Treatment of Wastewater by Cement Kiln Dust

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Abstract

The use of cement kiln dust (CKD) as an inexpensive, abundant adsorbent for removing Cr^{+3} from simulated wastewater was investigated. CKD was obtained from Al-Kufa Cement Factory in Al-Najif Government.Experiments for adsorption/precipitation mechanism for Cr^{+3} were performed. The results show that substantial adsorption occur at pH values less than 4 for Cr^{+3}. A set of isothermal batch experiments were conducted in order to investigate the performance of CKD towered the adsorption of the Cr^{+3} ion from the aqueous solution. The results show that the best conditions for Cr^{+3}adsorption on CKD were 60 min, 200 rpm, 20 g/L CKD and pH value 4.

The adsorption isotherm curves show that the adsorption is of a favorable type. A good fitting was obtained between the adsorption isotherm Langmuir model and the Cr^{+3} experimental data. The maximum sorption capacity q_m for Langmuir model equal to 28.5mg/g CKD for Cr^{+3}.

1. Introduction

Cement kiln dust (CKD) is a solid waste which is regard as a problematical issue (USEPA 1993). Cement kiln dust is a fine powder produced as a byproduct from cement plant. It is gathered from the dust collectors during the manufacturing of cement clinker. The composition of cement kiln dust based on: first, the materials that used to make the clinker, second, at the type of fuel used in the rotary kiln. CKD consist mainly of CaO, SiO_2, Fe_2O_3, Al_2O_3 and MgO. The mixture of cement kiln dust with water is very basic; it has a pH of 12.

Cement kiln dust are used in soil stabilization, cement replacement, asphalt pavement and as a raw material for cement production.

Cr^{+3} is one of the unlikely toxic heavy metals which affects human health and can buildup in the food chain, which leads to several illness. Cr^{+3} are disposed of to the environment by many of industries mainly from leather tanning and chrome plating. The maximum levels for Cr^{+3}in wastewater are 5mg/L, [2].

Many methods exist for treatment of heavy metals, these methods comprises of ion exchange, adsorption, chemical precipitation, electro deposition and membrane filtration. Many of these methods undergo disadvantages, such as disposable of the residual sludge and a high capital and–or operational cost. For that the use of low cost and efficient adsorbent materials is desired.

Activated carbon considered as a universal adsorbent, it is undoubtedly been the most used adsorbent in wastewater treatment. Yet its higher cost restricts its application. Therefore attention has shifted towards the use of non-conventional naturally occurring materials, agricultural and industrial waste products as low-cost adsorbents for water remediation, [3];[4];[9].

Cr^{+3} in aqueous solution at different pHs originate a varieties of complexes.

Figure (1) shows a speciation diagram for Cr^{+3}, the diagram was constructed by using the below reactions, [10];[5].

$Cr^{3+} + H_2O \leftrightarrow Cr(OH)^{2+} + H^+$ $(\log K = -4)$ … (1)

$Cr^{3+} + 2H_2O \leftrightarrow Cr(OH)_2{}^+ + 2H^+$ $(\log K = -9.65)$ … (2)

$Cr^{3+} + 4H_2O \leftrightarrow Cr(OH)_4{}^- + 4H^+$ $(\log K = -27.4)$ …(3)

$Cr^{3+} + 3H_2O \leftrightarrow Cr(OH)_{3(s)} + 3H^+$ $\log K = -12)$ ...(4)

The mechanism of adsorption in equation (1) a proton and $Cr(OH)^{2+}$ were generated , they are responsible to make the solution of Cr^{+3} acidic. From this reaction it can be realized that if $Cr(OH)^{2+}$ adsorbs, the reaction directed to the right then the solution becomes more acidic, and if Cr^{+3} is adsorbed, the reaction directed to the left, cause a depletion of protons then the solution become more basic, [7].

According to Fig. 1, the prevailing species below pH 2 is Cr^{+3}. At this pH, almost no adsorption of Cr^{+3} were obtained. This result has been related to the competition between the Cr^{+3} and the protons for the present binding sites on the adsorbent.

At pH 4.0, the dominant species were $Cr(OH)^{2+}$ (50%) and Cr^{3+} (50%). At pH 5 the $Cr(OH)^{2+}$ species prevalent for nearly 70%, at pH 6.0 the dominant species were $Cr(OH)_2^{+}$ (65%) and $Cr(OH)^{2+}$ (35%). Thus the highest capacity of adsorption was reached at pH range (4–6), where the chromium species are cationic. If the adsorbent surface presented a negative charge then the adsorption of Cr^{+3} is an attractive electrostatic interaction between the acid sites on the adsorbent surface and the Cr^{+3}cations. [8]. reported that Cr^{+3} is not adsorbed at pH below 2, and it precipitate at pH values above 6.4.

