Investigation on the Removal of Chromium (III) from Tannery Wastewater by Cement Kiln Dust

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ABSTRACT:

Tannery effluent is characterized not only by heavy loads an organic sludge but also with toxic heavy metals especially chromium ions. Chromium is considered an important source of contamination due to large volume of exhaust liquid discharged. An effort has been made in the present study to evaluate the potential of Cement Kiln Dust (CKD) for the elimination of trivalent chromium from the tannery effluents wastewaters by adsorption technique. The effects of various parameters such as adsorbent dosage, initial concentration of Cr(III), agitation rate, contact time and solution pH on the adsorption efficiency were studied during batch experiment at room temperature. The results indicate to that, the removal was effective at low Cr(III) concentrations and low pH value, and the optimum conditions for chromium tanning removal operation wastewater investigated were 60 minutes and 1200 rpm as a contact time and agitation rate respectively, and pH value equals to 0.5 at the temperature 25 ± 2 °C. And adsorbent dose 16 g/L was sufficient for the high removal of Cr(III) from wastewater for two both batches which is reach up to 95.11 and 93.72% respectively for both different batches that which is studied. The experimental data obtained during the study fitted well with the Langmuir and Freundlich isotherms. The maximum adsorption capacity of CKD was found to be 303 mg/g. The results obtained from this study show that, the CKD can be used as low cost adsorbent for completely removal of trivalent chromium from tanning wastewater.

KEYWORDS: Adsorption Isotherms, Chromium (III), Cement Kiln Dust, Tannery Wastewater.

INTRODUCTION:

Environmental pollution due to the development in technology is one of the most important problems of the century and the future. Heavy metals like chromium, copper, lead, cadmium, ..etc. in wastewater are hazardous to the environment. These metals cannot be degraded or readily detoxified biologically and have tendency to accumulate in living materials. Bedside that, heavy metals discharge in the wastewater can be toxic to aquatic life and render natural waters unstable for human consumption, (Khan and Mohamad, 2007).

Chromium is priority metal pollutant introduced into water bodies from many industrial processes such as pigments and paint manufacturing, wood preservative, leather tanning and finishing, water resistance plating and metal processing. Chromium is also used in explosive, ceramic and photography. Chromium exist in the aquatic environment in two stable oxidation states hexavalent Cr(VI) and trivalent Cr(III). Hexavalent chromium, which is primarily present as an anion in chromate (CrO_4^-), and dichromate ($Cr_2O_7^-$), possesses significantly higher levels of toxicity than other valence states, (Sharma and Forster, 1995). Chromium (III) is less health concern, because its low water solubility than Chromium (VI), but has a widely used as a tanning agent in the leather industry, and is an important source contamination due to the large volume of wastewater discharges and solid sludge's produced. There is no doubt that its compounds at higher concentrations are both accurately and chronically toxic and can generate serious trouble and diseases (nausea, skin ulceration's, lung cancer), (International Agency for Research on cancer, 1990).

The various compounds of chromium are found to be both corrosive to flesh and carcinogenic (Ajmal, et al., 1996). The presence of chromium compound in aquatic environment at high concentrations (coming from wastewater industries such as tanning wastewater) is lethal to marine species. Hexavalent chromium can affect the reproduction of fish and can accumulate in the tissue of fish even if it is present at low concentrations, (Haung, et al., 1978). Chromium is not biodegradable like organic compounds and trends to accumulate in living organisms causing various dieses and disorders (Bailey et al., 1999). The increase in chromium concentration above the consent or standard limits (5×10^{-5} $g.L^{-1}$ for drinking water) is becoming an important subject of public health (Environmental Protection Agency, 1998). It is needed therefore to developed suitable treatment processes to remove chromium from wastewater before being discharged into aquatic systems.

Various treatment techniques available for the removal of chromium from wastewater include solvent extraction, micro-filtration, precipitation and ion exchange. Most of these methods require high investment of capital and also expensive chemicals, making them unsuitable for treating chromium containing wastewater. Thus, there is a need to develop low cost and easily available materials that can effectively remove and recover chromium.

Adsorption is by far the most effective and widely used technique for the removal of toxic heavy metals from wastewater. (Selvi et al., 2001). Owing to the high cost and difficult procurement of activated carbon, efforts are being directed towards finding efficient and low cost adsorbent materials. A variety of low cost materials like fly ash (Grover and Narayansamy, 1982), wood charcoal (Deepak and Gupta, 1991), bituminous coal (Kannan and Vanangamudi, 1991), bagasse and coconut jute (Chand et al., 1994), rice husk carbon (Srinivasan et al., 1998), peat (Brown and Allen, 2000), red mud (Gupta et al., 2001), Used black tea leaves (Houssain et al., 2005), activated carbon from sugar industrial waste (Fahim et al., 2006) and sugarcane bagasse (Khan and Mohamad, 2007) and have been tried.

As shown recently, research efforts have been directed towards the use of industrial waste as an adsorbent material in an attempt to minimize processing costs and with the protection of the environment and public health. Therefore, the present objective of this study to investigate the potential use of Cement Kiln Dust (which is solid wastes, creates an air pollution problems) as an adsorbent for the removal of chromium (III) from wastewater under kinetic and equilibrium conditions. The effects of various parameters such as adsorbent dosage, initial metal concentration, agitation rate, contact time and pH on the adsorption process have been studied. Adsorption studies will also be carried out to evaluate the adsorptive capacity of the adsorbent.

1. MATERIAL AND METHODS:

1.1 Adsorbent:

The experiment was carried out to remove chromium (III) from tanning wastewater effluents by using by-pass cement kiln dust (CKD) as an adsorbent. The cement kiln dust was collected from the (Al-Mergheb Portland Cement Factory, Al-Khoms). The analysis of the sample of CKD was made by X-ray fluorescence, the composition of the constituents is shown in Table.1.

Table.1 Results of the chemical analysis of CKD

Constituents	Composition*, (*wt%*)
SiO_2	19.58
Al_2O_3	3.6
Fe_2O_3	2.97
CaO	54.89
MgO	2.12
K_2O	2.32
SO_3	0.83
Cl	0.54
Na_2O	0.109
Other residues	13.041

- ***Average values for four different batch samples.***

The wastewater obtained from national tanneries effluents, this wastewater obtained after the tanning operation was filtered to obtain a tanning solution, which is to be further treated for the chromium (III) removal.

