

EFFECT OF HEATING TREATMENT ON SYNTHETIC CERIC OXIDE AS INORGANIC RESIN FOR IMMOBILIZATION OF CADMIUM-(II).

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ABSTRACT

EFFECT OF HEATING TREATMENT ON SYNTHETIC CERIC OXIDE AS INORGANIC RESIN FOR IMMOBILIZATION OF CADMIUM-(II).

The immobilization of cadmium-(II) following nuclear reaction of ^{114}Cd (n,γ) $^{115}\text{Cd} \rightarrow ^{115\text{m}}\text{In}$ or ^{112}Cd ($p,2n$) ^{111}In is absolutely needed concerning to the quality of resulting radioisotope products and the efficiency of enriched target utilization. This presented study is based on hypothesis that heating treatment on synthetic ceric oxide resin will increase the binding capacity of Cd(II) over the resin. The aim of the presented study is to gain more experimental data supporting utilization of synthetic ceric oxide resin as separating agent in production of indium radioisotopes using enriched cadmium targets. Experiments in cold system using natural cadmium, instead of high-enriched cadmium, have been carried out, wherein a method of UV-spectrophotometry was performed for quantitative determination of Cd(II). It was found that the synthesized ceric oxide dried at 90 - 100°C was in form of hydrate compound formulated as $\text{CeO}_2 \cdot q \text{H}_2\text{O}$ where $q = (0.8868 \pm 0.0026)$. The value of q descended to 0.1706 and to 0.0400 as influenced by heating treatment up to 400°C and 800°C respectively along with a change on the crystallinity of the ceric oxide from semi crystalline to purely crystalline. The binding capacity of Cd(II) on ceric oxide dried at 90 - 100°C was found to be $(4.71 \pm 0.08) \times 10^{-2}$ mg Cd(II)/mg resin and significantly increased to $(11.71 \pm 0.24) \times 10^{-2}$ and to $(14.70 \pm 0.17) \times 10^{-2}$ mg Cd(II)/mg resin due to heating treatment up to 400°C and 800°C respectively. It can be concluded that the synthetic ceric oxide resin enable to be exploited for immobilization of Cd(II).

Key words : Inorganic resin, ceric oxide, Cd(II)-binding capacity, immobilization of Cd(II), production of indium radioisotopes

INTRODUCTION

Ceric oxide, CeO_2 , has been reported to have behaviour and capacity as an inorganic ion exchanger [1,2,3]. It has been even used in parent-daughter radionuclidic separations, e.g. in separation of $^{99}\text{Mo} - ^{99\text{m}}\text{Tc}$ [4] and of $^{68}\text{Ge} - ^{68}\text{Ga}$ [5]. Some studies on the immobilization of Cd and other metal ions over ceric oxide [6,7] lead to possibility that ceric oxide can be utilized as Cd(II)-binding agent in recovery of high-enriched Cd (^{114}Cd or ^{112}Cd) and purification of resulting product of $^{115\text{m}}\text{In}$ or ^{111}In .

The indium radioisotopes, both $^{115\text{m}}\text{In}$ and ^{111}In , are medical radioisotopes enable to be reacted with various ligand compounds to produce radiopharmaceutical preparations used for tumors diagnosis or hematological damage detection [8]. The $^{115\text{m}}\text{In}$ radioisotope is produced in nuclear reactor followed by separation in a radioisotope generator based on ^{114}Cd

$(n,\gamma) {}^{115}\text{Cd} \rightarrow {}^{115\text{m}}\text{In}$ nuclear reaction [9], while the ${}^{111}\text{In}$ radioisotope is produced in cyclotron by ${}^{112}\text{Cd} (p,2n) {}^{111}\text{In}$ nuclear reaction [10]. However, the domestic application of those indium radioisotopes is not popular yet due to the lack of capability on the production technology.

