



# Fabrication of ceramic materials based on synthetic ivanyukite by spark plasma sintering for immobilization of cesium and strontium radionuclides

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## ABSTRACT

A hydrothermally synthesized sodium titanate (synthetic ivanyukite) was converted into a durable ceramic waste form via spark plasma sintering (SPS) to address the challenge of managing spent sorbents after aqueous decontamination. The material shows high sorption capacities for Cs<sup>+</sup> and Sr<sup>2+</sup> in aqueous media, with uptake values of 176.4 mg/g and 192.6 mg/g, respectively. These metrics are critical for treating cesium- and strontium-contaminated water. Following saturation with these cations, the loaded powders were densified by SPS at 600 and 1000 °C. The sintering temperature and the cation type both strongly affect the phase assemblage and microstructure of the final ceramics. At 1000 °C, chemically durable mineral-like phases emerge: the Cs-bearing material recrystallizes into hollandite and rutile, while the Sr-bearing counterpart transforms into a multiphase assemblage comprising SrTiO<sub>3</sub> perovskite (SYNROC-type), rutile, anatase, and clinopyroxene. Leaching tests demonstrate that the immobilization efficiency is exceptionally high, with Cs<sup>+</sup> and Sr<sup>2+</sup> release rates stabilizing at 10<sup>-7</sup>–10<sup>-8</sup> g·cm<sup>-2</sup>·day<sup>-1</sup>. Overall, this study establishes a proof-of-concept that integrates water treatment (using synthetic ivanyukite as a sorbent) with advanced spark plasma sintering (SPS) to produce highly durable, mineral-like waste forms, offering a sustainable pathway for the final disposal of radioactive contaminants removed from liquid effluents.

## 1. Introduction

Radioactive isotopes <sup>137</sup>Cs and <sup>90</sup>Sr are among the primary components of the liquid radioactive waste and waters generated as a result of emergency situations [1]. These isotopes are characterized by high migration activity in aqueous media and long half-lives (30.17 years for <sup>137</sup>Cs and 28.8 years for <sup>90</sup>Sr) [2], which makes their selective recovery from solutions using sorbents critically important [3]. The spent material must then be converted into a radiation-safe and environmentally stable form for subsequent disposal [4].

A wide range of sorbents is commonly used for this purpose, including natural (clinoptilolite) [5,6] and synthetic (NaA, NaA-U, NaX) zeolites [7,8], ferrocyanide sorbents (FNS, FSS, FSA, SFNS, FND,

Termoxid 35, etc.) [9–11], polymers, ion-exchange resins, zirconium phosphates (Termoxid 3A), titanium silicophosphates [12,13], zirconium hydroxides, hydrated titanium dioxide (Termoxid 5), carbon fibers, amorphous barium silicates (CPM-Sr) [14], mixed manganese oxides [15,16], wood (containing phosphate ion-exchange groups) [17], and synthetic analogues of zeolite-like titanate minerals [18–20]. For example, the mineral sitinakite, Na<sub>2</sub>KTi<sub>4</sub>(SiO<sub>4</sub>)<sub>2</sub>O<sub>5</sub>(OH)•4 H<sub>2</sub>O and its synthetic analogue IONSIV serve as prototype for creating a durable matrix for the immobilization of <sup>90</sup>Sr and <sup>137</sup>Cs radioisotopes.

The mineral sitinakite was first discovered on the Kola Peninsula, Russia [21]. Subsequently, a complete synthetic analogue of the mineral was produced by UOP LLC, Honeywell (USA) [22] and is currently used as the primary sorbent (CST or IONSIV-911) for the sorption of <sup>137</sup>Cs

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from liquid radioactive waste [23] in the nuclear industry. Such a sorbent is of particular interest because it allows the spent material to be transformed into SYNROC-type ceramics [24–30], offering a clear advantage over conventional disposal preparation methods, e.g., vitrification [31], cementation [32], and bituminization [33].

A key advantage of this material is its ability to be transformed into a dense and leach-resistant ceramic containing mineral-like phases (polucite, pyrochlore, leucite, hollandite, etc.) [34–37]. These phases provide high radiation stability [38,39], low leaching rates of radioisotopes from the matrix, and, consequently, technologically acceptable disposal conditions for secondary solid waste. In addition to the possibility of obtaining ceramics, IONSIV-type sorbents are interesting from the perspective of their high sorption activity ( $K_d$  value exceeding  $10^{-5}$  mL/g) [39,40] and the specific features of their crystal structure. The formation of a robust titanosilicate framework with a well-developed channel system allows the radioisotope to be localised directly in the interblock space (channels) of the main structure-forming framework [41]. Such a sorbent operates via the "host-guest" scheme typical of supramolecular chemistry, wherein the crystal structure acts as the host and the sorbed radioisotope as the guest [42].

Ceramic matrices offer a reliable route for immobilizing radioisotopes, and this approach is becoming increasingly attractive. For instance, cement matrices are problematic because ion exchange between radioisotopes and  $\text{Na}^+/\text{K}^+$  cations present in the cement can occur, and water contained in the cement may leach elements through pores [43]. Another critical issue associated with converting spent sorbents into solid waste is that the process often leads to an increase, rather than a decrease, in the final waste volume [44]. This effect occurs as a result of an increase in the volume of the final product. Polymer matrices, in turn, are limited by the risks of water radiolysis and  $\text{H}_2$  generation [45]. High-temperature glass-forming processes, such as vitrification, are technologically effective but they are energy-intensive and require precise control of the melt composition [46]. Consequently, ceramic and glass-ceramic approaches are being actively developed, including hot isostatic pressing (HIP) [47], cold sintering [48] and electrophysical sintering techniques [24,25,49,50]. These methods enable the production of dense monoliths with low leaching rates. For certain secondary wastes, these approaches substantially simplify disposal and enhance long-term storage safety.

