

# Nuclear decontamination of cementitious materials by electrokinetics: An experimental study

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## Abstract

The objective of this paper is to evaluate the efficiency of an electrokinetic method of radionuclide removal from a cement-based material. This work is a two-part process. In the first part, a sample of mortar was uniformly contaminated for use as a reference. In order to ensure a uniform contamination, the ingress of the radioelement (cesium) was controlled by an external electrical field. The second part of this work concerns the removal of cesium from the contaminated mortar samples. This second, decontamination, phase was driven by the same electrical field. No electroosmosis was detected. Both phases were characterized by analyses of cesium and calcium concentrations in cathodic and anodic solutions, and by measurements of cesium content in the samples at the end of each phase. In addition to the electrical current, pH, and conductivity were measured during the experiments. Finally, the efficiency of the electrokinetic method was described in terms of decontamination factor, leading to promising results.

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## 1. Introduction

Many nuclear facilities will have to be decommissioned or dismantled in the next decade. Radionuclides may be found in building materials, and particularly in cement-based materials. Because cementitious materials are porous media, the penetration of radionuclides may occur up to several centimeters from the surface of the material [1]. Most of the existing decontamination methods are destructive (e.g., pneumatic drilling), some require a long time treatment (e.g., biological methods [2]), or are limited to the material surface (e.g., chemical methods [3]). The method proposed in this work, electrokinetics, is promising because deeply contaminated material may be treated without major damage. This

would reduce the cost of treatment by decreasing the volume of solid waste.

Electrokinetic methods have been used for chloride removal from concrete [4], and in applications for soil treatment [5–8]. They have also been applied more recently to nuclear problems [9–12]. It was decided to investigate the feasibility of such a method for the cesium removal, because it is frequently encountered as a contaminating agent in concrete [13]. Furthermore, the relatively high diffusivity of cesium, amongst the major contaminants, makes it a good candidate for this study.

The principle of the electrokinetic method is the application of an external electrical field between two electrodes in a sample of saturated cementitious material. The ionic transport is accelerated by the current, meaning that the diffusion rate is increased in comparison with a transport driven by a concentration gradient. The electrical field drives the ionic species towards the electrodes. When the cathode is

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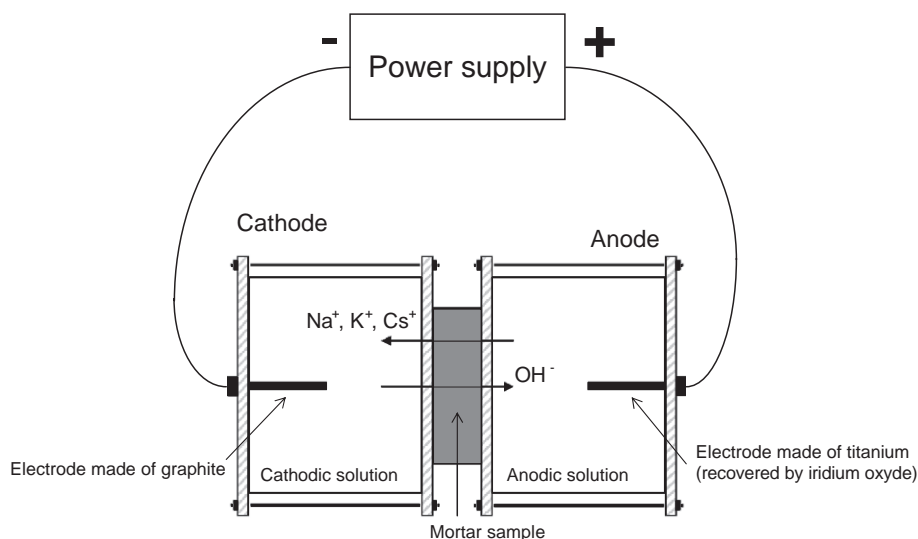


Fig. 1. Sketch of the decontamination cell.

positioned outside the material, cesium ions, cations, are carried away towards the outside.