As a part of the production technology of ${}^{115\text{m}}\text{In}$ or ${}^{111}\text{In}$, the processing technique for immobilization of Cd(II) is absolutely needed concerning to the efficiency of enriched target utilization and the quality of products. Related to the efficiency of enriched target utilization, the recovery of enriched targets from processing wastes is necessary because the enriched targets are very expensive and still must be imported. In order to find out a technique for recovery of enriched cadmium target, studies on dynamic sorption and chromatographic behaviour of Cd(II) in column of ceric oxide resin were performed [11,12]. Related to the quality of products, the indium radioisotopes should be chemically free from cadmium due to the toxicity of cadmium and, in case of ${}^{115\text{m}}\text{In}$, should be free from ${}^{115}\text{Cd}$ -radionuclidic impurity because the radiomedical behaviour of ${}^{115}\text{Cd}$ is very different from that of ${}^{115\text{m}}\text{In}$. In this case, immobilization of cadmium constitutes a technique of separation and purification in production of ${}^{115\text{m}}\text{In}$ and ${}^{111}\text{In}$. Such kind of works should be carried out to enable production of ${}^{115\text{m}}\text{In}$ and ${}^{111}\text{In}$ by facilities in BATAN Serpong, e.g. the G.A. Siwabessy Reactor and the modified CS-30 Cyclotron.

This study was based on hypothesis that heating treatment on ceric oxide resin will increase the binding capacity of Cd(II) over the resin. With this thought, the binding of Cd(II) is expected can be improved due to effect of heating treatment on the synthetic ceric oxide. The aim of the present study is to gain more experimental data supporting utilization of ceric oxide resin as separating agent in production technique of indium radioisotopes using enriched cadmium targets. Natural cadmium was used replacing high-enriched cadmium, because the former is much cheaper and easier to be procured locally than the later, but both have similar chemical behaviour. The experiment was carried out in cold system wherein a method of UV-spectrophometry was performed for quantitative determination of Cd(II) [11,12].

MATERIALS AND METHOD

Preparation of materials and equipments

Cadmium metal obtained from Merck (Cat. No. 139.A617501/102001.0250) was used for stock and standard solutions of Cd(II). Cerium sulphate tetra hydrate from Merck was used as basic material in the synthesis of ceric oxide resin. Other chemicals were also from Merck, dissolved or diluted using demineralized water obtained from purification water system of G.A. Siwabessy Reactor Facility.

Jasco FTIR Spectrophotometer (FTIR-410) and Shimadzu X-Ray Diffraction System (X-Ray Research Goniometer, VG-108R) were used for analysis of the synthesized resin. Blue M Furnace (Model No. E-514-3) was used for heating treatment up to 400°C and 800°C, whereas Fisher Scientific Oven (Model No. 630 F) was for heating treatment up to 90 – 100°C. Determination of cadmium content in solution samples was performed using Jasco UV/Vis Spectrophotometer (V-550) completed with Spectra Manager for Windows (961105 version) software program.

Synthesis and heating treatment of ceric oxide resin

The synthesis of ceric oxide was carried out based on the method previously reported [7,11,12]. An approximately 20 – 50 g of $Ce(SO_4)_2 \cdot 4 H_2O$ was dissolved with stirring in 250 mL of 0.05 M H_2SO_4 . Concentrate NH_4OH (25 %, 150 mL) was added drops wise to the stirred solution. After finishing the addition of the NH_4OH , the reaction mixture was stirred for about 15 minutes more and then kept overnight at room temperature. The precipitate was filtered and washed with 4×25 mL hot water, then with cold water until the pH of the washing was neutral. The solid product was dried at about 90 – 100°C for a few hours to give a relatively constant weight of the product.

The solid product, which was then assumed as ceric oxide hydrate, was divided into 3 portions. The first portion was used as control, whereas the second and the third were heated in furnace up to 400°C and 800°C respectively for about 1 hour. The ceric oxides were then carefully ground in a mortar and sifted using a 100-mesh-sieve. The fractions passing the sieve were used for further experiment.

Analysis of ceric oxide resin

An appropriate amount of ceric oxide resin was mixed and ground with KBr powder and pressed in a mold to give CeO_2 -KBr pellet suitable for FTIR-spectrophotometry. Another amount of the resin was sent to the Center for Research and Development of Material Science and Technology (CRDMST, BATAN) for X-Ray diffraction analysis using Cu- K_α target ($\lambda = 1.54056 \text{ \AA}$).

