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ORIGINAL ARTICLE

The combined effect of radiation and carbonation on the immobilization of Sr and Cs ions in cementitious pastes

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Abstract In the present study the effect of gamma irradiation on the transport of immobilized Sr and Cs ions in cementitious paste samples is described. During the irradiation process, the samples were exposed to atmospheric conditions. Hence, atmospheric carbonation of samples could takes place. Cementitious pastes, differing in their w/c ratio (0.3, 0.45), were exposed to an integral dose of 10^7 grays (Gy) of γ irradiation during a period of approximately 6 months. The effect of irradiation on the transport properties of the immobilized Sr and Cs ions was studied by leaching experiments. The carbonation depth of the different samples was measured and found to markedly increase in the case of irradiated samples compared to samples not exposed to irradiation. The enhanced carbonation is associated with the dehydration due to radiolysis of pore water in the paste. It was found that the irradiation resulted in a marked decrease in the leached fraction of Sr ions. This is related to the increased carbonation found for the irradiated samples. On the other hand, a small increase in Cs diffusivity was found for irradiated samples. This increase in leachability might be associated with the formation of microcracks in the irradiated sample.

Keywords Sr · Cs · Radiation · Carbonation · Immobilization

1 Introduction

During its lifetime, a solidified LLW (low level nuclear waste) form will be exposed to diverse degradation processes. The prediction of the long-term behavior of the immobilizing matrix requires the identification and the analysis of the various degradation mechanisms that will affect the structure.

One of these mechanisms is irradiation, originating from the immobilized radioactive waste elements. Irradiation might lead to matrix degradation which as a result will impair the immobilization of the waste ions in the cementitious paste [1, 2].

Cementitious materials and concrete are generally considered to be resistible to irradiation doses up to 10^8 Gy. Larger irradiation doses initiate processes that impair the physical properties of the cementitious matrix [2].

Irradiation can degrade cementitious materials through two main mechanisms. The first involves the relocation of atoms from their sites mainly due to

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the recoil energy of the radionuclides. This may result in micro-structural changes in the immobilizing material. When immobilization of radioactive waste is concerned, the irradiation sources that are dealt with include mainly beta and gamma emmiters.

The second and probably more important mechanism involves radiolysis of water, both interstitial as well as water in the pore structure of the sample. The two main outcomes resulting from water radiolysis are: (1) build up of internal overpressure that may lead to cracking of the wasteform and (2) the accumulation of hydrogen gas in a storage area, hence, build up of explosive gas mixture when mixed with atmospheric oxygen [1]. Irradiation would not change usually the macroscopic properties of the material; however, the interactions between the various radical species created during the radiolysis process might induce microscopic physical and chemical changes in the matrix [2]. The process of water radiolysis can be monitored by detection of gas products released.

There is very little published information on the effect of irradiation on the immobilization of waste ions. No conclusive results were obtained from leaching experiments performed using a series of cementitious paste samples with immobilized Cs, Sr and Co ions irradiated to total doses of up to 9 MGy [2].

Bouniol et al. [1, 3, 4] reported that oxygen disappears from the gaseous phase above the irradiated samples during their exposure to gamma radiation. To explain this result they suggested that the main reaction of the calcium ions in the cement is with H_2O_2 (assumed to be the main radiolysis product). This process was assumed to be responsible for the precipitation of calcium peroxide (CaO_2) thus fixing oxygen and reducing its release to the gaseous phase. The XRD of the irradiated samples shows a decrease in the peaks corresponding to portlandite and ettringite together with an increase of the calcite peaks. The formation of calcite crystals, which decrease both the size of pore space and also the strength of material was reported in another study devoted to understanding the effect of irradiation on concrete used as radiation shield [5].

Carbonation is probably the most common form of concrete environmental attack. Carbonation promotes changes to the cement chemical composition and physical properties that can affect the immobilization of waste ions. This process involves the chemical reaction of portlandite $Ca(OH)_2$ and calcium silicate hydrates (C–S–H) in the cement matrix with carbon dioxide gas leading to calcite ($CaCO_3$). Although the cement hydrate phases undergo carbonation, the carbonation rates of these phases are much smaller compared to the carbonation rate for portlandite. One of the main consequences of the carbonation reaction is the modification of the pH in the pore solution from a standard value between 12.5 and 13.5, to a value below nine in the carbonated zones [6].

Measurements show that the total porosity of hardened cement pastes decreases with carbonation. Thus, carbonation influences leaching of heavy metals by changing the solubility of components and the porosity of the matrix [6].