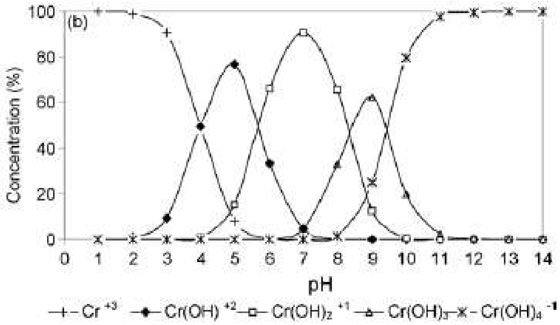

Fig 1. Speciation diagram illustrate the distribution of the chromium species at different pH values in aqueous solution, [5].

The objectives of this work are to study the possibility of using cement kiln dust (CKD) as inexpensive adsorbent for the removal of Cr^{+3} from aqueous environment.

The adsorption/precipitation experiments were studied for Cr^{+3} ion in aqueous solution. The effect of different conditions on the performance of adsorption in batch experiments, the contact time, pH, rpm, CKD dose and initial concentration were analyzed.

2. Experimental Arrangement and Procedures

2.1 Materials

A stock solution of chrome ions with concentration of 500 mg/L were prepared using $Cr(NO_3)_2.9H_2O$ salts by adding 3.926 g to 1 litter of distilled water . The prepared solution was kept at room temperature and used as stock solution to prepare the desired concentrations after dilution with distilled water.

Dissolved metal concentrations in solution were analyzed by atomic absorption spectrophotometer (AAS) (GBC 933 plus, Australia) available at Environmental Engineering Department/Collage of Engineering/University of Baghdad.

A Cement kiln dust was used as an adsorbent in the present work. It was obtained from Al-Kufa Cement Factory at Al-Najif Government. The chemical composition of the CKD constituents was performed in the lab of Geological Survey Company and is listed in Table. 1.

Scanning electron microscopy (SEM) technology shows the shape and surface morphology of the adsorbent. The test was done using the SEM device, (FEI, INSPECT S50, Japan), available at the Department of Applied Sciences Laboratory/ University of Technology/ Baghdad. Fig. 2 (a, b) shows different images of SEM test. From those images the SEM test showed that the CKD have irregular surface and many pores that may detained the adsorbate.

Table 1. X-ray analysis of CKD

SiO_2	9.92
Al_2O_3	5.22
Fe_2O_3	1.92
CaO	39.32
MgO	3.23
SO_3	9.15
Loss on Ignition	24.64
Total	93.4

3. Experimental procedure

3.1. Precipitation/ adsorption experiments

One gram of CKD was placed in 250ml conical flask, and then 50mL of distilled water was added. The inherent pH was measured by WTW pH meter (type 7110) to be 11.5. A drop of concentrated HNO_3 was added until the pH value of the solution dropped to a value of 4. This procedure may last for one hour. After that 50mL of Cr^{+3} solution was added to maintain the solution at a desired concentration (65mg/L), 10mL samples were taken every 60min to measure the concentration of the solution. Also the pH value was measured every 60min. To measure the concentration, the supernatant separated from the CKD by using filter paper of type Whatman No.1, then analyzed using AAS.

