

ORIGINAL RESEARCH ARTICLE

Synthesis and Characterisation of Magnesium-Doped Zirconolite-2M Ceramic

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ABSTRACT

Zirconolite has been considered a promising host matrix for plutonium immobilization because of its enhanced properties. In this work, a range of Zirconolite solid solutions with stoichiometric $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}xO_7$ were carefully explored to investigate magnesium solubility in zirconolite-2M ceramic. Energy-dispersive X-ray spectroscopy scanning electron microscopy (SEM-EDX), and X-ray diffraction (XRD) were used to characterize the materials. Theoretical formulae, Reactive Spark Plasma Sintering (RSPS), and air sintering were used to determine the samples' elemental compositions. Single-phase zirconolite-2M was discovered in $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}xO_7$ together with perovskite and zirconia phases in the air-sintered samples. As opposed to this, the RSPS shows a complete fit into the zirconolite-2M of $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}xO_7$. The elemental composition of the air sintering and RSPS samples is theoretically consistent with the XRD and SEM results, which demonstrated the full incorporation of magnesium on the Ti site of $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}xO_7$. Therefore, the results indicated the high capability of the Zirconolite-2M compound to immobilize divalent cations within the singlephase waste form.

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INTRODUCTION

Nuclear energy is a high-energy source with greater energy density, low cost, and lowest air pollution emission (Aliyu et al., 2025; Aliyu et al., 2024; Li et al., 2020). However, a significant amount of nuclear waste has been produced during the operation and decommissioning of nuclear facilities. Actinides (Pu and U), minor actinides, and fission products (Cs, Sr, I, Xe, and Ru) are among the radioactive elements found in nuclear waste that release alpha, beta, and gamma rays. Very short-lived waste (VSLW), very low-level waste (VLLW), low-level waste (LLW), intermediate-level waste (ILW), and high-level waste (HLW) are the five primary categories into which the International Atomic Energy Agency (IAEA) separates radioactive waste according to the radionuclide's half-life and radiotoxicity (Gera, 1974; Shuaibu et al., 2020; Vejmelkova et al., 2018). Thus, it is necessary to guarantee the long-term security and cost-effective handling of nuclear waste.

To properly immobilize highly radioactive waste, a sophisticated spent fuel management approach is required (Blackburn et al., 2020; Jafar et al., 2021). There is currently a significant amount of separated civil plutonium oxide (PuO₂) and magnesium non-oxidizing (Magnox) sludge discharge from reactors that can be either immobilized into a waste form and then disposed of in a multi-barrier geological repository or reprocessed as

Mixed Oxide (MOX) fuels. Waste loading, fabricability, criticality, radiation resistance, aqueous durability, and proliferation resistance must all be taken into account when using a waste form as an initial containment, which is a safe material intended primarily to prevent radioactive emission (Biswas et al., 2019; Blackburn, Bailey, et al., 2021; Bosbach et al., 2020). Cement, glass, ceramics, and glass-ceramic synthesised materials are examples of potential waste forms. Cement has been used to immobilize low and intermediate radioactive waste. However, its application is limited by the mechanical strength reduction of its matrix brought on by the creation of bubbles from hydrogen gas (Shuaibu et al., 2020; Singh et al., 2021). Around the world, borosilicate glasses are usually utilized for HLW encapsulation. Low solubility and reduced activity loading are its primary disadvantages for use in the future (Blackburn et al., 2020; Jafar et al., 2021).

Ceramic composites of mineral-comparable titanates, also known as SYNROC (synthetic rock), were proposed as an option for HLW immobilization due to their high waste loading and aqueous endurance (Foxhall et al., 2014; Jafar et al., 2016, 2021; Jafar et al., 2015). Among the possible waste forms for ceramics are pyrochlore (A2B2O7), zircon (ZrSiO4), zirconia (ZrO2), Zirconolite (CaZrTi2O7), brannerite (UT2O6), monazite (CePO4),

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perovskite (CaTiO3), and zircon (ZrSiO4) (Drey et al., 2020; Heuser et al., 2019; Huittinen et al., 2017; Kong et al., 2014; Orlova & Ojovan, 2019).