The work presented in this paper consisted of laboratory experiments (Fig. 1), which were conducted in two phases. First, several mortar samples were contaminated with Cs at initial concentrations of  $100 \text{ mol/m}^3$  and  $1 \text{ mol/m}^3$ . Although such high concentrations are unlikely to be found in buildings in the nuclear industry, they are convenient for an experimental study of the physical mechanisms and of the efficiency of electrokinetic remediation. These concentrations allowed the use of non-radioactive cesium. Lower concentrations would have required radioactive cesium, due to the detection limits of the measurement technique (AAS) for non-radioactive ionic species. The contamination was accelerated by an external electrical field. The resulting uniformly contaminated reference samples represent the initial state for the second phase of this study: the decontamination process. The electrical field applied to the mortar samples ( $667 \text{ V/m}$ ) allowed partial decontamination of the specimens within three weeks. Results on cesium profiles through the mortar during the decontamination tests are presented, along with the efficiency factor of the technique, a ratio representing the initial contamination over the final contamination.

## 2. Experimentals

### 2.1. Samples

The mix proportions of the mortar samples were chosen in accordance with the European Standard EN 196-1, with a water to cement mass ratio of 0.5. A CEM-I type cement (see Table 1 for the chemical composition) and standard siliceous sand were used. The samples were cast in 110

mm diameter by 220 mm long cylindrical moulds, and kept for 24 h at 100% relative humidity. The samples were then removed from the moulds and stored for 3 months at  $20^\circ \text{C}$  and a 100% relative humidity. The dry density of the material was  $2180 \text{ kg/m}^3$ . Because the mortar micro-

Table 1  
Cement characteristics

Chemical composition [%]	
Insoluble residue	0.23
SiO <sub>2</sub>	22.84
Al <sub>2</sub> O <sub>3</sub>	2.7
Fe <sub>2</sub> O <sub>3</sub>	1.84
CaO	67.41
MgO	0.81
SO <sub>3</sub>	2.23
K <sub>2</sub> O	0.23
Na <sub>2</sub> O	0.14
S <sup>-</sup>	<0.01
Cl <sup>-</sup>	0.01
Loss of ignition	1.72
Total	99.94
CO <sub>2</sub>	1.22
Free CaO	0.47
Bogue composition [%]	
C <sub>3</sub> A	4.04
C <sub>3</sub> S	65.55
C <sub>2</sub> S	16.02
C <sub>4</sub> AF	5.58
Gypsum	4.79
Carbonates	2.77
Physical characteristics	
Density [g/cm <sup>3</sup> ]	3.16
Blaine fineness [cm <sup>2</sup> /g]	3450
28 days shrinkage [μm/m]	580
28 days compressive strength [MPa]	63.2
Setting time [min]	195
Heat of hydration, 12 h [J/g]	190

structure is an extremely important parameter in transport phenomena through porous media, the material was characterized by mercury porosimetry (Fig. 2). The corresponding total porosity obtained from Fig. 2 was 12.9%, while water porosity measurements led to a value of 16.7%. Note that due to the high pressures needed in the mercury intrusion technique, some pores are not available to the mercury, leading to lower results compared to water porosity measurements. Mercury porometry gives information on the pore size distribution which in our case was concentrated around 100 nm.

## 2.2. Migration cells

The experimental program was conducted in electrodiffusion cells (Fig. 1). The terms migration and electrodiffusion are used interchangeably in this paper, and refer to ionic transport through a porous medium driven by an external electrical field. The experimental set-up was very similar to the one used in chloride transport under an electrical field [14], therefore only a brief description of the set-up is given in this paper. The mortar sample was sandwiched between two electrolytic compartments. The volume of each compartment was 2.8 l, and the electrical field was applied between electrodes placed in each compartment. The cathode was made of graphite and the anode was made of titanium covered by iridium oxide. In each cell two more electrodes, made of platinum, were directly in contact with the mortar sample. They allowed the measurement and control of the potential difference between the two faces of the specimen. These L-shaped electrodes were coated with an isolating paint except for the part in contact with the sample. In order to limit the effects on the pore solution of temperature increase due to the electrical current, the electrical field remained below 800 V/m [15]. An electrical field of 12 V between the L-electrodes was chosen, corresponding to 667 V/m. The cathodic and anodic compartments were filled with a NaOH solution ( $100 \text{ mol/m}^3$ ) in order to match the high pH value of the pore solution in cementitious materials. A volume control valve was located at the top of each compartment (see

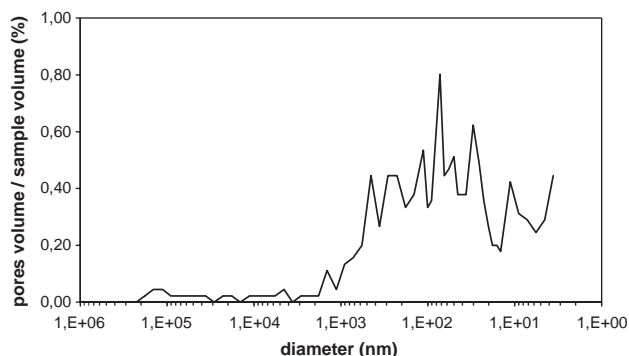


Fig. 2. Mercury porosimetry of the mortar.

