

## MATHEMATICAL MODELING OF Cs<sup>+</sup> TRANSPORT PHENOMENA FROM SOLIDIFIED SPENT ION EXCHANGE RESIN

by

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The aim of the study was to assess Cs<sup>+</sup> ions transport phenomena from solidified spent ion exchange resin by mathematical modeling. The experimental results comparison was obtained by Hesper's Standard Leaching Method. For the leaching prediction rate as a function of time, diffusion and semi-empirical models were used. Due to the presence of spent ion exchange resin, the cement matrix absorbed a larger amount of water, swelled, and degraded. This phenomenon caused a significantly lower value of mechanical resistance to pressure. Also, through the increase of bentonite and zeolite content, the cement matrix decreased its mechanical resistance. The retention of cesium ions in the cement matrix was low and they were leached during the early phase of the investigation. The diffusion coefficient,  $D_e$ , decreased by three orders of magnitude with the addition of zeolite and bentonite in the cement matrix. Linear regression of experimental Cs<sup>+</sup> leaching results, under static conditions, displayed that the semi-empirical parameter  $K_3$  absolute values were one to two orders of magnitude lower than the absolute values of the parameters  $K_2$  and  $K_1$ . Therefore, the contribution of matrix dissolution to the total radionuclides transport was irrelevant to the prevailing share of diffusion and surface washing processes.

*Key words: leaching, immobilization, cementation, sorption, radioactive waste*

### INTRODUCTION

Ion exchange processes represent one of the most important accompanying operations in nuclear power plant processes. The removal of <sup>137</sup>Cs, <sup>99</sup>Tc, and <sup>90</sup>Sr by ion exchange materials has been extensively utilized [1-3]. It is estimated that the annual consumption of ion exchange resins in nuclear plants amounts to tens of tons [4, 5]. After their useful life, spent resins have to be replaced. The end of useful life means that their regeneration ability is significantly reduced (for bead resins), or exchange capacity is dramatically decreased (for powdered or non-regenerable resins). Generally, spent ion exchange resins are rinsed out and collected in tanks, becoming thereby a source of a large amount of low-level or intermediate-level radioactive waste. The cementation process represents a common practice for disposal of this waste kind, where by the radioactive ions from resins are immobilized by binding to the inactive cement matrix [6].

Leaching of radioactive ions, immobilized in the cement matrix, refers to the extraction of a soluble

constituent from the solid phase by the solvent impact on the matrix. As follows, the solidification products free from the leaching of radioactive ions, are created. By Hesper Standard Leaching Test Method, accepted by the International Atomic Energy Agency (IAEA), the comparison of different author's results could be achieved [7]. In order to study the leaching of radionuclides from a cement matrix, two models are most commonly used: the diffusion and the semi-empirical model [8].

The diffusion model investigates the transport phenomenon to obtain the leaching level as a function of time. The hypothesis and borderline appliance requirements for the diffusion model request seem sensible in the experimental static conditions, if a consecutive change-renewal of the leachate solution is conceded, while ions concentration in the liquid phase is insignificant in comparison with the solid phase [8-10]. The diffusion could be considered as a dominant mechanism in the separation of the radionuclide from the matrix, presuming the experimental data are obeyed by linear dependence. Thus, the effective diffusion coefficient,  $D_e$  [cm<sup>2</sup>d<sup>-1</sup>], is calculated using the slope of the right,  $m$ , [8-10]

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$$D_e \frac{m^2}{2} \pi \quad (1)$$

from the graph of cumulative leaching fraction of Cs<sup>+</sup> ions as a function of time,  $t$

$$\frac{C_n}{C_0} \frac{V}{S} f(\sqrt{t}) \quad (2)$$

where  $C_0$  [mgdm<sup>-3</sup>] is the initial concentration of Cs<sup>+</sup> ions in the matrix,  $V$  [cm<sup>3</sup>] – the volume of the sample subjected to distilled water,  $S$  [cm<sup>2</sup>] – the total surface area subjected to distilled water, and  $C_n$  [mgdm<sup>-3</sup>] – a concentration of Cs<sup>+</sup> ions in the leachate solution according to its  $N^{\text{th}}$  variation.

