

## Treatment of radioactive liquid waste using Iraqi geological raw materials and zeolite

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### Abstract

A radioactive liquid waste contaminated with cesium-137 was found underneath a destructed radioactive waste treatment station in Tuwaytha site 25 km south of Baghdad - Iraq.

The appropriate sorbent materials found for the removal of radio cesium-137 were bentonite and modified bentonite with corresponding removal efficiencies of 85% and 91.8% respectively. The favorable batch experimental conditions were found to be 800 rpm stirring speed, 200 mg sorbent mass, 45 $\mu$ m sorbent particle size, and solution temperature of 30 °C for an initial radio cesium concentration of 0.44 mg/l.

The BET isotherm model was found to represent the experimental results very well.

**Keywords:** cesium-137, geological materials, sorption, isotherm models

### Introduction

The radioactivity of contaminants threatens the human environment seriously. As one of the most important fission radionuclide from the reactor, cesium-137 is always paid considerable attention due to its gamma radiation, half-life of 30 years. It is harmful for human health and environment [1-6]. There is an increasing effort for removing highly soluble radio- contaminants from aqueous waste solutions by fixing them onto solid waste forms that can be disposed of in a repository [7, 8]. Solvent extraction,

precipitation, volatilization, membrane technology, and inorganic ion exchange are usually used for the decontamination of cesium-137 from the radioactive wastewater [9]. Among these methods, inorganic ion exchange is more convenient and suitable candidate for the moderate or low radioactive wastewater because of its specific selectivity, radiation, and thermal stabilities [9]. Smectic clays, always known as bentonites and used in a variety of industrial applications, are the most

widely utilized and most interesting group of clay minerals. However, their real

potentialities can only be developed by subjecting them to chemical treatments. A montmorillonite clay mineral modified by alkaline hydrothermal could further improve the adsorption capacity of Cs<sup>+</sup> [10]. Montmorillonite has excellent sorption properties and possesses available sorption sites within its interlayer space as well as large surface area and more narrow channels inside. It was ever used for the removal of heavy metals and proved to have large adsorption capacity [11]. It was also showed more efficient adsorption for methylene blue [12]. However, few investigations have been involved in the adsorption of Cs<sup>+</sup> by montmorillonite in low radioactive wastewater until now, though it has been widely used for the adsorption of various heavy metal ions or organic matters with efficient sorption ability [13, 14].

The objective of this study is to decontaminate the radioactive liquid waste contaminated with cesium-137. The adsorption of Cs<sup>+</sup> and the effects of experimental conditions such as liquid-solid ratio, stirring speed, particle size, sorbent mass, and temperature on adsorption by zeolite and other Iraqi geological raw materials such as porcellanite, kaolin, and bentonite.

### Experimental arrangements and procedure

#### Materials

**Sorbents:** The sorption of radio cesium has been investigated onto three Iraqi geological raw materials (porcellanite, kaolin, and bentonite) as well as zeolite. The materials are analyzed using XRF at the Earth Science Institute, Mainz University, Germany; the chemical compositions of these materials are listed in Table (1):

oxide	porcellanite wt%	zeolite wt%	bentonite wt%	kaolin wt%
SiO <sub>2</sub>	40.89	36.96	54.26	49.50
Al <sub>2</sub> O <sub>3</sub>	12.59	29.19	14.87	32.00
CaO	18.58	1.35	5.53	0.75
Fe <sub>2</sub> O <sub>3</sub>	0.75	0.96	4.94	1.50
Na <sub>2</sub> O	0.27	12.24	0.98	0.40
MgO	1.48	0.14	3.80	1.20
K <sub>2</sub> O	0.20	0.10	0.38	0.15
TiO <sub>2</sub>	0.49	1.71	0.75	1.20
P <sub>2</sub> O <sub>5</sub>	0.40	0.02	0.30	0.24
SO <sub>3</sub>	0.30	0.67	1.39	1.52
LOI	23.16	17.05	12.80	11.54
Sum	99.86	100.01	100.00	100.00

Materials were pulverized using jaw crusher (type Retch-Germany) then grinded and sieved into various particle sizes (45, 63, and 75µm). The powder of each material is dried in an oven (type Heraeus-Germany), the temperature and time for drying sorbents are listed in Table (2):

Sorbent material	Drying temperature (°C)	Drying time (min)
porcellanite	100	480
kaolin	80	30
zeolite	100	480
bentonite	100	30

The results in Table (2) are found from the experiments conducted in the Iraqi state company for geological survey and mining to find the appropriate temperature for drying the sorbents.

