Research Square

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Leaching of Sr and Cs from geopolymers under radiation environments

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Abstract

The leaching rates of Sr and Cs from geopolymers used to immobilize municipal solid waste incineration ash as a simulated waste were investigated to ensure the stability to the radiation during long-term storage. The leaching rates were not varied by the dose rates from the natural environment up to 15 Gy/h. However, the temporal variation of the leaching rates exhibited different characteristics depending on the elements. Longer leaching times were required to reach a constant leaching rate for the sample with a larger distribution coefficient. We elucidated the main contributor to leaching by conducting a leaching test on each ingredient. The dose rates investigated in this work correspond to low-level radioactive waste below the approximate boundary level between L2 and L1 in the classification. The production of hydrogen gas by the radiolysis of water and the corrosion of the geopolymers by the effects of radiation were expected, depending on the dose rate of the waste; leaching, however, was not affected by the dose rate.

Article Highlights

- Leaching rates of Sr and Cs from the geopolymers were not varied by the dose rates lower than 15 Gy/h.
- Longer times were required to reach a constant leaching rate for the sample with a larger distribution coefficient.
- The production of hydrogen gas and corrosion of the geopolymer were expected depending on the dose rate.

1. Introduction

Low-level radioactive wastes such as filters, metals, spent resin, and sludge are discharged from nuclear power plants. The waste of fines is produced by processing concrete debris [1, 2]. These wastes are immobilized with cement in metal drums or containers. These wastes contain radioisotopes of ^{14}C, ^{60}Co, ^{63}Ni, ^{90}Sr, ^{137}Cs, and ^{129}I. Low leaching rates for the radionuclides are critical for the immobilization of these wastes. Compared with cement, geopolymers are believed to have lower leaching rates, greater compressive strength, and greater resistance to heat. Geopolymers are inorganic polymers solidified by kneading metakaolin or fly ash with an alkali solution such as NaOH solution [3, 4]. Polymerization occurs through a chemical reaction between Si–O–Al–O polymer chains and Al–Si oxides. We have focused on fly-ash-based geopolymers, which consist of industrial waste except for the alkali solution.

Our group has previously reported on trends in the physical properties (e.g., compressive strength and flowability) and adsorption properties of Co^{2+}, Sr^{2+}, and Cs^{+} ions on geopolymers prepared using 1–5 M NaOH solutions and cement pastes [5]. We found that strength increased with increasing concentration of the NaOH solution from 1 M to 3 M and became constant at 3–5 M. Flowability, which is an indicator of workability, showed the opposite trend. Co^{2+} ions exhibited a distribution coefficient (K_d), an indicator

of adsorption performance, of ~ 10^3 for both the geopolymers and cement pastes. Cobalt hydroxide, which is poorly soluble, would adsorb onto the surface of the geopolymers and cement pastes. The K_d value of Sr^{2+} increased from 10^2 to 10^4 and that of Cs^+ increased from 10^1 to 10^2 as the concentration of the NaOH solution was increased from 1 M to 3 M, then became almost constant at 3 M to 5 M. The K_d values for Sr^{2+} and Cs^+ on the geopolymers were 1–4 orders of magnitude greater than those on the cement pastes. The K_d values for Sr^{2+} and Cs^+ on the geopolymers were not related to the amount of NaOH solution used to prepare the geopolymer. In addition, we used ^{29}Si nuclear magnetic resonance (NMR), positron annihilation lifetime spectroscopy (PALS), and extended X-ray absorption fine structure (EXAFS) to elucidate the adsorption mechanism affecting the K_d values of Sr^{2+} and Cs^+ ions [6]. PALS experimentally identified numerous pores with radii of 1–4 Å in the geopolymers; these pores are related to the encapsulation of Sr^{2+} and Cs^+ ions. EXAFS showed that the Sr^{2+} and Cs^+ ions encapsulated in the pores are located 3.6–3.9 Å from the pore surface. NMR showed that the number of Al atoms bound to SiO_4 tetrahedra increased with increasing NaOH concentration. These results indicated that the K_d values for Sr^{2+} and Cs^+ were exponentially correlated with the coulomb force between the positive charge of the ion and the negative charge resulting from AlO_4^- tetrahedra at the surface pores in the geopolymers. The K_d values of the ions on the cement pastes resulted from the van der Waals force.