The objective of this work is to fabricate ceramic matrices from a synthetic analogue of IONSIV (which is similar in crystal structure [51]) that has been pre-saturated with  $\text{Cs}^+$  and  $\text{Sr}^{2+}$  ions, using spark plasma sintering (SPS) technology. We also aim to investigate the phase composition, microstructure, mechanical properties, and hydrolytic stability of the resulting materials. The main goals of this study are: (1) hydrothermal synthesis of a sodium-based synthetic ivanyukite powder; (2) its saturation with  $\text{Cs}^+$  and  $\text{Sr}^{2+}$  cations; (3) fabrication of ceramic matrices by SPS at 600 and 1000 °C; (4) investigation of the phase composition and microstructure depending on temperature and cation type; (5) evaluation of mechanical properties and  $\text{Cs}^+/\text{Sr}^{2+}$  leaching rates; and (6) comparison of the obtained leaching rates with the requirements of GOST standard R 50926–96 for solidified high-level waste. Notably, this study reports the first successful consolidation of ivanyukite by spark plasma sintering, demonstrating a previously unexplored pathway for fabricating dense, mineral-like waste forms from this titanosilicate.

## 2. Experimental section

### 2.1. Reagents

The following reagents were used to prepare the synthetic SIV sample:  $\text{Na}_2\text{SiO}_3 \cdot 5 \text{H}_2\text{O}$  (analytical grade, GOST/TU 2145–001–52257004–2002), NaOH (high purity, GOST/TU 4328–77),  $(\text{NH}_4)_2\text{TiO}(\text{SO}_4)_2 \cdot \text{H}_2\text{O}$  (obtained from sphene concentrate of JSC «Apatit» using the pilot plant «Sorbent»), CsCl (high purity, TU

6–09–4066–84) and  $\text{SrCl}_2$  (analytical grade, GOST 4140–74).

### 2.2. Synthesis of starting powders

The SIV powder was crystallized in a 7 L hydrothermal synthesis autoclave (Parr Instrument, USA, FRC KSC RAS) using the following procedure:

First, 1041 g of the reagent was dissolved in 4 L of distilled water to prepare an alkaline sodium metasilicate solution. 336.5 g of NaOH was added to compensate for the sodium deficiency and achieve the required molar ratio of components in the final reaction mixture ( $6.12 \text{ Na}_2\text{O} : 3.38 \text{ SiO}_2 : 1 \text{ TiO}_2 : 168.35 \text{ H}_2\text{O}$ ). To this solution with a pH of 13, 618.3 g of  $(\text{NH}_4)_2\text{TiO}(\text{SO}_4)_2 \cdot \text{H}_2\text{O}$  was gradually added under constant stirring until complete dissolution and homogenization of the mixture. The resulting reaction mixture was loaded into the autoclave (fill factor of 1.42) and treated at 120 °C for 4 days without stirring. The autoclave operating regime during the ramp-up to stable conditions is summarized in Table 1. Upon completion of the synthesis, the autoclave was gradually cooled with running water. The resulting precipitate was separated from the mother liquor by vacuum filtration and then dried at 70 °C for 5 h.

### 2.3. Sorption saturation with $\text{Sr}^{2+}$ and $\text{Cs}^+$ cations

To saturate the synthesized ivanyukite analogue with  $\text{Sr}^{2+}$  and  $\text{Cs}^+$  cations, the SIV powder was treated with 0.05 mol/L solutions of CsCl and  $\text{SrCl}_2$ , using a threefold excess of the cations relative to the sorbent's saturation capacity. The experiments were performed at a solid-to-liquid ratio of 1:180 (5 g: 900 mL) under static conditions at 24 °C with constant stirring. The sorption capacity of the initial sorbent was determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) using a Perkin Elmer Optima 2100DV instrument (USA, IHTREMS KSC RAS) and was found to be 176.4 mg/g and 192.6 mg/g for Cs and Sr, respectively.

### 2.4. Ceramic fabrication by spark plasma sintering (SPS)

Ceramic samples were fabricated by spark plasma sintering using an SPS-515S "Dr. Sinter-LAB™" apparatus (Japan). 1 g of the starting powder was placed into a graphite die (inner diameter 10.5 mm) and pre-pressed at 20.7 MPa. The preform was then transferred to a vacuum chamber ( $10^{-5}$  atm) and sintered. Heating was performed using unipolar low-voltage pulsed current in an On/Off mode with a periodicity of 12 pulses followed by 2 pauses (i.e., the pulse packet duration 39.6 ms and the pause duration 6.6 ms). The temperature was monitored using a thermocouple inserted into a blind hole in the side wall of the die. The sintering was performed at 600 and 1000 °C with a heating rate of 100 °C/min up to 600 °C. The sample was held at the target temperature for 5 min and then cooled to room temperature over 30 min. A uniaxial pressure of 50 MPa was applied throughout the sintering cycle. To prevent sticking and facilitate sample extraction, the die and punches were lined with 200 µm thick graphite foil. The die was additionally wrapped in thermal insulation fabric to minimize heat losses. The

**Table 1**

Temperature and pressure monitored during the autoclave ramp-up to stable operating conditions.

| Time, h <sup>a</sup> | Temperature, °C | Pressure, bar |
|----------------------|-----------------|---------------|
| 0.5                  | 76              | 1.0           |
| 0.4                  | 100             | 4.0           |
| 0.25                 | 120             | 7.5           |
| 0.25                 | 125             | 6.0           |
| 0.25                 | 127             | 3.5           |
| 0.25                 | 125             | 3.0           |
| 0.16                 | 121             | 2.5           |
| 96                   | 120             | 2.5           |

<sup>a</sup> Stable operating mode of the autoclave

resulting cylindrical ceramics had a diameter of 10.3 mm and a height of 2–3 mm, depending on the die type and sintering conditions.