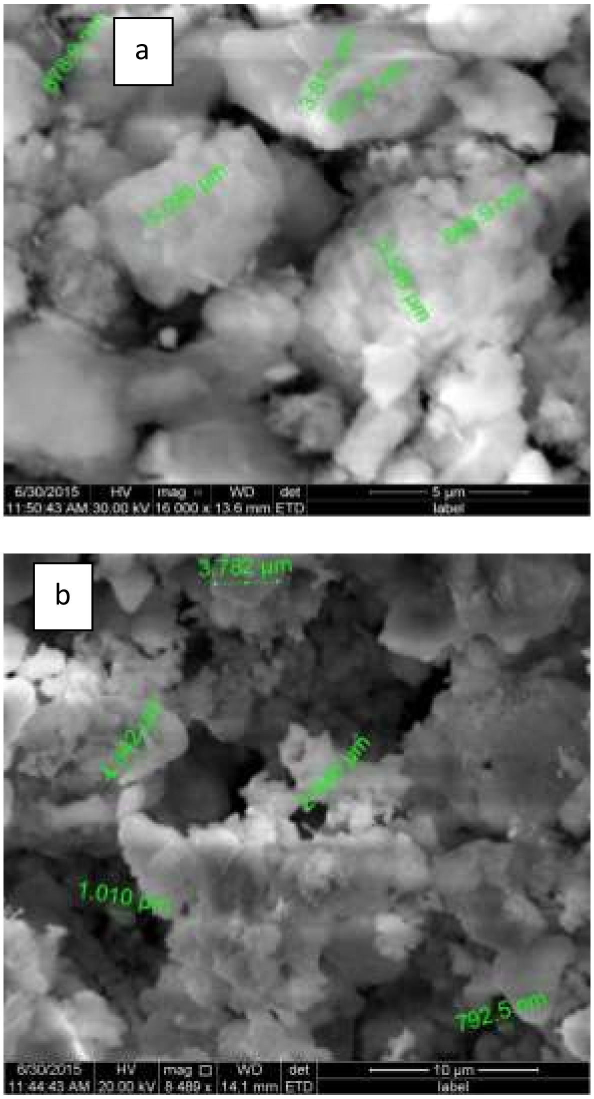

Fig 2. Different magnification images of SEM test.

The pure precipitation was measured by conducting other experiments without adding the CKD as follows:

100mL of Cr^{+3} solutions (65mg/L) was placed in five flasks of 250ml size. Each solution in the flasks was adjusted to the desired pH values, 4, 5, 5.5, 6, and 7 by adding few drops of HNO_3. The solution was shaken at 250rpm for 180 min, after that the concentration was measured by AAS.

3.2. Effect of contact time, agitation speed and CKD dosage

One gram of CKD was placed in 250ml conical flask, and then 50mL of distilled water was added. The pH of the solution was maintained at a value of 4 by adding drops of concentrated HNO_3. After that 50mL of Cr^{+3} solution was added to maintain the solution at a desired concentration (65mg/L), then the adjustment of the pH to a value of 4 was performed if needed. The mixture was shaken at 200rpm for 120min. 10mL samples were taken every 30min to measure the concentration and to see the effect of contact time.

The concentration of the supernatant separated from the CKD by using filter paper of type Whatman No.1 then analyzed by AAS.

The same procedure was conducted for the experiments used to examine the effect of applying different agitation speeds by varying the speed of agitation from 100 to 300 rpm. To examine the effect of using different CKD dosage on the percentage removal, the dosage was varied from 0.5 to 3 g.

3.3. Adsorption isotherm experiment

Adsorption isotherm curve was plotted between q_e vs. C_e at different concentration (10-200mg/L) of Cr^{+3} ion in aqueous solution. The pH of solution was maintained at 3.5 throughout the period of the experiments which lasted for 180min to insure adsorption mechanism.

Two gram of CKD was placed in five conical flasks each of 250mL. 50mL of distilled water was added to each flask. Drops of concentrated HNO_3 were added until the pH of solution adjusted to pH value of 3.5. After that 50mL of Cr^{+3} solution was added to maintain the solution at a desired concentration namely ; 10, 25, 50, 100 and 200mg/L, then the adjustment of the pH to a value of 3.5 was performed if needed. The mixture was shaken at 200rpm for 180min.

The pH of the solution in the flasks were measured at 60min intervals and adjusted to the initial pH 3.5 before the experiment was continued. After 180 min, the samples were taken and filtered using filter paper of type Whatman No.1 then analyzed to measure the concentration of the remaining Cr^{+3} ions by AAS.

q_e is calculated using the following equation:

$$q_e = \frac{V_L(C_o - C_e)}{W_A} \qquad \text{........ (5)}$$

Where, q_e is the weight of Cr^{+3} adsorbed per unit weight of CKD (mg/g), V_L is the volume of the sample (L) , W_A is the weight of the CKD (g) , C_o and C_e (mg/L) are the initial and equilibrium concentration respectively.

4. Results and Discussion

4.1. Precipitation test for chromium

Preliminary test was performed to examine the effect of pH on the precipitation of Cr^{+3} in aqueous solution. Solutions at different pH values 2, 4, 5, 6 and 10 were used. Cr^{+3} concentrations equal to 30mg/L without adding CKD were tested. The results were given in Fig. 3. From Fig. 3, it can be observed that the precipitation was dominant at pH greater than 5. It can be concluded that substantial adsorption may occurred at pH values less than those of precipitation. Fig. 4 shows the precipitation of Cr^{+3} at pH 6.