Fig. 1) in order to check whether electroosmosis occurred during the experiments.

The samples tested were 18 mm thick (by 110 mm diameter) sections sawn from the middle part of the 220 mm long cylinders. The specimens were surrounded by a coat of silicone in order to ensure a leak-proof assembly. Taking into account the silicone coating, the sample cross-section in contact with the electrolyte compartments solutions was  $65 \text{ cm}^2$ .

Atomic Absorption Spectroscopy (AAS) was used to measure the cesium and calcium concentrations in both electrolytic compartments. The calcium concentration was measured in order to obtain information on the possible carbonation of the samples which is an important factor in decontamination. In addition, Cs concentration profiles were measured after the contamination and decontamination tests using a profile grinder (PF 1100, Germann Instrument) was used to collect mortar powder. For the samples with higher contamination levels, grinding took place in 1 mm steps, but in the case of the lowest Cs contamination, 3 mm steps were required to obtain sufficient material to reach the lower measurement limit of AAS. 1 g of each powder sample was dissolved in 20 ml of nitric acid (52.5% by mass). The solution was completed to 100 ml, filtered, and analyzed by AAS. The results of the analysis represent the total cesium concentrations. Four samples from each cylinder were tested in four separate electrodiffusion cells in order to allow replicate concentration measurements to be performed. In addition the current, pH, and conductivity were measured in each electrolytic compartment.

## 3. Results and discussion

### 3.1. Initial phase of the experiments

An experimental program was implemented in order to detect the existence of an electroosmotic flow. 18 mm-thick samples, all coming from the same mortar specimen, were submitted to electrical fields ranging from 100 to 800 V/m, while the two electrolytic compartments were each filled with 1 or  $100 \text{ mol/m}^3$  NaOH solution. Each test was conducted for 40 days. The duration of the tests was chosen to be longer than the contamination and decontamination tests. The variation of solution volume in each compartment was measured daily and the results are shown in Fig. 3. The volume difference between the two electrolytes was between 0.5% and 0.9 %, and it was therefore concluded that no significant electroosmotic flow existed.

The mortar first had to be contaminated. An easy way to contaminate the samples would have been to introduce cesium directly in the mix water. However, adding cesium in the mixing water results in the incorporation of Cs in the hydration products [16]. Such a contamination would not represent what can be encountered in actual nuclear facilities because contamination comes during the use of

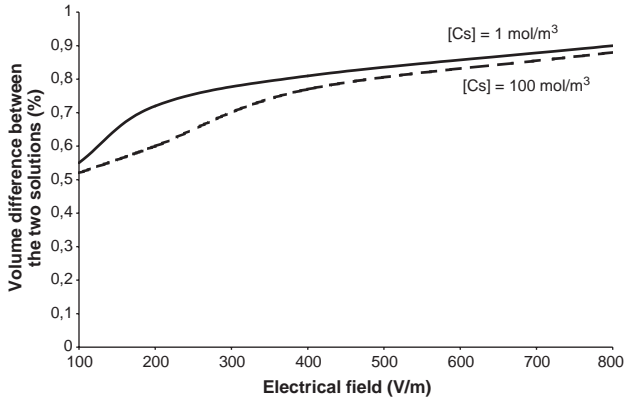


Fig. 3. Volume variations in the electrolytic compartments.

buildings (a laboratory inside a building where experiments on radionuclides are conducted for example), hence after the buildings are made. A more realistic method was chosen, which was to force cesium ions to migrate through the mortar samples by means of an external electrical field (natural diffusion of cesium would have taken too long). As shown later in this paper, this accelerated penetration of an ionic species allowed a uniform contamination of the samples.

Two different Cs concentrations, 100 and 1 mol/m<sup>3</sup>, were used in the anodic compartment. During the contamination phase Cs was added to the anolyte as cesium chloride.

