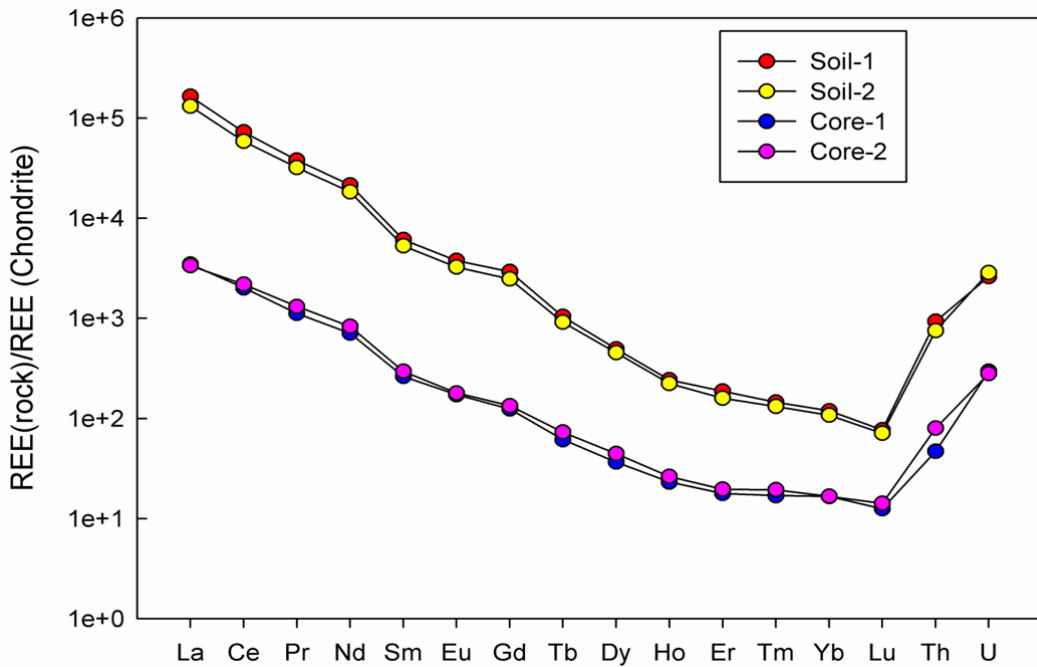
Figure 5. Patterns of the REE and radionuclides in the weathered and hard zones

The leaching and removal of intrusive formations by water activity and chemical processes led to the accumulation of igneous apa-tite, oxides, sulfides, and silicates. These phenomena were accompanied by replacement, decomposition, oxidation of the primary igne-

ous minerals and crystallization of the secondary minerals. In consequence, the overlying weathered zone is enriched in insoluble phosphates, clays, iron and manganese bearing oxides containing REE, U, Th, Nb, Ta, Zr, Ti, V, Cr, Ba and Sr (Hoatson et al., 2011). In the studied deposit, the REE minerals are parisite, bastnäsite, apatite, barite, fluorite and Celestine