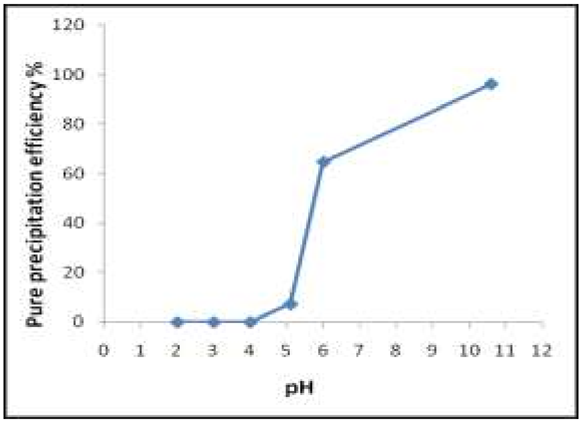

Fig 3. Relation between pure precipitation and pH.

Fig 4. Precipitation of Cr^{+3} at pH 6.

4.2 Adsorption /precipitation test for Cr^{+3}

Experiments were carried out in order to investigate the chromium removal from aqueous solution by adsorption/precipitation mechanisms.

Experiment was conducted at initial pH 4, Cr^{+3} concentration 65 mg/L, 250rpm, CKD dose10g/L and contact time 180min. Figure (5) shows the variation of the removal efficiency of Cr^{+3} and the pH value with time. As shown from Figure (5) the removal efficiency was increased until it reach 97% after 180min of contact time. Also it can be observed that the pH was increased from its initial value of 4 to reach 6.4 after 180min. This result means that the precipitation may have occurred at pH>4. To verify this observation additional experiments with different pH 4-7 at Cr^{+3} concentration 65mg/L, 250rpm, 180min contact time in the absence of CKD were performed. The result were tabulated in Table. 2 and plotted in Fig. 6. From Fig. 6 it can be observed that 100, 95 and 91% percent adsorption was occurred at pH 4, 5 and 5.5 respectively. After that at pH>5.5 precipitation was dominant, it represent 74 and 98% at pH 6 and 7 respectively. This indicates that about 80% adsorption and 20% precipitation was obtained based on the data used in the present investigation.

Fig. 7 shows a histogram illustrate the variation of adsorption /precipitation mechanism for Cr^{+3} removals from aqueous solution by CKD at different pH values.

It can be concluded that substantial adsorption may occurred at pH values less than 4. Also the pH must be maintained at this value throughout the period of contact time.

For isotherm and kinetics studies, care must be taken that no test to be performed at an initial pH>4 for Cr^{+3} treatment.

Table (2): Adsorption /precipitation data for Cr^{+3}

Initial pH	4	5	5.5	6	7
Final pH after 180min	4.1	4.5	5	5.5	6.9
Initial concentration	65	65	65	65	65
Final concentration after 180min	65	62	59	17	1.2
Pure precipitation after 180min	0	3	6	48	63.8
Pure precipitation %	0	5	9	74	98
Pure adsorption %	100	95	91	26	2

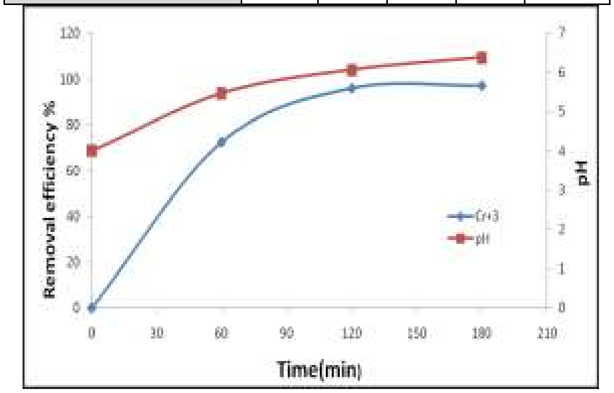

Fig 5. Relation between the removal efficiency of Cr^{+3} and pH with contact time. Cr^{+3} conc.=65mg/L, initial pH=4, rpm=250, 10g/L CKD and contact time =180min.