The initial contamination tests were performed on the four mortar samples with an initial cesium concentration of 100 mol/m<sup>3</sup>, with pH measured in both compartments (Fig. 4). The pH always remained higher in the cathodic compartment due to the formation of hydroxyl ions at the cathode. Potential losses exist at the cathode, the anode, and in the electrolytic solutions, therefore the potential difference between the cathode and the anode was periodically adjusted so that the two platinum L-electrodes in contact with the sample measured a constant value (12 V) between the two faces of the specimen.

To avoid carbonation, the two compartments of the experimental set-up were completely filled with their

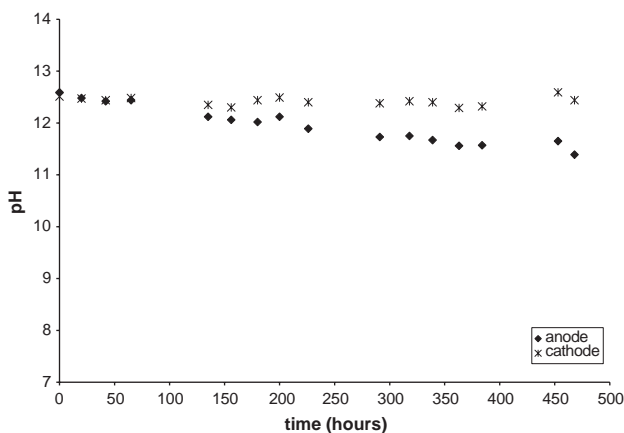


Fig. 4. pH during the initial phase of the experiments.

solution, and vertical valves were added which allowed the evacuation of the gas formed by electrode reactions, but prevented ingress of air.

### 3.2. Contamination

Fig. 5 shows the Cs concentrations variation in the cathodic compartment for an initial Cs concentration in the anodic side of 100 and 1 mol/m<sup>3</sup>. The diffusion coefficients of cesium were calculated by using the simplified Nernst–Planck equation [17]:

$$J = D_{\text{SNP}} z c \frac{F}{RT} E \quad (1)$$

where  $J$  represents the ionic flux (mol/m<sup>2</sup>s),  $D_{\text{SNP}}$  the Simplified Nernst–Planck diffusion coefficient (m<sup>2</sup>/s),  $c$  the concentration in the cathodic solution (mol/m<sup>3</sup>),  $z$  the valence of the ionic species,  $F$  the Faraday constant (96484.6 C/mol),  $R$  the ideal gas constant (8.314 J/mol K),  $T$  the absolute temperature (K), and  $E$  the local electric field (V/m) measured between the two faces of the specimen. The flux was approximated in steady state conditions by:

$$J = \frac{\Delta c V}{S \Delta t} \quad (2)$$

where  $V$  is the volume of the compartment,  $\Delta c$  the concentration variation in the cathodic compartment during a time interval  $\Delta t$ , and  $S$  the surface exposed to ionic transport.

Based on Eqs. (1) and (2), the Cs diffusion coefficient  $D_{\text{SNP}}$  was determined to be  $7.5 \cdot 10^{-12}$  m<sup>2</sup>/s for an initial cesium contamination of 100 mol/m<sup>3</sup>, and  $1.59 \cdot 10^{-11}$  m<sup>2</sup>/s for the 1 mol/m<sup>3</sup> initial contamination. This increase in  $D_{\text{SNP}}$  with the decrease in contamination level agrees with the work of Arsenault [18] who showed that the  $D_{\text{SNP}}$  for chloride varies in the same fashion for decreasing values of the initial chloride concentration.

The results of Lorente and Ollivier [19] show that the cesium contamination due to electromigration should be

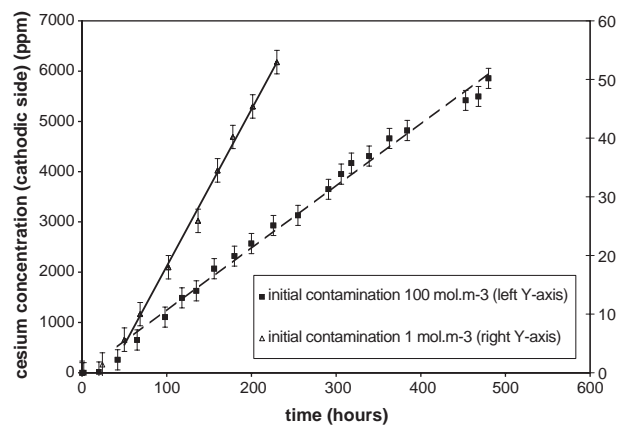


Fig. 5. Cs concentration in the cathodic compartment during a contamination phase (initial contaminating Cs concentration of 1 and 100 mol/m<sup>3</sup>).

