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Adsorption and Utilization of Cerium (III) Ions from Aqueous Solutions by Iraqi Rice Husk

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Abstract:

Iraqi rice husk (IRH) is used for cerium removal from aqueous solutions using different design parameters by adsorption process. The design parameters studied to adsorb cerium using IRH as an adsorbent material were initial concentration of cerium, absorbance material packing height which was IRH, pH of cerium feed inlet, treatment time, feed flow rate and feed temperature. The samples of IRH remaining after cerium adsorption were using it as a promoter in preparing type Y-zeolite catalyst, compared these type prepared above with normally type Y-zeolite catalyst and tested in the process of n-heptane catalytic cracking under different operating conditions.

Key words: Rice husk, cerium, SSAS, Y-zeolite and catalytic cracking

1. INTRODUCTION

Large quantities of nuclear wastes are generated from reprocessing of nuclear fuel and charged particle accelerators due to operation of large number of nuclear power plants. These nuclear wastes are often underground buried in stainless steel containers. Toxic radioactive materials may be transferred to the surrounding soil and groundwater as a result to the probability seepage of some of these containers even in distant future. Also, these radionuclides pollutants my prevalence to the surrounding due to tests of nuclear weapon, nuclear episode such as Chernobyl nuclear power plant disaster in 26 April 1986, forbidden discharge or puffs and the possible influx of certain toxic industrial effluents **[1]**. Amongst these, Lanthanides such as cerium (Ce) are commonly found in the waste streams of nuclear power plants and research centers **[2]**. Radioisotopes of cerium are marked products of nuclear fission and hence, they are a common constituent of liquid radioactive wastes arising from nuclear facilities. Due to the similarity in chemical behaviour with radioisotopes of transuranic elements, radioactive or non-radioactive isotopes of cerium are customarily used to substitute dangerous (highly radio-toxic) transuranic elements solution, prepared for the study of the liquid radioactive waste treatment or/and of the partitioning of transuranic elements **[3]**. Therefore, reduce the concentration of Ce ions from radioactive waste solution in economical and safe methods has been paid serious solicitude **[1].** Since ion-exchange by selective inorganic cation-exchangers (i.e. zeolites) is a very perspective (and in many cases widely used) separation method, the study of the exchange ability of cerium ions from the solution with sodium ions from zeolites is very important in order to find the possibility of the removal of the radioisotopes of cerium and actinides from radioactive solutions using zeolites **[3]**. The adsorption technique is one of the most commonly and efficacious methods in radioactive waste treatment and it represented the green chemistry direction to reduce the use of toxic and carcinogenic solvents in remediation processes of wastewater **[1]**. Recently, rice husk which is a type of agriculture waste is used as adsorbent media for adsorption process of many pollutants such as heavy metals [4,5,6,7,8,9,10] ,dyestuffs **[11]**, pesticides **[12]**, phenols [13 and 14] and non-organic toxins [15 and 16] because it is available, very inexpensive and ability to remove different types of contaminants, thus the rice husk usage will decrease the cost of wastewater treatment considerably and would represent practical benefit of agriculture waste residue in disposal toxic matter in save way **[1]**. Therefore, the aim of this study is to investigate the adsorption behaviour of cerium Ce^{+3} ions on Iraqi rice husk from simulated synthetic aqueous solution (SSAS) similar to nuclear waste and utilize from the husk remaining in eco-friendly method.

2 EXPRIMENTAL WORK

2.1 Materials

2.1.1 Iraqi Rice husk (adsorbent media)

Rice husk was collected from Al-Shanafia fields for rice in the Southern of Iraq. The rice husk was washed three times with doubled distilled water. Excess distilled water was used to remove the soluble materials present in the rice husk bringing from the field, boiled to remove colour and other fine impurities may be found in the rice husk, and then dried at 105°C for 24 hours and the adsorbent thus processed was used in its original piece size. The surface area of IRH was measured by BET (Brunauer – Emmett – Teller nitrogen adsorption technique). Characteristics of IRH were presented in **Table 1**. When the IRH was heated in an oven, most of the water had been removed from the rice husk while the second major mass loss of about 45-65% was attributed to the breakdown of cellulose constituent char, which is a carbonaceous residue.