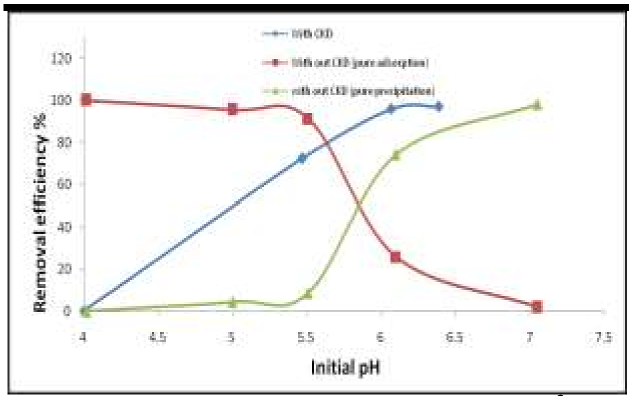

Fig 6. Adsorption /precipitation for Cr^{+3} in aqueous solution. Cr^{+3} conc.= 65mg/L, 250rpm, 180min contact time, with 10g/L and without CKD.

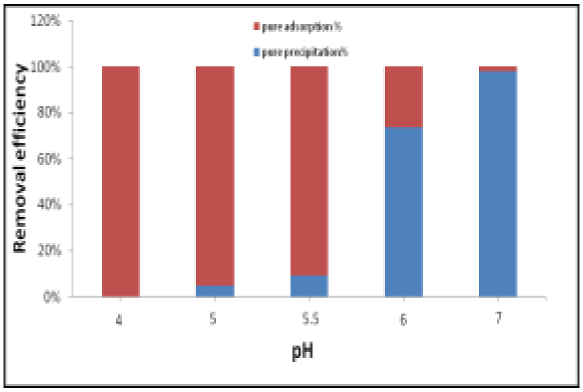

Fig 7. Adsorption /precipitation for Cr^{+3} in aqueous solution.Cr^{+3} conc. = 65 mg/L, initial pH=4, rpm=250, 10g/L CKD and contact time =180min.

4.3 Effect of contact time for Cr^{+3}

The removal of Cr^{+3} from aqueous solution was tested as function of contact time in the time interval (0-120) min. Initial Cr^{+3} concentrations 50 mg/L, pH 4, CKD dose 10g/L and 200rpm were used. Fig. 8 shows the effect of contact time with the removal efficiency of chromium ion. Fig. 8 shows a rapid increase in the removal efficiency within the first 20 min, and then stabilization was reached after 60 min. A contact time of 60min was chosen for the next experiments.

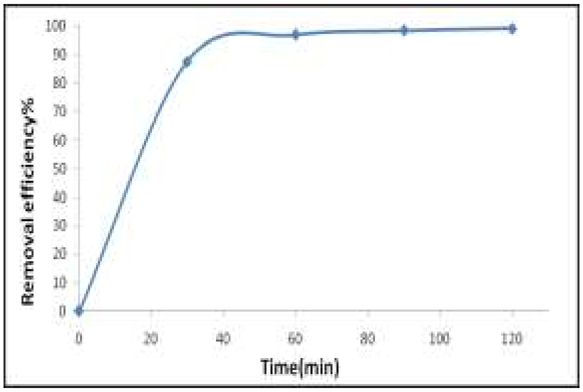

Fig 8. Effect of contact time on removal efficiency of Cr^{+3}. Cr^{+3} conc.= 50mg/L, pH=4, dose=10g/L, rpm=200.

4.4 Effect of agitation speed

The effect of agitation speed on adsorption of Cr^{+3} on CKD was studied using different agitation speeds 100-300 rpm. Initial Cr^{+3} concentrations 50 mg/L, pH 4, CKD dose 10g/L and contact time equal 60min were used. The results were shown in Fig. 9. This figure pointed that the removal efficiency of Cr^{+3}increases from 71 to 94% when increasing the agitation speed from 100 to 200 rpm, then there no observed increase in the removal efficiency above 200rpm.

This can be explained by: the boundary layer resistance was decreased as the agitation speed decreases which in turn enhance the driving force of Cr^{+3} ions. That is mean the film diffusion does not controlling the overall adsorption process, as agreed with [6].