Zirconolite ceramic has been chosen as a host matrix for immobilizing U, Np, and Pu due to its great chemical stability, radiation tolerance, and compositional flexibility (Blackburn, Bailey, et al., 2021; Blackburn, Cole, et al., 2021; Wei et al., 2021). Monoclinic (2M) zirconolite's anion-deficient structure is between the CaZrxTi3-xO7 compositional range (0.8 < x < 1.3). The Ca site is accommodated after incorporating lower valence charges (Mg²⁺, Al³⁺, Ti³⁺, Fe³⁺, and Nb⁵⁺) on the Ti⁴⁺ site to balance the species. This is known as charged compensated substitution. Alternatively, a variety of tri and tetravalent rare earth and actinide elements are accommodated within its solid solution by direct substitution through actinides accommodation (e.g. Ca1-_xPu_xZrTi_{2-2x}Cr_{2x}O₇) (Bettini, 2008; Blackburn, Bailey, et al., 2021; Lv et al., 2019; Ma et al., 2018; Tanti & Kaltsoyannis, 2021; Wei et al., 2021) Charge compensation species of zirconolite-2M with Cerium (Ce) as a Pu surrogate targeting both Ca and Zr sites (Ca₁₋ xCexZrTi2-2xCr2xO7 and CaZr1-xCexTi2O7) have been the subject of extensive research recently (Blackburn, Cole, et al., 2021; Wei et al., 2021). Furthermore, according to (Vance et al., 2002; Ji et al., 2018), magnesium (Mg) may accommodate both the Ca and Ti sites. Numerous studies have examined the phase development and solubility of trivalent inclusion on the Ti site of Zirconolite (Aliyu et al., 2025; Zhou et al., 2022). Nevertheless, little is known about how divalent (Mg2+) substitution affects the Ti site of Zirconolite. Aliyu et al. (2025) computationally investigated Mg doping into Zirconolite, which indicated complete incorporation into the structure. The goal of the research is to examine the Mg²⁺ incorporation on the Ti site in Zirconolite-2M and the effect of processing conditions for Ca(1-x)Zr(1+x)Ti4+(2-x)Mg2+xO7 the zirconolite-2M ceramic.

EXPERIMENTAL METHODS

2.1 Materials synthesis

Zirconolite ceramics that target Ca_(1-x)Zr_(1+x)Ti⁴⁺₍₂₋ $_{x}Mg^{2+}xO_7$ (0.00 $\leq x \leq 0.25$) were made by solid-state synthesis using stoichiometric amounts of CaTiO3 (Sigma Aldrich, 99.9% trace metals basis), ZrO2 (Sigma Aldrich, 99.9% trace metals basis), TiO2 (anatase, Sigma Aldrich, 99.9% trace metal basis), and MgO (Sigma-Aldrich 95%). The resulting mixture was then homogenized by planetary milling at 400 rpm for 20 minutes using isopropanol to dissolve the precursors. The slurries were then dried in an oven by heating for 12 hours at 90°C to evaporate the bulk of the water solvent. The synthesized powder was compressed into green pellets that were $20 \text{ mm} \times$ 10 mm (diameter \times height) and sintered in air for 20 hours at 1350°C. The oxide precursors were also sinterable using reactive spark plasma sintering (RSPS). 3 g of the powder mixture was put into a 20 mm-diameter

UMYU Scientifica, Vol. 4 NO. 2, June 2025, Pp 067 – 075 cylindrical graphite die using an HP-D 1050 SPS system. After consolidation, the die was sintered at 1300°C for 30 minutes while being subjected to 50 MPa of applied pressure.

2.2 Materials characterisation

Powder X-ray diffraction (XRD) with a D2 Phaser, Bruker, Cu-ka radiation, and Ni-filter was used to investigate the samples' phase identification (Sun et al., 2018). A 1-second count rate and a step size of 0.03°20 were used for the sensitive detector analysis, which was conducted in the 5-70°20 range. The XRD pattern was matched using CaZrTi2O7 (zirconolite-2M) with PDF cards #00-048-0072 PLUS20:412083. A quantitative phase analysis was conducted using Rietveld scale factors. In addition to EDX maps and spectra accumulation (SEM-EDX), the Hitachi TM3030 scanning electron microscope (SEM; Inspect F50, FEI) fitted with a Bruker Quantax 70 EDX map was utilized for the microstructure inspection of the samples. Bruker Quantax 70 software was also employed to compute the elemental compositions (Ca, Zr, Ti, and Mg) at each doping level. The legendary Archimedes notion of immersion in water was applied to measure the bulk density of the pellets.

RESULTS AND DISCUSSION

3.1 Phase assemblage

The low densification observed in sintered specimens was avoided by employing RSPS to process the samples of milling precursors $(0.00 \le x \le 0.25)$ instead of consolidation via air sintering. The phase assemblage of powder XRD data from air-sintering and RSPS samples is contrasted in Figures 1 and 2. Using powder XRD data, zirconolite-2M was determined to be the predominant phase for each nominal composition in the range $0.00 \leq$ $x \leq 0.25$. Zirconolite-2M reflections are referenced based on experimental data (PDF cards #00-048-0072 PLUS20:412083). The XRD pattern for Air Sintering at x = 0.00 showed almost single-phase zirconolite-2M (Fig. 1), but the RSPS pattern showed characteristic peaks with zirconolite-2M with perovskite appearance (peak at $2\theta=33^{\circ}$) and low porosity and impurity because of RSPS high densification (Fig. 2).

