

Sorption and migration of Cs, Sr, and Eu in gypsum-groundwater system

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Abstract. The distribution of ^{137}Cs , ^{152}Eu , and ^{85}Sr in a solid/aqueous system, gypsum in contact with groundwater, was investigated using γ -spectrometry. The sorption and migration of the radionuclides were investigated. The aqueous phase was characterized using instrumental neutron activation analysis (INAA) and high performance liquid chromatography (HPLC). The solid phases were characterized using X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS). The investigations included three kinds of gypsum: mineral, commercial, and own prepared gypsum. The influence of some parameters on sorption/migration of the radionuclides were studied, such as contact time, pH, and concentrations of concurrent elements. The effect of element concentration was also investigated. The results show the ability of gypsum to keep Sr and Eu in the solid phase in all three cases. The incorporation of Cs in the solid phase depends on the conditions and used materials, and varies between 93 and 97%.

Key words: distribution • cesium • europium • strontium • gypsum • groundwater

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Introduction

The distribution of elements between solid and aqueous phases has been investigated in many works. Some of them have been focused on transport mechanisms and thermodynamic aspects of element transport [24, 31], others have been concentrated on possible applications such as element separation [3], purification of contaminated waters [1], and deposition of nuclear and chemical wastes [28]. The solid phase could consist of natural materials, such as soil and clay minerals [7, 14], prepared materials such as ion exchangers [3, 23], or mixtures of natural and prepared materials [2, 26].

Soil and clay minerals from different localities, because of the variation in their composition, may behave differently; therefore, each soil or clay deposit, if it to be used in such applications requires specific studies [11, 13, 15, 20, 22, 25]. Organic and inorganic components have been used in various investigations [4, 5]. Organic materials may have the shortcoming of low resistance to ionizing radiation. For inorganic materials, high thermostability, resistance to swelling in solvents, physical strength, and radiation stability are the basic properties that, together with chemical and physical properties, determine their sphere of application [1].

Gypsum (calcium sulfate) exists in the nature in two different crystalline phases, CaSO_4 , and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ [16, 21]. It can be found in many locations around the world. Gypsum is used in many fields such as: medicine, agriculture, construction, cosmetic, decoration, and in

building materials [9, 29, 30, 33]. For its applications, especially in building materials, the plaster (di-hydrate phase: $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) is usually heated to $\approx 130^\circ\text{C}$ for several hours to obtain the hemihydrate phase $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$. On the contrary, the treatment of this phase (hemihydrate) with water leads to di-hydrate phase, which is called "solidification process" [9].



The mechanism, which is currently recognized to be responsible for plaster setting, is dissolution followed by precipitation [10, 21].

The sorption of some elements on mineral gypsum has been investigated in some works [8, 12, 17, 18, 32]. Gypsum removed less than 15% of thorium from solution according to Landa *et al.* [19]. The distribution coefficient K_d of Cs and Sr are 0.003 and 0.006, respectively [8]. More investigations about coprecipitation into gypsum have been done including: Sr^{+2} , Mg^{+2} , Na^+ and K^+ [18], Sr^{+2} [17, 18], and Cd^{+2} [32]. Factors such as partition coefficient and saturation index have been studied in these works.

The problem of nuclear waste is still unsolved in many countries. More research is required in order to find out the most stable geological position associated with sediments and composites, having large capability to keep radionuclides in normal and abnormal conditions. Gypsum and phosphogypsum could be used as a backfill or for the disposal of low-level radioactive waste into the ground. Therefore, in the present study, the sorption and migration of the radionuclides ^{137}Cs , ^{152}Eu , and ^{85}Sr in a solid/aqueous system, gypsum in contact with groundwater, was investigated. Different phases of gypsum have been studied. The effect of contact time, pH, the concentration of concurrent elements (Na, Ca and Ce), and the effect of element concentration have been investigated.