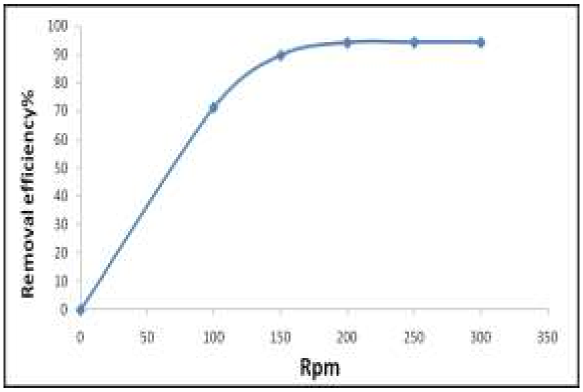

Fig 9. Effect of agitation rate on removal efficiency of Cr^{+3}. Cr^{+3} conc.=50mg/L, pH=4, dose=10g/L, contact time=60min.

4.5 Effect of CKD dosage

The CKD dosage was changed from 5-30g/L to test its effect on the adsorption of Cr^{+3}. Initial Cr^{+3}concentrations 50mg/L, pH 4, CKD 200rpm and 60min contact time were used. The results were plotted in Fig. 10. From this figure it was indicated that the removal efficiency for Cr^{+3} increased with the increase in adsorbent amount, this can be referred to the increase in the surface area of CKD and the number of active sites. Also, increasing adsorbent dosage may leads to increasing the linkage between surface active sites and this will reduce the uptake of Cr^{+3}, this observations were also demonstrated by [11].

The removal efficiency of Cr^{+3} ion reaches its maximum value at CKD dosage of 20g/L then it appeared to maintain constant value above 20g/L. The CKD dosage of 20g/L was selected for the next experiments.

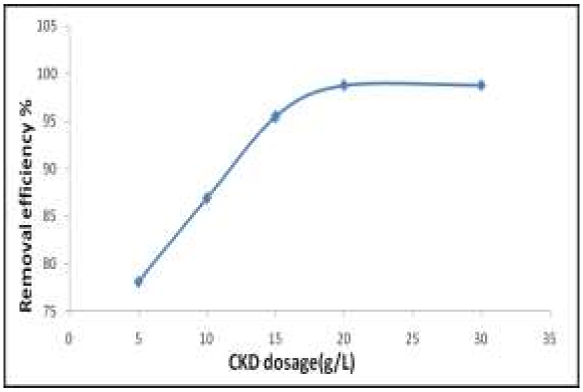

Fig 10. Effect of CKD dosage on removal efficiency of Cr^{+3}. Cr^{+3} conc.=50mg/L, pH=4, rpm=200, contact time=60min.

4.6 Adsorption isotherm for Cr^{+3}

Adsorption isotherm was plotted between q_e vs. C_e at different concentrations 10-200mg/L of Cr^{+3}. The pH of solution was kept at a value of 3.5 during the experiments to insure adsorption mechanism. Table. 3, shows that there is a significant variation in the pH throughout the experiment; adjustment was done whenever the pH changed. CKD dosage =20mg/L and 200rpm were used. The results were plotted in Fig. 11. From this figure it can be noticed that a sharp rise in the isotherm in the early stages for low C_e values. This means that a high uptake of the metal was obtained indicating that there are plenty of radially accessible sites. After that a decrease in the uptake and considerable increase in the C_e values was noticed. This can be related to a less available active sites at the end of the adsorption process and/or the difficulty of the Cr^{+3} molecules in passed through the adsorbent due to the partly coverage of the surface sites, this observation coincide with those presented by [1].

Additional experiment was conducted by changing the pH of the Cr^{+3}solution to 2.5, keeping the other parameters and the dosage constant. Table (4) shows the variation of the pH throughout the experiment. The adsorption isotherm was plotted in Fig. 12 and the comparison between the two isotherms was plotted in Fig. 13. Fig. 13 shows that the uptake of Cr^{+3} ion by the CKD at pH=2.5 is very small, also from Table. 4 it can be observed that there is no significant change in the pH during the experiment. This can be explained as follows: the smaller adsorption values observed is related to the competition between the protons H^+ and Cr^{+3} for the obtained binding site of CKD at acidic environment. In the pH range 4-6 all Cr^{+3} species (Cr^{+3}, $Cr(OH)^{+2}$ and $Cr(OH)_2^+$) are cationic, and the CKD surface have a negative charge, therefore the adsorption of Cr^{+3} is an attractive electrostatic interaction between the ionised acid sites on the CKD surface and the Cr^{+3} cations. Also if the Cr^{+3} is adsorbed, the reaction proceed to the left, leading to the depletion of protons and hence a rise in pH, (Eq. 1).