### 2.5. Characterization methods

Scanning electron microscopy (SEM) was performed using a Cross-Beam 1540 XB (Carl Zeiss, Germany) equipped with an energy-dispersive X-ray spectroscopy (EDS) attachment (Bruker, Germany). X-ray diffraction (XRD) analysis was carried out on a D8 Advance (Bruker AXS, Germany). Vickers microhardness (HV) was measured under a load of 0.2 N using an HMV-G-FA-D microhardness tester (Shimadzu, Japan).

The composition of solutions was determined by mass spectrometry using a Perkin Elmer ELAN 9000 DRC-e quadrupole mass spectrometer (PerkinElmer Inc., Shelton, USA).

The hydrolytic stability of the was evaluated by measuring the leaching rates of  $\text{Cs}^+$  and  $\text{Sr}^{2+}$  ions over 30 days of static contact with distilled water (pH 6.8) at 25 °C, following GOST R 52126–2003 (analogous to ANSI/ANS 16.1), which is based on an earlier IAEA-recommended method (ISO 6961:1982). The ion concentrations were determined by inductively coupled plasma mass spectrometry (ICP-MS) using an iCAP 7600 Duo spectrometer (Thermo Scientific, USA).

## 3. Results and discussion

### 3.1. Characterization of the synthesized powder

Fig. 1 shows the X-ray diffraction pattern of the synthesized ivanyukite analogue. Comparison with the reference pattern (ICSD No. 81366,  $(\text{Na,K,Cu})_{1-3}[\text{Ti}_4(\text{O,OH})_4(\text{SiO}_4)_3] \cdot 6-9\text{H}_2\text{O}$ , cubic, space group  $P-43m$ ) confirms that the synthesised material is a phase-pure sodium ivanyukite. A slight shift of the peaks towards lower angles is observed, which most likely reflects an increase in the unit-cell parameter of the synthesized product.

Tables 2 and 3 summarize the mass spectrometry data for the mother liquor and the autoclave bottom condensate, respectively. The mother liquor composition indicates near-complete consumption of the titanium introduced into the reaction mixture, along with residual Si and Na. Thus, the mother liquor can be reused in subsequent syntheses after appropriate adjustments of the Si and Na source quantities. The condensate composition reveals only negligible losses of the elements required for the synthesis, which do not affect the quality of the final product.

**Table 2**

Chemical composition of the mother liquor.

| Element | Content, g/L |
|---------|--------------|
| Na      | 81.1         |
| Si      | 21.8         |
| Ti      | 0.008        |
| Al      | 0.017        |

**Table 3**

Chemical composition of the autoclave bottom condensate.

| Element | Content, g/L |
|---------|--------------|
| Na      | 0.10         |
| Si      | 0.21         |
| Ti      | < 0.001      |
| Al      | not detected |

### 3.2. Characterization of ceramic samples

The XRD data (Fig. 2) reveal that the ceramic sample obtained by SPS at 600 °C without added Cs or Sr consists predominantly of an amorphous glass-like phase with an admixture of quartz phase (ICSD No. 079634, space group  $P3_121$ ) (Fig. 2). This behaviour is consistent with the thermal response of ivanyukite, which undergoes amorphization and framework rearrangement at temperatures around 600 °C. The cesium-containing ceramic prepared under the same conditions exhibits a more complex phase assemblage, comprising ivanyukite, a separate  $\text{SiO}_2$  phase, and a glass-like phase (Fig. 2). Unlike the Cs-free sample, the cesium-containing material does not fully amorphize at 600 °C; instead, the ivanyukite structure is partially retained, indicating that cesium stabilizes the framework to some extent. In contrast, the strontium-containing sample sintered at 600 °C shows no glass-like phase, but clearly contains the quartz phase (Fig. 2). This difference suggests that strontium has a more disruptive effect on the ivanyukite framework, promoting more extensive destruction and structural rearrangement. The absence of an amorphous phase in the SIV 600 Sr sample, along with the faster transformation kinetics compared to the pure and cesium-containing samples, supports this interpretation.

The diffraction patterns of the samples sintered at 1000 °C reveal more pronounced differences. For the cesium-containing ceramic, the formation of rutile (ICSD 009161, tetragonal,  $P4_2/mnm$ ) is observed