The XRD pattern at x = 0.05 indexed fully into singlephase zirconolite-2M, which is comparable to x = 0.00for RSPS and Air Sintering, respectively; for Air Sintering, a single-phase monoclinic zirconolite was identified at x =0.10 doping level, which is nearly identical to RSPS with perovskite intensity increase (peak at $20=33^{\circ}$); furthermore, the XRD pattern at x = 0.25 for Air Sintering revealed a high concentration of perovskite and zirconia as second phases, indicating high co-doping of Zr and Mg into Ca_(1-x)Zr_(1+x)Ti⁴⁺_(2-x)Mg²⁺_xO₇, which cannot be indexed into zirconolite-2M. Peaks close to $20=29.5^{\circ}$, $20=31.9^{\circ}$, and $20=33.9^{\circ}$ helped produce the perovskite and zirconia phases.



Fig. 1. Powder X-ray diffraction of $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}xO_7$ (0.00 $\leq x \leq 0.25$) compositions prepared by Air Sintering at $T = 1350^{\circ}$ C, for t = 20 h in total.



Fig. 2. Powder X-ray diffraction of $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}xO_7$ (0. 00 $\leq x \leq 0.25$) compositions prepared by RSPS at $T = 1300^{\circ}C$, for t = 30 minutes in total.

Additionally, RSPS has a similar tendency with two peaks at $2\theta=29.9^{\circ}$ and $2\theta=31.5^{\circ}$ that do not correlate with single-phase zirconolite-2M at the same doping level x = 0.25. As previously noted elsewhere, there was no indication of polymorphic transitions to other phases, and in both doping level scenarios, the XRD patterns were

fully indexed into the zirconolite-2M polytypes (space group C2/c) (Aliyu, 2019; Blackburn et al., 2020). Under $0.00 \le x \le 0.20$ doping level in air sintering, the Ca_(1-x)Zr_(1+x)Ti⁴⁺_(2-x)Mg²⁺_xO₇ powder XRD pattern also indexed into single phase zirconolite-2M. For $x \ge 0.25$, Ca_(1-x)Zr_(1+x)Ti⁴⁺_(2-x)Mg²⁺_xO₇ cannot index into single-

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phase zirconolite-2M. RSPS samples of $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ with $0.00 \le x \le 0.25$ are consistent with zirconolite-2M in a single phase. However, if x > 0.25, the $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ samples may not be indexed into zirconolite-2M.

3.2 Microstructural analysis

For each doping level, the polished pellet surfaces were analyzed using SEM-EDX. A micrograph representation of samples created with both RSPS and air sintering is displayed in Figures 3 and 4. The back-scattered SEM image at x = 0.00 for air sintering shows the presence of perovskite (CaTiO₃) and zirconia (ZrO₂), which are usually present in the synthesis of zirconolite ceramics. Minor porosity and impurity (carbon) were detected as a result of low densification. A similar SEM image with higher densification was discovered for RSPS. The presence of single-phase zirconolite-2M with minimal oxygen sensitivity is confirmed by air sintering at x =0.05, which also displays homogenous matrix formation in addition to the SEM image from RSPS. In agreement with SEM from RSPS with dense compositions, air sintering demonstrated single-phase zirconolite-2M with little porosity and perovskite and zirconia phases at a doping level of x = 0.10. A homogenous single-phase zirconolite-2M with porosity and trace quantities of perovskite at x = 0.15 was observed by SEM for air sintering. However, denser single-phase zirconite-2M was discovered for RSPS. The back-scattered SEM revealed the presence of perovskite, zirconia, and single-phase zirconolite-2M in addition to a high appearance of magnesium at x = 0.20 in air sintering.

UMYU Scientifica, Vol. 4 NO. 2, June 2025, Pp 067 – 075 Furthermore, the uniform enrichment of magnesium in the single-phase zirconolite-2M was confirmed by the EDX test, showing Mg corporation on the Ti site. Backscattered SEM at x = 0.25 demonstrates the inhomogeneous composition of single-phase zirconolite-2M with enhanced perovskite appearances in air sintering. Furthermore, the SEM in RSPS revealed perovskite with a high Mg content and partly single-phase zirconolite-2M.