Table 3. pH variation through the experiment of the adsorption isotherm for initial pH=3.5

Cr+3 conc.(mg/L)	10	25	50	100	200
PH at t=0	3.8	3.8	3.8	3.8	3.8
pH at t= 60 min	5.5	5.5	5.5	5.5	5.5
pH after modification	3.7	3.2	3.6	3.6	3.6

pH at t=120min	5.7	5.7	5	5.4	5.6
pH after modification	3.6	3.7	3.6	3.6	3.8
pH at t=180min	5.8	5.9	5.9	5.8	5.5

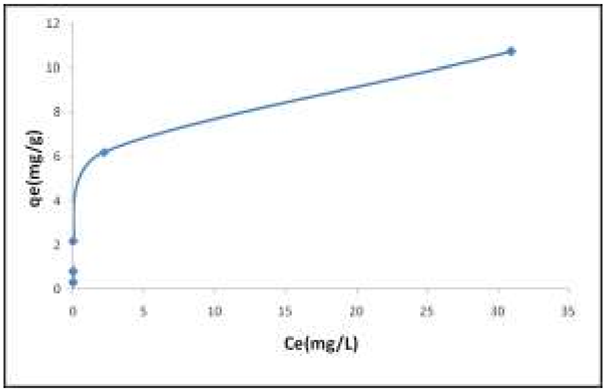

Fig 11. Adsorption isotherm for Cr^{+3}at initial pH=3.5.

Table 4. pH variation through the experiment of the adsorption isotherm for initial pH=2.5

Cr^{+3} concentration (mg/L)	10	25	50	100	200
pH at t=0	2.5	2.4	2.3	2.2	2.3
pH at t=180min	2.7	2.5	2.4	2.3	2.4

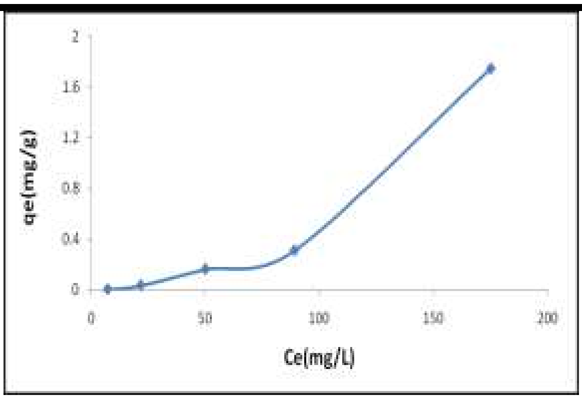

Fig 12. Adsorption isotherm at initial pH=2.5

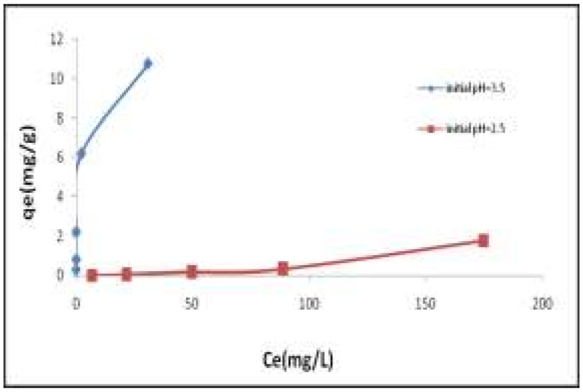

Fig 13. Comparison between adsorption isotherms at initial pH=3.5 and 2.5.

4.7 Adsorption isotherm models for Cr^{+3}

Different equilibrium isotherm models, Freundlich, Langmuir, BET, Redlich-Peterson, Toth, Radke-Praunsitz, Sips and Khan, were used to simulate the experimental adsorption isotherm. Table. 5 shows the parameters of each model and their correlation coefficient (R^2). The parameters for each model obtained from non-linear regression analysis using statistica version 10 software. Fig. 14 shows a comparison between these models and the experimental data. A good agreement was obtained between the models and the experimental data.