Experimental

Characterization of the phases

The water sample was taken from a borehole from the gypsum area. The cation and anion concentrations were determined using HPLC technique. Reference solutions were obtained from Merck. The elemental composition was analyzed, after evaporation to dryness at low temperatures, using instrumental neutron activation analysis. Soil 7 from International Atomic Energy Agency (IAEA) was used as reference material. Details of used analyzing procedures are published in [6]. The precision of concentration determination depended on element and concentration and has a value less than 8% (lowest value 0.3% for Na, exception 19% for Rb). The accuracy of all measurements is between 5 and 8%.

Commercial Syrian gypsum was characterized and compared with two other kinds of gypsum (mineral, and own prepared) using two techniques. The composition has been studied by X-ray photoelectron spectroscopy (XPS, SPECS UHV-system). X-ray diffraction (XRD, Stoe StadiP Transmission X-ray diffractometer) was applied to investigate crystal structures.

Distribution of Cs, Sr, and Eu between the phases

Case I

0.5 ml of groundwater, containing a certain activity of the radionuclides ^{137}Cs , ^{152}Eu , and ^{85}Sr , was mixed with 0.5 g of gypsum. This water/gypsum/mixture was kept for a time period of about one week at ambient temperature to become solid. Then, the mixture was brought in contact with groundwater (5 ml) for a certain time under defined conditions (pH value, temperature, concentration of various elements...). After phase separation, the element ratios of Eu, Sr, and Cs were determined using γ -spectrometry (HPGe-detector, 60% Eff., full width at half maximum (FWHM) = 0.998 at 122 keV and 1.88 at 1332 keV, Canberra 35 plus). The uncertainties of all γ -measurements were estimated as about 5%. ^{137}Cs was taken from an IAEA-standard. ^{152}Eu and ^{85}Sr were produced by irradiation of their nitrates with a neutron flux of $10^{11} \text{ cm}^{-2}\text{s}^{-1}$.

In this kind of experiments, the investigated radionuclides could be incorporated in the crystal of gypsum or adsorbed at the surface of gypsum and then migrated to the liquid phase, when the solid phase was brought into contact with groundwater. Three different kinds of gypsum were investigated: commercial gypsum (gypsum I), mineral gypsum (gypsum II), and own prepared gypsum (gypsum III).

Case II

0.5 ml of the used groundwater was mixed with 0.5 g of gypsum. After a solidification time of one week, the solid phase was dried and then brought into contact with groundwater (5 ml, containing a certain activity of the radionuclides ^{137}Cs , ^{152}Eu , and ^{85}Sr) for a certain time under defined conditions (pH value, temperature, concentration of various elements...). After phase separation, the element ratios of Eu, Sr, and Cs were determined using γ -spectrometry.

Case III

5 ml of groundwater containing a certain activity of the radionuclides ^{137}Cs , ^{152}Eu , and ^{85}Sr was mixed with 0.5 g of gypsum. In this case, the distribution of the radionuclides between the phases could take place during and after the solidification process, which could contain migration and sorption processes. After the phase separation, the element ratios of Eu, Sr, and Cs were determined using γ -spectrometry.

In all cases, the element ratio in the liquid phase was calculated according to the following equation:

$$(2) \quad D_w = (A_w / (A_w + A_s)) * 100 = C_w / C_o * 100$$

where D_w is the element ratio in the liquid phase; A_w is the activity of the liquid phase after phase separation; A_s is the activity of the solid phase after phase separation; C_w is the element concentration in the liquid phase after phase separation, and C_o is the initial element concentration.

The pH value was changed using LiOH and HClO_4 solutions, only in the pH-dependent experiments. Cal-

cium, cerium and sodium concentrations were changed using their nitrates only in the experiments of concurrent elements.