were observed. The picture of BSE (Back Scattered Electron) and the spectrum of energy diffraction scattered (EDS) of the parisite mineral are shown in the Figure 6 and Figure 7 respectively. The contribution of the REE in the parisite is (in wt.%) 14.89 for La, 23.36 for Ce, 2.19 for Pr, 6.59 for Nd and near 2 for remaining heavy REE.
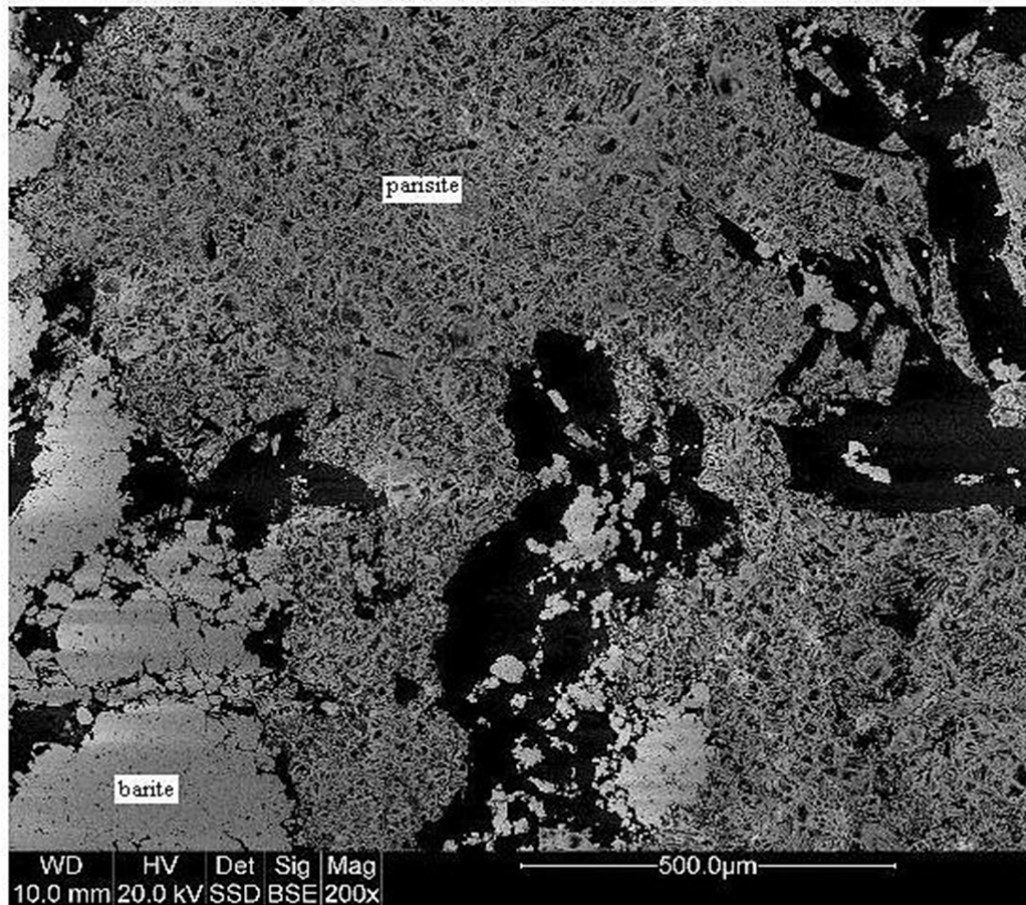

Figure 6. BSE picture

The mineralization of the stream and natural tap waters is equal to above two hundred mg/dm^3, the magnesium in tap and thermal waters are twice comparing to the stream ones (Table 5). The radium isotopes concentration in the stream water range from 100 to 300 mBq/dm^3, while in the thermal and natural tap waters amount only to several tens mBq/dm^3. The phenomenon is quite different for uranium isotopes, their concentration in the thermal water (groundwater) are equal to 110 and 78 mBq/dm^3 for 238U and 234U respectively.