All materials were not pretreated except bentonite, which was modified by washing with 6 wt% soda ash (Na<sub>2</sub>CO<sub>3</sub>); the mixture was left for three days (a proposed experiment conducted in the Iraqi state company for geological survey and mining).

The specific surface area for sorbent materials is influenced by several parameters such as particle size, sorbent structure, sorbent type, etc., the specific surface area for porcellanite, kaolin, zeolite, and bentonite are taken from the Iraqi state company for geological survey and mining to be 27, 203, 670, and 700 m<sup>2</sup>/gm respectively.

**Sorbate:** Ten liters of contaminated liquid were taken from a place located underneath the destructed RWTS in Tuwaytha site (February 2009). The liquid was analyzed using gamma spectroscopy system (type Canberra-USA) with a high purity Germanium detector. The sample showed an activity concentration of 278 Bq per liter (0.44 mg/l) which lies within the allowable concentration for cesium up to 1.2 mg/l [15].

### Equipments:

Gamma spectroscopy system based on high purity Germanium detector (HpGe) is used to determine the activity concentration of cesium-137. This is connected with multi-channel analyzer with 8192 channel (type Tandy-USA) which allows data acquisition, storage, and display with an online analysis using Genie 2000 program [15].

Atomic absorption system, flame emission type is used to determine cesium-137 concentration. Standard solutions were prepared from 1000 ppm cesium chloride (Stockes' solution) at which 1.267 gm CsCl was dissolved into 100 ml deionized water with an electrical conductivity of 2.1 µSiemens/cm then diluted up to 1000 ml and placed in polypropylene vial [16].

### Experimental procedure

#### Sorption isotherm

The batch method was used to find the sorption of cesium-137 ions onto porcellanite, kaolin, zeolite, bentonite, and modified bentonite sorbents. The sorption behaviors were investigated using various experimental parameters such as sorbent particle size, sorbent mass, and stirring speed.

A hundred ml of radioactive liquid waste was diluted with 400 ml deionized water. The solution of certain mass of specified particle size of sorbent was placed in a flask and stirred for 3 hours. Equilibrium was attained at 20 minutes for all sorbents except porcellanite was 120 minutes. After stirring, the solution was left to settle and the supernatant was filtered, the filtrate was placed into a centrifuge running at 10000 rpm for 30 minutes, the clear liquid was analyzed with gamma spectroscopy and atomic absorption systems.

The milligrams of sorbate per milligrams of sorbent can be calculated from the following equation [17]:

$$x/m = (C_o - C_e) V/m \quad (1)$$

## Distribution coefficient

Gamma radioactivity remaining in the supernatant was measured. The distribution coefficient was calculated from the difference in gamma counting rates using the following equation [18]:

$$K_d = \frac{A_0 - A_f}{A_f} \frac{V}{m} \quad (2)$$

## Results and discussion

### Kinetics of sorbent types and masses

The plot of cesium concentration versus stirring time with masses 150, 300, 450, and 600 mg for porcellanite and with masses 50, 100, 150, and 200 mg for kaolin, zeolite, bentonite, and modified bentonite are shown on Figures (2-6) respectively.

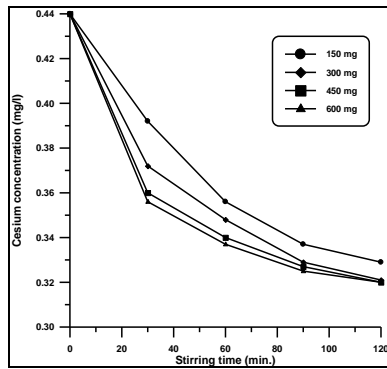


Fig (1) Effect of porcellanite mass on cesium concentration reduction,  $N = 800$  rpm and  $P.S = 45 \mu m$

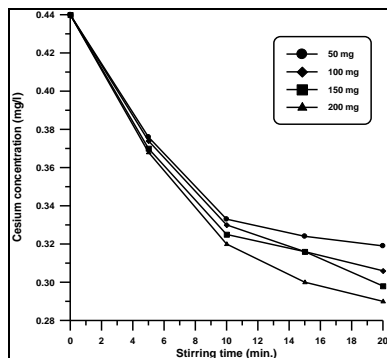


Fig (2) Effect of kaolin mass on cesium concentration reduction,  $N = 800$  rpm and  $P.S = 45 \mu m$