1.2 Methods:

1.2.1 Reduction of hexavalent chromium to trivalent chromium:

The most of chromium is discharged into aqueous solution from tannery effluents as Cr(III) and small content of Cr(VI). Also, CKD had no effect on the removal of hexavalent chromium, while it adsorbs trivalent chromium from solutions. Thus, before the addition of CKD to tanning solution, reduction of hexavalent chromium was carried out by using sodium bisulfite as a reducing agent and sulfuric acid (0.1 N), (Ayres, et al., 1994). The sodium bisulfite was added in different amounts 0.5, 1 and 1.5 g to 50 ml of the tanning solutions sample, while sulfuric acid was gradually added. The addition of sulfuric acid was continued until the reduction was completed. As detected by oxidation reduction potential which was used as a sensor in the device titroprocessor (Type: Metrohm 682).

1.2.2 Experimental Procedure:

The experimental was carried out by batch process for the tanning solution obtained from tanneries effluents. The Cr(III) content in the tanning solution was determined using UV visible spectrophotometer (Unicam 8700), at a wavelength of 540 nm. Each sample was digested by adding 5 ml of nitric acid to 50 ml of the sample before measuring its chromium content by the spectrophotometer. Then the solution was evaporated on a hot plate to its volume becomes 15 ml. Further, 5 ml of concentrate nitric acid and 10 ml of concentrate sulfuric acid (95% wt.) were added and the solution was evaporated until dense white fumes of SO_3 appeared. The solution was cooled and diluted to about 50 ml with distilled water. Then, the solution was heated top almost boiling to dissolve gradually salts and then cooled and diluted with distilled water to 100 ml after that, the solution was ready for chromium (III) measurement.

The initial concentrations of Cr (III) in tanning solutions were treated in an experimental were 2336 and 4320 mg/l respectively. These two tanneries batches are taking from two different national tanneries effluents wastewater.

All working of different concentrations were prepared by diluting the stock solution (tanning solutions) with distilled water. The pH of the test solutions was adjusted using reagent grade dilute sulfuric acid. A pH meter (Model: HI 8417, HANNA Instrument) was used to measure the pH of solutions. The effect of agitation rate was studied by agitated by a mixer with different rates. After agitation, the adsorbate and adsorbent were separated by filtration, and then the concentrations were analyzed by the spectrophotometer.

The adsorption studies were carried out by batch technique. For this investigation, a series of conical flasks containing equal volume (250 ml in each case) of adsorbate solutions of varying concentration were employed, at desired pH and temperature. A suitable dose of adsorbent was added into each conical flask. The samples were adjusted to the desired pH by using reagent grade sulfuric acid solution. The samples were then agitated using an agitator operation at variable speed (50-2800) rpm at room temperature (25 ± 2 °C) for pre-selected contact time. After agitation, the solutions were filtered and the concentrations of Cr(III) in the solution were analyzed by spectrophotometer according to the previous mentioned above. The effects of adsorbent dose, initial metal concentration, agitation rate, and contact time and pH values on the adsorption efficiencies were studied.

2. RESULTS AND DISCUSSION:

The chromium (III) removal was studied under different conditions, viz. dose of CKD, initial concentration of Cr(III) ions, the agitation rate, the contact time and pH. Additionally to, the equilibrium isotherms have been determined for the adsorption of chromium ions on CKD and determination of the adsorption capacity of CKD. These parameters affected on the adsorption efficiency illustrated as follow:

2.1 Effect of Adsorbent Dose:

The effect of adsorbent doses on removal of Cr(III) has been shown in Figure(1), for two both batch samples. All experiments were done under the following conditions (25 ± 2 °C, 1200 rpm and 60 min. as a contact time). The results shown in the figure indicate that the percentage removal of Cr(III) increases with an increase in adsorbent dose. This is due to increase in the surface area of CKD and hence more active sites are available for the adsorption of the metal ion.

It is also evident from the figure that, at an adsorbent dose 20 g of adsorbent per each one liter of tanning solution the removal efficiency for the first batch is completely removal of Cr(III) from the solution under the operating. While the completely removal of Cr(III) for the second batch is 22 g/l.