This level is a few times higher than that in the stream and natural tap waters, which range near a few mBq/dm^3. Though the uranium and radium concentration in natural tap water are lower than permit maximum contaminant level for drinking water (180 mBq/dm^3 for 238U and 185 mBq/dm^3 for total radium isotopes (226Ra + 228Ra)), but significantly higher than the concentrations of these isotopes in Red River and tap waters (WHO 2006; Nguyen et al., 2016). Such high concentration of the uranium and radium in the water can be the diagnostic feature of the region, where the rock formations are rich in the natural radioactive elements.
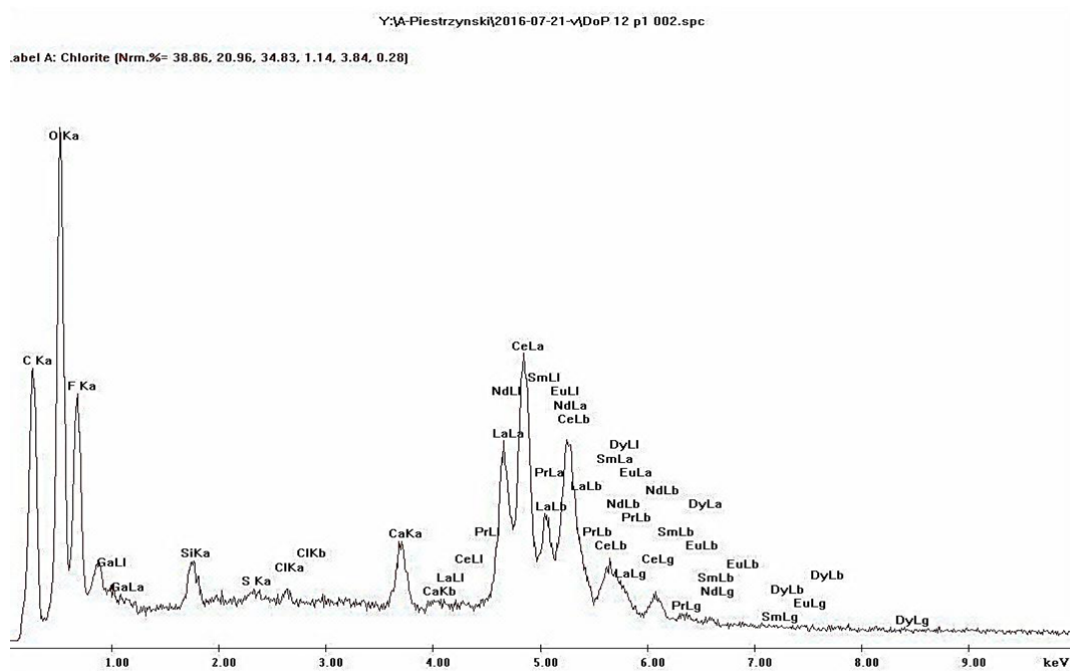

Figure 7. EDS spectrum of parisite

Table 5. Measured concentrations of main ions (mg/dm^3) and natural radioactive isotopes (mBq/dm^3) in the water samples

Water sample	F3	P1	F9	TM
Na$^+$	3.4	2.7	2.3	10.6
K$^+$	1.6	0.7	4.2	6.9
Ca^{2+}	38.0	54.8	37.0	99.8
Mg^{2+}	3.5	5.2	9.5	14.4
Sr^{2+}	0.9	0.8	2.5	8.9
Cl$^-$	5.1	4.2	3.4	4.2
SO$_4^{2-}$	3.0	2.3	6.3	184
HCO$_3^-$	137	187	159	185
TDS	206	271	256	560
pH	7.8	8.0	8.0	7.9
^{226}Ra	123	336	29	12
^{228}Ra	105	309	33	18
^{234}U	14	38	28	78
^{238}U	21	16	30	110
Hydro-chemical type of water	HCO$_3$-Ca	HCO$_3$-Ca	HCO$_3$-Ca-Mg	HCO$_3$-SO$_4$-Ca

5. Conclusions

The Dong Pao deposit is rich in light rare earth elements such as La, Ce, Pr and Nd, their total concentration approximates 10 wt.% in the weathered zone.

The mass contents of uranium and thorium in the studied REE deposit are comparable reaching about 0.01 wt.% and 0.001 wt.% in the upper weathered and lower limestone and dolomite zones respectively.

The pattern of U, Th and REE in the ore bodies occurring in the weathered zone is similar to that in the lower zone, but in the weathered zone the contents of these elements are higher by a few orders of magnitude in comparison with the lower dolomite and limestone zone.

The ΣLREE/ΣHREE ratio suggests that the mineralization processes in the Dong Pao deposit could be described as follow: the REE, uranium and thorium elements firstly came from the Mantle together with the Dong Pao alkali syenite and porphyritic syenite; next, the ores were precipitated from hydrothermal fluids and finally enriched by the surface weathering processes.

The radium and uranium concentration in the surface and natural tap waters are a consequence of the leaching processes of the elements from the weathered rocks and can be a geological indicator in the prospecting of deposits rich in the natural radionuclides.

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