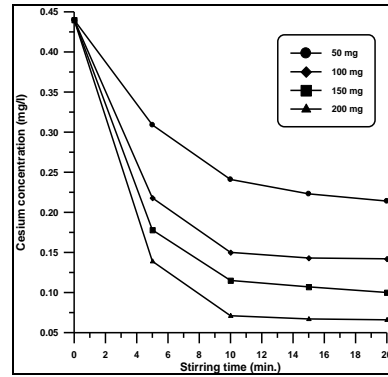


Fig (3) Effect of zeolite mass on cesium concentration reduction,  $N = 800$  rpm and  $P.S = 45 \mu m$

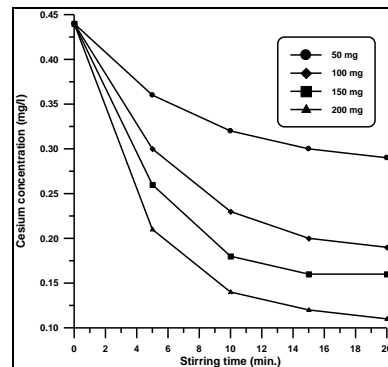


Fig (4) Effect of bentonite mass on cesium concentration reduction,  $N = 800$  rpm and  $P.S = 45 \mu m$

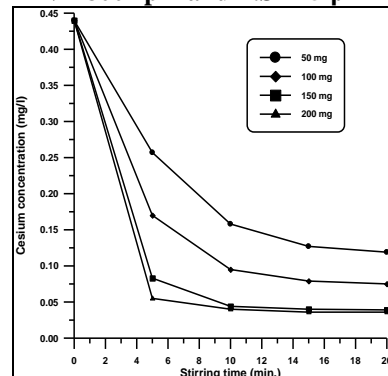


Fig (5) Effect of modified bentonite mass on cesium concentration reduction,  $N = 800$  rpm and  $P.S = 45 \mu m$

Figure (1) shows a decrease in cesium concentration with an increase in stirring time until 120 minutes for porcellanite while Figures (2-5) show a decrease in cesium concentration with an increase in stirring time until 20 minutes for kaolin, zeolite, bentonite, and modified bentonite. Table (3) shows the cesium removal efficiencies for various sorbents:

**Table (3): Cesium removal efficiencies for various sorbents at 800 rpm and P.S = 45  $\mu\text{m}$**

Sorbent	porcellanite	kaolin	zeolite	bentonite	modified bentonite
Mass (mg)	% Cesium removal				
50	-	26.0	34.0	51.4	72.9
100	-	30.3	57.0	67.7	83.0
150	26.0	32.0	64.0	77.3	91.1
200	-	34.0	75.0	85.6	91.8
300	27.0	-	-	-	-
450	27.0	-	-	-	-
600	27.0	-	-	-	-

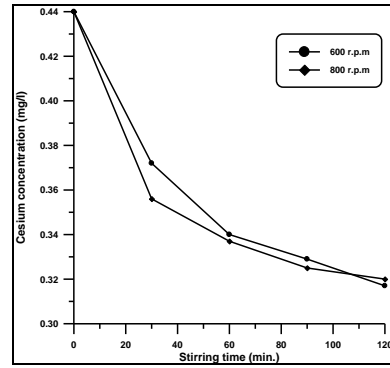
Examining Figures (1-5) and Table (3), it can be seen that the order of cesium removal efficiency for various sorbents is: porcellanite < kaolin < zeolite < bentonite < modified bentonite.

This is due to:

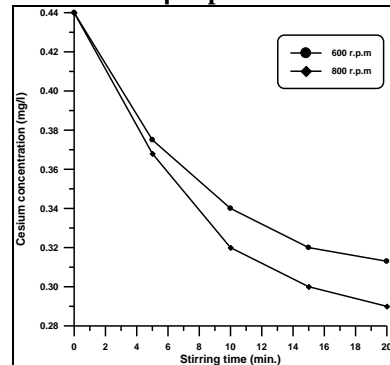
1. Different specific surface area for sorption which are 27, 203, 670, and 700  $\text{m}^2/\text{gm}$  for porcellanite, kaolin, zeolite, and bentonite respectively. The larger surface area means the higher capacity for successful cesium removal.
2. The increase in sorbent mass will consequently increase the sorption capacity.
3. Montmorillonite constituent in the clay which has had high affinity for cesium.