2.1.2 Stock solutions

In order to avoid interference with other elements in wastewater, the experiments in this study were carried out using simulated synthetic aqueous solution (SSAS) of different cerium concentrations. 1000 mg/l stock solution of cerium was prepared by dissolving known weight of cerium nitrite Ce(NO3)3.6H2O in one litre of double distilled water, all solutions using in the experiments were prepared by diluting the stock solution with double distilled water to the desired concentrations for the experimental work of this investigation.

The cerium concentrations were measured using spectrophotometer method using spectrophotometer thermo – genesys 10 UV, USA.

Table 1: Characterization properties of Iraqi rice husk

Chemical Composition	
Compound	Composition wt %
SiO_2	90.7
Al_2O_3	0.13
Fe_2O_3	0.06
TiO_2	0.015
CaO	0.61
MgO	0.25
Na_2O	0.09
K_2O	2.64
P_2O_5	0.73
LOI	4.71
S.A (m^2/g)	17.5

2.2 Sorption unit

Fixed bed column of continuous mode experiments were conducted in order to examine cerium removal by treated SSAS of above cerium at desired concentration with the various bed heights of the IRH (adsorbent media) using different flow rates of SSAS of cerium at various pH. The pH value was adjusted using 0.1 N NaOH and 0.1 N HCl solutions. The sorption unit consists of two glass container for SSAS of cerium one for inlet feed and another for outlet each of (1 liter) capacity. Glass column has 2.54 cm ID and 150 cm height. The sorption column packed with adsorbent media to a height of (10, 20, 30, 40, 50, 60, 70, 80, 90 and 100 cm) supported from the top and the bottom by glass hollow cylinder layer, each cylinder have (0.5 cm ID, 0.1 cm thickness and 1 cm long).

Before starting the runs, the packed bed sorption column was rinsed by double distilled water down flow through the column. The adsorbent media is packed in the column to the desired depth, and fed to it as slurry by mixing IRH with distilled water in order to avoid the formation of air bubbles inside the media. After the packed bed sorption column was accommodation and putting the required amount of adsorbent media, the adsorption process started by allowing the cerium SSAS of required concentration and pH down flow through the sorption column from inlet container by gravity at a precise flow rate in experiment which is adjusted by rotameter. To determination the best operational conditions, the experiments were carried out at a temperature between (20-45°C), various pH values which are (1-8) and initial feed concentrations of SSAS of different cerium which are between (1-100) mg/l each one alone and at different flow rates which are between (5-100) ml/min for SSAS of cerium initial feed concentration. Outlet samples after treatment in each experiment were collected every 10 minutes from the bottom of packed column and the unabsorbed concentration of cerium in SSAS was analyzed by spectrophotometer.

3 RESULTS AND DISCUSSION

The ability of IRH to remove cerium from SSAS in fixed bed column of continuous mode at various parameters which are pH's of SSAS of cerium (pH), height bed of adsorbent media IRH (l), flow rates of SSAS (F), SSAS temperature (T_{feed}) and time of treatment (t) was investigated. The experiments were achieved by varying all above parameters for different initial concentrations (Co) of SSAS of cerium. Thus, the results obtained are explained below.

3.1 Effect of Initial Concentration

The results showed that using adsorbent material, the percent removal of cerium was decreased when the initial concentration (Co) of SSAS of cerium was increased at constant other variables as shown in Figure 1. This can be explained by the fact that the initial concentration of cerium had a restricted effect on cerium removal capacity; simultaneously the adsorbent media had a limited number of active sites, which would have become saturated at a certain concentration. This was lead to the increase in the number of cerium molecules competing for the available functions groups on the surface of adsorbent material. Since the solution of lower concentration has a small amount of cerium than the solution of higher concentration of it, so the percent removal was decreased with increasing initial concentration of cerium. For adsorbent media, higher percent removal was 95.44% for cerium at initial cerium concentration of 1 mg/l, so adsorbent material was found to be efficient to cerium removal from SSAS and wastewater.

3.2 Effect of pH

The results showed that using adsorbent material IRH, the percent removal of cerium was increased when the pH of SSAS of cerium increased too, at constant other variables as shown in Figure 2. This increase can be explained as follows: the pH of the solution affects the surface charge of the adsorbent, degree of ionization along with speciation of different pollutants. The effect pH was very less pronounced in adsorption but had a marked effect on the stability of thorium. At low pH values cerium exists as a weak soluble in water. This affinity of water at low pH prevents its adsorption onto IRH. Also at a higher pH the deprotonation on IRH surface provides functional groups, for chemisorption, on its surface that can undergo ion exchange type of interaction with cerium. In the alkaline conditions free cerium is present predominantly in hydroxide form. Equilibrium adsorption between IRH and cerium indicates that extent of adsorption is not dependant on pH in high pH ranges (Abbas, 2014).