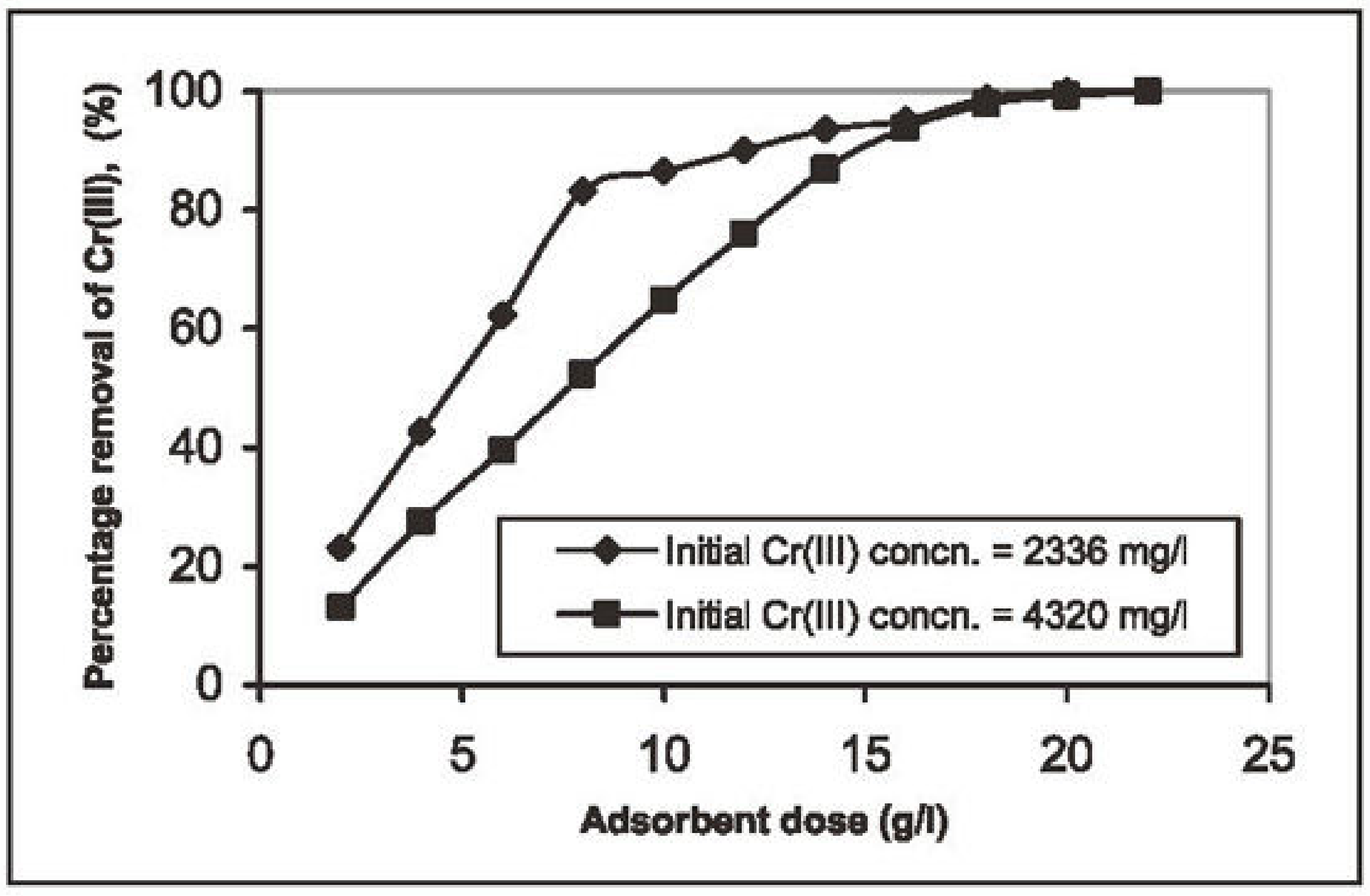

Figure1: Variation of Cr(III) removal versus adsorbent dose for both batches, at (25 ± 2 °C, 1200 rpm and contact time is 60 min)

Generally, using 16 g of CKD per 1 liter of tanning solution resulted in a relatively high percent removal (95.11% and 93.72% for both tanneries batches respectively). The pH values of the solution increases as the amounts of CKD increase. Figure (2) show the effect of pH on the removal of Cr(III) on the second batch (with initial concentration 4320 mg/l) at initial pH value 3.11. Therefore, at pH 3.11 Cr(III) ions is dissolved due to low pH upon adding CKD, then the value of pH increases gradually, consequently the precipitation of Cr(III) ions in form of $Cr(OH)_3$ is started. This can be explained by the effect of soluble alkalis and *CaO* present in CKD. When caustic is added to water containing heavy metals, a metal hydroxide precipitate is formed, (Ayres, et al., 1994). So, *CaO* in the solution reacted with ions of Cr(III) to gives a gray-green precipitate of chromium hydroxide. And we investigated of this precipitate by chemical analysis.

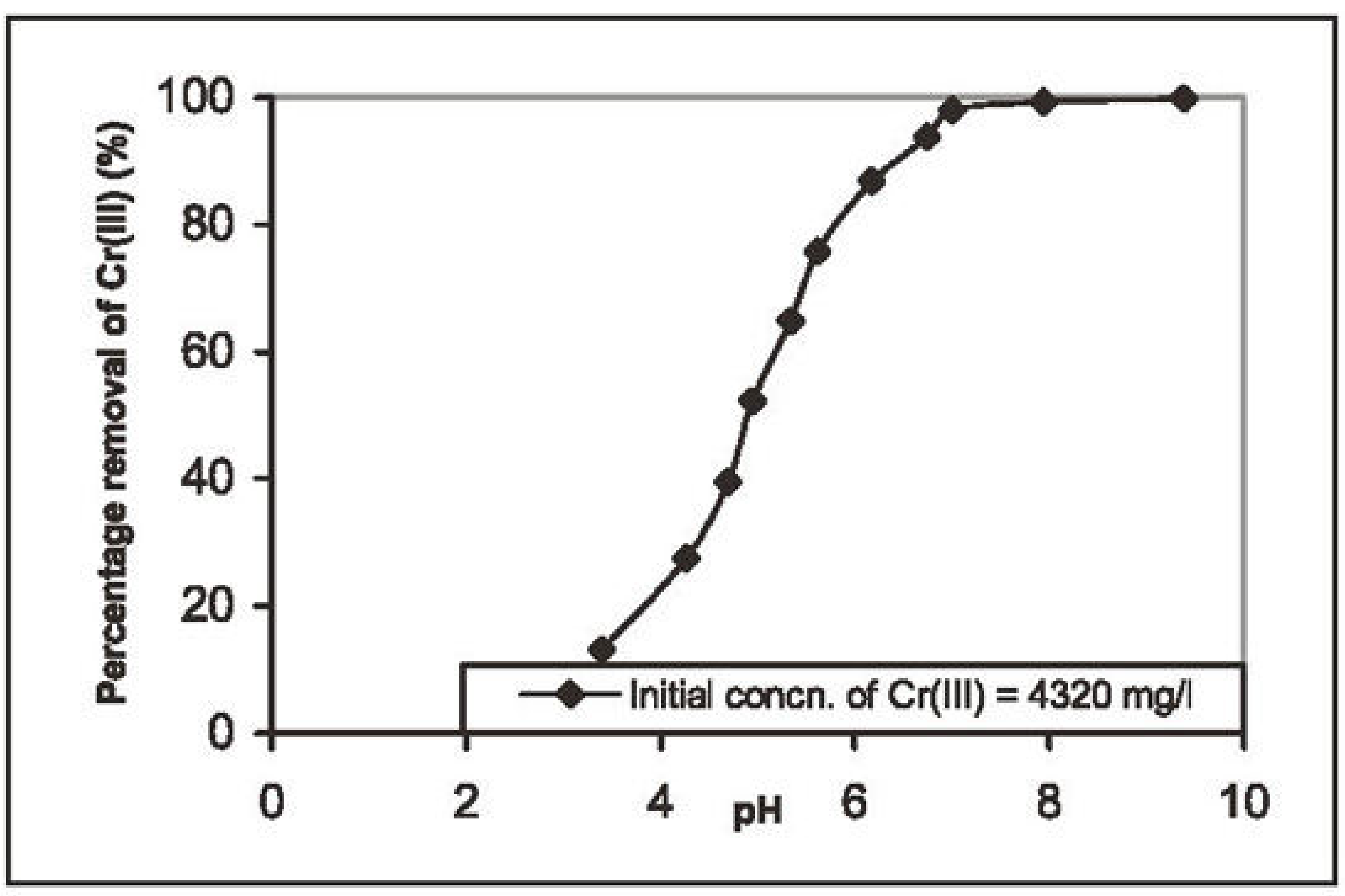

Figure2: Effect of pH on the removal percentage of Cr(III), at (25 ± 2 °C, 1200 rpm and contact time is 60 min)

The investigation included that the precipitate dissolves in excess hydroxide solution to gives green solution, this solution oxidized from Cr(III) ions to Cr(VI) ions when addition a solution of hydrogen peroxide, then confirmed the oxidation by addition Ba^{2+} solutions precipitate the yellow chromate ion as yellow barium chromate. This chemical investigation insure that precipitate was *$Cr(OH)_3$*. It seems that, at the pH value equal to 7.94 about 99% removal is achieved and complete removal of Cr(III) was at pH equal to 9.38. So, the increasing of percentage of removal of Cr(III) referred to the increase in the adsorbent dose not to the increase of pH values of the solution.