### Effect of stirring speed

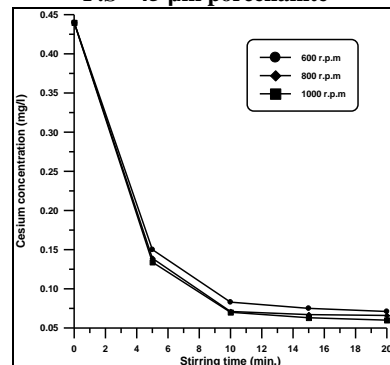
Experimental tests were carried out to find the appropriate stirring speed for the cesium concentration reduction, it is obvious from Figures (6-8) that the appropriate stirring speed are 800 rpm with 600 mg porcellanite, 800 rpm with 200 mg kaolin, 1000 rpm with 200 mg bentonite with corresponding cesium removal efficiencies of 27, 34, and 85.6% respectively; similarly 800 rpm with 200 mg zeolite and 1000 rpm with 200 mg modified bentonite are the appropriate stirring speed with corresponding cesium removal efficiencies of 75 and 91.8% respectively. The higher stirring speed affects the rate of mass transfer from liquid phase to solid phase; at which a decrease in boundary layer thickness that surrounds the solid i.e. decreasing the resistance to mass transfer and consequently causing maximum mass loading from the bulk of solution into the solid and hence both surface diffusion and pore diffusion will be more dominant other than film diffusion.



**Fig (6) Effect of stirring speed on cesium concentration reduction, mass 600 mg and P.S= 45  $\mu\text{m}$  porcellanite**



**Fig (7) Effect of stirring speed on cesium concentration reduction, mass 600 mg and P.S= 45  $\mu\text{m}$  porcellanite**



**Fig (8) Effect of stirring speed on cesium concentration reduction, mass 200 mg and P.S = 45  $\mu\text{m}$  bentonite**

Table (4) shows cesium removal efficiency for various sorbents:

**Table (4): Cesium removal efficiency for different particle sizes and various sorbents at 800 rpm stirring speed and 200 mg sorbent mass**

Sorbent	porcellanite	kaolin	zeolite	bentonite	modified bentonite
Particle size (µm)	% Cesium removal				
75	-	15.5	-	36.1	-
63	-	19.1	-	41.6	-
45	27.0	34.0	75.0	85.6	91.8

It is obvious that decreasing sorbent particle size will increase cesium removal efficiency due to the increase in surface area of small particles compared with a large one for a given unit mass.

Examining all Figures and Tables discussed above, it can be concluded that the appropriate sorbents for cesium removal is bentonite and modified bentonite, and the favorable experimental conditions are 1000 rpm stirring speed, 200 mg sorbent mass, and 45 µm sorbent particle size (For initial cesium concentration of 0.44 mg/l). Therefore, these sorbents and their experimental parameters are recommended and utilized for the removal of cesium from the radioactive liquid waste.

#### Batch equilibrium isotherms:

The most common equilibrium isotherm models used in sorption experiments are:

- (i) Langmuir sorption model:

$$\frac{x}{m} = \frac{abCe}{1 + bCe} \quad (3)$$

Linearization of equation (3) is:

$$\frac{Ce}{(x/m)} = \frac{1}{ab} + \frac{1}{a}Ce$$

- (ii) Freundlich sorption model:

$$\frac{x}{m} = K(Ce)^{1/n} \quad (n > 1) \quad (4)$$

Linearization of equation (4) is:

$$\ln\left(\frac{x}{m}\right) = \ln K + \frac{1}{n} \ln Ce$$

- (iii) BET sorption model:

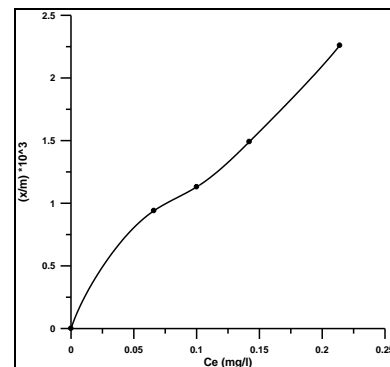
$$\frac{x}{m} = \frac{\alpha\beta Ce}{(C_s - Ce) \left[ 1 + (\beta - 1) \frac{Ce}{C_s} \right]} \quad (5)$$