Preparation of stock and standard solutions of Cd(II)

An approximately 20 g of Cd metal was accurately weighed and dissolved in appropriate volume of concentrate HNO_3 . The resulting solution was diluted with water and then evaporated to dryness under its boiling point. The residue was dissolved in water and again evaporated to dryness as before. Finally, the residue was dissolved again, quantitatively transferred into 100 mL volumetric flask and diluted to the mark to get Cd(II)-stock solution with Cd(II) content of about 200.000 ppm. Standard solutions of

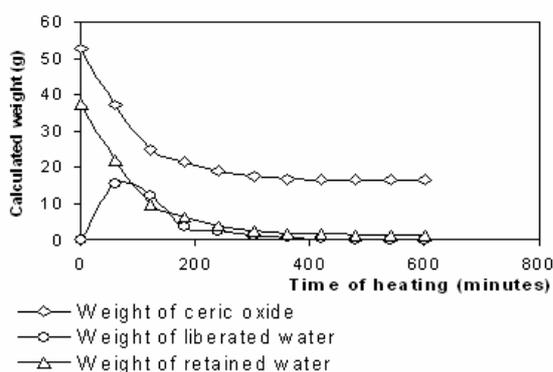
Cd(II) with various concentrations were prepared as required by suitable dilution of the stock solution.

Determination of Cd(II)-binding capacity over the resin

A 10 mL of Cd(II) standard solution was treated with a certain amount of resin and stirred at room temperature for 15 minutes and then kept quiet for 15 minutes more. The mixture was then transferred into centrifuge tube for separation of the resin and the supernatant. The supernatant was accurately cited into 10 mL volumetric flask and diluted to the mark. The Cd(II) content of diluting supernatant was measured by means of UV-spectrophotometry at 301 nm [11,12]. The resulting value, after being corrected with the corresponding dilution factor, represents Cd(II) content of the solution after being treated with the resin. Subtraction from Cd(II) content of the initial standard solution by that of the corresponding supernatant gives the value of Cd(II)-amount bound over the resin. This procedure was applied repeatedly by using various standard solutions (about 40,000 – 60,000 ppm) and various amounts (about 400 – 800 mg) of control and heat-treated resins. It is necessary to make sure that no change occurs on pattern of UV absorption spectra of the supernatant as compared to that of the initial standard solution. This indicates that during the treatment with the resins no change on chemical behaviour of Cd(II) has occurred in the solutions.

RESULTS AND DISCUSSION

The reaction between $\text{Ce}(\text{SO}_4)_2 \cdot 4 \text{H}_2\text{O}$ and NH_4OH apparently gives a hydrate compound of ceric oxide as the result of heating at about 100°C to the reaction product [11,12]. A typical pattern of water liberation during the heating at $90 - 100^\circ\text{C}$ as shown in Fig. 1 indicates that some water was still retained on the ceric oxide molecule as a hydrate that is not liberated up to 100°C .



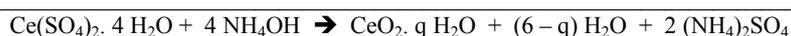
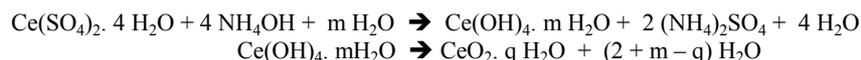
Weight of ceric oxide : the weight of synthesized product after each of heating

Weight of liberated water : the difference between two successive weighing of synthesized product.

Weight of retained water : the amount of gram that is equivalent to "q" in $\text{CeO}_2 \cdot q \text{H}_2\text{O}$ corresponding to weight of the synthesized product after each of heating.

Figure 1. Typical pattern of water liberation from ceric oxide during heating treatment up to $90 - 100^\circ\text{C}$.

The existence of retained water in the resulting ceric oxide was also indicated by yield of synthesis that was obtained to be more than 100 % as compared to calculation based on the production yield of CeO₂. The result shown in Table 1 indicates that the synthesis product is a hydrate compound, formulated as CeO₂. q H₂O, which is formed through the following reactions :



It is shown in Table 1 that the water content bound in the structure of resulting ceric oxide, thus the value of “q” in CeO₂.q H₂O, was found to be (0.8868 ± 0.0026) mol/mol. This value is in accord with those reported previously [11,12] i.e. (0.885–0.886) mol/mol. It may be necessary to observe whether the hydrate water bound in ceric oxide can be liberated by vacuum evaporation instead of drying at 90 – 100°C.

Table 1. The chemical yields on the synthesis of ceric oxide resin.