Results and discussion

Characterization of the phases

Element-, anion-concentrations, pH, and redox conditions of the liquid phase can influence radionuclide distribution in several ways [27]. Therefore, physical and chemical properties of the investigated water samples were investigated. Tables 1–3 show the properties of the used aqueous phase (groundwater). From the analyses, it is to notify that the pH value is near neutral, the redox conditions are normal, and the concentration of complexing anions (e.g. phosphate) is very low. The concentration of calcium and sodium is very high, which can be very important, because these elements can play the role of concurrent elements for cesium and strontium.

Table 1. Elemental composition of the used groundwater

Element	Concentration (mg/l)
Al	230 ± 14
As	nd
Ba	9.4 ± 1.5
Br	7.8 ± 0.6
Ca	43 500 ± 3 000
Ce	0.35 ± 0.04
Cr	5.6 ± 0.09
Fe	21.8 ± 2.6
Mg	24 000 ± 600
Na	283 000 ± 800
Rb	0.26 ± 0.05
Nb	2 ± 0.14
Se	0.3 ± 0.01
Sr	529 ± 2.7
Th	0.0097 ± 0.002
U	1.11 ± 0.17
Zn	43 ± 0.6

Table 2. Main characteristics of the used groundwater

Depth	170 m
pH	8.2
Temperature	18.5°C
Conductivity	1550 µs
EH ^a	-0.722 Mv
TDS ^b	761 mg/l
Density	1.006 g/ml
TC ^c	48.6 ppm

^aElectrochemical potential.

^bTotal dissolved solids.

^cTotal carbon.

Table 3. Main anion concentrations in the used groundwater

Anion	Concentration (mg/l)
F ⁻	0.55 ± 0.01
Cl ⁻	163.9 ± 3.5
Br ⁻	0.62 ± 0.01
NO ₃ ⁻	13 ± 0.08
PO ₄ ³⁻	< 0.25
SO ₄ ²⁻	250.9 ± 3.9

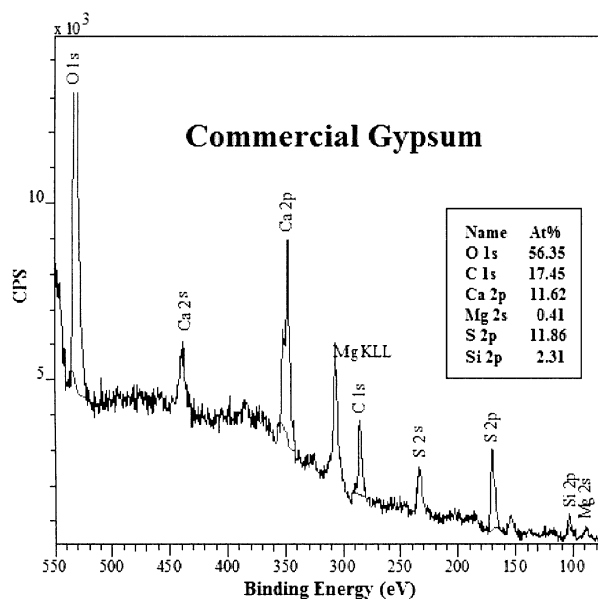


Fig. 1. XPS pattern of gypsum I.

Table 4. Atomic ratios calculated with XPS for three kinds of gypsum

	O/Ca	O/S	Ca/S
Gypsum I	2.7	2.7	1.0
Gypsum II	4.4	4.3	1.0
Gypsum III	3.7	3.5	0.9

Gypsum I is commercial gypsum. It was brought from the local market (Alsharq Company). Its preparation procedures from natural gypsum contain heating to 140°C for several hours. Gypsum II is raw gypsum without any treatment. It has been brought from the northern region of Damascus. After cleaning of gypsum II, it was purified, crushed, and grilled at 130°C for 1 h to get gypsum III.