2.2 Effect of Initial Concentration:

The effect of initial metal concentrations was studied by diluting a solution having an initial concentration 4320 ppm (second batch) by adding 450, 400, 350, 300, 250, 200, 150, 100 and 50 ml of distillated water to obtain several concentrations of Cr(III) representing (264, 869, 1304, 1728, 2173, 2607, 3041, 3476 and 3910 ppm) respectively. Figure (3) show the effect of variation of initial concentration Cr(III) on the percentage removal. The adsorption of Cr(III) decreases from 99.2% to 11% when the initial metal concentration was increased from 434 to 3910 ppm at an adsorbent dose of 16 g/l for agitation rate 1200 rpm, 60 minute contact time, pH value is 0.5 and temperature 25 ± 2 °C.

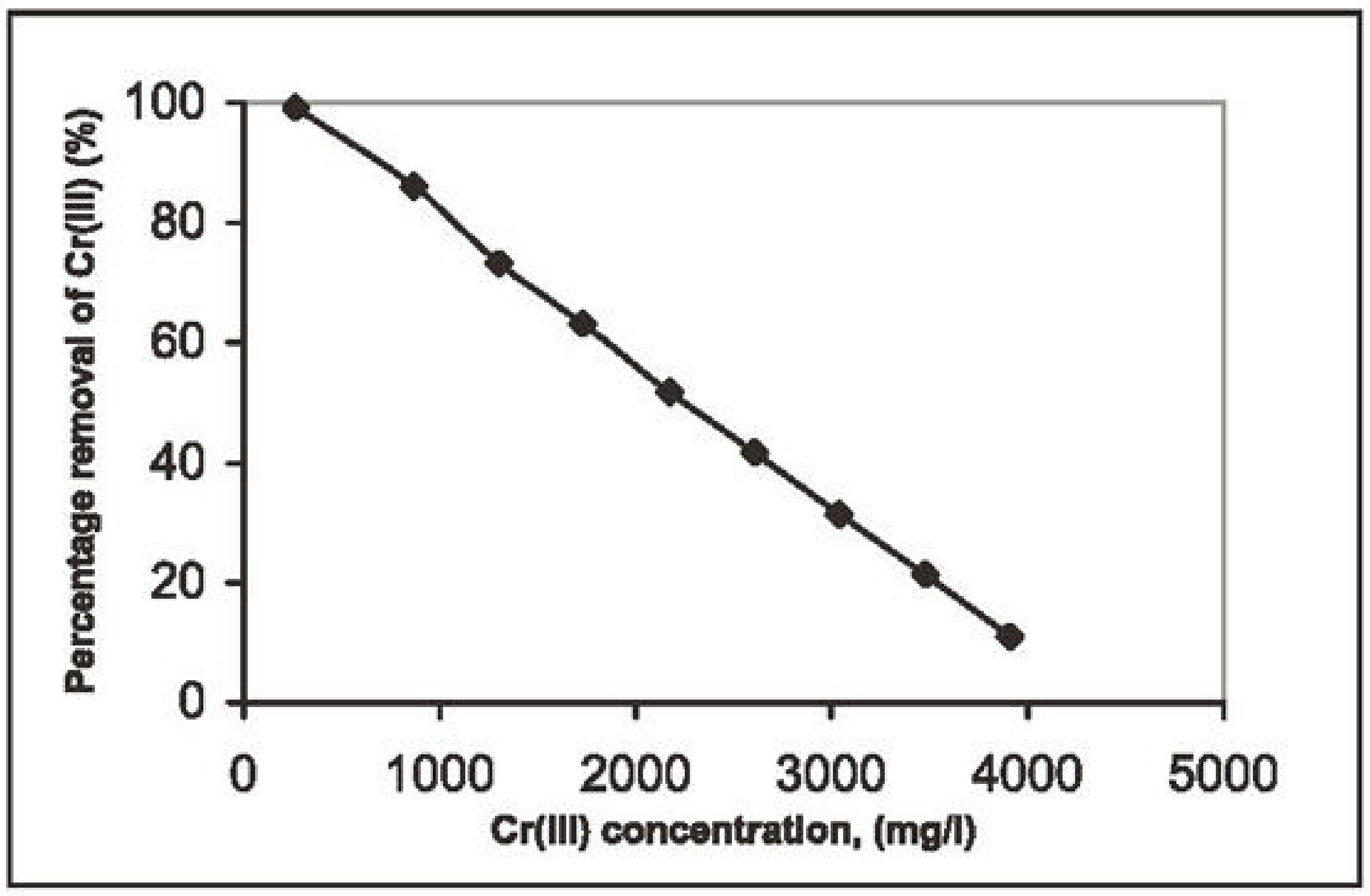

Figure3 : Effect of initial metal concentration on the removal percentage of Cr(III), at (25 ± 2 °C, 1200 rpm, contact time is 60 min., adsorbent dose 16 g/l and pH=0.5)

CKD adsorbed Cr(III) ions best at lower Cr(III) concentrations in the range (264-1728) ppm. At lower concentration, the ratio of initial number of moles of metals ions to available surface area is larger and subsequently the fractional adsorption becomes independent of its minimum initial concentration (Yu, et al., 2002).