3.3 Effect of Adsorbent Media Bed Height

The results elucidated that when the adsorbent media bed height was increased, the percent removal of cerium was increased too at constant other variables as shown in Figure 3. The increased of bed height meaning increased in the amount of adsorbent media IRH, thus increasing the surface area of adsorbent material, hence increased the number of active sites in the adsorbent material surface i.e. increased the availability of binding sites for adsorption and consequently increase cerium removal capacity on IRH. This lead to increase the ability of adsorbent media to adsorb greater amount of cerium from SSAS at different initial concentrations and ultimately the percent removal of cerium increased.

3.4 Effect of Flow Rate

The results illustrated that when the flow rate of SSAS of cerium was increased, the percent removal of cerium was decreased at constant other variables as shown in Figure 4. This may be due to the fact that when the flow of SSAS of cerium increasing, the velocity of SSAS in the column packed with the adsorbent media IRH was increasing too, so the solution spend shorter time than that spend in the column while at low flow rate, and the SSAS of cerium resides in the column for a longer time, therefore undergoes more treatment with the adsorbent media, thus the adsorbent media uptake low amount of cerium from SSAS of cerium for high flow rate, therefore the percent removal of cerium was decreased when the flow rate was increased.

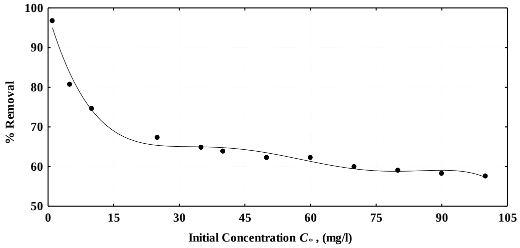

Fig. Effect of initial concentration ($C_{\circ}$) on the percent removal of cerium @ T_f =45°C, l = 1 m, pH=8, t=60 min. and F=5 ml/min

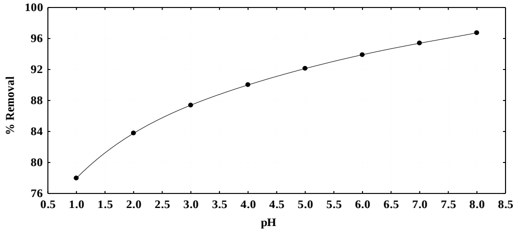

Fig. 2: Effect of pH on the percent removal of cerium @ C_{o}= 1 mg/l, Tf =45°C, l = 1 m, t=60 min. and F=5 ml/min

3.5 Effect of Feed Temperature

The results demonstrated that when the temperature of feed which was SSAS of cerium was increased, the percent removal of cerium was increased too at constant other variables as shown in Figure 5. The effect of temperature is fairly common and increasing the mobility of the acidic ion. Furthermore, increasing temperatures may produce a swelling effect within the internal structure of the adsorbent media enabling cerium ions (Ce+3) to penetrate further. It was indicated that cerium adsorption capacity increased with increasing feed temperature from 20 to 45°C. This effect may be due to the fact that at higher temperature an increase in active sites occurs due to bond rupture.

3.6 Effect of Treatment Time

The results demonstrated that when the treatment time of SSAS of cerium increased the percent removal of cerium increased at constant other variables as shown in Figure 6. This may be due to the fact that when the treatment time of cerium SSAS increasing and the velocity of SSAS in the column packed with the adsorbent material was remaining constant, the solution spend longer time than that spend it when the time of treatment decreased, so the adsorbent material uptake more amount of cerium from SSAS, therefore the percent removal of cerium from SSAS was increased.

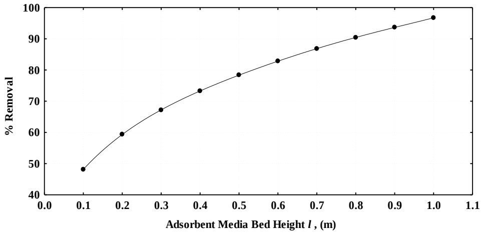

Fig. 3: Effect of adsorbent media bed height (l) on the percent removal of cerium @ $C_\circ$= 1 mg/l, pH=8, Tf =45°C, t=60 min. and F=5 ml/min.