Contradictory leaching results were reported for carbonated samples [6]. These contradictions originate from the fact that in some cases carbonation might improve waste ion retention either by coprecipitation as insoluble carbonates, or due to the reduced porosity obtained in the matrix, while on other cases ion retention was reduced. This reduced retention may reflect microcracking due to shrinkage during carbonation or rearrangement in pore connectivity [6, 7].

It has been shown that the leachability of Sr ions is reduced for the carbonated samples due to the coprecipitation of $SrCO_3$ with $CaCO_3$ which was found to be an effective solubility-limiting process for Sr release from the matrix [8]. The effect of carbonation was studied for other waste ions including Cs. For ions that are not coprecipitated as carbonates it was found that carbonation increases the fraction of the leached ion, probably due to formation of micro-cracks [7, 9].

As reported in several investigations, the quantity of water present in the pores is of great importance when the carbonation process is studied [10]. Although the carbonation reactions need the presence of water, in high relative humidity conditions, the diffusion of both carbon dioxide and oxygen is reduced and even inhibited by the water filling the pores. For lower degrees of humidity the rate of carbonation increases with decreasing humidity, until reaching a peak at a certain point. On the other hand, for very low humidity values the carbonation rate starts to decrease due to lack of water for the process. Therefore, the most suitable range of internal relative humidity for the carbonation of concrete seems to be 40–80% [7, 10, 11]. Increasing the w/c ratio of the cementitious matrix induces an increased degree of carbonation due to higher diffusivity of carbon dioxide through the more porous matrix.

In the present study the effect of gamma irradiation on the transport of immobilized Sr and Cs ions in cementitious paste samples is presented. During the irradiation process, carried out during a few months, the samples were exposed to atmospheric conditions. Hence, while irradiated, atmospheric carbonation of samples could occur. In the followings we shall try to couple between these two phenomena and investigate the combined effects on the immobilization of Sr and Cs ions.

2 Experimental

2.1 Sample preparation

The cement used was ordinary Portland cement (OPC) CEM I 52.5N conforming EN 197 (manufactured at "Nesher", Israel). The chemical composition of the OPC is presented in Table 1.

Cementitious pastes of different w/c ratios (0.3, 0.45, 0.6) were prepared. Paste with w/c ratio of 0.6 is very porous, thus it was used solely for the evaluation of gas release. Non radioactive Sr and Cs ions (representing the waste ions to be immobilized) were added as a solution of the nitrate salts, $Sr(NO_3)_2$ and $CsNO_3$ (Aldrich chemicals), during the paste preparation. The concentrations of the immobilized ions in the paste were 2.9 mg Cs/gr paste and 3.5 mg Sr/gr paste. All components were mixed in a 5 l Hobart mixer. Specimens were cast in plastic molds (diameter of 40 mm and height of 100 mm), sealed and rotated for the first 24 h to prevent bleeding and segregation. After setting, the samples were removed from the molds, inserted into polypropylene bags, sealed again (to prevent exposure to the atmosphere) and allowed to self-cure at room temperature for 28 days. After curing, the specimens designated to leaching experiments were sliced into 15 mm thick disc shaped samples. Additional samples, identical to the samples used for leaching were prepared for the carbonation tests. For the gas evolution experiments the cured samples were cut into cubes $1 \times 1 \times 1$ cm^3, and sealed in small glass ampoules under Ar atmosphere at sub-atmospheric pressure.

Table 1 Chemical composition of the Portland cement (wt%)

	wt%
CaO	63.23
SiO_2	19.15
Al_2O_3	5.47
Fe_2O_3	2.89
MgO	1.32
TiO_2	0.40
K_2O	0.46
Na_2O	0.20
P_2O_5	0.51
Mn_2O_3	0.04
SO_3	2.97
Cl	0.01
LOI total	3.17

2.2 Sample irradiation

2.2.1 *Irradiation of samples for leaching experiments*

Discs of 40 mm in diameter and 15 mm in height, were prepared for the leaching experiments, differ in their w/c ratio (0.3, 0.45). The disks were subjected to gamma irradiation to an integral dose of 10^7 Gy (at Sor-Van Radiation Ltd.) during a period of approximately 6 months. The irradiation was performed under atmospheric conditions at room temperature.

Following irradiation, the samples were subjected to leaching experiments according to ANSI procedure [12] described in the followings.