NUMBER OF SYNTHESIS	AMOUNT OF Ce(SO ₄) ₂ .4H ₂ O (g)	AMOUNT OF CERIC OXIDE (g)	YIELD BASED ON CeO ₂ (%)	WATER CONTENT (mol/mol)	AVERAGE VALUE OF “q” (mol/mol)
1	23.5547	10.9660	109.28	0.8876	(0.8868 ± 0.0026)
2	35.3364	16.4419	109.30	0.8890	
3	47.1425	21.9243	109.24	0.8839	

The descent of water content bound in CeO₂ molecule, due to heating treatment up to 400°C and 800°C, can be observed from Fig. 2 showing FTIR spectra of the resulting hydrous oxide compounds. The broad absorption in the range of 3000 to 3500 cm⁻¹ is due to contribution from interstitial water and hydroxyl group, whereas the absorption band appears at the range of 1630 to 1650 cm⁻¹ is due to bending vibration of water molecule [7,11,12]. Comparison of absorption bands at 3000 to 3500 cm⁻¹ and at 1630 to 1650 cm⁻¹ respectively to the third one, i.e. in the range of 900 to 1150 cm⁻¹ representing the presence of Ce–O–H group [13], shows highest value in the spectra of control CeO₂ (A) and lowest value in the spectra of CeO₂ heated up to 800°C (C). The data indicate that water molecule was liberated from the molecule of synthesized ceric oxide by heating treatment in agree with the descent of the oxide’s weight. Further calculation on the weight of the oxide shows that the structure of the hydrate compound changed from CeO₂. 0.8868 H₂O (control) to be CeO₂. 0.1706 H₂O (on heating up to 400°C) and to be CeO₂. 0.0400 H₂O (on heating up to 800°C).

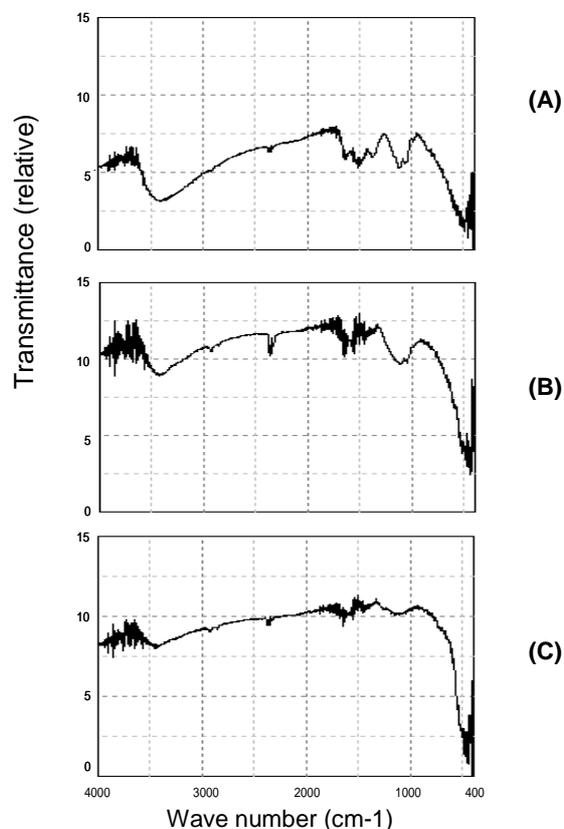


Figure 2. FTIR spectra of the resulting hydrous ceric oxide : dried at 90 – 100°C (A), heated up to 400°C (B) and heated up to 800°C (C).

The heating treatments up to 400°C and 800°C also influenced the crystalline of hydrous ceric oxide revealed by the change on the sharpness of the X-Ray diffraction spectra as indicated in Fig. 3 and the change on the values of FWHM as presented in Table 2. The crystal behaviour is growing increase from semi crystalline (control CeO₂) to purely crystalline (CeO₂ heated up to 800°C) in a line with the descent of water content. However, the crystal lattice structure appears to be unchanged, that is similar to that of *cerianite* (ceric oxide) as per reference [14]. Table 2 exhibits the conformities of the strongest 5-peaks of the X-Ray diffraction spectra in sequence of intensity indicating that the analyzing samples have a crystal structure resembling one to another.