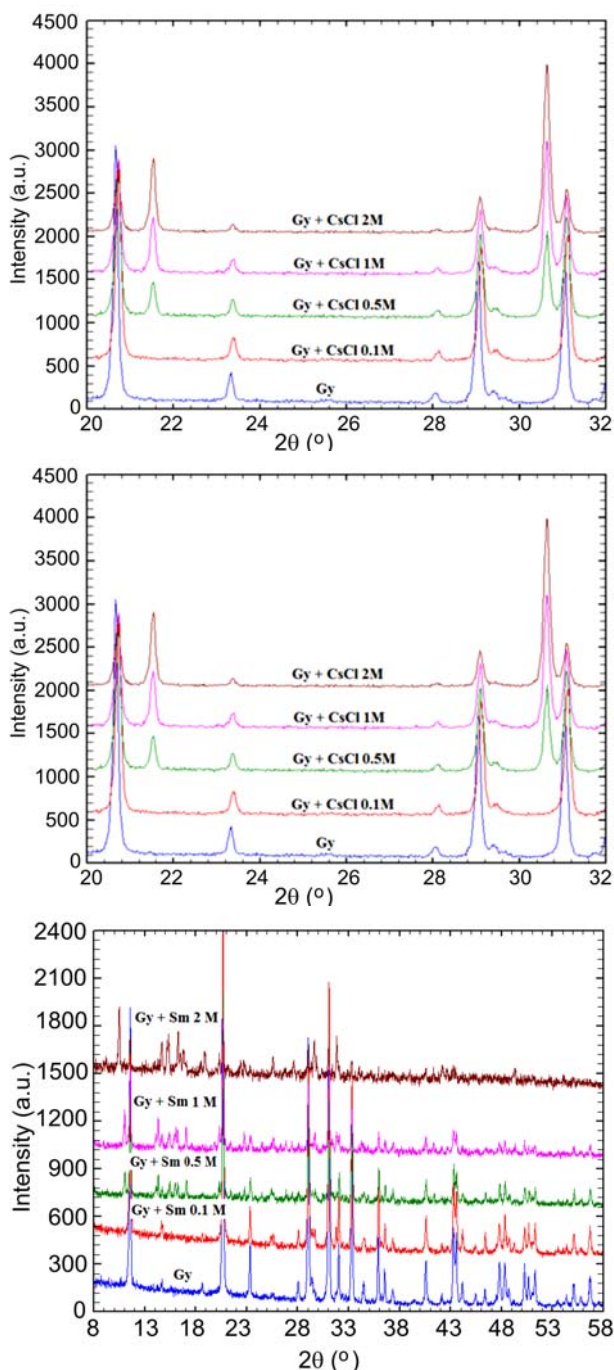
The elemental composition of gypsum was investigated using XPS. Figure 1 shows a typical survey scan for gypsum I. The peaks correspond to the three main elements in gypsum Ca ($\approx 12\%$), S ($\approx 12\%$) and O ($\approx 56\%$); in addition, C ($\approx 1.7\%$), Mg and Si ($\approx 2.5\%$) were found as contaminations.

Table 4 summarizes the O/Ca, O/S and Ca/S ratios for the three used kinds of gypsum, which are calculated from concentration values for O, Ca and S measured by XPS. The ratios Ca/S ≈ 1 corresponds with calcium sulfate formula (for the three phases). It is to note that the larger oxygen quantity exists in gypsum II, decreases in gypsum III, and become lesser in gypsum I.

The proportions of crystalline phases in the gypsum types, which are calculated from XRD patterns, are listed in Table 5. The CaSO₄·2H₂O phase was dominant in gypsum II, below 20% in gypsum III, and it disappeared finally in gypsum I (Table 5, Fig. 2). These results correlate with XPS analysis, where the maximum ratio of oxygen exists in gypsum II (augmentation of water molecules). So, it is to expect that the ability of crystalline transformation of gypsum after treatment with solution (water) will be maximal in the case of gypsum I and minimal in mineral one (gypsum II), if the solidification mechanism is as prophesies in [10, 21] dissolution followed by precipitation.

