Article

Coal Fly Ash as Raw Material for Immobilization of Sr-Contaminated Soil by Microwave Heating: Mechanism and Performance

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Abstract: In this work, coal fly ash, hereinafter CFA is proposed to work as raw material for immobilization of Sr-contaminated soil by microwave sintering in the path towards resource utilization of solid waste. The immobilization mechanism and performance was systemically investigated through phase evolution, microstructure, elemental distribution, and physical properties. The results shown that the Sr could be incorporated into feldspar strontian ($\text{SrAl}_2\text{Si}_2\text{O}_8$) at 1300 °C for 30 min. Moreover, the maximum solid solubility of SrSO_4 was more than 30 wt.%. The Sr was homogeneously distributed in the sintered matrices without substantial enrichment. The sintered matrix exhibited high density (2.53 g/cm³). Thus, microwave heating coupled with CFA could provide a new method for immobilization of Sr-contaminated soil in case of the spent nuclear reprocessing cycle in nuclear power plants or a nuclear accident emergency.

Keywords: Sr-contaminated soil; microwave heating; feldspar strontian; microstructure

Citation: Xie, Y.; Zhu, A.; Chen, M.; Dai, B.; Wang, B.; Liu, Y. Coal Fly Ash as Raw Material for Immobilization of Sr-Contaminated Soil by Microwave Heating: Mechanism and Performance. *Crystals* **2022**, *12*, 55. <https://doi.org/10.3390/cryst12010055>

Academic Editors: Dawei Wang, Andrey Prokofiev and Zhao Zhang

Received: 12 November 2021

Accepted: 27 December 2021

Published: 31 December 2021

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1. Introduction

Strontium (⁹⁰Sr) with a long half-life of approximately 30 years is a typical fission product with high radioactivity and radiotoxicity generated from the spent nuclear reprocessing cycle in nuclear power plants [1,2]. Importantly, once the nuclide migrates into water or air through the soil, it will pose a great threat to human health and the surrounding environment. In recent years, especially after the nuclear accident in Fukushima, the safety of nuclear energy has been a greater public consideration [3,4]. In the Fukushima nuclear accident, large amounts of radioactive ⁹⁰Sr were released and deposited in the soil, resulting in chronic soil pollution [4,5]. Therefore, it is urgent to safely dispose of Sr-contaminated soil for the sustainable development of the nuclear industry. In the last decades, borosilicate glass is the only waste form applied at industrial scale [6,7]. Due to superior stable nature, extensive researches suggest that ceramic waste forms could improve the long-term aqueous performance in comparison with the vitrified matrices [8,9]. Meanwhile, CFA, the major solid waste from the coal-fired thermal power plants, also has caused a lot of environmental problems, such as decreasing the agricultural acreage and polluting the groundwater and atmosphere [10]. It is reported that approximately 1000 million tons of CFA are produced annually around the world. Unfortunately, its utilization ratio remains below 50% [11]. Abundance in silica (SiO_2) and alumina (Al_2O_3), in the path towards resource utilization of solid waste, CFA can be selected as a potential source of raw materials to sinter glass and ceramic [12,13]. As an emerging heating method, microwave sintering has the advantages of volumetric energy absorption, rapid heating rate, and low energy consumption when compared to conventional sintering methods [14]. Extensive previous

researches have been carried out to explore immobilization of radionuclide-contaminated soil via microwave heating, and the curing mechanism and feasibility are proven [15–18]. Furthermore, microwave sintering substantially improved the microstructural uniformity and macroscopic properties of the final cured product [19].

Inspired by the above, herein, microwave heating coupled with CFA was employed to dispose of Sr-contaminated soil. Phase evolution and microstructure with the elevation of sintering temperature were systematically studied. The existing form, ultimate solid solubility, and immobilization mechanism of strontium in the sintered matrix was investigated through X-ray diffraction (XRD, X' Per Pro, Almelo, Netherlands), Fourier transform infrared spectroscopy (FT-IR, IRAffinity-1S, Kyoto, Japan), scanning electron microscope (SEM, TESCAN MIRA4, Brno, Czech Republic), and transmission electron microscope (TEM, Talos F200X G2, Brno, Czech Republic).