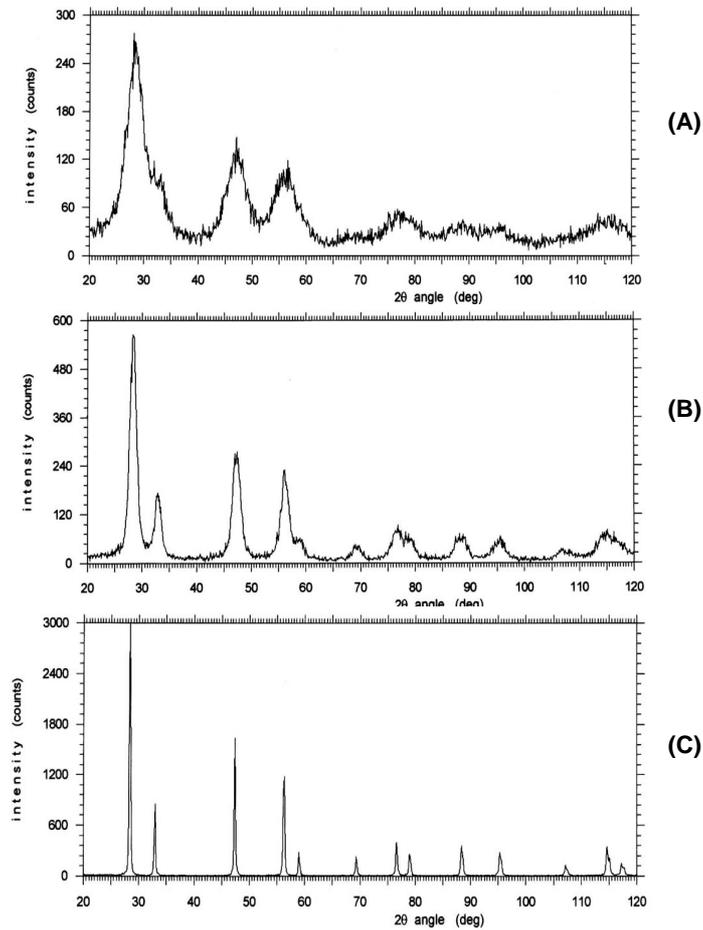


Figure 3. The pattern of X-Ray diffraction spectra of CeO₂ dried at 90 – 100°C (A), heated up to 400°C (B) and heated up to 800°C (C).

Table 2. Strongest 5-Peaks on X-Ray diffraction analysis (Sequence of intensity).

2θ (°)	I		II			III			IV		
	I/I ₁ (%)	d (Å)	I/I ₁ (%)	d (Å)	FWHM (relative)	I/I ₁ (%)	d (Å)	FWHM (relative)	I/I ₁ (%)	d (Å)	FWHM (relative)
± 28	100	3.124	100	3.162	3.622	100	3.138	1.475	100	3.138	0.428
± 47	51	1.913	61	1.929	3.685	62	1.917	1.750	76	1.917	0.432
± 56	44	1.632	33	1.681	2.540	53	1.642	1.719	59	1.636	0.441
± 33	29	2.706	25	2.672	1.734	27	2.723	1.374	29	2.714	0.432
± 76	15	1.241	23	1.238	2.385	18	1.241	2.168	25	1.243	0.431

NOTES : I. Ceric oxide as per reference [14].

II. Ceric oxide, control (heated up to 90 – 100°C).

III. Ceric oxide, heated up to 400°C.

IV. Ceric oxide, heated up to 800°C.

The Cd(II)-binding capacity over the synthesized ceric oxide resin can be expressed by the following equation [12,16] :

$$B_c = (C_0 - C_s) / W_r \dots\dots\dots(i)$$

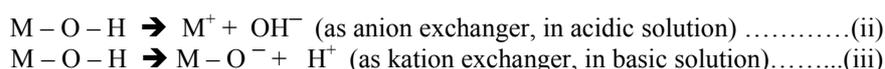
where B_c = Binding capacity [mg Cd(II) / mg resin]
 C_0 = Cd(II) content in standard solution prior to treatment with the resin [mg]
 C_s = Cd(II) content in standard solution after the treatment with the resin [mg].
 W_r = Total weight of the resin used [mg].

In this experiment, C_0 and W_r values were determined as variables of experiment, whereas C_s was calculated from UV-spectrophotometric measurement at maximum wavelength of 301 nm by considering the respective dilution factors. The calibration curve was found to have mathematical equation of $A = 0.00012 C + 0.00884$, where A is absorbance and C is Cd(II) concentration (ppm), with a correlation coefficient of 0.9985. The data presented in Table 3 containing values of variables in equation (i) show replication of the experiment related to determination of Cd(II)-binding capacity over the synthetic cerium oxide resin.

Table 3. Experimental data on determination of Cd(II)-binding capacity (B_c) over synthetic ceric oxide resin with various heating treatment.

