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Investigating Cold Sintering of Natural Clay Minerals for Applications in Nuclear Waste Management

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1. Introduction

The safe management of radioactive waste generated during nuclear decommissioning remains one of the most critical environmental challenges. Among various waste streams, volatile radionuclides such as Cs are considered particularly hazardous due to their potential mobility and environmental dispersion. Numerous studies have focused on the adsorption and immobilization of radionuclides, and materials containing silicate or aluminosilicate phases, which are the key constituents of the natural clay minerals, have been widely recognized as promising candidates for stable waste forms [1,2]. This study proposes the use of hydrous aluminosilicates (phyllosilicates) as an effective immobilization matrix for nuclear waste.

We investigate the cold sintering behavior of four different natural clay minerals without the use of external liquid additives or transient liquid phases. This low-temperature, pressure-assisted consolidation approach enables the formation of dense and stable clay-based ceramics suitable for long-term containment in nuclear decommissioning applications.

2. Material and Method

Cold sintering of natural clay minerals, including kaolin, kaolinite, bentonite, and montmorillonite, was performed at 250 °C under a uniaxial pressure of 400 MPa, with a holding time of 40 min. Densification evaluation, water immersion test, microstructural investigation, phase analysis, and thermogravimetric analysis (TGA) were performed to evaluate the sintering behaviour and stability of different clays. The shortlisted candidate was further explored in depth to be used in the future waste management phase of this research.

3. Results and Discussion

3.1 Densification and Microstructural Analysis

All four sintered clay samples showed significant densification, with relative densities of ~96% for kaolin and kaolinite, 97% for bentonite and up to 99% for montmorillonite. However, their behavior in aqueous conditions differs due to their unique crystal structures. Only kaolinite retained its structural integrity during the water immersion test, while the other clays dissolved rapidly. This is because kaolinite has a stable structure with strong interlayer bonding that restricts water penetration and prevents degradation. In contrast, kaolin contains impurities and is therefore less stable, while bentonite and montmorillonite are swelling clays with expandable layered structures that readily absorb water, leading to expansion, and eventually disintegrate despite their higher densification.

The fractured surface (SEM images) of the sintered clay samples, as shown in Fig. 1, demonstrates significant microstructural densification compared to the typical morphology of raw clay. Kaolin and kaolinite exhibit small nanosheet-like structures with fused particles and visible neck formation, indicating effective pressure-assisted bonding during sintering. Bentonite forms relatively more compact particle clusters, while Montmorillonite displays compressed lamellar nanosheets with closer stacking compared to its typical loose structure. Overall, cold sintering enhances interfacial contact and significantly improves densification in natural clay systems.

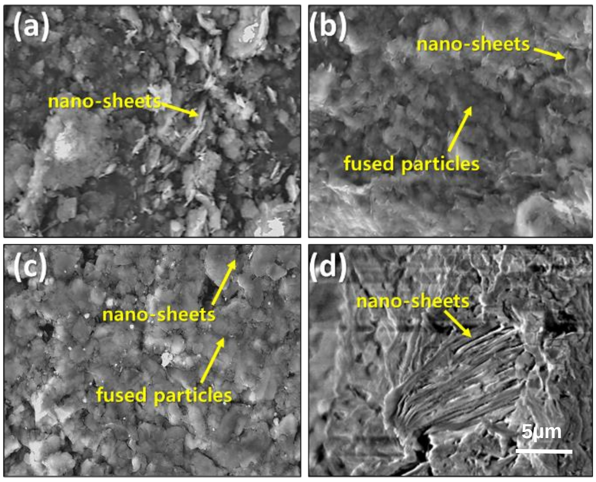

Fig.1 SEM micrographs of fractured cold-sintered clay samples showing distinct morphology: (a) kaolin (b) kaolinite (c) bentonite, and (d) Montmorillonite.