Lowering the RSPS condition results in perovskite appearances that stabilize during RSPS. Accordingly, the partial reduction of Ti4+ to Ti3+ is the cause of the zirconolite-2M variation in the perovskite (Clark et al., 2017). Since the sample's density and mass (molarity) are closely related, it can be inferred that the density of the RSPS and Air Sintering samples increases with the doping level (Fig. 5). However, RSPS created a denser matrix than Air Sintering, which leads to the development of high porosity in Air Sintering. Furthermore, the backscattered SEM images of the samples Ca_(1-x)Zr_(1+x)Ti⁴⁺_(2-x)Mg²⁺_xO₇ revealed homogenous single-phase zirconolite-2M with minor traces of perovskite and zirconia at $0.00 \le x \le$ 0.20 in Air Sintering, which serves as a solubility limit. However, SEM revealed significant porosity and impurity in $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}{}_{(2-x)}Mg^{2+}{}_{x}O_{7}$ with $x \ge 0.25$. The SEM results for RSPS show a homogenous single-phase zirconolite-2M of $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ with minimal perovskite because of its reducing condition within $0.00 \le x \le 0.25$. But when x > 0.25, zirconolite-2M might shift phases.



Fig. 3. Representative backscattered SEM Image for $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}xO_7$ (0.00 $\leq x \leq$ 0.25) compositions prepared by Air Sintering at T = 1350°C, for t = 20 h in total.



Fig. 4. Representative backscattered SEM Image for $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ (0.00 $\leq x \leq$ 0.25) compositions prepared by RSPS at $T = 1300^{\circ}C$, for t = 30 minutes in total.



Fig. 5. Samples Density Measurement for $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ (0. 00 $\leq x \leq 0.25$) prepared by both Air Sintering and RSPS.

3.3 Sample Composition Computation

Fig. 6–7 and 8 display the compositional computation results of $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ generated by Air Sintering and RSPS, respectively, and the theoretical formula with $0.00 \le x \le 0.25$ (Oxygen is omitted due to

inadequate sensitivity). The results demonstrated that magnesium was soluble on the zirconolite-2M's Ti site. The sample's mean composition converges approximately to 100%, or the entire theoretical molarity of 4 mol (%), for all doping levels (Fig. 8). Theoretically, the molarity ratio of titanium and magnesium should equal 50% (the

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molarity of each element adds up to 25%). Air Sintering demonstrated higher molarity of Mg at both lower and higher doping levels because it densifies less than RSPS. Both the theoretical calculations and the compositional

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computations from RSPS and Air Sintering agree. Mg was encapsulated and soluble on the Ti site of $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_7$ zirconolite-2M, according to the results of the Powder XRD and SEM.



Fig. 6. Air-Sintered Samples Composition Computation for $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ (0. 00 $\leq x \leq$ 0. 25).



Fig. 7. SPS Samples composition computation for $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ (0. 00 $\leq x \leq 0.25$).



Fig. 8. Theoretical Samples composition computation for $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ (0.00 $\leq x \leq 0.25$).

CONCLUSIONS

The incorporation of magnesium inside CaZrTi₂O₇ zirconolite was investigated for the Ca(1-x)Zr(1+x)Ti4+(2-_{x)}Mg²⁺_xO₇ system. Reactive Spark Plasma Sintering (RSPS) at T=1300°C for t=30 minutes and Air Sintering at T=1350°C for t=20 hours were the two synthesis techniques that were effectively employed. Examining the Mg incorporation on the Ti site of the zirconolite-2M structure. Powder XRD and SEM revealed that the Ca(1-_{x)}Zr_(1+x)Ti⁴⁺_(2-x)Mg²⁺_xO₇ solid solution had single-phase zirconolite-2M within $0.00 \le x \le 0.20$ for air-sintering samples. However, additional crystalline phases like perovskite and zirconia were also present. When $x \ge$ 0.25, there was no single-phase zirconolite-2M in the Ca(1-_{x)}Zr_(1+x)Ti⁴⁺_(2-x)Mg²⁺_xO₇. The XRD and SEM results for the RSPS samples showed that single-phase zirconolite-2M was present in the Ca_(1-x)Zr_(1+x)Ti⁴⁺_(2-x)Mg²⁺_xO₇ solid solution within $0.00 \le x \le 0.25$. At x > 0.25, a solid $Ca_{(1-x)}Zr_{(1+x)}Ti^{4+}_{(2-x)}Mg^{2+}_{x}O_{7}$ solution might not integrate into single-phase zirconolite-2M. The presence of magnesium on the Ti site of Ca_(1-x)Zr_(1+x)Ti⁴⁺_(2-x)Mg²⁺_xO₇ was determined by the sample composition calculations for both Air Sintering and RSPS. The theoretical sample composition computation complements the XRD and SEM data.

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DATA AVAILABILITY

The data used to support this study's findings are available from the corresponding author upon request.

DECLARATIONS

Conflict of Interest The authors declare that they have no conflict of interest.

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