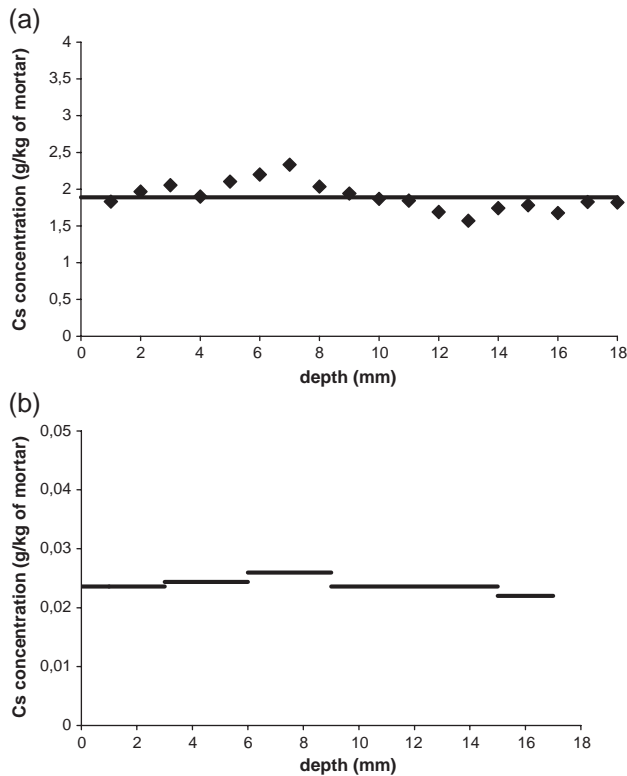


Fig. 6. Cs distribution through the mortar sample at the end of the contamination phase, initial contaminating Cs concentration of  $100 \text{ mol/m}^3$  (a) and  $1 \text{ mol/m}^3$  (b).

uniform in the mortar samples once steady state is reached. In natural diffusion, the flux of ionic species is driven by a concentration gradient and the concentration profile in a

porous medium can be expressed as first approximation by an error-function. When the ionic transport is controlled by an external electrical field, the concentration profiles are characterized by a penetration front as the contaminating ionic species enters the porous medium. This penetration front can be predicted by a multi-species approach model [19], which is in good agreement with experimental results dealing with chloride migration through cement-based materials [20,21]. Fig. 6 shows the total Cs concentration profiles measured in the mortar samples after contamination. For an initial contamination of  $100 \text{ mol/m}^3$ , the total Cs content was  $724 \text{ mg} (\pm 20 \text{ mg})$ , which represents an average value of  $1.90 \text{ g/kg}$  of mortar (Fig. 6-a). When the initial contamination was  $1 \text{ mol/m}^3$  the total Cs content was  $9 \text{ mg} (\pm 3 \text{ mg})$  (Fig. 6-b). Based on these measurements, the mortar samples were considered to be uniformly contaminated and therefore became reference samples. Fig. 7-a shows the calcium concentrations measured in both electrolytic compartments. The regular increase in Ca in the cathodic compartment suggests that no carbonation occurred during the experiments. The conductivity of the solutions was measured throughout the experiments and the results are given in Fig 7-b. Conductivity increased regularly in the anodic compartment due to both the decrease in hydroxyl ions (chemical reaction at the anode), and the migration of cations (cesium, calcium, and sodium) from the anolyte through the mortar. A slight decrease in conductivity occurred in the catholyte during the first few hours of the experiments because of the hydroxide transport to the anodic compartment. This decrease was followed by a linear increase, the onset of which corresponds to the