The b- and g-rays from the radionuclides would cause long-term radiation effects on the geopolymers immobilizing actual radioactive waste. The radiation environment is known to promote corrosion in water via the radiation effect [7]. For example, the number of colloid particles with sizes in the nanometer range increased when Cu foil immersed in water was irradiated with g-rays. According to the Eh–pH diagram prepared using the FACTSAGE program, Sr is a water-soluble element at pH levels less than ~ 13 and Cs is soluble at any pH, in contrast to Cu [8]. Therefore, the radiation from the radioactive waste would increase the leaching rate of the radioisotopes.

In the present work, we investigated the leaching rates of Sr and Cs from geopolymers immobilizing municipal solid waste incineration ash (MSWIA). Compared with geopolymers immobilizing sludge and spent resin, the geopolymers immobilizing MSWIA would show degraded compressive strength in previous studies because of the production of hydrogen gas resulting from a chemical reaction with metallic Al contained in the waste during curing [9, 10]. Therefore, in terms of physical properties, MSWIA would be an inappropriate waste for immobilization. By contrast, the leaching expected in the worst case can be obtained by immobilizing MSWIA. In the present study, we performed leaching tests on the geopolymers under different dose rates to ensure their stability toward the radiation. However, we limited the irradiation time to ensure that the experiment was safe. Therefore, we obtained the temporal variation of the leaching over 1 week in the natural radiation environment to predict the long-term leaching using short-time leaching data collected in the radiation environment. In addition, we considered not only the radiation effect on the leaching in the scenario where the waste is accidentally immersed in water, but also the radiation effect on other issues expected during long-term storage.

2. Materials and methods

2.1. Preparation of geopolymers

Coal fly ash (FA) satisfying standard JIS A 6201, which was acquired from a thermal power plant, and granulated blast-furnace slag (BFS) that satisfies standard JIS A 6206 and contains gypsum were used as geopolymer ingredients. MSWIA crushed to a particle diameter smaller than 1 mm was used as simulated waste. The concentrations of the major elements in the ingredients, as well as those of Sr and Cs, were determined by X-ray fluorescence (XRF) analysis (Table 1, quoted from Kinoshita et al. [5]). FA, BFS, NaOH solution with a concentration of 1–5 M, and MSWIA were kneaded in a weight ratio of 7.0 : 3.0 : 10.0 : 24.4 for preparation of the geopolymers. After curing at 20°C for 3 months, the geopolymers were pulverized into particles with a diameter smaller than 1 mm.

Table 1
Chemical composition and Sr and Cs concentrations in the geopolymer ingredients.

Ingredient	Ca (%)	Si (%)	Al (%)	Fe (%)	Sr (ppm)	Cs (ppm)
FA	6.0	26.0	10.5	5.8	589	0.969
BFS	39.9	11.6	5.1	0.4	311	0.176
MSWIA	17.5	2.3	1.9	0.8	728	0.685

2.2. Leaching test under radiation environment

Each pulverized geopolymer and water in an amount equal to 10 times the weight of each geopolymer were transferred to a glass bottle. The samples were stirred for 2 h under g-ray irradiation at various distances from the radiation source in a ^{60}Co irradiation facility (Institute for Integrated Radiation and Nuclear Science, Kyoto University). Approximately 15 min after irradiation was stopped, supernatants were collected by centrifugation and filtration using a polytetrafluoroethylene (PTFE) syringe filter with a pore size of 0.22 μm. The Sr and Cs contents in the supernatants were determined by inductively coupled plasma mass spectrometry (ICP-MS). The same leaching test was performed on each ingredient as well. We obtained the leaching rates at dose rates from 0.17 Gy/h to 15 Gy/h based on the nominal dose. In addition, the leaching experiment was conducted outside the irradiation room, where the dose rate was measured to be ~ 0.02 μSv/h with a NaI scintillation survey meter. The dose rate corresponds to ~ 3×10^{-8} Gy/h using a conventional conversion factor.