C_0 (mg)	DRIED AT 90-100°C			HEATED TO 400°C			HEATED TO 800°C		
	W_r (mg)	C_s (mg)	B_c (mg/m)	W_r (mg)	C_s (mg)	B_c (mg/mg)	W_r (mg)	C_s (mg)	B_c (mg/mg)
400.10	400.3	308.8	0.0482	400.9	351.8	0.1205	400.6	340.8	0.1480
	600.4	371.8	0.0471	600.8	329.8	0.1170	600.5	311.8	0.1470
	800.5	362.8	0.0466	800.3	307.8	0.1153	800.4	282.8	0.1465
480.12	400.4	460.8	0.0483	400.2	428.8	0.1207	400.8	419.8	0.1505
	600.2	451.8	0.0472	600.3	409.8	0.1171	600.4	391.8	0.1471
	800.1	442.8	0.0466	800.1	387.8	0.1154	800.4	363.8	0.1453
560.14	400.1	541.8	0.0458	400.4	512.8	0.1182	400.5	500.8	0.1482
	600.5	531.8	0.0472	600.1	490.8	0.1156	600.2	472.8	0.1455
	800.2	522.8	0.0467	800.2	468.8	0.1142	800.2	443.8	0.1454

Ceric oxide, as generally inorganic resins, may have behaviour of either ion exchanger or adsorber, or even both [15,16]. In particular, the ion exchange behaviour of hydrous oxide metal is influenced by pH as generally shown below [1] :



According to (iii), if the binding of Cd(II) from aqueous solution over ceric oxide resin was happened through ion exchange mechanism, it should

be followed by the decrease of pH due to liberation of H^+ in the solution. But in this presented experiment, there was no significantly change on the pH of the Cd(II) solution treated with ceric oxide resin. The previous report [16] showed that the pH of Cd(II) solution treated with ceric oxide was relatively unchanged as observed up to 30 minutes of treatment. This case indicates that the binding of Cd(II) on ceric oxide resin is not based on ion exchange mechanism.

Assuming that the binding mechanism is adsorption, the presented experiment can be compared to other works on immobilization study of Cd(II) on titanium oxide and zirconium oxide [17]. This study indicates that zirconium oxide was able to bind Cd(II) from an aqueous solution while titanium oxide was not. This case was slightly amazing because both zirconium and titanium are members of Group IV B in the Periodic Table. It was presumed that the interaction of Cd(II) with titanium oxide and zirconium oxide is influenced by the radius and mol volume of Ti and Zr as compared to those of Cd. The radius and mol volume of titanium atoms (1.32 Å and 10.64 cm³/mol) are smaller than those of cadmium atoms (1.41 Å and 13.07 cm³/mol) [18], so that there is space hindrance of TiO₂ molecules preventing adsorption of Cd(II) on titanium oxide resin. On the contrary, those of zirconium atoms (1.45 Å and 13.97 cm³/mol) [18] are bigger than the cadmium's, and there is no space hindrance hindering adsorption of Cd(II) on zirconium oxide resin. In the case of cerium oxide resin, the radius and mol volume of cerium atoms (1.65 Å and 21.0 cm³/mol) [19] are bigger than those of zirconium and, thus, than the cadmium's so that Cd(II) can be bound on zirconium oxide resin.

Further calculation for the Cd(II) binding capacities by means of T-test method is presented in Table 4. It shows that the values of calculated T were found to be higher than that of T from table [20]. This calculation result indicates that the values of Cd(II)-binding capacity shown in Table 3 were significantly different as influenced by heating treatment. This is presumably due to the decrease of hydrate water content bound in the ceric oxide resins. The typical correlation between hydrate water content with Cd(II) binding capacity is shown in Fig. 4. Although it might be necessary to have more data supporting this correlation, it can be stated that the descent of water molecules bound in ceric oxide molecules due to heating treatment will increase the active site of ceric oxide molecules to bind Cd(II), instead of hydrate water, by adsorption mechanism.

Table 4. Matrix calculation of T-test for Cd(II)-binding capacity as influenced by heating treatment.

