

## Preparation of SiO<sub>2</sub>-KCoFC composite ion-exchanger for removal of Cs in the soil decontamination waste solution

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### 1. Introduction

The soil decontamination process has been developed for remediate the soil wastes excavated from the TRIGA research reactor sites. Even though the process was proven to be very effective for decontaminate the radioactive nuclides such as cesium and cobalt, the secondary spent solution should be treated with an appropriate method to minimize the waste volume. There are mainly two components in the spent decontamination solution of Cs and Co. The Co in the waste solution can be removed easily by precipitation under a basic condition. However, since the Cs is hardly removed by precipitation, an appropriate selective removal method should be employed.

In this study, an inorganic composite ion exchanger of SiO<sub>2</sub>-KCoFC was prepared by sol-gel method for a removal of Cs in the decontamination waste solution. An optimum condition for a preparation of the composite ion exchanger and the adsorption performances of the prepared composite ion exchangers were evaluated.

### 2. Materials and method

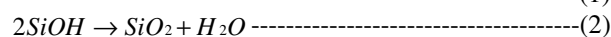
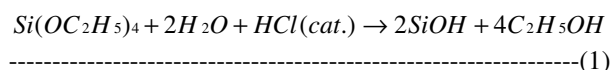
#### 2.1. Sample descriptions

##### 2.1.1. Preparation of KCoFC

In order to prepare KCoFC, 480 mL of 0.3 M Co(NO<sub>3</sub>)<sub>2</sub> solution was added to 200 mL of 0.5 M K<sub>4</sub>Fe(CN)<sub>6</sub> solution and stirred for 24 hours. After the filtration of mixed solution, deionized water was used for washing until less than 1 mg/L of Co conc. in filtrated water. Finally, KCoFC was dried in dry oven at 115 °C and kept in desiccater.

##### 2.1.2 Sol-Gel process

Chemical reactions of sol-gel process by using tetraethyl orthosilicate (TEOS; Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>) was shown below.



From equation (1), when the powder state of KCoFC is added, KCoFC can be physically fixed between SiO<sub>2</sub> particles. In conclusion, the powder state of KCoFC can be granulized (SiO<sub>2</sub>-KCoFC). In sol-gel process, the

addition ratio of H<sub>2</sub>O and HCl are the key parameters affecting the characteristics of composite ion exchanger.

Table 1. Experimental conditions of sol-gel reactions on the various KCoFC content

KCoFC content in SiO <sub>2</sub> -KCoFC (wt %)	KCoFC (g)	TEOS (g)	TEOS : H <sub>2</sub> O	TEOS : HCl
20	0.5	7.07	6	0.6
40		2.65		
50		1.77		
60		1.18		
70		0.76		
80		0.44		
85		0.31		
90		0.20		
92		0.15		

Table 2. Experimental conditions of sol-gel reactions on the various TEOS vs. H<sub>2</sub>O and HCl ratio

Cs conc. (mg/l)	108	
V(ml)	100	
Kinds of exchanger	H <sub>2</sub> O	4, 5, 6
	HCl	0.4, 0.5, 0.6
Mass of exchanger (g)	0.1	
L/S ratio (mL/g)	1000	
Shaking speed (rpm)	150	
Sampling time (hr)	0.17, 0.5, 1, 2, 3.5, 8, 18, 24	

##### 2.1.3 Stability test of synthesized SiO<sub>2</sub>-KCoFC

Various amounts of TEOS were added to 0.5 g of KCoFC. KCoFC content in the prepared SiO<sub>2</sub>-KCoFC composite were 20, 40, 50, 60, 70, 80, 85, 90 and 92 %. Gelation time was measured. After gelation, each sample was dried in dry oven (70 °C) and washed with deionized water to remove the excess of sol-gel reactants such as HCl. After drying, synthesized SiO<sub>2</sub>-KCoFC was examined whether KCoFC redissolves in acidic and alkali solutions. From the dissolution test, possible maximum KCoFC content to make SiO<sub>2</sub>-KCoFC was determined. Experimental conditions were shown in Table 1.