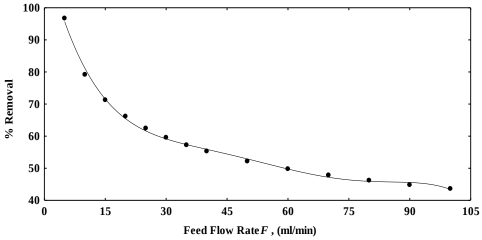

Fig. 4: Effect of SSAS flow rate (F) on the percent removal of cerium @ $C_\circ$= 1 mg/l, pH=8, Tf =45°C, l = 1 m and t=60 min.

4 STATISTICAL MODEL

A statistical model was carried out to the experimental results obtained from this study. Regression analysis and π Theorem was adopted to maintain a relation between the percent removal of cerium and the feed temperature, flow rate, pressure, pH of feed solution, initial concentration of cerium, adsorbent media of IRH bed height, treatment time, column diameter and other parameters. These relations are shown in Table 2 below, which has a correlation coefficient (R^2) 0.9994.

5 PROFITING FROM TREATED RICE HUSK

Huge amount of IRH was lingered after using it in removal of cerium from SSAS as explained above. Utilization from IRH uses can be achieved as a promoter of zeolite catalyst follows:

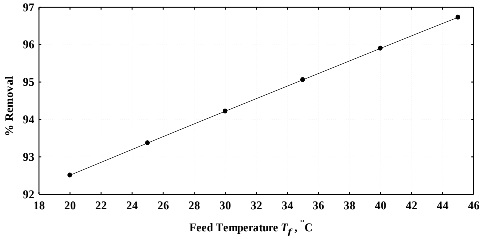

Fig. 5: Effect of feed temperature (Tf) on the percent removal of cerium @ $C_\circ$= 1 mg/l, pH=8, l = 1 m, t=60 min. and F=5 ml/min.

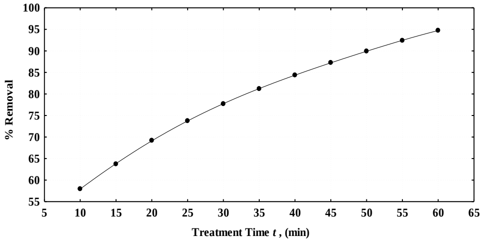

Fig. 6: Effect of treatment time (t) on the percent removal of cerium @ $C_\circ$= 1 mg/l, Tf =45°C, pH=8, l = 1 m, and F=5 ml/min.

Table 2: Statistical equation

metal	Equation	R²
cerium	$\%\mathrm{R} = 1.6751 \times 10^{-7} \left(\frac{\rho_{sol}}{C_\circ}\right)^{0.1225} . \left(\frac{l}{d}\right)^{0.2436} . \left(\frac{T_f . Cp_{sol} . t}{\vartheta}\right)^{0.2555} . \left(\frac{K_{sol} . T_f}{\gamma . u}\right)^{0.250} . (\mathrm{pH})^{0.1021}$	0.9994

Where :-

$\%\boldsymbol{R}$	Percent Removal of Cerium from SSAS
$\boldsymbol{\rho_{sol}}$	Density of SSAS, $(\mathrm{kg/m^3})$
$\boldsymbol{C_\circ}$	Initial Concentration of Cerium , $(\mathrm{kg/m^3})$
$\boldsymbol{l}$	Adsorbent Material Bed Height, (m)
$\boldsymbol{d}$	Internal Diameter of Sorption Column, (m)
$\boldsymbol{T_f}$	Feed Temperature, (K)
$\boldsymbol{C_{P_{sol}}}$	Heat Capacity of SSAS, (kJ/kg. K)
$\boldsymbol{t}$	Treatment Time, (s)
$\boldsymbol{\vartheta}$	kinematic viscosity of SSAS, $(\mathrm{m^2/s})$
$\boldsymbol{K_{sol}}$	Thermal Conductivity of SSAS , (W/m. K)
$\boldsymbol{\gamma}$	Surface Tension, (N/m)
$\boldsymbol{u}$	Velocity of SSAS, (m/s)

5.1 Synthesis of promoted zeolite type Y catalyst from rice husk loaded with Ce^{+3} ions

Iraqi rice husk samples which were adsorbed cerium ions Ce+3 from SSAS at different operating conditions aforementioned was segregated and classified according to its contain of cerium and utilization from these samples as a raw material in the synthesis of promoted catalyst and tested in n-heptane catalytic cracking which is zeolite type Y. The samples give different ratios of cerium ions Ce+3 to IRH.