2.2.2 *Irradiation for gas release experiments*

The samples used for the gas release measurement were cementitious cubes of size $10 \times 10 \times 10$ mm^3 with w/c ratio of 0.3 and 0.6. The cubes were sealed in glass ampoules. These ampoules were placed in a ^{60}Co irradiation source (dose rate of 24 Gy/min). The number of irradiated samples was limited by the size of the irradiation source cavity. Therefore, most of the ampoules contained samples prepared from the paste with w/c = 0.6, which was expected to give a higher volume and better resolution of the gaseous products. Duplicate Ampoules were removed from the irradiation source at six different times, representing different irradiation doses (0, 10^6, 3×10^6, 6×10^6, 8×10^6, 10^7 Gy) for the w/c = 0.6 samples and at three different irradiation doses (0, 5×10^6, 10^7 Gy) for the w/c = 0.3 samples. The gas phase composition in the ampoules was analyzed by Mass Spectrometry.

2.3 Leaching tests

The leaching tests were performed according to ANSI/ANS-16.1 (1986) procedure [12], using deionized water as the leachant. According to this procedure [12], samples are immersed in the leachant for 90 days, with replacement of leachant at predetermined intervals of 2, 7, 24, 48, 72, 96, 120, 456, 1,128, and 2,136 h. Analysis of the concentration of leached Cs and Sr ions was preformed using Inductive Coupled Plasma-Mass Spectroscopy (ICP-MS) for samples from each leaching interval. Leaching tests were performed only with samples of w/c ratio of 0.3 and 0.45.

Since the ions were added during the paste preparation and the pastes were cured prior to the leaching tests, it was assumed that the ions were evenly dispersed. Moreover, the ions were allowed to interact with the paste, to become chemically bound or incorporated in the matrix. Duplicate samples of each of the pastes were used in the leaching tests.

The apparent diffusion coefficient was calculated using Eq. 1 derived from solution of the diffusion equation for semi-infinite media. It can be shown that this equation can be used for a finite sample that has leached less than 20 wt% of the contaminant [11]:

$$D_a = \pi \left[\frac{(a_n/A_0)}{(\Delta t)_n} \right]^2 \left[\frac{V}{S} \right]^2 T \tag{1}$$

where D_a, the apparent diffusion coefficient (cm^2/s); a_n, the concentration of the ion released from the specimen during leaching interval n; A_0, total concentration of the ion in the specimen at the beginning of the test; $(\Delta t)_n$, duration of the nth leaching interval (s); V, volume of the specimen (cm^3); S, geometric surface area of the specimen (cm^2); T, cumulative leaching time representing the mean time of the nth leaching interval (s) calculated from Eq. 2:

$$T = \left[\frac{t_n^{1/2} + t_{n-1}^{1/2}}{2} \right]^2 \tag{2}$$

For cases when the leached fraction exceeds a value of 20%, the apparent diffusion coefficient can be calculated from a specific solution of the mass transfer equations for the sample geometry. The standard [12] provides correction tables for finite cylinders of diameter d and height h. The apparent diffusion coefficient is calculated using a dimensionless time factor (G) from Eq. 3:

$$G = \frac{D_a t}{d^2} \tag{3}$$

where G, dimensionless time factor (given by the standard for each h/d value); D_a, apparent diffusion coefficient (cm^2/s); t, leaching interval (s); d, sample diameter (cm).

2.4 Carbonation tests

To check the degree of carbonation, samples were split using a chisel and hammer and the pH of the inner surface of the paste was determined using a phenolphthalein solution. As noted above, carbonation leads to a reduction with the pH of the paste, thus, carbonated area in the fractured face is indicated by low pH. Phenolphthalein is a pH-sensitive dye that appears reddish-purple at the noncarbonated regions and clear at carbonated matrix. Carbonation depth was estimated by averaging the measured depth from at least four different points in each sample.

3 Results

3.1 Leaching results

The results of the leaching experiments are presented in Figs. 1–4 as the accumulated leached fraction of the different ions. The leaching experiment was performed using both irradiated and non-irradiated duplicate samples differing in their w/c ratio (w/c = 0.3, 0.45). Both irradiated and non irradiated samples were exposed to atmospheric conditions during the whole irradiation period (6 months).