TEMPERATURE OF HEATING (°C)	Cd(II)-BINDING CAPACITY (mg/mg)	N ¹⁾	T _{CALC.} ²⁾ AND T _{TABLE} ³⁾	
			3-Groups test	2-Groups test
90 to 100	$(4.71 \pm 0.08) \times 10^{-2}$	9	Reference	Not tested
400	$(11.71 \pm 0.24) \times 10^{-2}$	9	T _{CALC.} = 25.150 T _{TABLE} = 1.746	Reference
800	$(14.70 \pm 0.17) \times 10^{-2}$	9	T _{CALC.} = 36.644 T _{TABLE} = 1.746	T _{CALC.} = 30.498 T _{TABLE} = 1.746

NOTES : 1). N = Number of replicates for each of heating treatments.

2). T_{CALC.} = Value of calculated T based on mathematical equation [21] :

$$T_{\text{CALC}} = \frac{|(B_c)_1 - (B_c)_2|}{[\{(S_1)^2 \cdot N_2 + (S_2)^2 \cdot N_1\} / (N_1 \cdot N_2)]^{1/2}}$$

(B_c) : Cd(II)-Binding capacity of CeO₂.

S : Standard deviation.

Index 1 and 2 represent sample and reference respectively.

3). T_{TABLE} is for One-tail T-test with degree of freedom (N₁ + N₂ - 2) = 16 and probability level P = 0.05 [20].

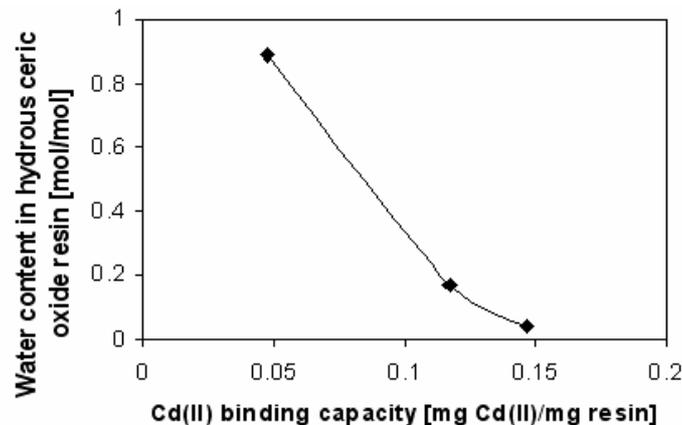


Figure 4. Correlation between water hydrate content with Cd(II) binding capacity.

On the other side, Table 3 shows that a slight decrease of Cd(II)-binding capacity was generally obtained on increasing the amount of resin used. Although it was statistically insignificant, but it might have correlation with the probability of uniformity in the particle size of the resin used in each experiment batch. The more of amount of the resin used is the less of uniformity in its particle size. Consequently that is the less of adsorption and, thus, the less of binding capacity.

The all presented experiment results indicate that heating treatment up to 400°C and 800°C did not influence the crystal lattice structure of ceric oxide resin but the crystallinity of the resin, the water content as binding hydrate molecule and the capacity to bind Cd(II) as well. The Cd(II) binding mechanism is apparently not a phenomenon of ion exchange, but that of adsorption. The higher temperature of heating treatment is the higher of the Cd(II)-binding capacity, giving good prospect to use ceric oxide resin in the immobilization of high-enriched cadmium target in production of indium radioisotopes.

CONCLUSION

The synthesized ceric oxide resin dried at 90 – 100°C is in form of hydrate compound of $\text{CeO}_2 \cdot q \text{H}_2\text{O}$ where “q” is (0.8868 ± 0.0026) . The value of “q” decreases to 0.1706 and to 0.0400 as influenced by heating treatment up to 400°C and 800°C respectively.

The heating treatment up to 400°C and 800°C did not influenced the crystal lattice structure, but the crystalline was growing as stated from semi crystalline on ceric oxide dried at 90 – 100°C to purely crystalline on ceric oxide heated up to 800°C. The crystalline behaviours of the synthesized ceric oxide resemble that of cerianite (CeO_2).

The binding capacity of Cd(II) over ceric oxide dried at 90 – 100°C was found to be $(4.71 \pm 0.08) \times 10^{-2}$ mg Cd(II)/mg resin and significantly increased to $(11.71 \pm 0.24) \times 10^{-2}$ and to $(14.70 \pm 0.17) \times 10^{-2}$ mg Cd(II)/mg resin due to heating treatment up to 400°C and 800°C respectively. The binding of Cd(II) is presumably based on adsorption mechanism.

The synthetic inorganic resin of ceric oxide, thus enable to be exploited for immobilization of Cd(II) and applied as separating agent in production of indium radioisotopes from high-enriched cadmium target.

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