The ratios were calculated according to mass balance on the fixed bed adsorber and were between (0.05 to 5.5 wt %). IRH samples with different ratios were firstly treated with 10% phosphoric acid (H3PO4) for 24 hours for preliminary removing all impurities. Then they were well washed with double distilled water, filtered, dried in air, and calcined at 750°C for 6 hours. 12 g of calcined IRH were then subjected for dissolution in sodium hydroxide NaOH (4 M) followed by refluxing at 90°C for 12 hours, then concentrated hydrochloric acid (HCl (37%)) was added to the aforementioned base dissolved IRH to complete precipitation. IRH were filtered, washed with excess distilled water to be freeing from chloride ions and finally dried in an oven at 120°C for 6 hours.

Zeolite type Y was synthesized using above prepared IRH as a silica source in the following method. A 500 ml Teflon beaker containing a magnetic stirrer was washed with deionized water. Sodium hydroxide of 1.6616g was added slowly to deionized water and stir until clear and homogenous solution appeared for about 5 minutes. The aqueous solution of sodium hydroxide was ready for the preparation of seed gel. The gel was prepared according to the following molar chemical composition: 10.67 Na2O: Al2O3: 10 SiO2: 180 H2O. Two millilitre aqueous solution of sodium hydroxide was added to 0.7515g sodium aluminate oxide until a homogenous mixture was formed; 1.5361g of above prepared IRH was added separately to 5.5 ml sodium hydroxide aqueous until homogenously mixed. Both of the preparations were heated under vigorous stirring to obtain a homogenous mixture. The sample was aged for 24 hours at room temperature in the Teflon bottle. The aluminate and silicate solutions were mixed together in the polypropylene beaker, subsequently stirred for 2 hours with the purpose of making it completely homogenized. This combined solution was used as the feed stock gel.

The synthesized zeolite type Y which was in sodium (Na+) powder form i.e. Ce+3/NaY-Zeolite, mixed with 15 % montmorillonite clay as a binder then mixed with water to form a paste. A spherical shape (0.5 cm) was formed, dried overnight at 110°C. In order to make a promoted catalyst ready for test in any process, hydrogen zeolite (H-Zeolite) form must be prepared. The Ce+3/HY-Zeolite were prepared by exchanging Na+ ions in the sodium form (Ce+3/NaY-Zeolite) with ammonium chloride solution NH4Cl. In order to obtain ideal degree of ion exchange the technique of multi-steps (three times repeating) was used. Thus, the first step, 2N of ammonium chloride solution (26.75 g of NH4Cl in 250 ml of distilled water) contacted with 90 g of prepared Ce+3/NaY-Zeolite with stirring for 2 hours. In the second step, the procedure in the first step was repeated under the same conditions but on about 60 g of zeolite, which was taken from the total zeolite amount produced in the first step. Finally, in the third step, the procedure under the same conditions was repeated again but on about 30 g of zeolite, which was taken from the total zeolite amount produced in the second step.

The exchanged ammonia zeolite were filtered off, washed with deionized water to be free of chloride ions dried overnight at 120oC and then calcined initially at 150°C for two hours. The temperature was increased 75°C per hour until it reached 550°C and it was held constant for 5 hours at this temperature. During calcination, ammonia and water were liberated and promoted zeolite which is Ce+3/HY-Zeolite was formed [17 and 18].

5.2 Synthesis of normally zeolite type Y catalyst from rice husk only

In the same way illustrated in above section exactly, normally zeolite type Y catalyst was prepared but the raw material was (normally IRH) i.e. IRH without treating with SSAS containing cerium ions Ce+3 [17 and 18].

5.3 Catalytic Activity of Synthesized Catalysts

The promoted and normally type Y zeolite catalyst activity was studied by applying n-heptane catalytic cracking reaction. The n-heptane catalytic cracking reaction was carried out in catalytic cracking unit which is operated under atmospheric pressure and the experiments of catalytic cracking were performed in an experimental fluidized bed unit. The unit consists of n-heptane storage tank, gas flow meter, dosing pump, evaporator, condenser/separator, cooler with appropriate control, and power supply box.

