

## Cold immobilization of as-spent 3D printed ceramic filter

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### 1. Introduction

The developments in the field of ceramic 3D printing and the use of this technology for the manufacturing of ceramic filters have suggested the use of these filters in the nuclear industry for adsorbents applications. This technology aims to use both printability and adsorption properties of the ceramic materials such as zeolites and their composites. The proposed uses of these filters are in the Chemical and Volume Control System (CVCS) as well as the purification of process water.

Cs, Ni and Co are among the elements which should be adsorbed and removed. Lee et al. reported the adsorption of these ions using the developed 3D filters and the precursor ceramic powders in the dried form [1].

The as-spent filters are porous and structurally not suitable to be disposed of as it is. Therefore, volume reduction technique such as compaction and consolidation is required to develop for the final immobilization. We investigated a non-volatile technique namely cold sintering with an aim to reduce the volume and obtain a durable waste matrix for the final immobilization of as spent ceramic filters.

### 2. Methods/Experimental

#### 2.1. Materials

Sigma Aldrich supplied zeolite 13X powder. Amorphous aluminosilicate gel (AG) was synthesized using an organic free synthesis method. Concentrated sulfuric acid and ammonia solution were used for controlling pH during the synthesis process.

For making the zeolite-based inks, we followed Lee's work, specifically adding bentonite, methylcellulose, poly vinyl alcohol as a solid form and deionized water as a liquid form [1-2]. Additionally, the AG was added for the possible improvements in structural strength and adsorption capacity.

#### 2.2. Fabricating filter

For making the filter for adsorption testing, we followed these three steps: 1) layered slurry deposition by dispenser extrusion 2) Drying and heat treatment 3) Weighing for making dosage compared to powder forms.

#### 2.3. Cold sintering

We investigated the cold sintering of dried amorphous aluminosilicate only with adsorbed

simulated radioactive ions. We followed the already published procedure for cold sintering [3]. The sintering parameters were optimized as 200 °C, 500 MPa and 10 min holding time.

#### 2.4. Characterization

Standard characterization methods and tools were used for the analysis of the synthesized materials and the samples prepared at each step of the experiment. We performed the X-ray diffraction (XRD) and Scanning electron microscopy (SEM) of the synthesized and sintered samples.

### 3. Results and Discussion

The synthesized aluminosilicate gel (AG) was X-ray amorphous. The use of AG for the printing of zeolite based 3D ceramic filters showed good results in improving its physical properties as well as adsorption capacity. The optimized composition of prepared ink was 1 (zeolite) : 2.5 (AG). The composite filter showed 4% higher removal efficiency than the pure zeolite filters. In case of zeolites, the cold sintering did not produce significant densification that may due to the absence of amorphous phase. The cold sintering of amorphous aluminosilicate produced a highly dense and durable matrix with apparent density and microhardness values of  $2.05 \times 10^3 \text{ kg/m}^3$  and 3 GPa, respectively. The cold sintered 10 wt.% amorphous aluminosilicate and zeolite composite sample has shown higher volumetric density as compared to the pure zeolite under same conditions. In order to immobilize the as-spent filter, the contents of amorphous aluminosilicate can work as a binding agent and can facilitate the higher densification. Fig. 1 shows the filters printed with the zeolite and amorphous aluminosilicate gel.

These results are being used for the consolidation of the whole filter and the detailed results are under investigation.



Fig. 1. A 3D printed composite filter

### 4. Conclusions

The study demonstrated that filter precursors with adsorbed simulated radioactive ions can successfully be cold sintered with good physical durability. Based on these results, the effective compaction and immobilization of as spent ceramic filters can be envisaged. The final step of whole filter immobilization is under investigation.

#### **Acknowledgments**

This study is supported by KAI-NEET, KAIST and the National Research Foundation, MSIT, Korea (NRF-2018M2B2A9065746\_

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