2.3. Leaching tests under a natural radiation environment

Each geopolymer sample and 10 times its weight of water were stirred in a general experimental room under a natural radiation environment. Approximately 1 mL of the solution was collected at a certain time. The Sr and Cs contents in the solution were determined by ICP-MS after filtration using a 0.22 μm PTFE syringe filter. We obtained the temporal variation of the leaching rates in 1 week.

3. Results and discussion

3.1. Feature of leaching rates

3.1.1. Leaching in a radiation environment

Figure 1 shows leaching rates from the geopolymer samples under different dose rates. The leaching rates of Sr and Cs did not vary when the dose rate was between 3×10^{-8} and 15 Gy/h. The leaching rates for both elements decreased with increasing concentration of the NaOH solution used to prepare the samples. As mentioned in Sec. 1, the K_d values of Sr and Cs on the geopolymers prepared using FA, BFS, and NaOH solution increased by approximately two orders of magnitude for Sr and by approximately one order of magnitude for Cs when the NaOH concentration was increased from 1 M to 3 M; the K_d value was constant when the NaOH concentration was 3–5 M. By contrast, the leaching rates decreased by approximately two orders for Sr and by 50% for Cs with increasing NaOH concentration. The K_d value can be obtained by dividing the concentration in the solid phase by that in the liquid phase under the assumption that adsorption–desorption reaches equilibrium. The concentrations of Sr and Cs in each geopolymer sample should be the same because the geopolymers were prepared by kneading the same material with the same chemical composition. Therefore, the two-order difference for Sr and twofold difference for Cs in the concentrations in the liquid phases are consistent with the trends predicted from the K_d values. Most of the Sr and Cs ions would remain in the geopolymers after leaching. The approximate trend of the leaching can be explained in terms of the K_d values.

Numerous investigations on colloid formation and corrosion of metals, including their mechanisms, have been performed at high dose rates at the kGy/h level. In the first step of the radiation effect, radicals, hydrated electrons, and metal ions are produced by irradiation of the metals immersed in water [11–13]. The metal ions are reduced to atomic metals by interaction with the hydrated electrons. The atomic metals then form nanoparticles through nucleation. The radiation effect should cause corrosion even on the geopolymers by the same mechanism responsible for the corrosion on the metals.

In general, inorganic materials exhibit much higher durability against radiation than organic materials. For example, concretes and cement pastes did not show a substantial difference in compressive strength in materials irradiated by total dose of less than ~ 10^8 Gy of ^{60}Co g-rays [14–16]. The porosity varied when the total dose was greater than 10^4 Gy. By contrast, the organic materials were clearly decomposed when the total dose was greater than a few hundred grays [17, 18]. The changes in the characteristics result from the breaking of chemical bonds and the displacement of atoms forming the structure. The corrosion induced by the radiation would not only cause colloid formation but also destroy the absorption site of ions that exhibit larger K_d values. Therefore, water-soluble metals such as Sr and Cs immobilized in the geopolymers would be eluted by the corrosion. The radiation effect related to corrosion proceeds on a picosecond timescale [19]. Corrosion should terminate immediately after irradiation stops.

In the present work, we examined the leaching behavior at absorbed doses lower than 30 Gy. This dose was insufficient for us to observe a clear difference induced by the radiation effect. According to other studies of differences in the characteristics, most of the geopolymer structure would not be destroyed by the radiation effect. The radiation effect terminates immediately after irradiation stops. By contrast, the eluted elements can interact with the geopolymer unless the solid phase is removed. The solid phase was removed approximately 15 min after the irradiation was stopped in the leaching tests in this work. Chemical interactions during this period may affect the leaching rates.

3.1.2. Temporal variation of leaching rate in a natural radiation environment

Figure 2 shows the temporal variation of the leaching rates for Sr and Cs from the geopolymer samples in a natural radiation environment. The leaching rates of both elements increased rapidly with time during the first few hours. The leaching rate of Sr increased slightly even at 168 h. The rates at 168 h were greater than those at 2 h by a factor of 1.3, 2, and 2.7–2.8 for the samples prepared using NaOH concentrations of 1 M, 2 M, and 3–5 M, respectively. The samples prepared using higher-concentration NaOH solutions, which show larger K_d values, required longer times to become constant in the leaching. By contrast, the rate of Cs became nearly constant in 2 h for all of the samples. The leaching rates of both Sr and Cs at 2 h were approximately equivalent to those observed in the radiation environment described in Sec. 3.1.1.