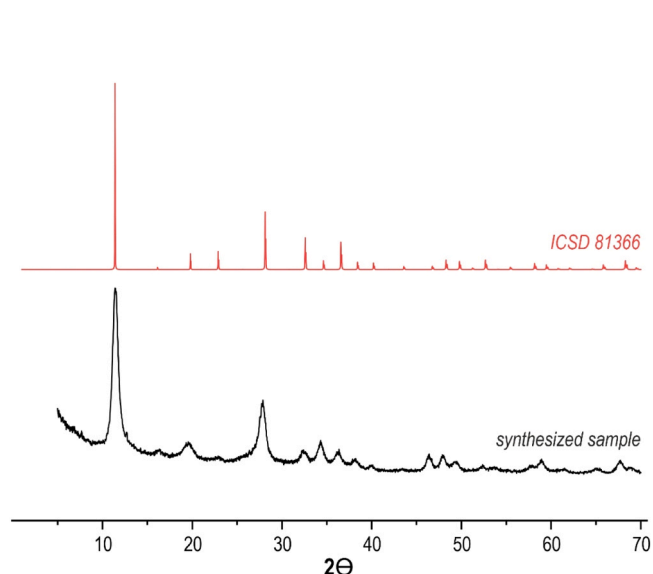


**Fig. 1.** X-ray diffraction pattern of the synthesized ivanyukite analogue (SIV).

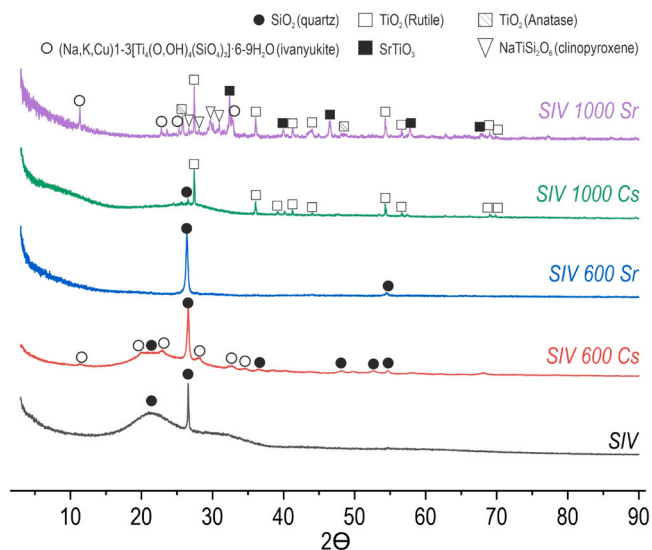


**Fig. 2.** X-ray diffraction patterns of ceramic samples obtained by spark plasma sintering (SPS).

alongside the ivanyukite phase (Fig. 2). The strontium-containing ceramic exhibits a multiphase assemblage consisting of ivanyukite, rutile, anatase (ICSD 202242, tetragonal,  $I4_1/amd$ ), tausonite ( $SrTiO_3$ , ICSD 065089, cubic,  $Pm-3m$ ), and clinopyroxene ( $NaTiSi_2O_6$ , ICSD 039194, monoclinic,  $C2/c$ ). This phase assemblage indicates that the transformation of the original ivanyukite matrix is essentially complete at 1000 °C, resulting in ceramics suitable for effective immobilization of  $Cs^+$  and  $Sr^{2+}$ . Notably, the strontium-containing sample contains a SYNROC-type phase, tausonite ( $SrTiO_3$ ) with the cubic perovskite structure. This phase is well established as an effective host matrix for safe and durable immobilization of radionuclides [52].

Metallographic examination of the cross-sections (Fig. 3) reveals that the ceramics have a composite microstructure. Heterogeneous regions containing both coarse and fine inclusions are clearly visible. Energy-dispersive X-ray spectroscopy (EDS) analysis of individual zones (Table 4) corroborated the XRD data, enabling more precise phase identification (Table 5). The amorphous phase present in the samples

sintered at 600 °C is consistent with pyrochlore-type phases. Residual pyrochlore-type phases are also detected in the strontium-containing sample, which predominantly consists of quartz according to XRD (Fig. 2). The cesium-containing ceramics sintered at 1000 °C contains a ivanyukite phase, indicating that the phase transformation is complete and a crystalline ceramic material has been formed. The strontium-containing sample sintered at 1000 °C exhibits numerous mineral phases, many of which are difficult to identify unambiguously; these include various titanosilicate phases and a SYNROC-type phase.

SEM analysis shows that the ceramics sintered at 600 °C have a monolithic structure (Fig. 4a, b). In contrast, the strontium-containing sample exhibits porosity (Fig. 4c), which is probably attributable to the accelerated formation of a new ceramic framework and concurrent destruction of the original ivanyukite structure. The microstructure of the samples sintered at 1000 °C is markedly different. The cesium-containing ceramic retains a monolithic character but contains large agglomerates composed of structured acicular crystallites (Fig. 4d).