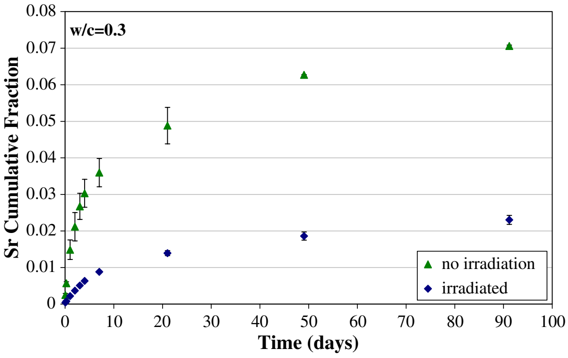

Fig. 1 Leaching of Sr ions from irradiated and non irradiated cementitious paste samples, w/c = 0.3. The total irradiation dose was 10^7 Gy

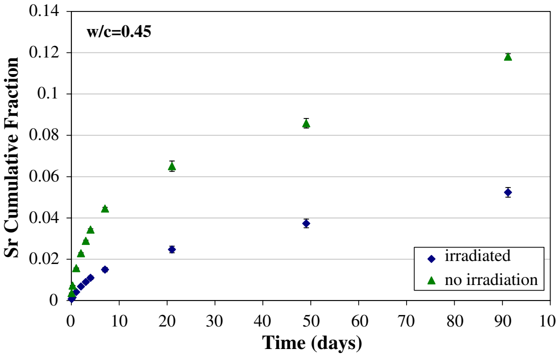

Fig. 2 Leaching of Sr ions from irradiated and non irradiated cementitious paste samples , w/c = 0.45. The total irradiation dose was 10^7 Gy

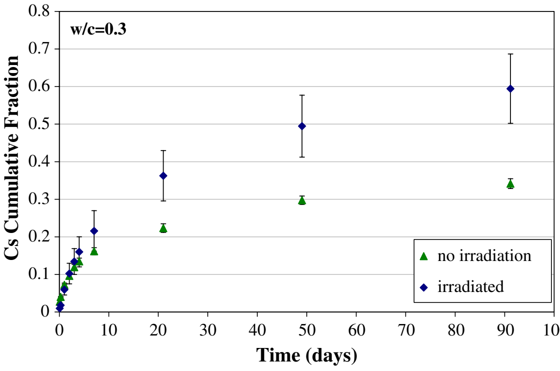

Fig. 3 Leaching of Cs ions from irradiated and non irradiated cementitious paste samples , w/c = 0.3. The total irradiation dose was 10^7 Gy

The accumulated leached fraction of the Sr ions was noticeably reduced as a result of the irradiation process. For both the w/c = 0.3 and 0.45 samples it

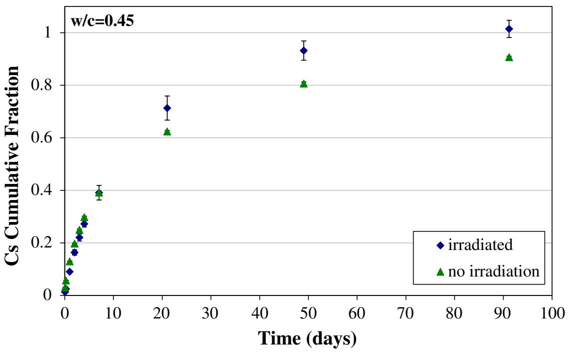

Fig. 4 Leaching of Cs ions from irradiated and non irradiated cementitious paste samples, w/c = 0.45. The total irradiation dose was 10^7 Gy

can be seen that the difference in the leaching curves of the irradiated and non irradiated samples is mainly related to the early experimental times (up to 7 days), suggesting that this effect is related to chemical or microstructural changes near the surface area of the sample induced by the irradiation process.

A markedly different picture is observed for the Cs ions. In this case, the irradiation seems to have a much smaller affect, and for both samples (w/c = 0.3, 0.45) no marked difference is noticed at the initial times of the experiment. For longer leaching times irradiation results in some increase in the accumulated leached fraction of the ion compared to the accumulated leached fraction of the non-irradiated sample. For the sample prepared at w/c = 0.45 the leached fraction was approximately 1, meaning that all the amount of immobilized Cs ion has leached out during the experiment.

Increasing the w/c ratio is known to increase the porosity of the sample [13, 14] leading to higher ion mobility and higher cumulative leached fractions of both ions as seen for w/c = 0.45 samples compared to the w/c = 0.3 samples (cumulative fraction of 0.9 versus 0.3, respectively, before irradiation, and 1.0 versus 0.6 after for Cs ions and cumulative fraction of 0.12 versus 0.07, respectively, before irradiation, and 0.05 versus 0.02 after for the Sr ions).