We estimated the leaching rates from the geopolymer ingredients to demonstrate the effect of immobilization. The leaching rates, R, were obtained using Eq. 1:

$$R = C_{\mathrm{FA}} R_{\mathrm{FA}} + C_{\mathrm{BFS}} R_{\mathrm{BFS}} + C_{\mathrm{MSWIA}} R_{\mathrm{MSWIA}} \qquad (1)$$

where C_{FA}, C_{BFS}, and C_{MSWIA} denote composition percentages for FA, BFS, and MSWIA used to prepare the geopolymer, respectively, and R_{FA}, R_{BFS}, and R_{MSWIA} are the individual leaching rates from each ingredient. Figure 3 shows the temporal variation of the leaching rates for each component, as estimated using Eq. 1. The leaching rates of both Sr and Cs from all of the ingredients increased rapidly in the first few hours. The leaching rates of Sr from FA, BFS, and MSWIA became nearly constant in 96 h. The leaching rate of Cs from FA reached a constant value after 72 h, whereas the leaching rates from BFS and MSWIA continued increasing even after 72 h. The fractions of the leaching rates of Sr correspond to 66% from FA, 2% from BFS, and 32% from MSWIA after 2 h and to 52% from FA, 24% from BFS, and 24% from MSWIA after 168 h. The fractions of Cs correspond to 1% from FA, 1% from BFS, and 98% from MSWIA after 2 h and to 4% from FA, 24% from BFS, and 72% from MSWIA after 168 h.

A comparison of the temporal variations shown in Fig. 2 with those shown in Fig. 3 reveals that the leaching of Sr was equal to or suppressed by immobilization using the geopolymers, depending on the NaOH concentration. By contrast, the leaching of Cs was degraded by immobilization. As previously mentioned, most of eluted Sr resulted from FA and BFS. Via a chemical reaction with the NaOH solution, FA and BFS form a geopolymer structure with a larger K_d for Sr; this structure consists of an

aluminosilicate chain. Therefore, the leaching of Sr from the reaction product of FA and BFS is suppressed compared with the leaching of Sr from each geopolymer ingredient. However, most of the eluted Cs resulted from MSWIA. In addition, the eluants of the geopolymers show an alkaline pH of ~ 12 [5]. The pH values of FA, BFS, and MSWIA prepared by shaking each sample with water at a solid-to-liquid ratio of 1 : 10 were 9.3, 11.5, and 10.3, respectively. The pH values became constant within 2 h for all of the samples. Zhang et al. [20] reported that the leaching behavior is affected not only by materials but also by pH. The greater alkali concentration from the geopolymers may induce greater leaching from MSWIA than from each ingredient.

The particles of MSWIA are surrounded by an aluminosilicate chain structure. The structure would be removed from the surface of the MSWIA by pulverization. Therefore, the geopolymers that actually immobilize the radioactive waste would suppress the leaching more than the geopolymers used in the experiments in the present work.

3.2. Implication of use for immobilizing actual wastes

The distribution of the dose rate in a 200-liter drum filled with the geopolymer was simulated using PHITS code [21]. We assumed that the waste immobilized with the geopolymer contained only ^{90}Sr or ^{137}Cs as a radionuclide. The geopolymer was presumed to have the same weight ratio of FA, BFS, NaOH solution, and MSWIA as described in Sec. 2.1. In addition, the density of the geopolymer was assumed to be 2.1–2.3 g/cm^3. Figure 4 displays the dose-rate distribution in the drum containing the geopolymer with a unit concentration of either ^{90}Sr or ^{137}Cs. The waste containing ^{90}Sr exhibited a homogeneous distribution because ^{90}Sr emits only b-rays that are stopped within a few millimeters. The dose rate in the drum containing ^{137}Cs was higher at more than ~ 10 cm inside from the drum surface because the g-rays penetrated more deeply than the b-rays. On the basis of the simulation, a dose rate of 15 Gy/h, the maximum in the present work, corresponds to concentrations of (4.8–5.3)×10^7 Bq/g for ^{90}Sr and (6.9–7.5)×10^7 Bq/g for ^{137}Cs.