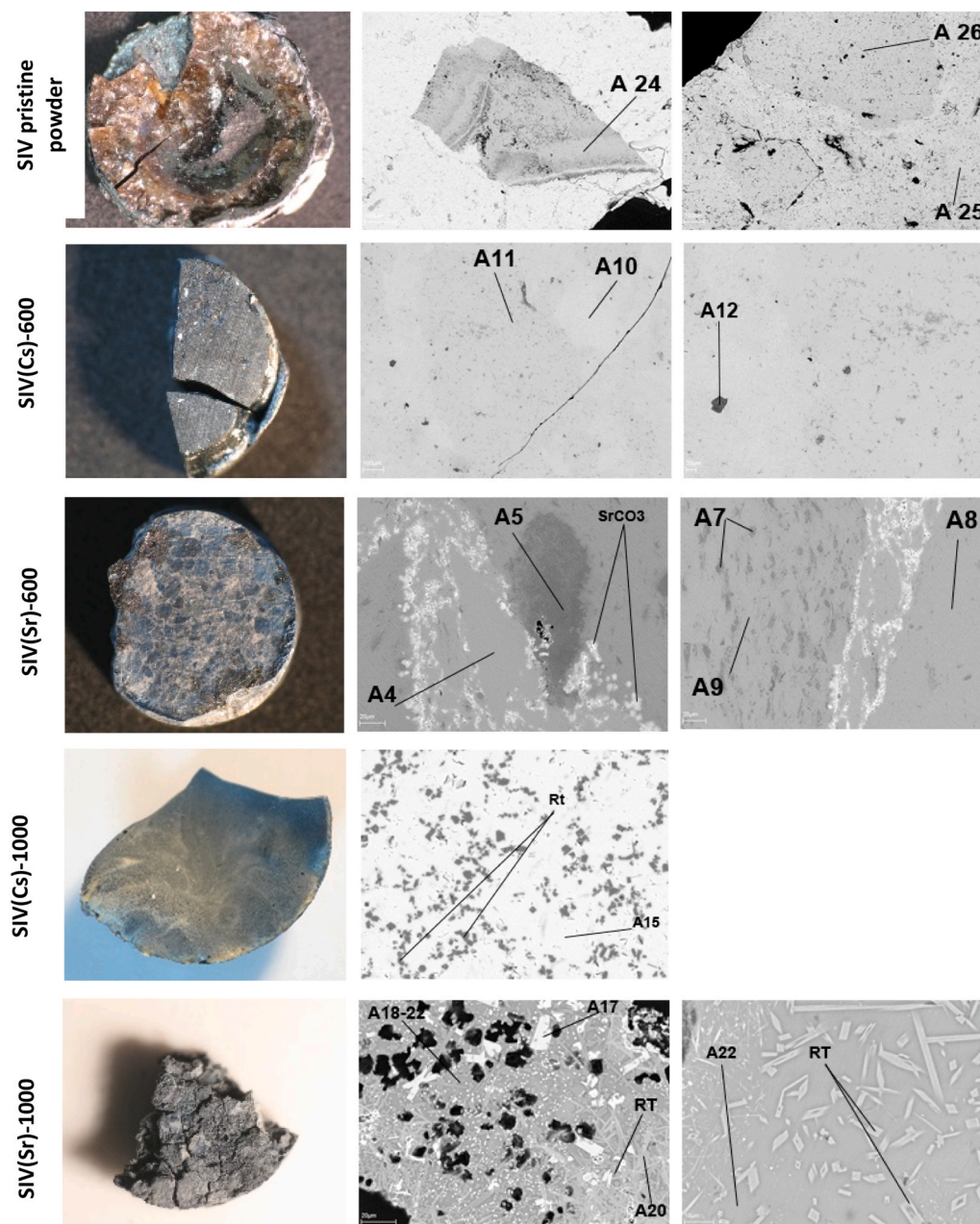


Fig. 3. General appearance and optical images of polished cross-sectional surfaces of the ceramic samples fabricated by SPS.

**Table 4**

Energy-dispersive X-ray spectroscopy (EDS) data for the phases present in the ceramics fabricated by SPS.

| Element, oxide    | Analysis zone |       |       |       |       |       |       |       |       |       |       |       |       |       |       |
|-------------------|---------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
|                   | A4            | A5    | A7    | A8    | A9    | A10   | A11   | A12   | A17   | A18   | A20   | A22   | A24   | A25   | A26   |
| Na <sub>2</sub> O | 1.98          | 7.93  | 6.18  | 3.69  | 2.07  | 1.40  | 2.40  | 0.94  |       | 6.44  | 1.67  | 5.39  | 21.31 | 14.55 | 12.40 |
| SiO <sub>2</sub>  | 28.69         | 41.84 | 46.66 | 29.50 | 27.82 | 22.79 | 22.28 | 18.49 | 20.77 | 47.18 | 39.14 | 47.14 | 32.93 | 29.94 | 34.32 |
| K <sub>2</sub> O  | 3.20          | 4.86  | 6.08  | 4.46  | 2.94  | 0.84  | 0.83  | 0.62  | 0.07  | 7.50  | 1.60  | 6.98  | 0.16  | 7.35  | 7.96  |
| TiO <sub>2</sub>  | 45.94         | 37.58 | 29.07 | 46.77 | 47.22 | 35.93 | 38.27 | 30.55 | 40.56 | 26.68 | 31.53 | 24.67 | 37.21 | 46.16 | 43.47 |
| SrO               | 19.22         | 6.58  | 11.90 | 15.34 | 19.69 |       |       | 12.94 | 37.41 | 9.45  | 25.45 | 12.28 |       |       |       |
| CsO <sub>2</sub>  |               |       |       |       |       | 38.87 | 36.06 | 13.15 |       | 1.07  |       | 1.04  |       |       |       |
| H <sub>2</sub> O  | 0.83          | 0.18  | 0.10  | 0.14  | 0.26  | 0.18  | 0.17  | 22.28 | 1.20  | 1.35  | 0.60  | 2.37  | 8.08  | 1.09  | 1.28  |

**Table 5**

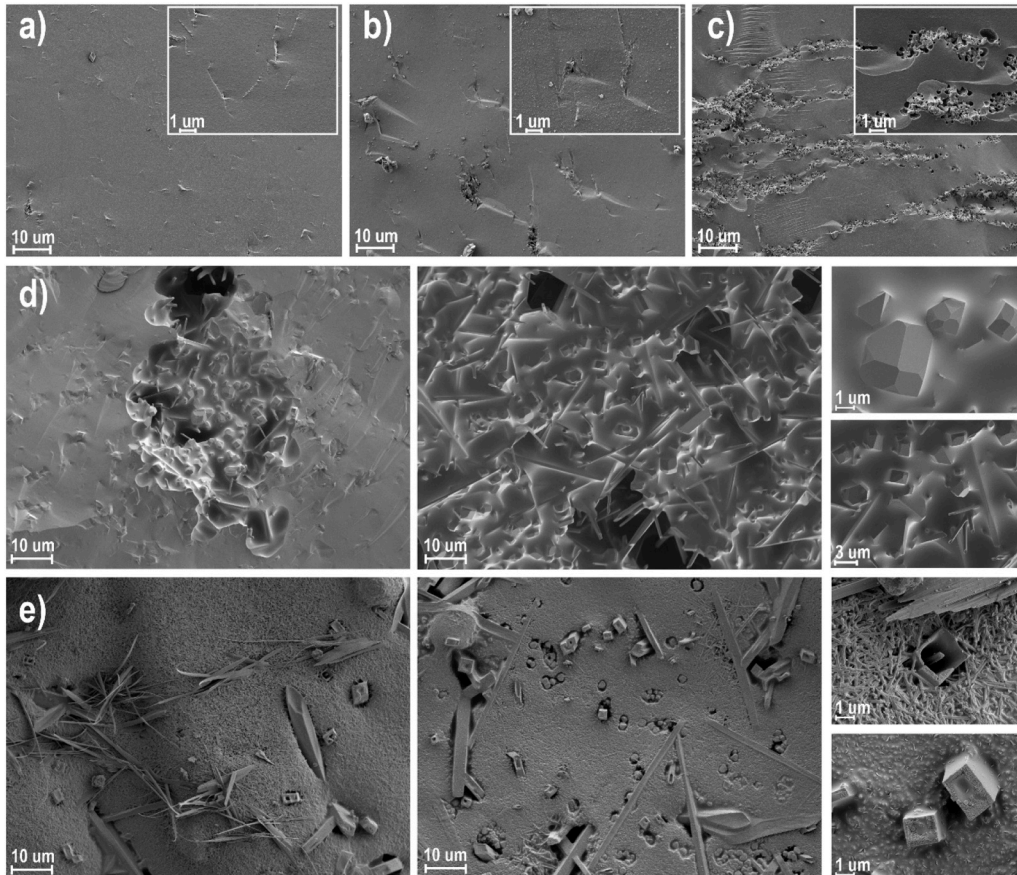
Mineral-like phases identified in the ceramics based on EDS data.