The three mechanisms might be distinguished using the leaching cumulative fraction as a function of time: surface wash-off, dissolution, and diffusion [8, 11, 12]. The mathematical model regarding the entire three leaching mechanisms was rather multifaceted, although achieved results had scarce practical use [8]. To overcome this subject, the semi-empirical model was accepted, established on an orthogonal-polynomial equation that defines leaching waste components, immobilized in a solid matrix for a long time. The leaching cumulative fraction was conveyed rendering to model

$$\frac{C_n}{C_0} K_1 K_2 t^{1/2} K_3 t \quad (3)$$

where  $K_1$  is a constant which represents the surface wash-out contribution to radionuclide transport,  $K_2$  [s<sup>-1/2</sup>] represents the diffusion contribution to the radionuclides transport,  $K_3$  [s<sup>-1</sup>] represents the contribution of a kinetically controlled solution to the radionuclides transport, and  $t$  [s] – the duration of the experiment.

## EXPERIMENTAL PART

### Sample preparation

The distinctive cement-resin matrix composition, R, with proportion variations of bentonite, RB<sub>x</sub>, and zeolite, RZ<sub>x</sub>, with the initial Cs<sup>+</sup> ions concentration,  $C_0 = 965$  mgdm<sup>-3</sup> is given in tab. 1.

Materials of the cement matrix were:

- portland cement (PC-35, Lafarge),
- sand (2 mm, Moravac),
- spent mix bed ion exchange resin, Lewatit SM600, density  $\rho = 1.13$  gcm<sup>-3</sup>,
- bentonite, 200  $\mu\text{m}$  – 250  $\mu\text{m}$ , origin Koceljjevo mine, and
- zeolite, 200  $\mu\text{m}$  – 250  $\mu\text{m}$ , origin Zlatokop mine.

A similar matrix composition already showed satisfactory Cs<sup>+</sup> and Co<sup>2+</sup> ions immobilization properties in previous investigations with sludge-based simulated radioactive waste [8, 13].

The materials were processed in a planetary mixer (Pulverisette 5, Fritsch, Germany) to accom-

**Table 1. Cement matrix composition for the samples  $V = 1$  dm<sup>3</sup> production**

Composition	Cement matrix						
	R	RB <sub>2</sub>	RB <sub>5</sub>	RB <sub>10</sub>	RZ <sub>2</sub>	RZ <sub>5</sub>	RZ <sub>10</sub>
Cement [g]	1000	1000	1000	1000	1000	1000	1000
Resin [g]	330	330	330	330	330	330	330
Sand [g]	340	320	290	240	320	290	240
Bentonite [g]	0	20	50	100	0	0	0
Zeolite [g]	0	0	0	0	20	50	100
Water [mL]	305	307	309	313	306	308	311

plish viscosity that provides the cement matrix transmission to the formed molds. The mixing duration was limited to 15 minutes, usually 5 minutes after achieving the required viscosity degree. The mixer speed rate was limited to  $n_1 = 75$  rpm and batch counter to  $n_2 = 35$  rpm.

## Procedures

### Mechanical resistance test

After the drying period of cement matrices in laboratory conditions for 28 days, their mechanical resistance was determined by measuring the compressive strength. A hydraulic press, in the range of 0-140 MPa, was used. The compressive strength of each sample was determined according to the modified ASTM C109 standard [14] in triplicate (the results are presented as mean values with corresponding standard deviation).

### Leaching test

Leaching study in static conditions for Cs<sup>+</sup> ions, immobilized in the cement matrix, was implemented by the standard Hesse method [7]. The ortho-cylinder,  $H = 2r = 4.5$  cm sample was entirely dipped in 0.4 L distilled water at temperature  $T = 25 \pm 5$  °C. The leachate solution was replaced after 1<sup>st</sup>, 2<sup>nd</sup>, 3<sup>rd</sup>, 4<sup>th</sup>, 5<sup>th</sup>, 6<sup>th</sup>, 7<sup>th</sup>, 14<sup>th</sup>, 21<sup>st</sup>, 28<sup>th</sup>, 35<sup>th</sup>, 42<sup>nd</sup>, 50<sup>th</sup>, 63<sup>rd</sup>, and 93<sup>rd</sup> day and any variation represent an incremental contribution to the overall ions release. The leached Cs<sup>+</sup> ions concentrations were determined by the AAS method using Perkin Elmer Analyst 200 [8]. Leaching tests were conducted in duplicate and the results are presented as mean values with corresponding standard deviation.