Linearization of equation (5) is:

$$\frac{Ce}{\left(\frac{x}{m}\right)(C_s - Ce)} = \frac{1}{\alpha\beta} + \left(\frac{\beta - 1}{\alpha\beta}\right) \frac{Ce}{C_s}$$

The sorption of cesium onto bentonite and modified bentonite at concentration  $C_0 = 0.44$  mg/l,  $N = 800$  rpm, and 45 µm particle size has been conducted at constant temperature 30 °C. Plotting  $x/m$  versus  $Ce$  for bentonite and modified bentonite is shown on Figures (9 and 10). These Figures show that the sorption rate increases significantly when cesium concentration at equilibrium approaches its saturation value i.e. maximum mass loading occurs near saturation concentration of 0.40 and 0.17 mg/l (values were determined using trial and error for BET isotherm equation when successive substitutions for  $Ce/C_s$  corresponding to maximum linear correlation coefficient) for bentonite and modified bentonite respectively.

The three equilibrium isotherm models were fitted with the experimental data as shown on Figures (11- 16):



**Fig (9) Sorption of cesium onto bentonite**

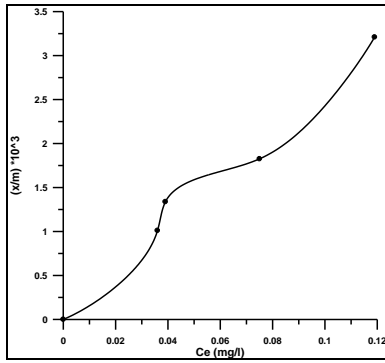


Fig (10) Sorption of cesium onto modified bentonite

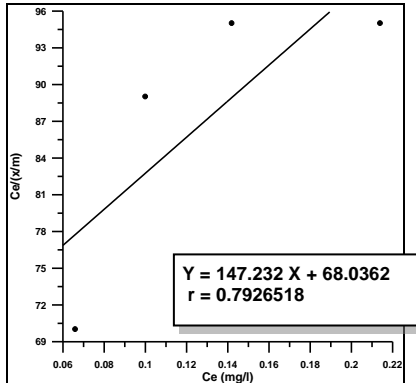


Fig (11) Langmuir plot of  $Ce/(x/m)$  vs.  $Ce$  for the sorption of cesium onto bentonite

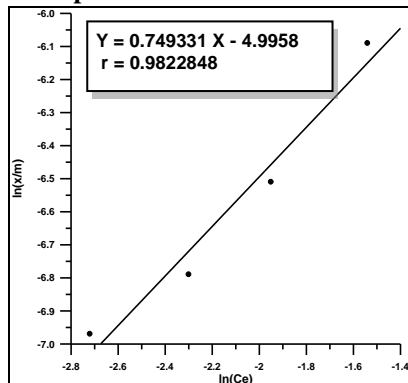


Fig (12) Freundlich plot of  $\ln(x/m)$  vs.  $\ln(Ce)$  for the sorption of cesium onto bentonite

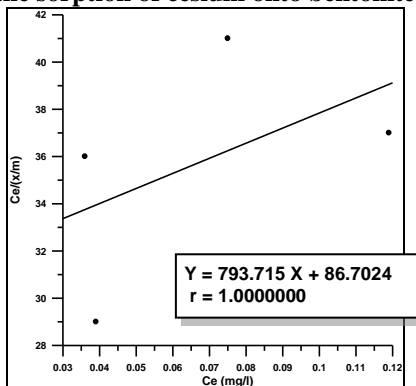


Fig (13) BET plot of  $Ce/((Cs-Ce)(x/m))$  vs.  $(Ce/Cs)$  for the sorption of cesium onto bentonite

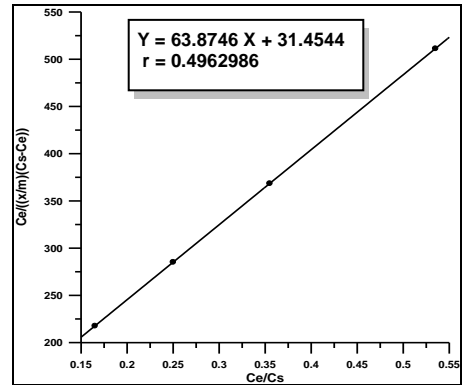


Fig (14) Langmuir plot of  $Ce/(x/m)(Cs-Ce)$  vs.  $Ce/Cs$  for the sorption of cesium onto modified bentonite