A suitable amount of Ce/HY-zeolite catalyst prepared above was charged in the reactor from the top of it. At preheating section, the feed was preheated at 400°C before entering to catalytic bed. The temperature controlled was setting at desired reaction temperature between (400-550°C) in the reaction zone. The first condenser was supplied with water from chiller and second charged with ice.

A suitable quantity of water was charged to the preheater to remove air from the reactor. This water changed to steam used for air stripping from the reactor.

The temperature in preheater kept at 400°C. After stripping air from the reactor, the flow of water was stopped and began to n-heptane flow at a rate 4.5 ml/min to the reactor for ten minute period. The reaction products were flown through double pipe heat exchanger, internal tube ice bath, separation and collection for liquid storage, while the gaseous products passed through the liquid products and were collected in a vessel over water.

The gas volume is determined by water displacement. A sample of gaseous product was collected and then analyzed by gas chromatography device (GC) (Dani, Model GC 1000 DPC), to determine the chemical composition of the gaseous product results as shown in Table 3. Cracking liquid products also collected in a tube and then analyzed by gas chromatography, to determine the chemical composition of the liquids product. The catalyst was replaced after the end of each experiment.

Table 3: n-heptane catalytic cracking using prepared zeolite catalysts

Catalyst	Temp., °C	Substances (wt%)														
		Methane	Ethane	Propane	Butane	Pentane	Ethene	Propylene	1-Butene	2-Butene	1-Pentene	2-Pentene	1-Hexene	2-Hexene	3-Hexene	n-Heptane (unreacted)
HY-Zeolite	400	7.77	–	–	26.1	–	–	31.9	–	6.57	–	–	6.19	4.41	–	17.1
	425	7.87	–	–	24.2	–	–	32.8	–	6.65	–	–	6.29	4.44	–	17.8
	450	7.96	–	–	22.2	–	–	33.8	–	6.72	–	–	6.39	4.48	–	18.5
	475	8.06	–	–	20.3	–	–	34.7	–	6.79	–	–	6.48	4.51	–	19.2
	500	8.15	–	–	18.3	–	–	35.5	–	6.86	–	–	6.57	4.55	–	20.1
	525	8.24	–	–	16.4	–	–	36.5	–	6.93	–	–	6.66	4.58	–	20.7
	550	8.33	–	–	14.5	–	–	37.4	–	7.00	–	–	6.75	4.62	–	21.4
Ce+3/NaY-Zeolite	400	–	0.13	0.15	–	0.41	0.11	–	0.24	31.8	0.26	33.8	0.25	0.24	0.34	32.3
	425	–	0.14	0.15	–	0.43	0.12	–	0.56	32.1	0.24	35.1	0.28	0.27	0.37	30.2
	450	–	0.16	0.15	–	0.45	0.12	–	0.22	32.1	0.21	36.1	0.25	0.23	0.39	29.6
	475	–	0.19	0.12	–	0.47	0.13	–	0.27	31.5	0.25	37.5	0.23	0.26	0.31	28.8
	500	–	0.18	0.14	–	0.45	0.15	–	0.25	33.1	0.28	38.1	0.22	0.23	0.30	26.6
	525	–	0.17	0.13	–	0.42	0.18	–	0.21	31.8	0.21	32.8	0.29	0.21	0.38	33.2
	550	–	0.11	0.17	–	0.41	0.16	–	0.23	30.3	0.28	20.3	0.25	0.22	0.31	47.3

6 CONCLUSIONS

The following conclusions can be drawn:

1. IRH showed a good ability to remove cerium from SSAS using fixed bed adsorption unit. So, it could be recommended for removal of cerium from wastewater instead of other material because it is valid, cheaper, economical, easy and simplicity for using, and has a high ability to adsorb cerium, can be used several times by costly regeneration method and can be used finally in another benefit use.
2. Maximum removal of cerium was 95.44% at initial cerium concentration of 1 mg/l.
3. The percentage removal of cerium was increased with decreeing flow rate of SSAS, and initial concentration of cerium while the percentage removal was decreased

with increasing of pH, treatment time and the height of adsorbent material IRH.

4. It can be prepared a catalyst promoter for cracking process of n-heptane from the residual samples of IRH that adsorb cerium from aqueous solution to produce a fuel of higher octane number and remove the hazards radioactive waste in economic and eco-friendly method.

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