## RESULTS AND DISCUSSION

Obtained cement matrix mechanical resistance values are given in tab. 2.

Solidified spent ion exchange resin shows a detrimental effect on the structure of the cement matrix due to its organic-based structure. Also, the cement

**Table 2. Cement matrix mechanical resistance values [MPa] obtained by determining compressive strength**

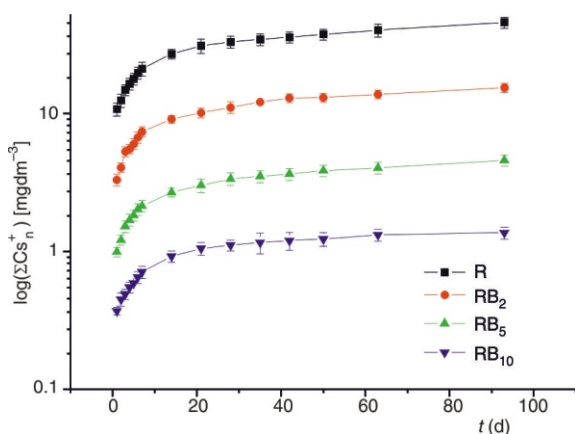
Composition	Cement matrix															
	RF*		R		RB <sub>2</sub>		RB <sub>5</sub>		RB <sub>10</sub>		RZ <sub>2</sub>		RZ <sub>5</sub>		RZ <sub>10</sub>	
Compressive strength [MPa]	25.3	0.7	23.5	0.8	22.4	0.7	20.2	0.7	17.5	0.7	22.8	0.8	20.7	0.7	23.7	0.8

\*resin-free cement matrix

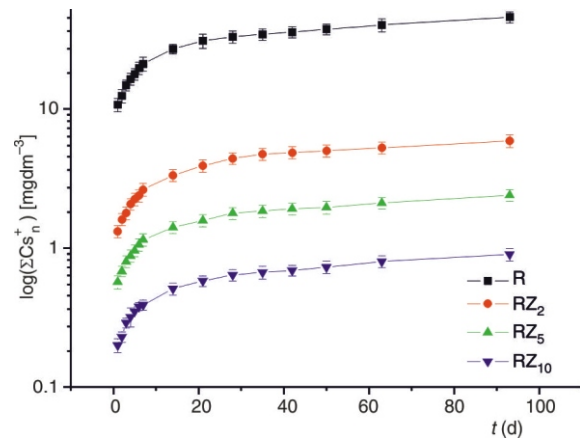
matrix absorbs a larger amount of water, due to the properties of resin and it swells and degrades its structure. This caused a lower value of mechanical resistance to pressure compared to the resin-free cement matrix which, according to previous research, amounted to 25.3 MPa [13]. Through the increase of bentonite and zeolite content, the cement matrix decreased its mechanical resistance, as expected [14-18]. Also, zeolite as a solid is durable, denser than bentonite, does not swell in water occurrence, and therefore does not damagingly distress the cement matrix structure. As a result, the cement matrix containing zeolite had a slightly higher mechanical resistance.

The leaching of Cs<sup>+</sup>, immobilized in all cement matrix types under static conditions, is shown in figs. 1 and 2.

The retention of cesium ions in the cement matrix was low [19]. Cesium is an element of a large atomic radius and weak electric field. Therefore, it does not hydrolyze in solution, thus, it is present in the free cationic form at all pH values. Besides not being precipitated, Cs<sup>+</sup> ions rarely enter into the composition of complex salts that are retained in the matrix structure [20]. Moreover, cesium ions were leached during the early phase of the investigation. The reasons were the initial, rapid leaching of Cs<sup>+</sup> ions from surfaces exposed to contact with the surrounding liquid medium and the exchange reaction with soluble alkaline elements (Na<sup>+</sup> and K<sup>+</sup>), originated from the cement-based matrix, and consequently present in the leachate solution. The result of contact of water or aqueous solution with the cement matrix, solvation effects, and relatively high pH values of the leachate solution (pH > 10) due to the high alkali-



**Figure 1. The leaching of Cs<sup>+</sup> immobilized in the cement-resin matrix, R, and cement-resin-bentonite matrices, RB<sub>x</sub>, under static conditions**



**Figure 2. The leaching of Cs<sup>+</sup> immobilized in the cement-resin matrix, R, and cement-resin-zeolite matrices, RZ<sub>x</sub>, under static conditions**

linity of the cement-based matrix, also enabled the formation of solution-cesium hydroxide or CsNaSO<sub>4</sub> [21].