#### 2.2. Determination of optimum addition ratio of TEOS : H<sub>2</sub>O : HCl

Experimental conditions to determine the optimum addition ratio of H<sub>2</sub>O and HCl were shown in Table 2. The nine kinds of SiO<sub>2</sub>-KCoFC composite ion exchangers were prepared with different addition ratio of H<sub>2</sub>O and HCl (4, 5, 6 : 0.4, 0.5, 0.6). The removal

efficiencies of the prepared SiO<sub>2</sub>-KCoFC for Cs ion were investigated. 100 mL of Cs solutions (108 mg/L) were mixed with 0.1 g of SiO<sub>2</sub>-KCoFC under stirring for 24 hours. From the measurement of Cs conc. in solutions, optimum addition ratio of TEOS : H<sub>2</sub>O : HCl was determined.

### 3. Results and discussions

#### 3.1 Possible max. KCoFC content in SiO<sub>2</sub>-KCoFC

Table 3 shows the result on the dissolution tests for the SiO<sub>2</sub>-KCoFC composite ion exchangers prepared with various KCoFC contents by using acidic and basic solutions in the pH range of 2 and 10. There was no observation of KCoFC leaching from the composite ion exchanger (SiO<sub>2</sub>-KCoFC) when the KCoFC contents are less than 80 %. However, KCoFC were released from the composite ion exchanger in the d.w., acidic and basic solutions when the KCoFC contents are more than 85 %. Therefore, it was decided that the KCoFC content in the SiO<sub>2</sub>-KCoFC should not exceed 80 % to prepare a stable composite ion exchanger composite for Cs removal.

Figure 1 shows the photo of synthesized KCoFC and SiO<sub>2</sub>-KCoFC (80 % KCoFC), respectively. As shown in this figure, the very fine particles of KCoFC are composited successfully by SiO<sub>2</sub> to form SiO<sub>2</sub>-KCoFC composite ion exchanger (80 % KCoFC).

#### 3.2 Optimum addition ratio of H<sub>2</sub>O and HCl

Figure 2 shows that the Cs uptake curves reached almost their equilibrium state after 8 hours. The composite ion exchanger prepared with the condition of 4 : 0.4 for the ratio of H<sub>2</sub>O and HCl shows the best performance for Cs removal.

The equilibrium study will be carried out for characterizing the adsorption capacities of the composite ion exchanger.

Table 3. Results of sol-gel reaction in various KCoFC content

KCoFC content in SiO <sub>2</sub> -KCoFC (wt%)	Gelation time (hr)	Dissolution test	
		d.w.	pH 2 ~ 10
20	2.2	O	O
40	15	O	O
50	43	O	O
60	72	O	O
70	120	O	O
80	154	O	O
85	163	X	X
90	172	X	X
92	183	X	X

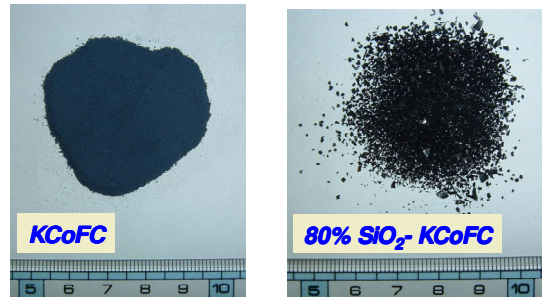


Figure 1. Photo of synthesized KCoFC and SiO<sub>2</sub>-KCoFC (80 % KCoFC)

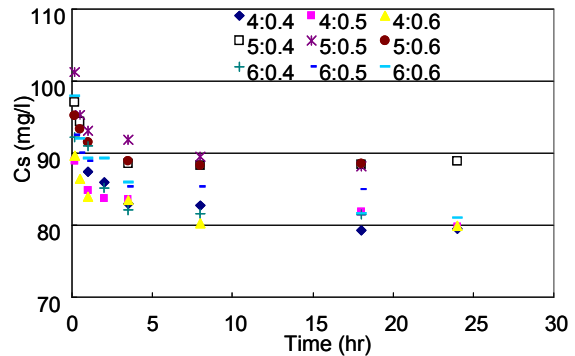


Figure 2. Cs removal behavior with time in SiO<sub>2</sub>-KCoFC (80 % KCoFC) ion exchanger prepared with different H<sub>2</sub>O : HCl addition ratio

### REFERENCES

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