In Japan, low-level radioactive waste is classified into clearance, L3, L2, or L1 depending on its activity level [22, 23]. The concentration range of ^{90}Sr in each level is less than 1 Bq/g for clearance, 1–10 Bq/g for L3, 10^1–10^7 Bq/g for L2, and greater than 10^7 Bq/g for L1. The range of ^{137}Cs is less than 0.1 Bq/g for clearance, 0.1–100 Bq/g for L3, 10^2–10^8 Bq/g for L2, and greater than 10^8 Bq/g for L1. The present work indicates that no substantial effect was observed in the leaching at dose rates lower than 15 Gy/h. A dose rate of 15 Gy/h corresponds to lower L1 for ^{90}Sr and higher L2 for ^{137}Cs. That is, no substantial difference would be observed in the leaching from the geopolymers that immobilized even radioactive waste with an approximate boundary level between L2 and L1. Corrosion via colloid formation would occur as a result of the radiation effect.

Here, we consider the worst case that the waste is accidentally immersed in water. Based on the leaching test described in Sec. 3. 1. 2., the concentrations of soluble radionuclides in the water would nearly reach the maximum within a few hours if the waste is in a fine form. Actually, much longer time

would be required for the concentrations to reach the maximum because the waste should be immobilized in the drum. On the other hand, the concentrations should be lower comparing to the waste immobilized using the cement because the geopolymer has a few orders larger K_d than the cement [5].

The radiation not only induces corrosion in the case where the waste is immersed in water but also causes hydrogen production by radiolysis of water present in the waste. The geopolymer includes water originating from the alkali solution. We note that hydrogen was produced during long-term storage. The amount of hydrogen produced by radiolysis is linearly proportional to the dose [24]. The same trend has been observed for hydrogen production from hydrates [25]. Comparing the radiolysis of the hydrate with water reveals no substantial difference in the production rate of hydrogen. The production rate is affected by linear energy transfer (LET) of the radiation and dissolved materials in the water [26]. The geopolymers prepared in the present work contain 20% water. The amount of hydrogen gas produced is estimated to be on the order of 10^3 M when the geopolymer is continuously irradiated at a dose of 15 Gy/h for 300 years, presuming the storage scenario reported by Nakarai et al. [27] for L2 waste for this period. The hydrogen produced from L3 waste is estimated to be more than 5–8 orders of magnitude lower than the production at the 15 Gy/h level. Because radionuclides decay according to their half-lives, the amount of produced hydrogen gas should be smaller. We note that hydrogen was produced from the geopolymer immobilizing the radioactive waste with a higher dose rate, although the geopolymer showed higher performance for containment depending on the K_d value.

4. Summary

The leaching rates of Sr and Cs from geopolymers that immobilized MSWIA were investigated to ensure their stability under irradiation. The geopolymers were prepared by kneading FA, BFS, MSWIA, and a NaOH solution with a concentration of 1–5 M. The leaching tests for each geopolymer were performed at different dose rates ranging from the natural radiation environment dose rate of 3×10^{-8} Gy/h to 15 Gy/h. The leaching rates of Sr and Cs irradiated for 2 h were not affected by the dose rates. The rates were lower in the geopolymers prepared using NaOH solutions with higher concentrations. The magnitude of the leaching rates could be quantitatively explained in terms of the absorption related to the K_d values reported by Kinoshita et al. [5].

The 1-week leaching test performed in a natural radiation environment revealed different characteristics in the leaching rates depending on the elements. Leaching times longer than 1 week for Sr and 2 h for Cs were required for the leaching rates to become constant. In addition, most of the eluted Sr resulted from FA and BFS and most of the eluted Cs resulted from MSWIA, as indicated by the leaching test for each ingredient. The aluminosilicate chain forming the geopolymer was removed from the surface of the MSWIA particles by pulverization of the samples used for the leaching test in this work. Therefore, the actual geopolymers that immobilize the radioactive waste would suppress the leaching more than in the experiments in the present work.