3.2 Carbonation tests

Carbonation tests were performed to: (a) samples that were irradiated at atmospheric conditions to a total dose of 10^7 Gy during a 6 month period; (b) reference non irradiated samples that left in free atmospheric conditions during the same period; and (c) sealed samples that were not subjected to either free atmospheric conditions or irradiation during the whole period. All samples were prepared with a constant w/c ratio of 0.45.

No carbonation was detected for the samples in group c. The reference samples, exposed to free atmospheric conditions but not irradiated, showed average carbonation depth of approximately 0.5–0.6 mm. For the irradiated samples a much larger carbonation depth was detected (average of around 3 mm), reaching, in some cases, depths of up to 6 mm.

3.3 Gas release results

The average amount of H_2 gas released from cementitious paste cubes (w/c = 0.3, 0.6) irradiated to different integral irradiation doses is presented in Fig. 5. The relative volume of the hydrogen gas released, increases as a function of both the irradiation dose and the increased porosity in the sample (higher w/c). At the low irradiation doses the results were obtained with a good repeatability (small deviation between the two samples examined), however, for the highest irradiation doses a larger deviation is obtained.

Figure 6 presents the ratio between volume % of the hydrogen (H_2) and the oxygen (O_2) gases released during irradiation. The ratio of H_2/O_2 obtained in the experiment does not correspond exactly to the atomic molar ratio of the elements in water (2:1) but is somewhat lower (the dotted line represents the ratio $H_2/O_2 = 2$). This reduced amount of oxygen generated was already reported in a number of publications [1, 3].

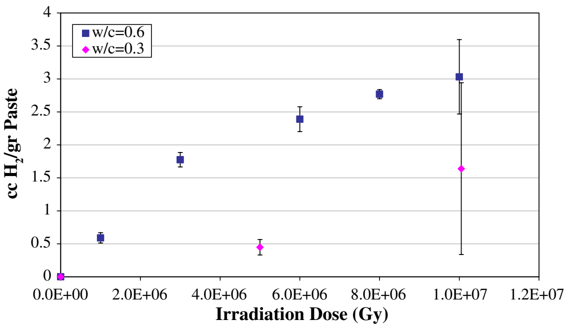

Fig. 5 Amount of hydrogen gas released (ccH_2/gr paste) during irradiation of cementitious paste samples (w/c = 0.3, 0.6) as a function of irradiation dose

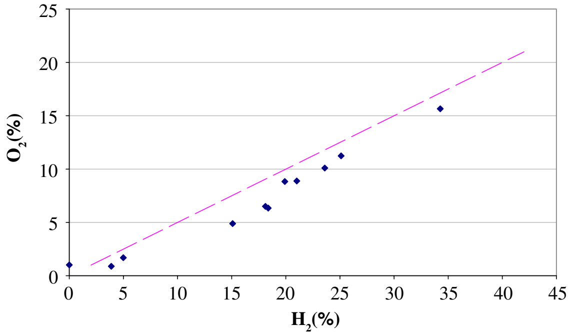

Fig. 6 The released oxygen percentage (%O_2) versus released hydrogen percentage (%H_2) during irradiation of cementitious paste samples (w/c = 0.6) to different doses. The dashed line represents the theoretical O_2/H_2 ratio of 1:2 corresponding to water radiolysis

4 Discussion

Irradiation of cementitious paste samples affected the immobilization of Sr and Cs ions differently. For Sr ions the irradiation resulted in a marked decrease in the leached fraction. This can be related to the increased carbonation found for the irradiated samples. The Sr ions are probably coprecipitated in the matrix as the highly insoluble $SrCO_3$ [8]. This coprecipitation process improves the retention of this ion in the cementitious matrix. The large difference found between the leaching rates of the irradiated and non irradiated samples during the initial experiment time can be related to the carbonation process that takes place in the outer shell of the sample and hence affects the amount of mobile Sr ions near the sample surface. It can be expected that if the irradiation process would proceed for a longer time, it will lead to higher carbonation depth; hence the difference in the leaching rate between the irradiated and non irradiated samples would remain large for longer experimental time.

The apparent diffusion coefficients for the various pastes were calculated according to Eq. 1 for all points with a leached fraction below 0.2. For all the experimental points exceeding a total leached fraction of 0.2 the apparent diffusion was calculated using the tabular method and Eq. 3. Since the apparent diffusion coefficient can be calculated independently from each experimental point and since the experimental results of the leaching experiments imply that there might be a difference in the values of the apparent diffusion coefficients obtained from the initial experimental times (first 7 points) and the final experimental times (last 3 points), a separate average apparent diffusion coefficient was calculated for the initial and the final experimental times. Table 2 presents the average calculated apparent diffusion coefficients (D_a) for Sr and Cs ions from irradiated and non irradiated samples with different w/c ratios (0.3, 0.45) for the initial and final experimental times.