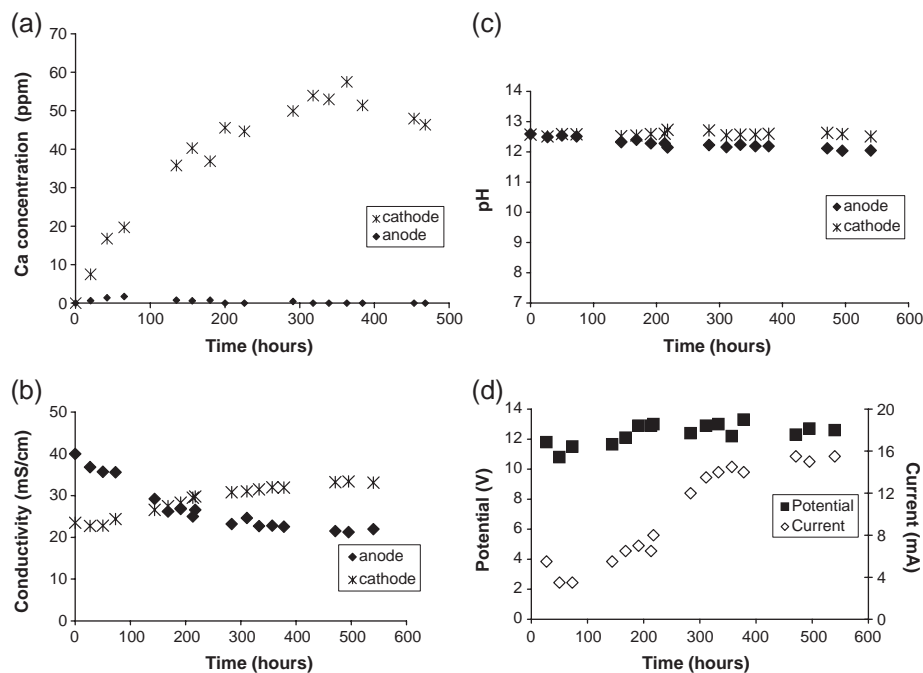


Fig. 7. Measurements of calcium (a), conductivity (b), pH (c), potential, and current (d) during a contamination phase (initial contaminating Cs concentration of  $100 \text{ mol/m}^3$ ).



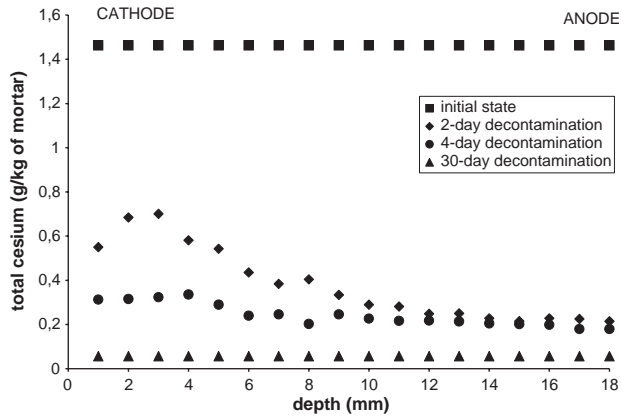


Fig. 8. Cs profiles through the mortar sample after different decontamination periods (initial contaminating Cs concentration of  $100 \text{ mol/m}^3$ ).

moment when the cations reached the compartment. The pH was again measured in each compartment, as shown in Fig. 7-c, which shows similar behaviour to the initial tests when carbonation occurred (Fig. 4). Plotted in Fig. 7-d are the potential and current values measured during the contamination test. The potential difference between the two faces of the sample was maintained at 12 V. The current, after an initial decrease, increased with the ionic fluxes according to the current law [22]:

$$I/S = F \sum_i z_i J_i \quad (3)$$

where  $I$  is the electrical current (A).

### 3.3. Decontamination

The mortar samples contaminated by cesium represented qualitatively what may be found in in situ conditions. The contaminated samples were considered therefore as the initial conditions of the decontamination phase. In the migration cells the electrical field was stopped for two days and the electrolytic solutions were replaced by a NaOH solution ( $100 \text{ mol/m}^3$ ). Thus the initial boundary conditions were the same in both compartments. Decontamination used the same electrical field as in contamination ( $667 \text{ V/m}$ ).