2.3 Effect of Agitation Rate:

To study the effect of the agitation rate on the Cr(III) removal from the tanning solution is illustrated in Figure (4). The used rates 50, 450, 800, 1200, 1600, 2200, 2400 and 2800 rpm; at a temperature 25 ± 2 °C, the contact time 60 minutes using the adsorbent dose 16 g/l at the tanning solution with pH value is equal to 0.5; then the Figure (4) show that, the removal of Cr(III) from tannery wastewater for first batch (with initial concentration 2336 mg/l) increase from 68 to

100% when the agitation rate increase from 50 to 2400 rpm. The same trend for the second batch (with initial concentration 4320 mg/l), which the removal percentage increase from 54 to 100% when increase of agitation rate from 50 to 2800 rpm. These results for both batch results indicate that, Cr(III) removal is controlled by degree of stirring. Increasing of the agitation rate, decrease the boundary layer resistance to mass transfer surrounding CKD particles. From, the results we find that the optimum agitation rate that gives a high removal efficiency for both tanneries batches is 1200 rpm, which the removal efficiency are 96 and 93.1% for both tanneries batches respectively.

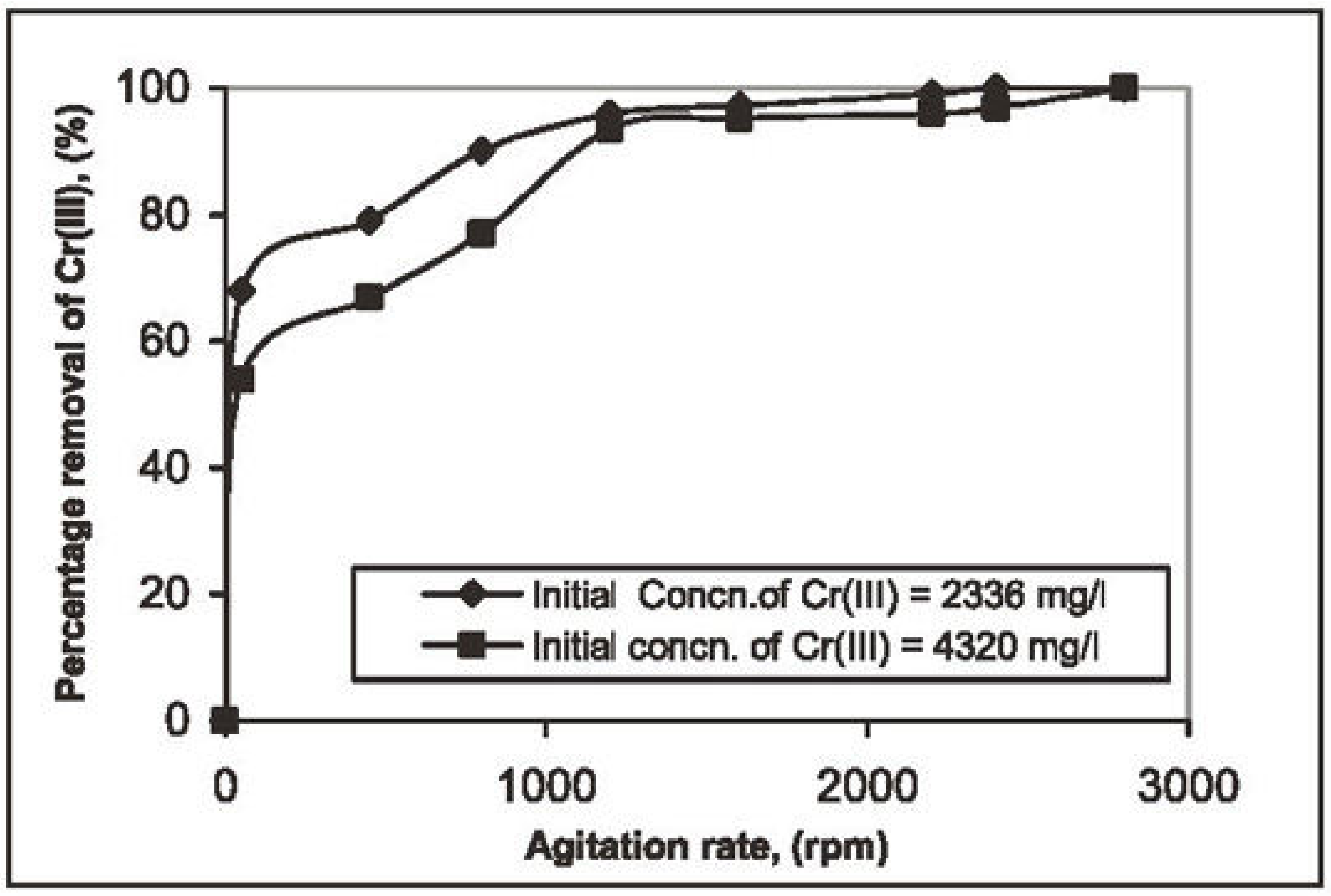

Figure 4: Variation of Cr(III) removal percentage versus agitation rate for both batches, at (25 ± 2 °C, contact time is 60 min, adsorbent dose 16 g/l and pH=0.5)

2.4 Effect of Contact Time:

To study the effect of contact time on Cr(III) removal from wastewater for both tanneries batches, the experiments were carried out at different contact time (5, 10, 15, 20, 25, 30, 35, 45, 45, 50, 55, 60 and 75 min.) with a fixed adsorbent dose 16 g/l at temperature (25 ± 2 °C), pH value is 0.5 and agitation rate 1200 rpm (Fig 5).