2. Materials and Methods

2.1. Materials

The CFA and SrSO_4 powders (AR grade) were utilized as the starting materials. The CFA was sourced from the thermal power plant in Hunan, China. After being oven-dried at 105°C for 24 h to remove absorbed water, the CFA was passed through a 200-mesh screen. The phase composition of the CFA was characterized by X-ray diffraction (XRD), as shown in Figure 1. Moreover, the chemical composition of the preprocessed CFA characterized by X-ray fluorescence (XRF) was present in Table 1. It can be observed that the CFA has high contents of SiO_2 (57.06 wt.%) and Al_2O_3 (29.13 wt.%) with only trace amounts of other oxides and carbon.

Figure 1. XRD pattern of the CFA.

Table 1. Chemical composition of coal fly ash determined by XRF (wt.%).

	Al_2O_3	SiO_2	CaO	K_2O	Fe_2O_3	Na_2O	MgO	TiO_2	SO_3	P_2O_5	ZnO
CFA	29.13	57.06	3.05	2.88	2.36	1.15	1.11	1.11	1.02	0.522	0.465

2.2. Experiment Procedures

The refined CFA was mixed with SrSO_4 in equal proportion (0 wt.%, 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.%, 25 wt.%, and 30 wt.%) to obtain Sr-contaminated soil, denoted as S0, S5, S10, S15, S20, S25, and S30, respectively. Subsequently, the mixtures were wet ground in a mortar with alcohol as the medium. After being ground uniformly, the homogenized mixtures were dried again and then pressed into pellets of 10 mm in diameter and 3 mm in thickness by the cold isostatic pressing under 200 MPa. Finally, the samples held by

alumina crucibles were sintered at 1100 °C, 1200 °C, and 1300 °C for 30 min in a microwave muffle furnace under air atmosphere. The samples were firstly heated at 800 °C for 60 min to burn out the carbon and remove volatile components. As the temperature below 1000 °C, the heating rate was set to 20 °C/min and then reduced to 10 °C/min as the temperature beyond 1000 °C. After being maintained at target temperatures for 30 min, the sintered matrices were naturally cooled down to room temperature. The whole experimental process was shown in Figure 2.

Figure 2. Schematic of the experimental process.

2.3. Characterization

The compositions of the sintered compacts were determined by X-ray diffraction (XRD, X' Per Pro, Almelo, Netherlands) with Cu-K α radiation operating at 45 kV and 40 mA in the 10°–80°(2 θ) range. The detailed structure of the sintered forms was further confirmed by a Fourier transform infrared spectroscopy (FT-IR, PE Spectrum One, Waltham, MA, USA). The micromorphology and elemental distribution of the sintered sample was observed by a scanning electron microscope (SEM, TESCAN MIRA4, Brno, Czech Republic).

The bulk density of the sample was measured by a densitometer (MDJ-200S, Dongguan, China) with the Archimedes method at 24 °C, and deionized water was used as the immersion medium. The open porosity was calculated using the following Formula (1) [20]:

$$P_o = \frac{M_3 - M_1}{M_3 - M_2} \quad (1)$$

where M_1 is the dry mass of the sample, M_2 is the mass of sample who is suspended in deionized water, and M_3 is the mass of the sample who is fully immersed in deionized water and saturated water absorption.

3. Results and Discussion

3.1. Phase Evolution Analysis

Figure 3 presents the XRD patterns of as-sintered samples doped with 0–30 wt.% of SrSO₄ in the temperature range of 1100–1300 °C for 30 min. Figure 3a displays the XRD patterns of the sintered forms loading 0–30 wt.% SrSO₄ fabricated at 1100 °C. Evidently, the SrSO₄-related phase can be found in all the sintered matrices, indicating that the SrSO₄ could not be immobilized in the sintered samples at 1100 °C. As demonstrated in Figure 3b, the diffraction peaks of feldspar strontian (SrAl₂Si₂O₈) are detected with the elevation of sintering temperature. Moreover, the main crystalline phase transforms from mullite and sillimanite to feldspar strontian with increasing SrSO₄, indicating that the SrSO₄ could be incorporated in the sintered matrix. Analogous results are shown in Figure 3c. The difference is that, as the firing temperature increases from 1200 °C to 1300 °C, the number of diffraction peaks of feldspar strontian increase. The diffraction peaks of quartz and sillimanite both decrease, which may be ascribed to the involvement of SiO₂ and Al₂O₃ in the synthesis of SrAl₂Si₂O₈. What is interesting in Figure 3b is that a few diffraction peaks of SrSO₄ are detected in the sintered samples at 1200 °C as the doping amount of SrSO₄ increases to 20 wt.%, which suggest that the maximum solid solubility of SrSO₄ at 1200 °C is between 15 wt.% and 20 wt.%. As the sintering temperature elevates to 1300 °C,

the SrSO_4 -related phase has not been found in all the sintered compacts, implying that the maximum solid solubility of SrSO_4 at 1300°C is over 30 wt.%.