Among the samples tested for decontamination, four were dedicated to the study of transient regime with the  $100 \text{ mol/m}^3$  initial contaminating solution. Cesium content profiles were measured before starting the decontamination test, then after two, four, and thirty days of decontamination. The results are shown in Fig. 8 where the sample surface in contact with the cathodic compartment corresponds to the origin of the abscissa axis, and the 18 mm depth represents the surface in contact with the anodic compartment. The profile, measured before the electrical field was applied to the contaminated sample, represents the initial state, where cesium was uniformly distributed ( $1.47 \text{ g/kg}$  of mortar). After two days of decontamination, the total cesium concentration decreased to an average value of  $0.38 \text{ g/kg}$  of mortar, which represents 25% of the initial contamination

in the sample. However, the cesium profile was no longer uniform: a peak value of Cs ( $0.7 \text{ g/kg}$  of mortar) was measured at 3 mm from the cathodic compartment. The Cs profile was shaped by two different fronts. The decontamination front on the right side (see Fig. 8) moves from the anodic compartment towards the cathodic compartment, and represents Cs transport due to migration. The left side of the curve shows the existence of a leaching front due to the concentration gradient between the mortar sample (left-hand side of Fig. 8) and the cathodic compartment. The leaching front progresses towards the anodic side; which would represent (in situ) the healthy core of the material. This front corresponds to an accelerated leaching (or diffusive) phenomenon, leaching being enhanced by the external electrical current. The combined effect of two fronts, decontamination and leaching, accounts for the removal of cesium from the mortar sample.

The existence of the two different fronts can still be seen after four days of decontamination. Due to the progression of these two fronts in opposite directions, the maximum content of cesium ( $0.34 \text{ g/kg}$  of mortar) was found at about 4 mm from the cathodic side. On average,  $0.24 \text{ g}$  of Cs per kg of mortar remains in the sample, which is 16% of the initial contamination. Note that from 12 to 18 mm, the Cs content had not change compared to the value obtained after two days.

After 30 days of decontamination, the average residual contamination was  $0.06 \text{ g/kg}$  of mortar (4% of the initial contamination). A thirty-day decontamination was longer than the time needed to reach steady state (as shown in Fig. 9). It was therefore considered that the total amount of Cs measured represented the residual contamination. This indicates that a certain amount of Cs was not available for electrokinetic decontamination with the experimental conditions which were used. The remaining contamination was uniformly distributed throughout the mortar sample. The observation that no local accumulation occurs is important, because a concentration of cesium at the

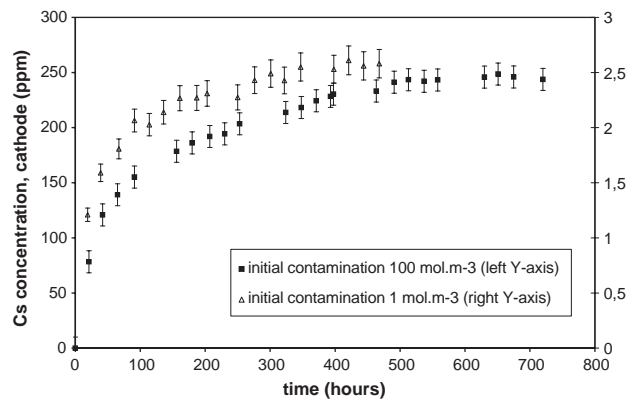


Fig. 9. Cs concentration in the cathodic compartment during the decontamination phase (initial contaminating Cs concentration of 1 and  $100 \text{ mol/m}^3$ ).

cathodic side of the material could represent a potential danger to workers during decontamination.

The Cs concentrations in the cathodic compartment are given in Fig. 9 (initial contamination of 100, and 1 mol/m<sup>3</sup>). In the case of an initial contamination of 100 mol/m<sup>3</sup>, the increase in Cs concentration reached a plateau after 480 h. Electrokinetic decontamination was more efficient in the early stages: it took 120 h to reach 74% of the maximum Cs concentration, while a further 360 h were necessary to reach the maximum concentration in the compartment. The total Cs content in the contaminated sample was 724 mg ( $\pm 20$  mg). The analysis of the cathodic solution indicated that 694 mg ( $\pm 10$  mg) were removed, suggesting that 30 mg  $\pm 30$  mg of cesium remained in the material. These results were confirmed by the measurement of the total Cs content (Fig. 10): 31 mg ( $\pm 3.6$  mg) of Cs still in the sample, with an average content of 0.081 g/kg of mortar (4% of the initial contamination).

