

# Co<sup>2+</sup> adsorption performance of Zeolite powder and 3D printed filter

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## 1. Introduction

The technology of 3D printing for ceramic materials has been boosted nowadays because of its feasibility and cost-effectiveness, which the manufacturing cost does not depend on the number of products [1]. ~~Lee et al.~~ Our previous works [2-3] investigated 3D printed zeolite filter to enhance the mechanical properties by post-treatments because Hawaldar mentioned the limitation of printed filter [4].

Cobalt is one of the main elements of Chalk River Unidentified Deposition (CRUD) in the primary water coolant system of nuclear power plants. In addition activated Co<sup>2+</sup> emits hazardous radiation to cause employees' exposure at work [5]. Therefore, ~~Co<sup>2+</sup>~~ adsorption performance of Zeolite powder and its 3D printed filter was studied to investigate the adsorption mechanisms and the effects of material and processing parameters.

## 2. Methods/Experimental

### 2.1. Materials

Zeolite 13X powder and Cobalt nitrate were supplied by Sigma Aldrich. 0.1 M of nitric acid and 0.05 M of ammonia solution were used for controlling pH of test solutions. 125 mL of polypropylene bottle, 15 mL of a syringe, and 0.45 μm of PTFE filter were used. For making the zeolite-based printable inks, we followed ~~Lee's work~~ our previous work, specifically adding bentonite, methylcellulose, poly vinyl alcohol as a solid form and deionized water as a liquid form [2-3].

### 2.2. 3D Printing of filter

For making the filter for adsorption testing, we followed these three steps: 1) Extrusion printing 2) Drying and heat treatment 3) Weighing for making dosage compared to powder forms.

### 2.3. Adsorption test with Co<sup>2+</sup> ion

We had investigated batch adsorption test for finding the test condition for comparing adsorption capacity between a powder and a filter. Batch adsorption was obtained at 100 mL of the 50 ppm, pH 7 ionic solution.

### 2.4. Characterization

The concentrations of ions in the solution were measured by Inductively Coupled Plasma Optical Emission Spectroscopy (Agilent ICP-OES 720). The Scanning Electron Microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS, TEAM<sup>TM</sup>) were used to investigate sorption sites on the adsorbents after adsorption test.

## 3. Results and Discussion

### 3.1. Adsorption Test with the powder

The adsorption trend of zeolite 13X for cobalt ions was characterized by the controlling the factors of contact time, dosage, and pH. It reached to a saturation point after 10 min and 0.12 g/ 100 mL. A range of pH 5 to 7 did not affect the total amount of removal %. The removal % was calculated by equation (1).

$$\text{Removal \%} = \frac{(C_i - C_e)}{C_i} \times 100 \quad (1)$$

Where C<sub>i</sub> is the initial concentration of ions and C<sub>e</sub> is the equilibrium concentration of ions after adsorption [6]

### 3.2. Printed samples

Through 3D printing, zeolite 13X inks were printed well with 0.6 mm of a nozzle through air pressure as shown in Fig 1.



Fig. 1. A 3D printed zeolite filter

### 3.3. Comparing powder and a filter form.

In order to compare the adsorption capacity, with removal percent factor, the adsorption test of a filter form was done as same as the powder adsorption test, at pH 7. The powder form was 3 times larger than filter form with removal percent as shown in Fig 2. In the case of bulk sample, we could not measure the specific surface area through the BET so we can make a virtual factor which can explain the relative difference of specific surface areas between the powder and bulk form.

Not only lower specific surface areas but also the permeability of printed body could affect to adsorption capacity. Based on SEM-EDX mapping, we observed the outer and inner surface of the printed body after adsorption test and found out that the inner area of the bulk body did not act as sorption sites as shown in Fig 3.

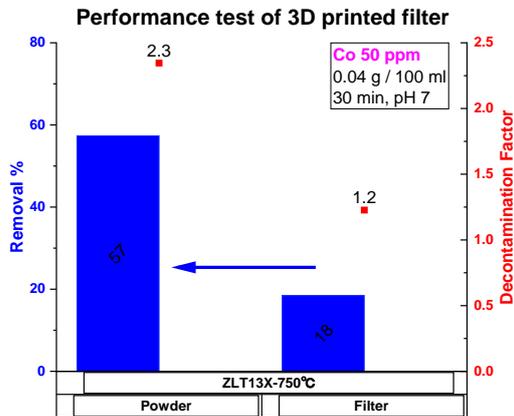


Fig. 2. Comparing of powder and a filter's adsorption capacity

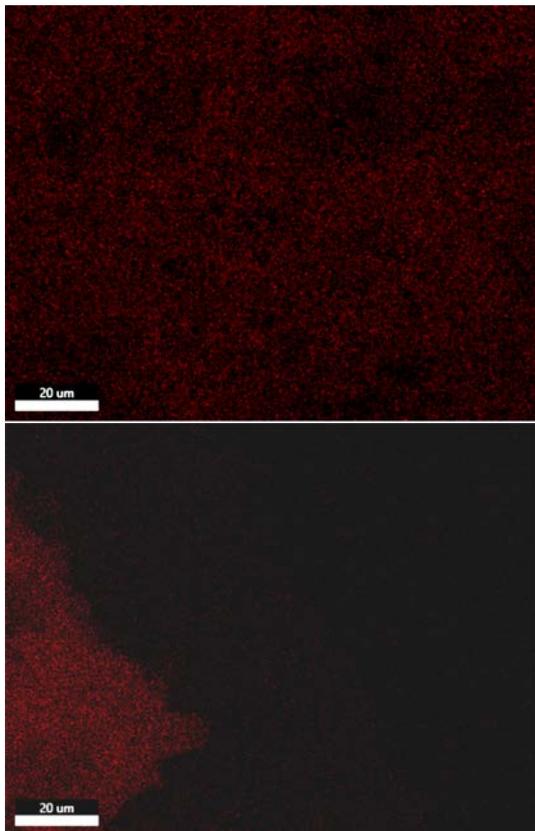


Fig. 3. Comparing at% of  $\text{Co}^{2+}$  on an outer (top) and an inner (bottom) of the printed filter after adsorption

The study demonstrated that zeolite-based ink were established by controlling the rheology-related parameters such as the solid particle volume fraction and binder fraction. As a result of adsorption tests of powder and a printed filter, we found that the specific surface area is one of the controlling factors of the adsorption performance of zeolite-based ceramics. Moreover, we observed that the permeability of  $\text{Co}^{2+}$  ions into the printed filter was limited.

#### Acknowledgments

This study is supported by the KAI-NEET, KAIST

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#### 4. Conclusions