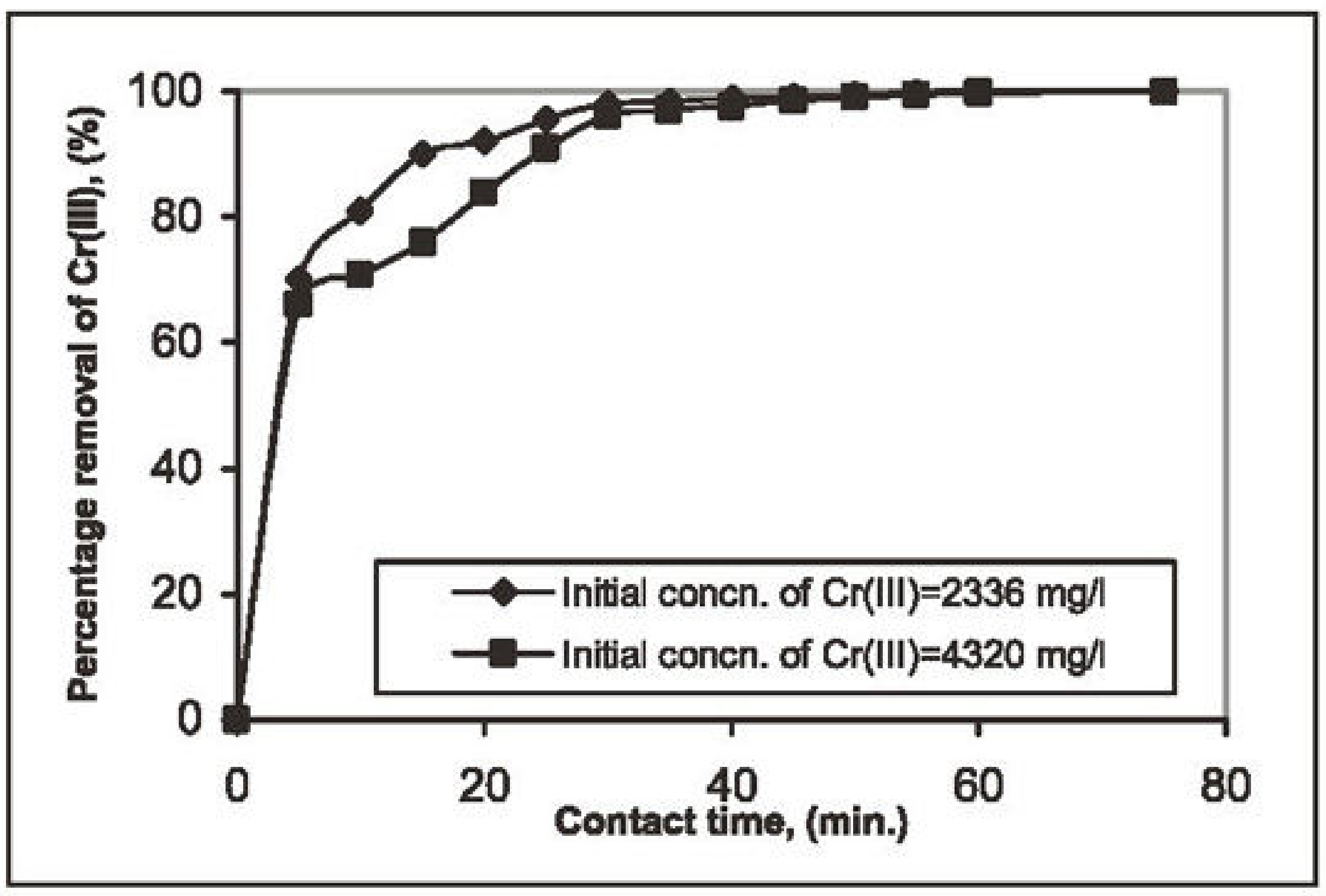

Figure5: Variation of Cr(III) removal percentage versus contact time for both batches, at (25 ± 2 °C, 1200 rpm, adsorbent dose 16 g/l and pH=0.5)

The response of contact on Cr(III) removal increased from 70% to 100% when the contact time was increased from 5 to 60 minutes for the first batch. While an increased in the removal efficiency was noted from 66 to 100 % when the contact time was varied from 5 to 75 minutes for the second batch at the same conditions as shown in Figure (5).The optimum contact time was 60 minutes for both samples, which is the percentage removal of Cr(III) is reach up to 100 and 99.8% for two both tanneries batches respectively.

2.5 Effect of pH:

Variation of Cr(III) removal versus pH at different metal concentration is depicted in Figure (6). Chromium ions in the solution were transformed to chromium hydroxide by the effect of *CaO* which present in the CKD. The experiments were carried out on the both samples by treating the precipitate using sulfuric acid (95% *wt.*) was added to the precipitate in addition to 250 ml of distilled water at different pH values (0.5, 1, 1.5, 2, 2.5, 3, 3.5 and 4). With Adsorbent dose 16 g/l, contact time 60 minutes, agitation rate 1200 rpm and the temperature was 25 ± 2 °C. The percentage removal of Cr(III) increases with decrease in pH value. At an initial the percentage removal of Cr(III) for the first batch was 95.11% and the second batch is 93.72% at the pH value equal to 0.5. While at pH 4 the percentage removal efficiencies were found to be 62% and 76% for the first and second tanneries batches respectively. So, the optimum value of pH which that give higher values of the percentage removal of Cr(III) from both tanneries batches is found to be 0.5.