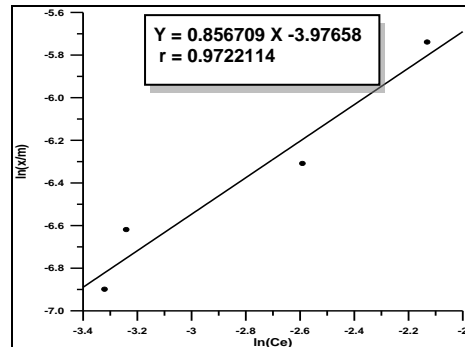


Fig (15) Freundlich plot of  $\ln(x/m)$  vs.  $\ln(Ce)$  for the sorption of cesium onto modified bentonite

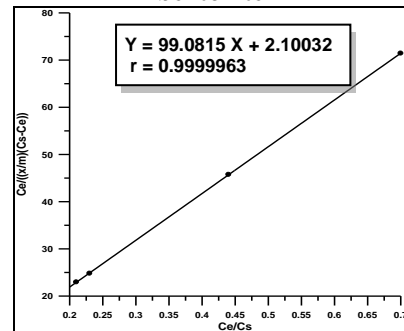


Fig (16) BET plot of  $Ce/((Cs-Ce)(x/m))$  vs.  $(Ce/Cs)$  for the sorption of cesium onto modified bentonite

The constants of each model with their correlation coefficients are listed in Tables (5) and (6)

**Table (5) Equilibrium isotherm model constants and correlation coefficients at  $N = 800$  rpm,  $P.S = 45 \mu m$ , mass 200 mg bentonite, and temperature  $30^\circ C$**

Isotherm model	Isotherm model constants						Linear correlation coefficient
	a	b	$\alpha$	$\beta$	K	n	
Langmuir	0.00687	2.1344	-	-	-	-	0.793
Freundlich	-	-	-	-	0.00676	1.34	0.982
BET	-	-	0.001136	10.15	-	-	1.000

**Table (6) Equilibrium isotherm model constants and correlation coefficients at N = 800 rpm, P.S = 45 μm, mass 200 mg modified bentonite, and temperature 30 °C**

Isotherm model	Isotherm model constants						Linear correlation coefficient
	a	b	$\alpha$	$\beta$	K	n	
Freundlich	-	-	-	-	0.01875	1.17	0.972
BET	-	-	0.009878	52.55	-	-	0.999

Tables (5 and 6) show that the BET isotherm model has a high correlation coefficient compared with the other models, therefore BET isotherm model is adopted and recommended for bentonite and modified bentonite

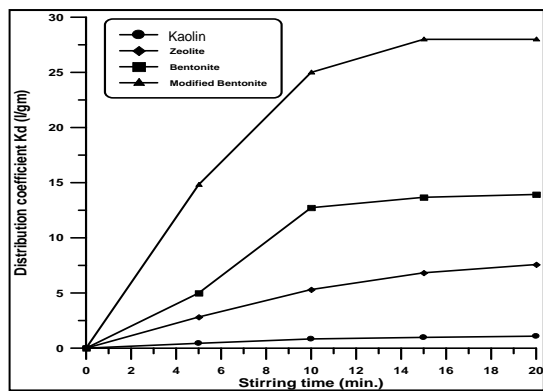
**Distribution coefficient for sorbent materials:**

The distribution coefficient for various sorbent materials used in the present experiments was calculated using equation (2). The results are listed in Table (7):

**Table (7) Distribution coefficient for various sorbent materials**

Material	porcellanite	kaolin	zeolite	bentonite	modified bentonite
Kd (l/gm)	0.336	1.083	7.600	13.900	28.00

Distribution coefficient was plotted against stirring time, Figure (17), for porcellanite, kaolin, zeolite, bentonite and modified bentonite at N = 800 rpm, particle size 45 μm, and 200 mg sorbent.