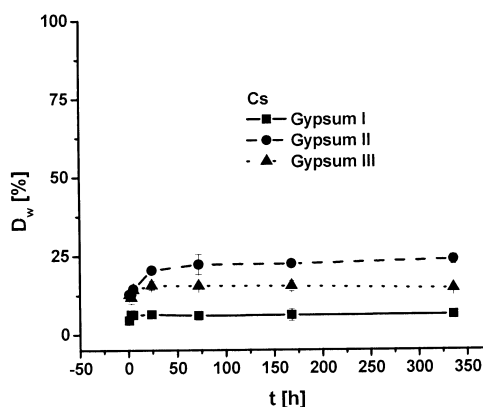
Table 5. Proportion of crystalline phases in gypsums (calculated from XRD patterns)

	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	$\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$	CaSO_4
Gypsum I	0%	67%	33%
Gypsum II	98%	2%	0%
Gypsum III	16%	84%	0%

**Fig. 2.** XRD spectra of gypsum I containing different concentrations of the investigated elements after equilibrium.

Contact time

The element ratios of Eu and Sr in the solid phase are 100% in all three gypsum kinds. Figure 3 shows the results for Cs ratio in the aqueous phase as a function of contact time.

**Fig. 3.** Element ratio of cesium in the aqueous phase as a function of contact time after phase separation using three kinds of gypsum.

For all next experiments, gypsum I was used. Figure 4 shows the element ratios in aqueous phase as a function of contact time of the phases for the investigated elements.

Case I represents the migration of the isotopes from gypsum to the liquid phase; the element ratios of Eu and Sr in the solid phase are 100%, and there is no change with respect to contact time. The element ratio of Cs in the solid phase is about 94% in the equilibrium, which can be reached after less than three hours (Fig. 4).

Case II demonstrates the sorption process of the isotopes on gypsum; the element ratio of Sr in the liquid phase decrease with increasing contact time (Fig. 4). The steady state can be reached after about 100 h. The element ratio of Sr in the solid phase is about 91%. The equilibrium of Eu and Cs in this case is reached after about 30 h (Fig. 4). The element ratios of Eu and Cs in the solid phase are 100 and 91%, respectively.

Case III shows that the element ratios of Sr in the liquid phase decrease with increasing contact time. The steady state has not been reached even after about 800 h (Fig. 4). Its ratio in the solid phase after 720 h is still higher than 21%. The equilibrium of Eu and Cs in this case is reached after about 180 h (Fig. 4). The element ratios of Eu and Cs in the solid phase are 100 and 91%, respectively.

Usually, it can be expected that migration and sorption processes are reversible processes, which is not the case here. In the first case, the change of gypsum from $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ to $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ is occurring in the presence of the investigated radionuclides. It seems that the investigated nuclides have been incorporated in the structure of gypsum during the change from $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ to $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, which leads to high element ratios in the solid phase in comparison with cases II and III.

In cases II and III, usually the time required to reach the steady state for the transport of radionuclides between the phases is determined by a rate-limiting step of processes, which are involved in the transport. Rapid