The effective diffusion coefficient values,  $D_e$ , and the regression coefficients,  $R$ , are given in tab. 3, calculated by the leaching diffusion model. Also, the constants  $K_1$ ,  $K_2$ , and  $K_3$  and the regression coefficients,  $R$ , are presented in tab. 4 (designed by a semi-empirical leaching model of Cs<sup>+</sup> ions immobilized in the cement matrix under static conditions).

The effect of 2 %, 5 %, and 10 % bentonite and zeolite content, as cement matrix constituents, on Cs<sup>+</sup> ions leaching under static conditions, was examined. From studying the experimental results, it could be established that an increase in the amount of added zeolite and bentonite in the cement matrix considerably decreases the leaching rate of Cs<sup>+</sup> ions (diffusion coefficient,  $D_e$ , decreases by three orders of magnitude). Moreover, zeolite has a higher sorption ability of Cs<sup>+</sup> ions than bentonite. Furthermore, bentonite and zeolite selectivity towards Cs<sup>+</sup> ions and their influence on the leaching reduction degree is long-established [3, 22]. The total sorption process in porous materials is controlled by the sorbate mass diffusion transport inside the channel and pore network, instead of the sorption kinetics itself. The sorbate ions diffusion is affected by sorbent geometric structure, ionic radius, valence, and hydration energy [23]. The sorption mechanism includes ion exchange, surface, external, and internal complexation/precipitation. The nature of the sorbent-sorbate interaction is inclined by the ion's hydration and their hydrolysis, hence, the mechanism of Cs<sup>+</sup> ion sorption on bentonite and zeolite was ion exchange.

Linear regression of experimental leaching results under static conditions Cs<sup>+</sup> displayed that the pa-

**Table 3. Effective diffusion coefficients values,  $D_e$  [ $\text{cm}^2\text{d}^{-1}$ ], and regression coefficients,  $R$ , calculated by leaching diffusion model of Cs<sup>+</sup> immobilized in the cement matrix**

Cement matrix							
Diffusion leaching model parameters	R	RB <sub>2</sub>	RB <sub>5</sub>	RB <sub>10</sub>	RZ <sub>2</sub>	RZ <sub>5</sub>	RZ <sub>10</sub>
$D_e$ [ $\text{cm}^2\text{d}^{-1}$ ]	$8.04 \cdot 10^{-6}$	$9.67 \cdot 10^{-7}$	$8.24 \cdot 10^{-8}$	$7.58 \cdot 10^{-9}$	$1.49 \cdot 10^{-8}$	$2.16 \cdot 10^{-8}$	$3.10 \cdot 10^{-9}$
$R$	0.985	0.983	0.985	0.968	0.984	0.985	0.990

**Table 4. Constants  $K_1$ ,  $K_2$  [ $\text{s}^{-1/2}$ ],  $K_3$  [ $\text{s}^{-1}$ ] vs. and regression coefficients,  $R$ , calculated by semi-empirical leaching model of ions immobilized in the cement matrix**

Cement matrix							
Parameters of the semi-empirical leaching model	R	RB <sub>2</sub>	RB <sub>5</sub>	RB <sub>10</sub>	RZ <sub>2</sub>	RZ <sub>5</sub>	RZ <sub>10</sub>
$K_1$	$4.46 \cdot 10^{-3}$	$1.12 \cdot 10^{-3}$	$3.97 \cdot 10^{-4}$	$1.10 \cdot 10^{-4}$	$3.13 \cdot 10^{-4}$	$2.50 \cdot 10^{-4}$	$9.48 \cdot 10^{-5}$
$K_2$ [ $\text{s}^{-1/2}$ ]	$7.03 \cdot 10^{-3}$	$2.59 \cdot 10^{-3}$	$7.33 \cdot 10^{-4}$	$2.69 \cdot 10^{-4}$	$1.01 \cdot 10^{-3}$	$3.71 \cdot 10^{-4}$	$1.28 \cdot 10^{-4}$
$K_3$ [ $\text{s}^{-1}$ ]	$-2.79 \cdot 10^{-4}$	$-1.11 \cdot 10^{-4}$	$-3.04 \cdot 10^{-5}$	$-1.39 \cdot 10^{-5}$	$-4.36 \cdot 10^{-5}$	$-1.51 \cdot 10^{-5}$	$-4.43 \cdot 10^{-6}$
$R$	0.997	0.998	0.998	0.998	0.998	0.997	0.997