| Analysis zone | Calculated formula                                                                      | Closest mineral-like prototype |
|---------------|-----------------------------------------------------------------------------------------|--------------------------------|
| A4            | (Na,K,Sr)Si <sub>2.4</sub> Ti <sub>3</sub> O <sub>12</sub>                              | pyrochlore                     |
| A5            | (Na,K,Sr,Zn) <sub>2.2</sub> Si <sub>3.5</sub> Ti <sub>2.4</sub> O <sub>13</sub>         | leucite                        |
| A7            | (Na,K,Sr) <sub>2.2</sub> Si <sub>4</sub> Ti <sub>1.8</sub> O <sub>13</sub>              | leucite                        |
| A8            | (Na,K,Sr) <sub>1.8</sub> Si <sub>2.4</sub> Ti <sub>3</sub> O <sub>12</sub>              | pyrochlore                     |
| A9            | (Na,K,Sr) <sub>1.6</sub> Si <sub>2.4</sub> Ti <sub>3</sub> O <sub>12</sub>              | pyrochlore                     |
| A10           | (Cs,Na,K) <sub>2.22</sub> Si <sub>2.5</sub> Ti <sub>3</sub> O <sub>12</sub>             | pyrochlore                     |
| A11           | (Cs,Na,K) <sub>2.22</sub> Si <sub>2.4</sub> Ti <sub>3</sub> O <sub>12</sub>             | pyrochlore                     |
| A12           | (Cs,Na,K,Sr) <sub>2</sub> Si <sub>2.3</sub> Ti <sub>3</sub> O <sub>12</sub>             | pyrochlore                     |
| A15           | Cs <sub>1.36</sub> □ <sub>0.64</sub> Ti <sub>8</sub> <sup>(3+,4+)</sup> O <sub>16</sub> | hollandite                     |
| A17           | Sr <sub>2</sub> Ti <sub>3</sub> Si <sub>2</sub>                                         | not a mineral                  |
| A18–22        | (Na,K,Sr) <sub>3</sub> Ti <sub>2</sub> Si <sub>5</sub>                                  | labuntsovite                   |
| A24           | not identified                                                                          | -                              |
| A25           | not identified                                                                          | -                              |
| A26           | not identified                                                                          | -                              |
| RT            | rutile TiO <sub>2</sub>                                                                 | -                              |

dispersed throughout the bulk. The remainder of the material consists of a monolithic hollandite phase with the nominal composition Cs<sub>1.36</sub>□<sub>0.64</sub>Ti<sub>8</sub><sup>(3+,4+)</sup>O<sub>16</sub>. The strontium-containing ceramic exhibits a structure comprising nanoscale acicular crystallites, together with larger acicular and cubic crystallites (Fig. 4e). The larger acicular crystallites are again rutile, whereas the cubic crystallites are most probably perovskite-type.

The EDS data (Fig. 5) corroborate the phase distribution observed by SEM. Titanium is strongly enriched in the acicular regions, where other elements are virtually absent. This confirms that these regions consist of rutile. Cesium and strontium are uniformly distributed across the scanned areas and are associated with regions containing both titanium and silicon, which correspond to the hollandite and perovskite phases, respectively. For the samples sintered at 600 °C, the elemental distribution is almost completely uniform (Fig. 5a, b, d). For the SIV and SIV (Cs)-600 samples, this uniformity is consistent with their amorphous phase composition and SEM microstructure (Fig. 4a, b). In contrast, the SIV(Sr)-600 sample exhibits the same elemental uniformity despite its highly porous structure, which is attributed to rapid phase

Metallographic analysis indicates that these crystallites consist of rutile

**Fig. 4.** SEM images of polished cross-sections of the SPS-fabricated ceramics: (a) SIV; (b) SIV(Cs)-600; (c) SIV(Sr)-600; (d) SIV(Cs)-1000; (e) SIV(Sr)-1000.