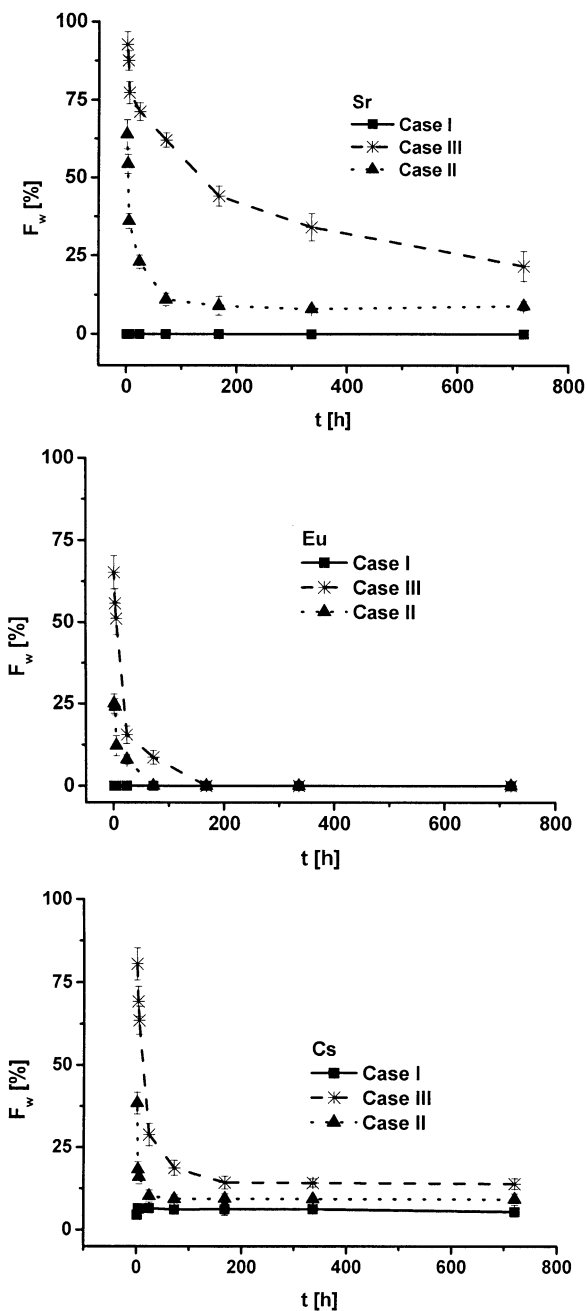


Fig. 4. Element ratios of strontium (upper panel), europium (medium panel), and cesium (lower panel) in the aqueous phase as a function of contact time after phase separation using gypsum I (case I – migration, case II – adsorption, case III – mixed experiment).

sorption processes (e.g. ion exchange) will be controlled by the diffusion of nuclides through surface to sorption sites, controlled by diffusion [15]. Slow adsorption or chemical reactions including speciation in solutions, alternation of the solid phase, etc. will be covered by the

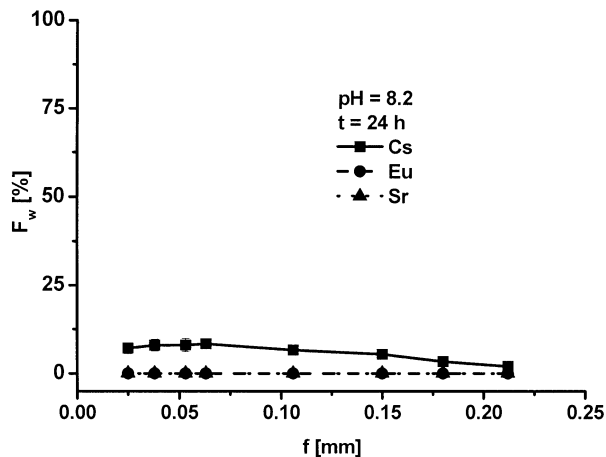


Fig. 5. Element ratios of strontium, europium, and cesium in the aqueous phase as a function of particle diameter after phase separation using commercial gypsum (case I).

law of kinetics. The behavior of Cs and Eu could be seen as rapid processes in comparison with Sr, which steady state require 100 and more than 800 h to be reached. The results for Sr and Cs as a function of time can be fitted to a second order exponential decay (Fig. 4). The values of the parameters are given in Table 6.

Effect of particle size

Figure 5 shows the element ratios of Cs, Eu and Sr in the aqueous phase as a function of particle diameter (case I). The ratios of Eu and Sr in the liquid phase do not show any variations with increasing particle diameter. The ratio of Cs in the liquid phase decreases with increasing particle diameter, which supports the hypothesis that the investigated nuclides have been incorporated in the structure of gypsum during the change from $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ to $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. The long pathway in the presence of big particles during the migration could reduce the amount of migrated Cs atoms and leads to lower element ratio in the liquid phase.