Figure 3. XRD patterns of the sintered forms doped with various amounts of SrSO_4 prepared at different temperatures: (a) S0-S30 at 1100°C , (b) S0-S30 at 1200°C , and (c) S0-S30 at 1300°C .

3.2. Microstructural Analysis

The FT-IR technique is utilized to further reveal the immobilizing modes of Sr element in sintered matrix. Figure 4a demonstrates the FT-IR spectra of the samples doped with various contents of SrSO_4 prepared at 1300°C . Obviously, the main absorption peaks of the sintered samples are concentrated in the range of 400 cm^{-1} to 800 cm^{-1} . The absorption peaks at $\sim 470\text{ cm}^{-1}$ and $\sim 456\text{ cm}^{-1}$ are attributed to the stretching vibrations of Si-O bonds [21,22]. The absorption peaks at $\sim 725\text{ cm}^{-1}$ and $\sim 673\text{ cm}^{-1}$ are ascribed to the stretching vibrations of Si-Al (Si) and Al (Si)-O bonds, respectively [23]. The remaining part of the spectrum contains the modes of the bending vibrations of O-Si (Al)-O bonds (around 610 cm^{-1} and 576 cm^{-1}) and O-Si-O bonds (near 540 cm^{-1} and 420 cm^{-1}), and the stretching vibrations of Sr-O bonds (near 540 cm^{-1} and 420 cm^{-1}) [3]. Combined with the XRD results, the existence of Sr-O bonds indicates that feldspar strontian is present in the sintered samples. Analogous results are shown in Figure 4b. The difference is that, the S-O bonds at $\sim 640\text{ cm}^{-1}$ are detected at 1100°C and 1200°C , indicating the existence of SrSO_4 [24]. As the firing temperature increases from 1200°C to 1300°C , the S-O bonds are disappeared. It could be deduced 30 wt.% SrSO_4 could not be fully incorporated in the sintered matrices at 1100°C and 1200°C , whereas the maximum solid solubility of SrSO_4 at 1300°C is over 30 wt.%.

Figure 4. FT-IR spectra of the typical samples: (a) S0~S30 sintered at 1300 °C, and (b) S30 sintered at 1100–1300 °C.

3.3. Micromorphology Analysis

The SEM and corresponding elemental mapping images of the typical samples are presented in Figures 5 and 6, respectively. As illustrated in Figure 5, many grains with dimensions of 5–10 μm are observed. The ceramic is inferred to be feldspar strontian ($\text{SrAl}_2\text{Si}_2\text{O}_8$) according to the above XRD results. The corresponding elemental mapping of the main elements (Sr, Al, Si, O, and S) is demonstrated in Figure 6. It is evident from Figure 6a,b,d,e that the S element is distributed with obvious enrichment. It implies that SrSO_4 could not be fully fixed under these conditions, which is consistent with the phase analysis results in Figure 3. On the contrary, all the elements are evenly distributed without obvious enrichment in Figure 6c,f. Combined with the above XRD analysis, the elemental mapping results are valuable to certify that at least 30 wt.% SrSO_4 could be successfully incorporated in the sintered samples obtained by microwave preparation at 1300 °C for 30 min.

Figure 5. Microscope analyzed by SEM: (a) 1100 °C, 20 wt.% SrSO_4 , (b) 1200 °C, 20 wt.% SrSO_4 , (c) 1300 °C, 20 wt.% SrSO_4 , (d) 1100 °C, 30 wt.% SrSO_4 , (e) 1200 °C, 30 wt.% SrSO_4 , and (f) 1300 °C, 30 wt.% SrSO_4 .

Figure 6. Elemental distribution of the sintered samples: (a) 1100 °C, 20 wt.% SrSO_4 , (b) 1200 °C, 20 wt.% SrSO_4 , (c) 1300 °C, 20 wt.% SrSO_4 , (d) 1100 °C, 30 wt.% SrSO_4 , (e) 1200 °C, 30 wt.% SrSO_4 , and (f) 1300 °C, 30 wt.% SrSO_4 .