3.2 TGA Analysis:

TGA was conducted to evaluate the mass loss behavior of the clay samples. Measurements were performed using a thermal analyzer over a temperature range of 20–500 °C under an argon atmosphere. The TGA curves exhibit the characteristic multi-stage weight loss of hydrous aluminosilicate clays. An initial minor mass loss around 100 °C is attributed to the removal of physically adsorbed moisture. Up to the cold sintering temperature of 250 °C, the weight loss remains minimal: approximately 0.11% for kaolin, 0.14% for kaolinite, 0.82% for bentonite, and 2.92–3% for montmorillonite, indicating the thermal stability of the clay frameworks under the processing conditions. The slightly higher losses observed in bentonite and montmorillonite are consistent with their greater interlayer water content [2]. Significant mass loss occurs only at higher temperatures (>400–500 °C), confirming that cold sintering at 250 °C does not induce structural decomposition.

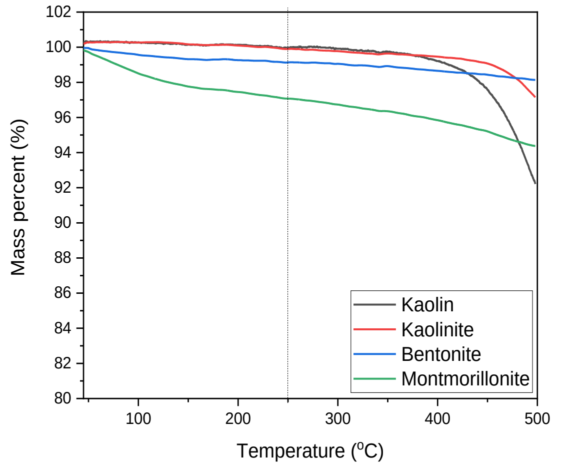

Fig.2 TGA curves of raw clays showing weight loss as a function of temperature.

3.4 XRD Analysis:

XRD analysis (Fig. 3) confirms that the clays remain predominantly crystalline after cold sintering, with no evidence of amorphization, lattice distortion, or major phase transformation. The preservation of sharp diffraction peaks demonstrates that densification occurs primarily through microstructural compaction rather than crystallographic modification. Maintaining the crystalline structure while enhancing particle packing contributes to increased density and overall consolidation of the clay.

4. Conclusion

This study demonstrates that the cold sintering method successfully consolidates natural clay minerals while preserving their crystalline structure. The process enhances the morphology through strong inter-particle bonding of nano-sheets, indicating improved densification without causing phase transformation or thermal degradation. Overall, cold sintering is a reliable low-temperature method for stabilising clay minerals without using any additives. Among all clays, kaolinite shows strong potential as a stable matrix for long-term radionuclide immobilization while maintaining structural functionality. Future studies will include detailed leaching tests and radionuclide retention analysis to establish the effectiveness of these materials further.

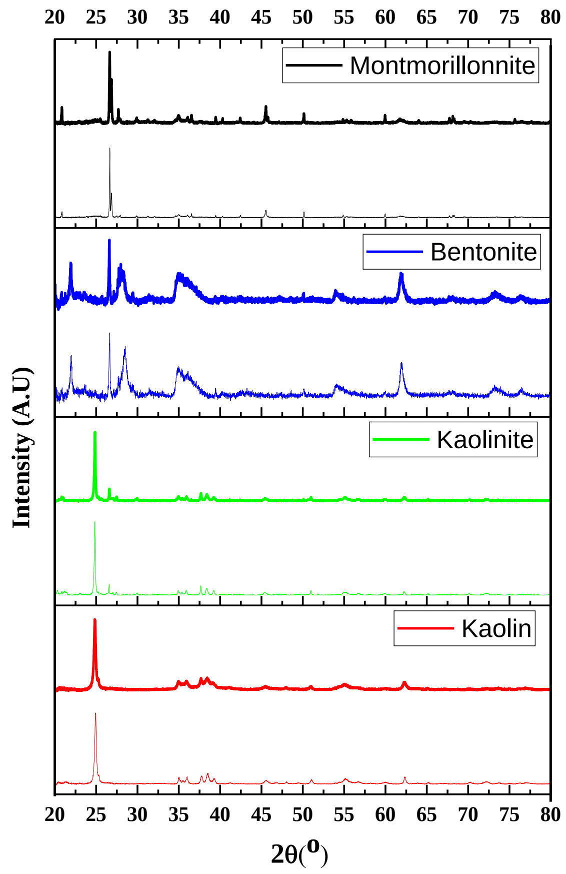

Fig.3 XRD patterns of raw (thin line) and cold-sintered clays (thick line) were recorded over the 20–80° (2θ) range.

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