Effect of pH

Sorption and migration ratios of elements depend on the pH value of solutions in a somewhat complex manner: (change of the chemical state and charge of element species, pH dependent variation of the potential between solid and aqueous phases, competition of H_3O^+ ions and charged element species in ion exchange processes, and electrostatic attraction or repulsion between the charged hydrous and element species in solution [7, 20]). The element ratios of all three elements do not show big variations as a function of pH

Table 6. Sorption parameters of Cs and Sr in time dependence fitted to a second order exponential decay ($D_w = y_0 + A_1e^{-t/X_1} + A_2e^{-t/X_2}$)

Element and case	y_0	A_1	X_1	A_2	X_2	R^2
Sr II	20.15 ± 6.0	23.45 ± 8.45	2.98 ± 1.7	57.13 ± 5.6	213.92 ± 59.42	0.992
Sr III	8.26 ± 0.81	25.97 ± 8.9	36.79 ± 16.15	47.34 ± 10.5	2.88 ± 1.22	0.995
Cs II	13.99 ± 20.80	55.64 ± 4.79	9.77 ± 6.30	16.38 ± 23.39	54.32 ± 2.4	0.999
Cs III	9.30 ± 0.28	11.21 ± 8.02	9.55 ± 1.04	94.91 ± 24.79	1.10 ± 0.62	0.999

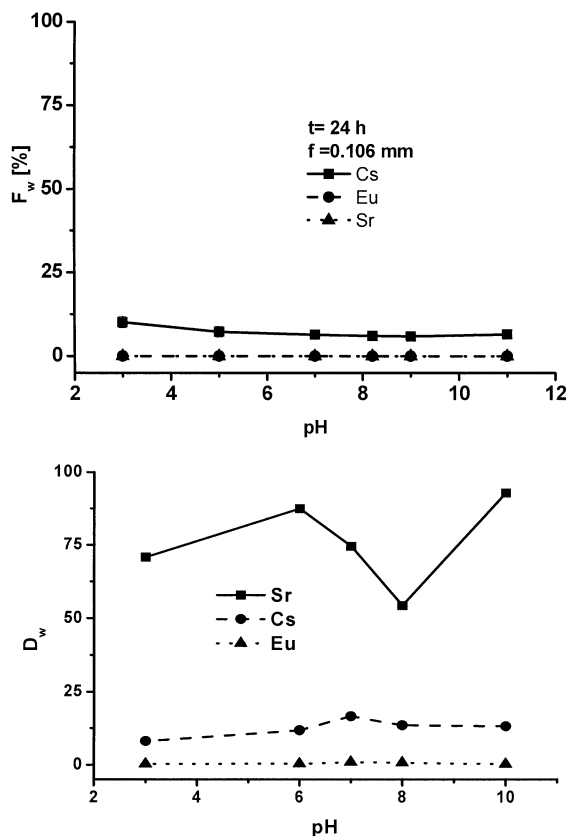


Fig. 6. Element ratios of strontium, europium, and cesium in the aqueous phase as a function of pH after phase separation using commercial gypsum (upper panel – gypsum, lower panel – mineral phosphate).

value (Fig. 6, upper panel), in spite of the behavior of the same elements on phosphate (Fig. 6, lower panel) [13], where the mentioned effects have a big influence, due to a different behavior and a different changing possibilities of the phosphate.

Effect of concurrent elements

Calcium has the highest element concentration in the groundwater samples. The concentration of sodium is also high (Table 1). For this reason, they were chosen to study the effect of concurrent elements on the distribution of the studied elements in the phases. Cerium has been used as a concurrent element for europium.