The maximum dose rate of 15 Gy/h in the present work corresponds to the approximate boundary level between L2 and L1 in the classification scheme for low-level radioactive waste in Japan. At dose rates less than 15 Gy/h, the leaching rates are not varied by the radiation effect, although corrosion is expected as a result of the radiation effect. However, the production of hydrogen gas by radiolysis of water cannot be ignored. We note that hydrogen is produced if the radioactive waste with a higher dose rate is immobilized using the geopolymer.

Declarations

Competing interests

The authors have no relevant financial or non-financial interests to disclose.

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Author Contribution

N.K. performed the experiment and wrote the main manuscript. M.I. assisted the experiments. All authors reviewed the manuscript.

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Figures

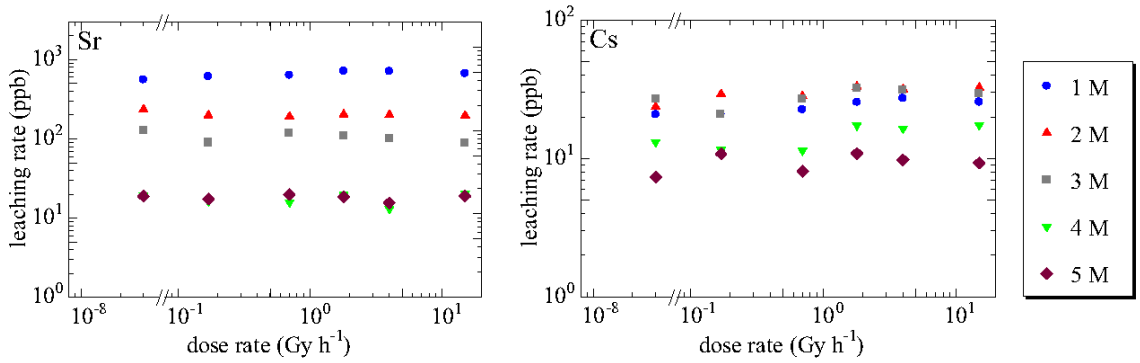


Fig. 1 Leaching rates of Sr and Cs from the geopolymer samples under different dose rates; the geopolymers were prepared using NaOH solutions with concentrations ranging from 1 M to 5 M.

Figure 1

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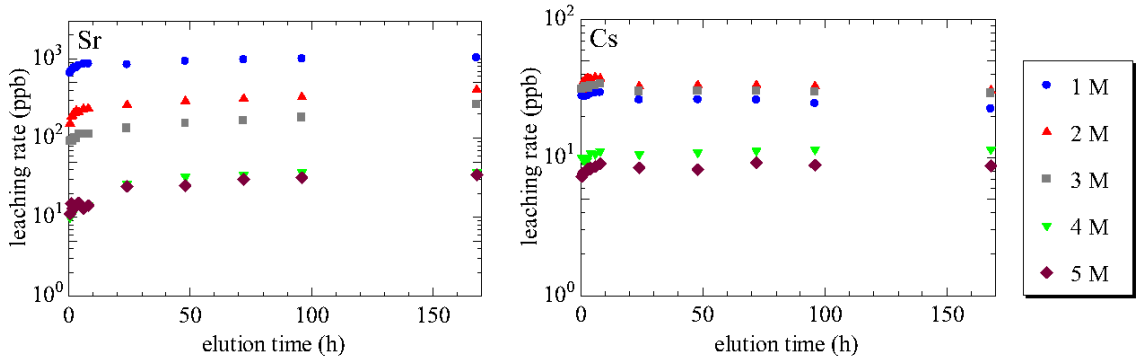


Fig. 2 Temporal variation of the leaching rates of Sr and Cs from the geopolymer samples prepared using NaOH solutions with concentrations ranging from 1 M to 5 M.