When the initial contamination was 1 mol/m<sup>3</sup>, a similar trend was observed: the Cs concentration reaches 81% of its maximum value after 120 h and the maximum concentration was obtained after 350 h. It seems that the concentration rise is slightly higher in the first 120 h when the contamination was 1 mol/m<sup>3</sup>. Due to the very low Cs content in the sample (see Fig. 6), only the total Cs content in the material was measured. The residual contamination was 1.68 mg ( $\pm 0.55$  mg), which represented between 10% and 37% of the initial Cs content. This is in good agreement with the data published by Castellote et al. [11], although the experimental conditions were not exactly the same: in their study, Cs was introduced in the mixing water. The residual contamination seems to be different depending on the initial amount of contamination. The uncertainties in the cesium content are very high when contaminating the samples with 1 mol/m<sup>3</sup> of Cs: the measurements obtained in these samples after decontamination are at the lower detection limit of the AAS technique. Therefore it is difficult to be conclusive on whether the residual contamination is concentration dependent or not.

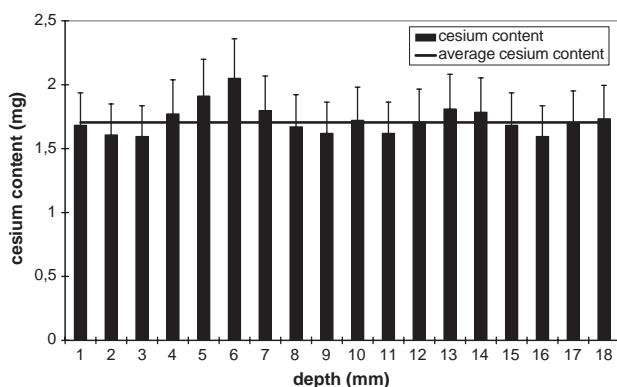


Fig. 10. Cs distribution through the mortar sample at the end of the decontamination phase (initial contaminating Cs concentration of 100 mol/m<sup>3</sup>).

Again, the calcium concentrations, pH, and conductivity were monitored in each compartment of the cells, along with the electrical current. No significant difference exists with the measurements obtained during the contamination tests were observed.

The efficiency of a nuclear decontamination method can be evaluated in terms of the decontamination factor, or efficiency factor. The decontamination factor represents the ratio between the initial level of contamination and that achieved after decontamination. Electrokinetic decontamination belongs to non-destructive techniques. Non-destructive techniques for decontaminating nuclear facilities are new, therefore the number of comparisons which can be made is limited. In a study [3] of chemical decontamination treatments, cesium was introduced in its radioactive form in the mixing water of cement pastes, leading to a cesium concentration of  $3 \cdot 10^{-7}$  g/kg of material. The chemical treatment achieved an efficiency factor of less than 2. Although the value of the decontamination factor shows the usefulness of chemical treatments, one has to remember that such a technique allows only surface decontamination, unlike an electrokinetic process.

The results which we have obtained gave decontamination factors of 23 when the initial contamination was 100 mol/m<sup>3</sup>; and 5 when the initial contamination was 1 mol/m<sup>3</sup>. Taking into account the uncertainties attributed to the measurement techniques, particularly for the 1 mol/m<sup>3</sup> contamination, the decontamination factor can be given in a range of 20.3–27 for the initial contaminating CsCl solution of 100 mol/m<sup>3</sup>, and 2.7–10.6 for the initial contaminating CsCl solution of 1 mol/m<sup>3</sup>. The decontamination factor seems to depend on the initial level of contamination, and this issue is currently being investigated.

#### 4. Concluding remarks

In this study an electrokinetic technique was used for cesium decontamination of cementitious materials. Firstly, an external electrical current was used to contaminate uniformly mortar samples. Then the samples were decontaminated by a reversal of the applied electrical current. Two different initial contaminating Cs concentrations were tested, namely 100 and 1 mol/m<sup>3</sup>.

Results were presented in terms of ionic concentrations in the compartments of the experimental cells and cesium profiles through the mortar samples. The concentration profiles shed light on the mechanisms of cesium transport during the decontamination by electrokinetics: leaching and migration, both having the external electrical field as a driving force. The experimental results indicate an order of magnitude for the decontamination factor: 5 for the 1 mol/m<sup>3</sup> and 23 for 100 mol/m<sup>3</sup> contamination solutions. This is significantly higher than in other non-destructive decontaminating techniques ( $\sim 2$  for chemical treatments).

Electrokinetic decontamination extracted 96% of the initial Cs contamination after three weeks, when the material was initially contaminated with 100 mol/m<sup>3</sup> solution of cesium; 63–90% with the 1 mol/m<sup>3</sup> contaminating solution. At this stage, the results suggest that electrokinetics appears to be an effective method for decontaminating cement-based materials.

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