rameters  $K_3$  absolute values were one to two orders of magnitude lower than the absolute values of the parameters  $K_2$  and  $K_1$ , tab. 4. This indicated that the effect of the variable  $t$  is insignificant, and the influence of the variable  $\sqrt{t}$  is predominant. Hence, the matrix dissolution contribution to the total radionuclide transport was irrelevant concerning the dominant contribution of diffusion and surface washing. Likewise, surface washing might be identified as an instantaneous phenomenon ( $t = 0$ ). Although the diffusion model cannot define the overall leaching process, it is suitable for anticipation due to its simplicity [24]. However, concerning the regression coefficients, a semi-empirical model provided the improved approximation of the Cs<sup>+</sup> static leaching process [25].

## CONCLUSION

The investigated cement-based matrix absorbed a substantial amount of water, due to spent ion exchange resin presence, which further influenced the swelling and its degradation. This caused a significantly lower value of mechanical resistance to pressure. Furthermore, the reduction of the mechanical resistance was caused by the presence of the resin, since it is composed of plastic microspheres, which do not create bonds, resulting in the reduction of mechanical strength.

The retaining of Cs<sup>+</sup> in the cement matrix was low and they leached during the early phase of the analysis. By increase of the content of bentonite and zeolite in the matrix, mechanical resistance to pressure was decreased, while cement matrix containing zeolite had slightly higher mechanical resistance. Furthermore, an increase in the amount of zeolite and bentonite in the matrix considerably reduced the Cs<sup>+</sup> leaching degree while zeolite had a higher sorption ability.

The contribution of the diffusion process to the total transport of Cs<sup>+</sup> in the cement matrix porous me-

dium under static leaching conditions, was dominant. The contribution of matrix dissolution was negligible in relation to the considerable contribution of diffusion and surface washing that could be considered an instantaneous phenomenon. Hence, the semi-empirical model showed a more acceptable approximation of the Cs<sup>+</sup> leaching process.

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## AUTHORS' CONTRIBUTIONS

The experiments were carried out by S. D. Dimović. All authors analyzed the results and participated in the preparation of the final version of the manuscript.

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## МАТЕМАТИЧКО МОДЕЛОВАЊЕ ФЕНОМЕНА ТРАНСПОРТА Cs<sup>+</sup> ИЗ СОЛИДИФИКОВАНЕ ИСТРОШЕНЕ ЈОНОИЗМЕЊИВАЧКЕ СМОЛЕ

Циљ рада је да се математичким моделовањем процене феномени транспорта јона Cs<sup>+</sup> из солидификоване истрошене јоноизмењивачке смоле. Хеспевом стандардном методом исљуживања остварено је поређење експерименталних резултата. За предвиђање брзине исљуживања, у функцији времена, коришћени су дифузиони и семи-емиријски модели. Услед присуства истрошене јоноизмењивачке смоле, цементни матрикс је апсорбовао већу количину воде, набубрио и разградио се. Овај феномен је проузроковао знатно нижу вредност механичке отпорности на притисак. Повећањем садржаја бентонита и зеолита у цементном матриксу такође је смањена његова механичка отпорност. Задржавање јона цезијума у цементном матриксу је слабо, те су ови јони излужени већ током ране фазе испитивања. Коефицијент дифузије,  $D_e$ , смањен је за три реда величине додатком зеолита и бентонита у цементни матрикс. Линеарна регресија експерименталних резултата излуживања Cs<sup>+</sup> у статичким условима показала је да су апсолутне вредности параметра  $K_3$  биле један до два реда величине ниже од апсолутних вредности параметара  $K_2$  и  $K_1$ . Услед тога, допринос растварања матрикса у укупном транспорту радионуклида може се сматрати ирелевантним у односу на доминантни допринос дифузије и површинског испирања.

Кључне речи: исљуживање, имобилизација, цементација, сорпција, радиоактивни отпад