Figure 7 shows the concentration effect of sodium (upper panel), calcium (medium panel), and cerium (lower panel) on the distribution of cesium, strontium and europium. Strontium is not affected in any way by the addition of all three concurrent elements. The similarities in the chemical properties between calcium and strontium as elements of the second group could lead to a very strong bounding in the structure of gypsum in such a way, that the composition of liquid phase does not play any role more.

The increasing sodium concentration leads, at high concentrations, to the exchange between sodium and cesium in the solid phase and to lowering the cesium concentration in the solid phase. The same effect can be seen by cerium and europium, whereas the high cerium concentration influences also the cesium ratio.

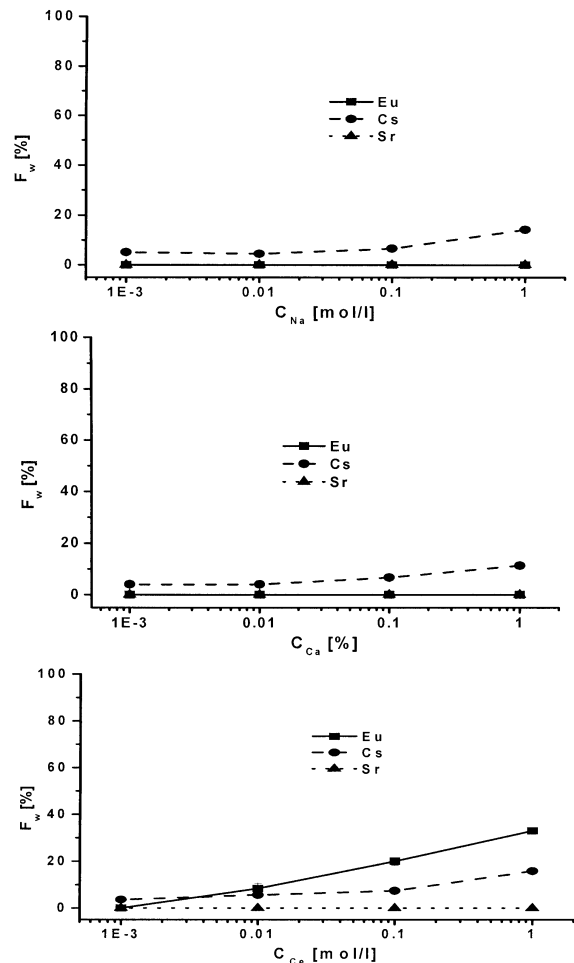


Fig. 7. Element ratios of strontium, europium, and cesium in the aqueous phase as a function of concurrent element concentration (upper panel – Na, medium panel – Ca, lower panel – Ce) after phase separation using commercial gypsum.

X-ray diffraction of the solid phase after equilibrium

To investigate the crystal structure of the solid phase after equilibrium, three experimental series with various concentrations of strontium and cesium, samarium (0.1, 0.5, 1, and 2 mol/l) were performed. Strontium nitrate, cesium chloride and samarium nitrate were used. Figure 2 shows the XRD-spectra. In the case of cesium (upper panel), the peaks in the spectra correspond to two different independent crystals: gypsum and CsCl, which lead to the hypotheses, that Cs is adsorbed on gypsum, and this leads to element ratios in the solid phase less than 100%. In the case of strontium (middle panel), the peaks in the spectra correspond to two different independent crystals: gypsum and SrSO₄, which is insoluble, and this leads to element ratios about 100% in the solid phase. Similar effect was observed by Kushner [18]. In the case of samarium (lower panel), the interpretation of the spectra is complex and it seems that there is a mixed crystal [12]. The detail interpretation needs further special crystal calculations.

Conclusion

The described work demonstrates the quantitative ability of gypsum to embrace europium and strontium is

all cases. The retention of cesium is dependent on the applied conditions, and varies between 93 and 97%. Structural studies such as XRD technique show different retention mechanisms. Strontium coprecipitates as sulfate, cesium is adsorbed as chloride, and europium seems to build mixed crystals with gypsum.

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