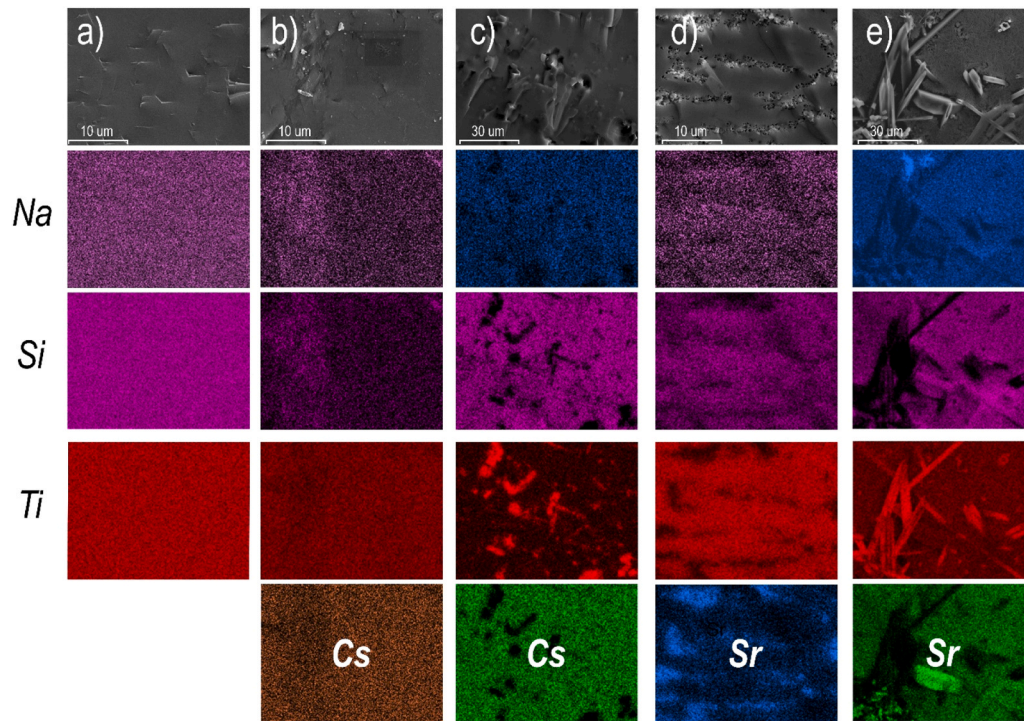


Fig. 5. Elemental distribution maps (EDS) of the SPS-fabricated ceramics: (a) SIV; (b) SIV(Cs)-600; (c) SIV(Cs)-1000; (d) SIV(Sr)-600; (e) SIV(Sr)-1000.

transformation (Fig. 4c).

Vickers microhardness could be measured only for the neat ceramics and the Cs-loaded sample sintered at 600 °C. For the other samples, values fell below the instrument's detection limit. The data (Table 6) show minimal scatter for the SIV 600 sample, indicating a homogeneous structure and uniform phase composition. The Cs-loaded sample exhibits a higher average microhardness but with outliers well above the mean. This suggests phase heterogeneity, consistent with the XRD (Fig. 2) and petrographic observations (Fig. 3). This behavior is not due to defects or porosity, as SEM reveals a monolithic structure (Fig. 4b). For the Sr-loaded sample, no reliable measurements could be obtained because of the highly porous structure (Fig. 4c), which resulted in values below the detection limit.

The hydrolytic stability of the SPS-fabricated ceramics was evaluated by measuring Cs<sup>+</sup> and Sr<sup>2+</sup> leaching rates (Fig. 6). After 14 days of contact with the leaching medium, the leaching rates stabilised at 10<sup>-7</sup>–10<sup>-8</sup> g·cm<sup>-2</sup>·day<sup>-1</sup> and did not increase further. This behavior is

consistent with the XRD data (Fig. 2) and the phase identification (Table 5). Effective Cs immobilization is attributed to incorporation of Cs<sup>+</sup> into both the ivanyukite framework and the amorphous pyrochlore-type phase, whereas Sr<sup>2+</sup> is retained primarily in amorphous leucite- and pyrochlore-type structures. The initially higher leaching rates are attributed to the washout of weakly adsorbed cations from the surface. (Table 7)

#### 4. Conclusions

A sodium-based synthetic analogue of the cubic ivanyukite was successfully synthesised via a hydrothermal route, as confirmed by XRD. The material exhibited high sorption capacities of 176.4 mg/g for Cs<sup>+</sup> and 192.6 mg/g for Sr<sup>2+</sup>, enabling effective pre-loading of the powder

Table 6

Vickers microhardness (HV) of the samples sintered at 600 °C.

| N <sup>o</sup> | SIV, HV     | SIV-Cs, HV    | SIV-Sr, HV  |
|----------------|-------------|---------------|-------------|
| 1              | 4.3 ± 1.49  | 44.0 ± 14.35  | 22.1 ± 5.12 |
| 2              | 4.5 ± 1.49  | 48.0 ± 14.35  | 27.5 ± 5.12 |
| 3              | 5.8 ± 1.49  | 52.5 ± 14.35  | 30.2 ± 5.12 |
| 4              | 9.9 ± 1.49  | 54.1 ± 14.35  | 34.8 ± 5.12 |
| 5              | 10.5 ± 1.49 | 55.8 ± 14.35  | 36.1 ± 5.12 |
| 6              | 10.6 ± 1.49 | 61.7 ± 14.35  | 39.4 ± 5.12 |
| 7              | 11.0 ± 1.49 | 63.1 ± 14.35  | 42.0 ± 5.12 |
| 8              | 12.0 ± 1.49 | 75.9 ± 14.35  | 45.7 ± 5.12 |
| 9              | 12.7 ± 1.49 | 84.3 ± 14.35  | 48.3 ± 5.12 |
| 10             | 13.0 ± 1.49 | 85.7 ± 14.35  | 51.9 ± 5.12 |
| 11             | 14.5 ± 1.49 | 89.0 ± 14.35  | 55.6 ± 5.12 |
| 12             | 15.0 ± 1.49 | 93.0 ± 14.35  | 60.2 ± 5.12 |
| 13             | 16.9 ± 1.49 | 94.9 ± 14.35  | 64.5 ± 5.12 |
| 14             | 18.1 ± 1.49 | 98.4 ± 14.35  | 70.9 ± 5.12 |
| 15             | 22.4 ± 1.49 | 100.9 ± 14.35 | 76.4 ± 5.12 |
| 16             | 22.6 ± 1.49 | 108.7 ± 14.35 | 82.1 ± 5.12 |
| 17             | 23.8 ± 1.49 | 114.0 ± 14.35 | 88.5 ± 5.12 |
| 18             | 25.1 ± 1.49 | 254.9 ± 14.35 | 94.0 ± 5.12 |