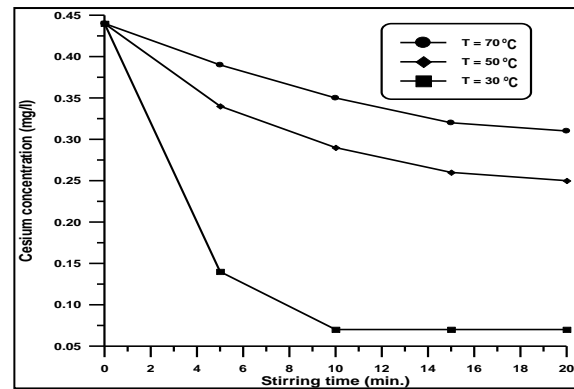
**Fig (17) Effect of stirring time on sorption of cesium onto various sorbents**

Figure (17) and Table (7) show the effect of stirring time on the distribution coefficient of cesium onto porcellanite, kaolin, zeolite, bentonite and modified bentonite. The sorption in terms of the distribution coefficient first increases with stirring time, and then eventually approaches an equilibrium value; this is because of the mass

transfer phenomenon at which the transfer is directly proportional to the driving force (Co-C) which initially attains its maximum value. This force decreases gradually with time, thus the rate of sorption is maximum at the beginning and then tends to level out at subsequent time due to the decrease of such driving force whenever concentration increases with time. It can be seen that sorption equilibrium was approximately attained after twenty minutes; therefore, a twenty minutes was employed throughout all batch experiments. Relatively larger Kd value was obtained for modified bentonite since this material contained montmorillonite having high affinity for cesium. The sorption of cesium onto bentonite was primarily due to its constituent of montmorillonite.

**Effect of temperature on sorption of cesium onto bentonite:**

Sorption behavior of cesium onto bentonite was studied by varying the experimental temperatures at N = 800 rpm stirring speed, 45 μm sorbent particle size, and 200 mg bentonite. Examining Figure (18) shows that the cesium concentration decreases rapidly with a decrease in temperature and the cesium removal decreased from 85% at temperature 30 °C to 30% at temperature 70 °C, this implied that sorption is exothermic and hence decreased with increasing temperature.



**Fig (18) Effect of temperature on sorption of cesium onto bentonite**

## Conclusions:

- Bentonite was found to be a preferable sorbent for cesium removal compared with porcellanite, kaolin, and zeolite.
- Bentonite when modified with soda ash lead to an increase in sodium cations at the expense of calcium cations and indicated significant increase in cation exchange capacity.
- The favorable batch experimental conditions were 1000 rpm stirring speed, 45  $\mu\text{m}$  sorbent particle size, 200 mg sorbent mass, and solution temperature of 30  $^{\circ}\text{C}$  for initial radio cesium concentration of 0.44 mg/l.
- BET isotherm model was found to represent the batch experimental results very well.

## Nomenclature

Symbol	Description	Unit
a	Adsorption equilibrium constant defined by Langmuir equation	mg/mg
$A_o$	Activity concentration before treatment	Bq/l
$A_f$	Activity concentration after treatment	Bq/l
$\alpha$	Sorption constant for BET equation	mg/mg
b	Langmuir constant	l/mg
$\beta$	Constant for BET equation	l/mg
Bq	Radioactivity unit	dps
Ce	Equilibrium concentration of sorbate	mg/l
Co	Initial concentration of sorbate	mg/l
Cs	Saturation concentration of sorbate	mg/l
K	Freundlich sorption coefficient	mg/mg
Kd	Distribution coefficient	l/gm
m	mass of sorbent	mg
n	Freundlich sorption intensity	mg/l
N	Stirring speed	rpm
r	Linear correlation coefficient	-----
V	Volume of solution	l
x	mass of solute sorbed onto a sorbent	mg

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## معالجة النفايات السائلة المشعة باستخدام مواد جيولوجية خام عراقية و الزيولايت

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### الخلاصة

هنالك نفايات سائلة مشعة تحتوي على السيزيوم - 137 وجدت تحت انقاض محطة معالجة النفايات المشعة المدمرة في موقع التويثة والذي يبعد 25 كم جنوب بغداد في العراق. ان البنتونايت و البنتونايت المحور هو من انسب المواد المازة والتي تقوم بإزالة السيزيوم المشع بكفاءة إزالة 85% و 91.8% على التوالي. ان الظروف التجريبية المفضلة هي 800 دوره في الدقيقة كسرعة خلط, 200 ملغم من المادة المازة, 45 مايكرومتر للحجم الحبيبي للمادة المازة, درجة حرارة المحلول 30 مئوي لتركيز أولي للسيزيوم المشع 0.44 ملغم لكل لتر. يعتبر نموذج (BET) من افضل النماذج التي تمثل النتائج التجريبية بشكل جيد.