To obtain immobilization mechanism of strontium in the sintered bodies, the S30 sample fabricated at 1300 °C is further studied by TEM. As depicted in Figure 7a, the sintered sample is composed of a well-developed crystal structure with a regular arrangement. The corresponding selected area electron diffraction (SAED) is presented in Figure 7b. According to the atom arrangement, the sample is identified as a single crystal under the high-resolution mode, which is in agreement with XRD results in Figure 3c. The SAED pattern (Figure 7b) indicates that the neighboring diffraction spots can be readily indexed to the (1 0 0) and (0 10 0) crystal planes of the $\text{SrAl}_2\text{Si}_2\text{O}_8$ phase respectively according to the XRD standard card (PDF# 70-1826, $a = 8.388 \text{ \AA}$, $b = 12.974 \text{ \AA}$, $c = 14.264 \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 115.2^\circ$, $\gamma = 90^\circ$, space group $I2/c$). Moreover, the angles measured between the (1 10 0) crystal plane and the (1 0 0) and (0 10 0) crystal planes are 80.3° and 9.7° respectively. The crystalline band axis of the sample can be identified as (0 0 1). Combined with XRD results and FT-IR results, it can be inferred that Al^{3+} could replace Si^{4+} in an ordered or non-ordered way as the ionic radius of Al^{3+} is similar with Si^{4+} in the sintering process. Sr^{2+} cations with positive charge could compensate the charge difference caused by the substitution of Al^{3+} for Si^{4+} as well as react with Al^{3+} and Si^{4+} to form $\text{SrAl}_2\text{Si}_2\text{O}_8$. It is worth to mention that the sintering temperature is conducive to improve the solid solution of SrSO_4 . The crystallographic information of $\text{SrAl}_2\text{Si}_2\text{O}_8$ is demonstrated in Figure 7c.

Figure 7. TEM image (a) and SAED pattern (b) of S30 sintered at 1300 °C, and (c) crystallographic information of SrAl₂Si₂O₈.

3.4. Physical Property Analysis

Figure 8 shows the density and open porosity of typical samples sintered at 1100 °C, 1200 °C, and 1300 °C. It can be found that the density of all sintered samples increases with an elevated doping amount of SrSO₄. When the doping amount is 30 wt.% and the sintering temperature reaches 1200 °C, the density reaches the maximum value (2.53 g/cm³), which satisfies the density requirement of radioactive waste packages (>2.5 g/cm³) [25]. However, when the doping amount of SrSO₄ is below 25 wt.%, the density of all sintered samples decreases with the increase of sintering temperature. It might be ascribed to the increasing involvement of Sr²⁺ in the reaction with Al³⁺ and Si⁴⁺ to form the SrAl₂Si₂O₈ as the temperature increases which leads to more vacancies. It is in agreement with above XRD and FT-IR results. The open porosity of all sample is maintained at a relative high level, which is attributed to the presence of carbon and volatiles in the CFA. With the increase of sintering temperature, the open porosity decreases, implying that the elevation of sintering temperature is conducive to densification.

Figure 8. Density and porosity of the typical samples sintered at various temperature.

4. Conclusions

In the present work, a series of Sr-contaminated soil were immobilized by using CFA as raw material via microwave sintering. The phase evolution, microstructural, and micromorphology analysis results show that the more than 30 wt.% SrSO₄ can be accommodated into feldspar strontian (SrAl₂Si₂O₈) at 1300 °C, and the elements are homogeneously distributed in the sintered matrices without substantial enrichment. The density and porosity tests show that the sintered sample fabricated at 1300 °C has good physical stability. Therefore, the CFA could be an ideal alternative raw material for the disposal of Sr-contaminated soil via microwave sintering.

Author Contributions: Y.X.: Methodology, Conceptualization, Writing-original draft, Funding acquisition. A.Z.: Validation, Data curation. M.C.: Software, Conceptualization. B.D.: Investigation, Resources. B.W.: Characterization, Formal analysis. Y.L.: Project administration, Funding acquisition. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the research and Development Program in Key Areas of Hunan Province (No. 2019SK2011), Hunan Provincial Innovation Foundation for Postgraduate (No. CX20210915), and the Natural Science Foundation of Hunan Province (No. 2019JJ50497).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are available upon reasonable request to corresponding authors at xieyupeng@usc.edu.cn (Y.X.)

Acknowledgments: We acknowledge financial support from China Hunan provincial science and technology department and Hunan provincial department of education. We are thankful to the reviewers for their valuable comments and suggestions.

Conflicts of Interest: All authors declare that they have no financial interest and personal relationships with other people or organizations that can influence the work reported in this paper. University of South China and the Hunan NO.4 Engineering Co., LTD have only scientific cooperation and no commercial cooperation.

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