For the Sr ions, irradiation reduces the diffusion coefficient by a factor of 28 for the initial experimental times and a factor of 10 for the final experimental times for the w/c = 0.3 paste. For the w/c = 0.45 pastes the apparent diffusion coefficient has been reduced due to irradiation by a factor of 9 (initial) and 4 (final). This reduction in the diffusion coefficient is even more significant than the reduction due to lowering the w/c ratio from 0.45 to 0.3. The effect of irradiation is more significant for the initial experimental times probably due to the enhanced carbonation of the irradiated samples.

For Cs ions a minor increase in the values of the diffusion coefficients for the w/c = 0.45 irradiated sample compared with the non irradiated samples was noticed. In contrary to our findings with the Sr ions, the effect of irradiation on Cs ions is much smaller than the effect of the w/c ratio. Since Cs ions are highly soluble and do not precipitate as a carbonate phase, carbonation has no "chemical" effect on the ion diffusivity. The small increase in Cs diffusivity due to irradiation, might originate from the formation of microcracks in the product due to the radiolytic process that, in part, led to the increased carbonation.

Thus, Sr ions are immobilized in the paste by a "chemical" mechanism involving their coprecipitation in the paste as $SrCO_3$ and as a result their reduced mobility. The coprecipitated Sr ions are less sensitive to microstructural changes in the matrix, and formation of microcracks does not affect their leachability. The formation of microcracks at the higher irradiation doses is a random process, leading to a reduced repeatability of the experimental results. This is clearly exhibited in the leaching experiment with Cs ions (the extended experimental times in Figs. 3 and 4) and more clearly in the gas release results (Fig. 5) where smaller samples were used.

The increased carbonation measured for the irradiated samples compared with non-irradiated samples that were exposed to atmospheric conditions can be the direct result of the radiolysis of the water in the cement as is demonstrated by the gas release experiment. As described above, the degree of carbonation depends on the degree of saturation of the sample. All samples start at fully saturated conditions after the curing period. For the reference samples, exposed to free atmospheric conditions, the carbonation rate depends on the relative humidity in their storage place, which also dictates the rate at which the sample is dried. For the irradiated samples, on the other hand, the radiolysis process is responsible for increased internal dehydration of the sample facilitating the carbonation process. For the irradiated sample, at a certain point, the radiolytic dehydration might bring the sample to a dehydration degree at which the rate of carbonation will be reduced due to the lack of water for the process.

Several publications have shown, using XRD measurements, that due to irradiation the fraction of

Table 2 Apparent Diffusion coefficient of Cs and Sr ions as obtained from the leaching experiments with paste samples irradiated to a dose of 10^7 Gy and non irradiated paste samples differing in their w/c ratio (w/c = 0.3, 0.45)

w/c	Irradiation	D_a Sr (cm^2/s) ($\times 10^{10}$)		D_a Cs (cm^2/s) ($\times 10^8$)	
		Initial	Final	Initial	Final
0.3	+	0.09 ± 0.04	0.04 ± 0.02	0.7 ± 0.6	0.6 ± 0.4
0.3	–	2.5 ± 1.8	0.4 ± 0.3	0.6 ± 0.6	0.6 ± 0.1
0.45	+	0.3 ± 0.1	0.3 ± 0.1	2.5 ± 1.7	8.9 ± 2.1
0.45	–	2.6 ± 0.7	1.1 ± 0.3	3.3 ± 1.3	4.9 ± 0.5

For each sample separate values were calculated for the initial and the final experimental times the calcite phase in the paste is increased. The increased fraction of the calcite phase was connected to the carbonation of Calcium Peroxide, one of the radiolysis products [1, 5]. The present study suggests that an additional reason for this increased carbonation rate is the enhanced radiolytic dehydration of the sample in addition to the formation of micro-cracks during the radiolytic process. In the case of the immobilized Sr ions the enhanced carbonation improves the retention of the ion in the matrix. The effect of carbonation on Sr immobilization is important when considering long-term service conditions in which large carbonation depth under natural conditions is expected. Enhanced carbonation of the wasteform, either by storage at high CO_2 levels or exposure to supercritical CO_2 could be considered as a method for improving the immobilization of the Sr ions.

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