Figure 2

See image above for figure legend

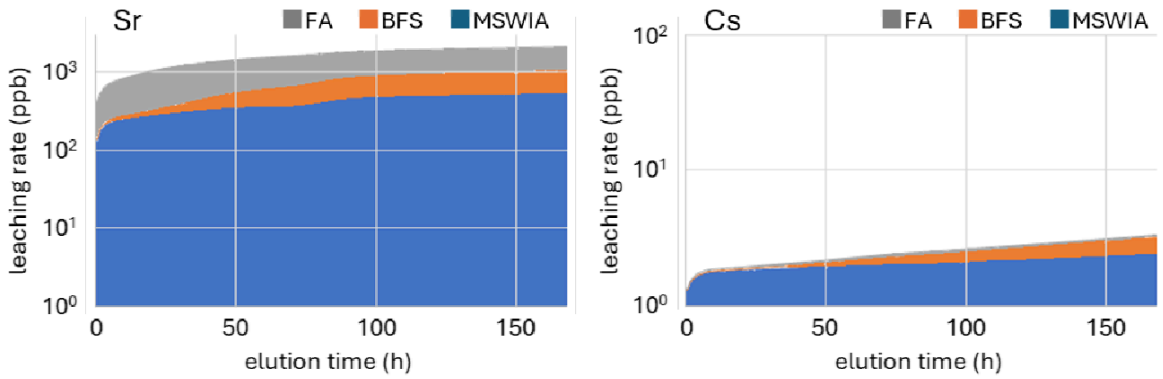


Fig. 3 Temporal variation of the leaching rates of Sr and Cs from each geopolymer ingredient.

Figure 3

See image above for figure legend

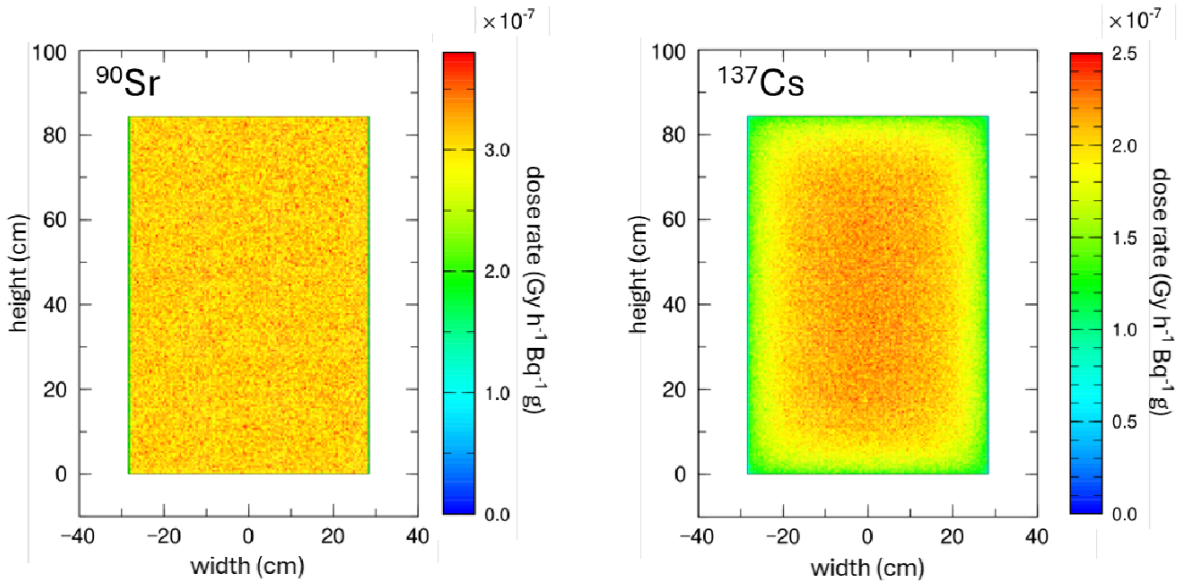


Fig. 4 Distribution of the dose rate in a drum in which ^{90}Sr and ^{137}Cs are immobilized in the geopolymer with a density of 2.1 g/cm^3.

Figure 4

See image above for figure legend