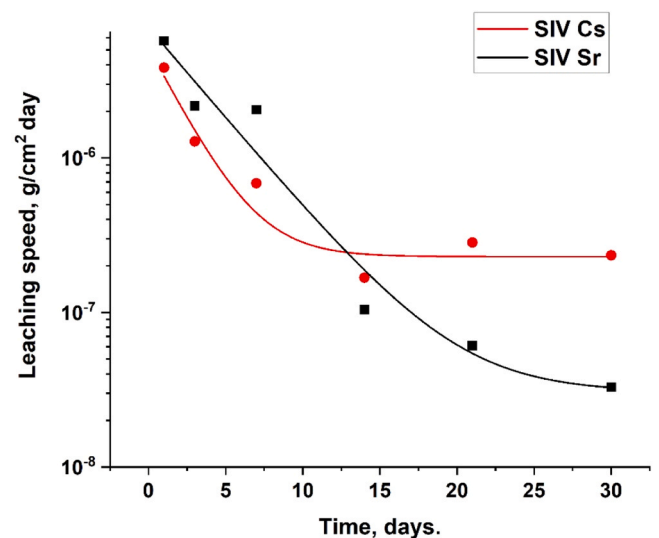


Fig. 6. Leaching rates of cesium and strontium ions from the ceramics sintered at 600 °C.

**Table 7**Kinetic fitting parameters for the Cs and Sr leaching data, obtained using an exponential decay model (ExpDec1) with a fixed baseline ( $y_0$ ).

| Radionuclide / Element | $y_0$ (g/cm <sup>2</sup> ·day)           | $A_1$ (g/cm <sup>2</sup> ·day)   | $t_1$ (day)     | Reduced $\chi^2$       | R <sup>2</sup> (COD) | Adj. R <sup>2</sup> |
|------------------------|------------------------------------------|----------------------------------|-----------------|------------------------|----------------------|---------------------|
| SIV Cs                 | $(3.55 \pm 1.32) \times 10^{-8}$ (fixed) | $(6.64 \pm 2.39) \times 10^{-6}$ | $3.21 \pm 0.54$ | 0.183                  | 0.840                | 0.733               |
| SIV Sr                 | $3.06 \times 10^{-8}$ (fixed)            | $(6.96 \pm 1.29) \times 10^{-6}$ | $3.70 \pm 1.20$ | $4.99 \times 10^{-13}$ | 0.918                | 0.898               |

with these cations prior to sintering.

Spark plasma sintering (SPS) at 600 and 1000 °C revealed that both the sintering temperature and the cation type profoundly influence the phase composition and microstructure of the final ceramics, a finding that, to our knowledge, has not been reported previously for ivanyukite-based materials. At 600 °C, the neat sample consisted predominantly of an amorphous glass-like phase. In contrast, the presence of Cs partially stabilized the ivanyukite framework, whereas Sr promoted more extensive destruction of the original structure and the formation of a highly porous material. Raising the temperature to 1000 °C completed the phase transformations. The Cs-loaded ceramic transformed into rutile and hollandite, whereas the Sr-loaded sample yielded a multi-phase assemblage comprising rutile, anatase, tausunite (SrTiO<sub>3</sub>, SYNROC-type perovskite), and clinopyroxene. SEM and EDS analyses confirmed that the 1000 °C ceramics are monolithic composites with uniform distribution of Cs<sup>+</sup> and Sr<sup>2+</sup> throughout the bulk. Strontium was predominantly incorporated into cubic perovskite crystallites, while cesium was retained in the hollandite phase.

Vickers microhardness measurements were feasible only for the neat and Cs-loaded samples sintered at 600 °C. The Cs-loaded material exhibited higher hardness than the neat ivanyukite, whereas the Sr-loaded sample could not be measured reliably because of its high porosity.

The most significant finding concerns the hydrolytic stability of the SPS-fabricated ceramics. After 14 days of immersion in distilled water, the leaching rates of Cs<sup>+</sup> and Sr<sup>2+</sup> stabilized at  $10^{-7}$ – $10^{-8}$  g·cm<sup>-2</sup>·day<sup>-1</sup>, values that fully satisfy the requirements for solidified high-level waste matrices.

Overall, this study demonstrates that spark plasma sintering of Cs- and Sr-loaded synthetic ivanyukite offers a viable route to chemically and mechanically durable, mineral-like ceramics suitable for the long-term and safe immobilization of <sup>137</sup>Cs and <sup>90</sup>Sr radionuclides.

#### CRedit authorship contribution statement

O.O. Shichalin: Writing - Review & Editing, Conceptualisation, Investigation, Writing - Original Draft. G.O. Kalashnikova: Investigation, mass spectrometry data curation, Writing. O.I. Siidra: Investigation, XRD analysis - reviewer, Writing - Review & Editing. E.A. Selivanova: Investigation, XRD analysis. Ya.A. Pakhomovsky: Investigation, SEM analysis. D.V. Gryaznova: Investigation, Sorption experiments. A.I. Nikolaev: Validation, Supervision. V.N. Yakovenchuk: Resources, Conceptualization. Ideas. O.F. Goychuk: Investigation, preparation of polished sections. A.A. Belov: Validation, Investigation, Writing - Original Draft, Visualization. I. V. Smirnov: Project administration, Funding acquisition. E.K. Papynov: Supervision, Project Administration, Funding Acquisition, Conceptualization. Tananaev I.G.: Conceptualization Ideas, Funding acquisition, Data curation.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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