Table 5. Parameters of adsorption isotherm models for Cr^{+3} onto CKD

Model	Parameters	Trivalent Chromium
Frendlich Model $q = KC_e^{1/n}$	K	5.27
	n	4.81
	R^2	0.963
Langmuir Model $q = \frac{q_m C_e}{1 + bC_e}$	q_m	28.57
	b	2.628
	R^2	0.997
BET model $q_e = \frac{q_m . K_B . C_e}{(C_s - C_e)[1 + (K_B - 1)^{(\frac{C_e}{C_s})}]}$	q_m	8.44
	K_B	237
	R^2	0.996
Redlich-Peterson Model $q_e = \frac{K_{RP} C_e}{1 + a_{RP} C_e^{\beta_{RP}}}$	K_{RP}	11.6
	a_{RP}	1.54
	β_{RP}	0.8868
	R^2	0.963
Toth Model $q_e = \frac{q_{max} b_T C_e}{\left[1 + (b_T C_e)^{\frac{1}{n_T}}\right]^{n_T}}$	q_{max}	15.262
	b_T	3.703
	n_T	2.5
	R^2	0.963
Radke-Praunsitz Model $q_e = \frac{K_{RP} C_e}{1 + \left(\frac{K_{RP}}{F_{RP}}\right) C_e^{1-N_{RP}}}$	K_{RP}	10.41
	F_{RP}	7.897
	N_{RP}	0.1
	R^2	0.963
Sips Model $q_e = \frac{K_s C_e^{\beta_s}}{1 + a_s C_e^{\beta_s}}$	K_S	7.014
	a_S	0.401
	β_S	0.403
	R^2	0.963
Khan Model $q_e = \frac{q_{max} b_K C_e}{(1 + b_K C_e)^{a_K}}$	q_{max}	7.75
	b_K	0.9
	a_K	1.11
	R^2	0.963

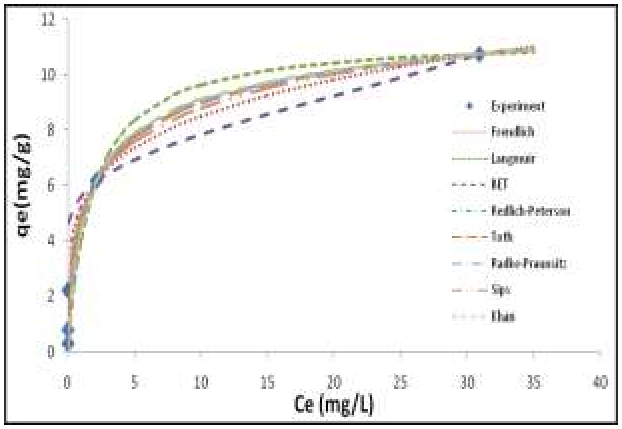

Fig 14. Comparison between the adsorption isotherm models and experimental data for Cr^{+3}.

5. Conclusions and Recommendations

The main conclusions that can be drawn from the experimental work of this study are as follows:

- The result of this investigation indicated that a substantial adsorption of Cr^{+3} were occur when the pH value is less than 4.
- It was found that the best conditions and amount of C^{r+3}adsorption on CKD were 60min, 200rpm, 20g/LCKD and pH 4.
- A good correlation was obtained between the adsorption isotherm models and the Cr^{+3} experimental data.
- q_m from Langmuir model equal to 28.5mg/g CKD for Cr^{+3}.

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معالجة المياه الثقيلة باستخدام غبار افران السمنت

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- استاذ مساعد
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- كلية الهندسة – جامعة النهرين

الخلاصة :-

تم دراسة استخدام غبار السمنت (CKD) كمادة رخيصة الثمن ومتوفرة كمادة مازة لازالة ايونات الكروم الثلاثية من المياه الملوثة وتم الحصول على غبار السمنت من معمل سمنت الكوفة في محافظة النجف الاشرف.

تم اجراء فحص لميكانيكية الامتزاز والترسيب لايونات الكروم الثلاثي حيث اظهرت النتائج ان الامتزاز التام وجد عند الدالة الحامضية pHللقيم اقل من 4 لايون الكروم الثلاثي.

اجريت مجموعة من التجارب بطريقة الوجبات لقياس اداء ال CKD لامتزاز ايون الكروم الثلاثي من محاليلها المائية. واظهرت النتائج ان افضل الظروف لامتزاز الكروم على ال CKD كانت 20 دقيقة 200 دورة بالدقيقة و 20 غم/لتر لغبار السمنت عند الدالة الحامضية 4.بينت منحنيات الامتزاز ان الامتزاز من النوع المفضل.وتم الحصول على تطابق جيد بين النتائج العملية وموديل لانكمير لامتزاز ايون الكروم الثلاثي. بلغ الحد الاقصى لطاقة الامتزاز qm لموديل لانكمير 28.5 ملغم/غم غبار السمنت.