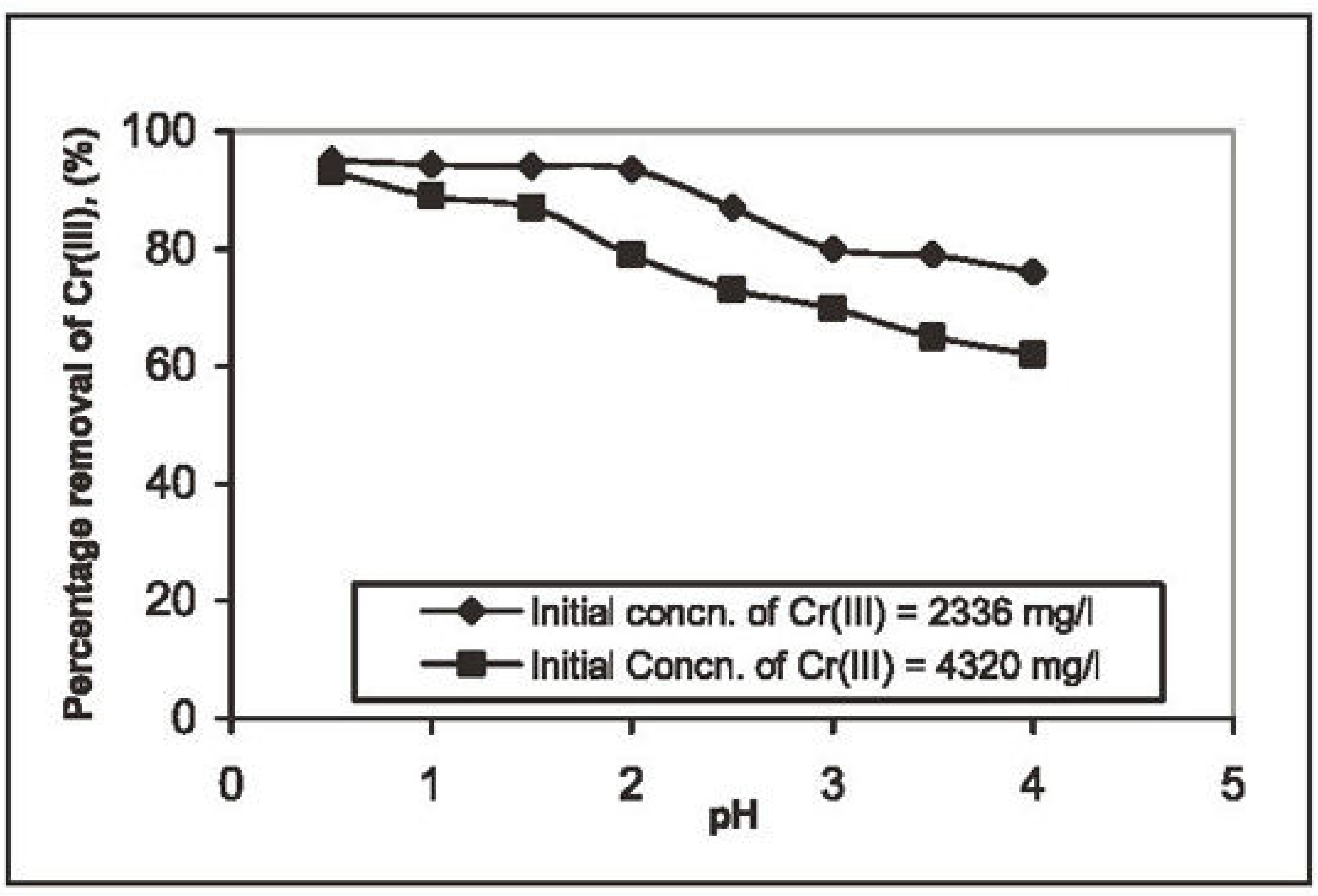

Figure.6: Variation of Cr(III) removal percentage versus pH for both batches, at (25 ± 2 °C, 1200 rpm, contact time is 60 min. and adsorbent dose 16 g/l)

2.6 Adsorption Isotherm

In order to model the adsorption behavior and calculate the adsorption capacity, the adsorption data obtained during the present study is analyzed by the Langmuir and Freundlich isotherms.

Langmuir isotherm is expressed as follow:

$$q_e = \frac{abC_e}{1+aC_e} \qquad \text{..........} \ (1)$$

where;
q_e : mg of Cr(III) adsorbed per gram of adsorbent (CKD).
C_e : Equilibrium of Cr(III) concentration, (mg of Cr/Liter of tanning solution).
a : Langmuir constant, (liter/mg of Cr(III)).
b : Monolayer coverage, (mg of Cr(III)/g of adsorbent "CKD").
The linear form of Langmuir represented by the following:

$$\frac{C_e}{q_e} = \frac{1}{b} * C_e + \frac{1}{ab} \qquad \text{.......... (2)}$$

Freundlich isotherm is expressed as follow:

$$q_e = K_f (C_e)^{1/n} \qquad \text{.......... (3)}$$

where:
K_f : Freundlich constant, (mg of Cr(III)/g of adsorbent "CKD")
n : Freundlich exponent, (g of adsorbent/liter of tanning solution)
The logarithmic form of Freundlich model is represented by the following equation:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \qquad \text{.......... (4)}$$

When the data from laboratory test are plotted the best straight line connecting the points is isothermal relationship between contaminant concentration and adsorbent equilibrium capacity. The conformity between experiential data and model predicted values was expressed by the correlation coefficient (R^2, values close to 1). A relatively high R^2 value indicates that the model successfully describes kinetics of Cr(III) adsorption.

Isothermal data was used to calculate the ultimate sorption capacity of the CKD by substituting the required equilibrium concentration in the Langmuir and Freundlich isotherm models equations (2 and 4) respectively. The data demonstrate that the CKD is an effective adsorbent for Cr(III). The equilibrium results of the batch using different amounts of CKD (0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, and 5) g added to 250 ml of tanning solution with pH value is 0.5, 1200 rpm agitation rate, 60 minutes contact time and the temperature was 25 ± 2 °C.

The values of constants of Langmuir isotherm model are obtained from the plot of C_e/q_e against C_e for the adsorption data of Cr(III), which is shown in Figure (7). It is evident that the data is well fitted to the Langmuir isotherm model and can be represented by the following mathematical model expression:

$$q_e = \frac{5.102 C_e}{1 + 0.01684 C_e}$$

The values of constants of Freundlich isotherm model K_f and $1/n$ are obtained from the plot of $\log q_e$ against $\log C_e$, for the adsorption data of Cr(III), which is shown in Figure (8). It is evident that the data is well fitted to the Freundlich isotherm model and can be represented by the following mathematical model expression:

$$q_e = 173.2208 (C_e)^{0.0661}$$

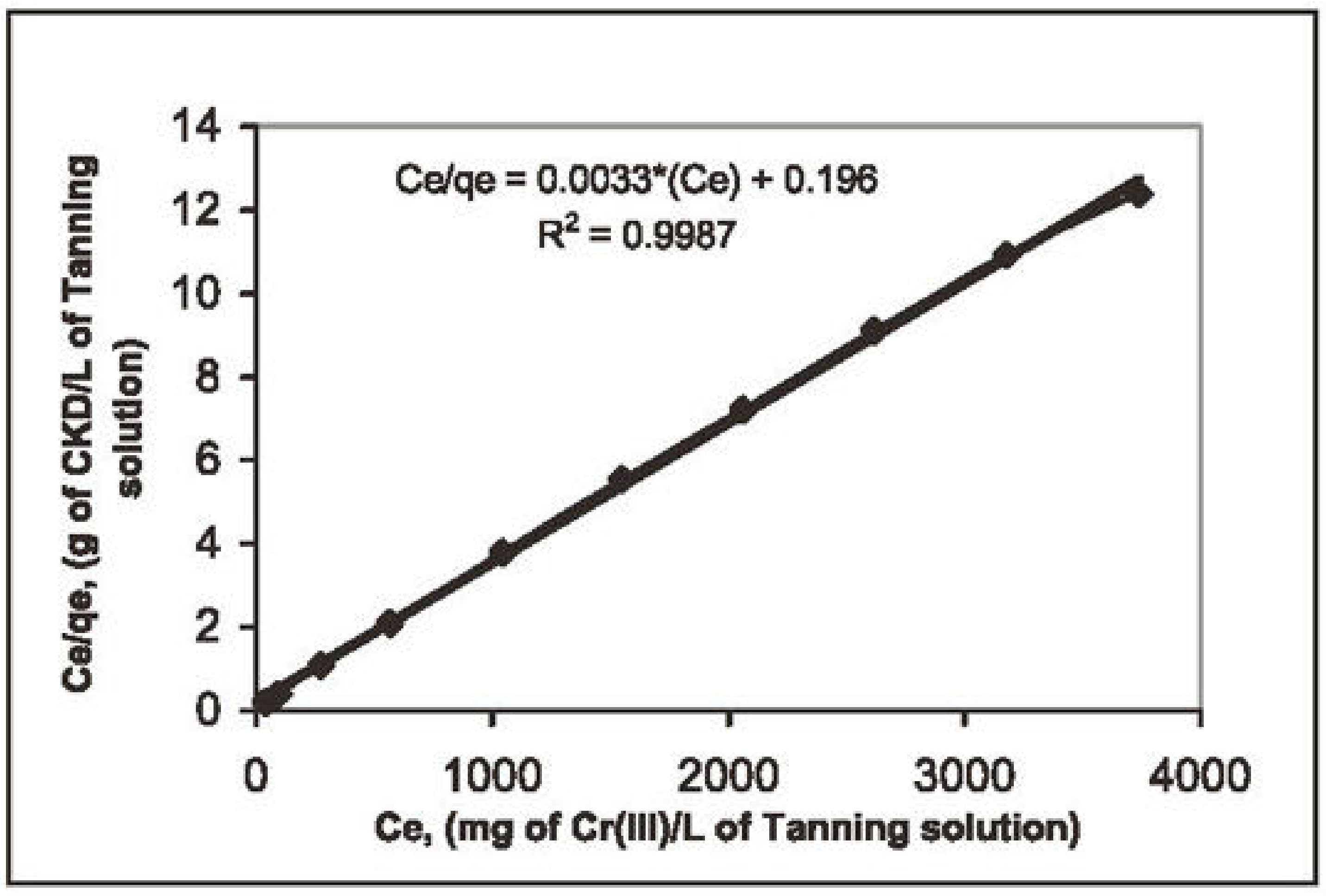

Figure.7: Langmuir isotherm for adsorption of Cr(III) by Cement Kiln Dust, at (25 ± 2 °C, 1200 rpm, contact time is 60 min. and pH=0.5)

From the experimental results have been fitted using Langmuir and Freundlich isotherms. The maximum adsorption capacity of CKD was found to be 303 mg/g. Also, the higher values of *ab* and K_F reflects the strong affinity of CKD for Cr(III) ions.

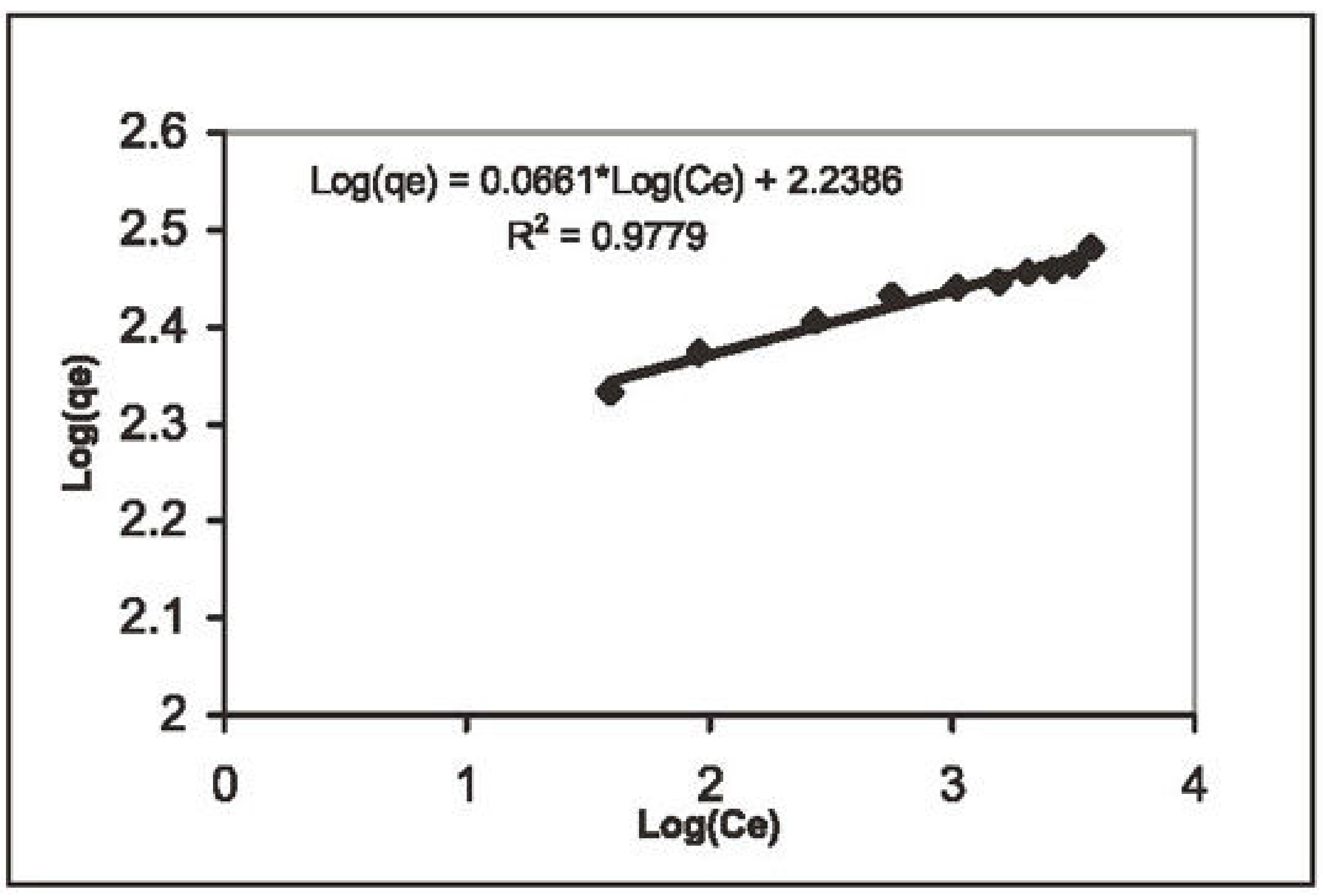

Figure.8: Freundlich isotherm for adsorption of Cr(III) by Cement Kiln Dust at (25 ± 2 °C, 1200 rpm, contact time is 60 min. and pH=0.5)

From the analysis of the tanning wastewater before and after treatment using the CKD, it was found that BOD was decreased from 420 to 68 mg/l, COD decreasing from 7296 to 2634 mg/l and the pH value of the solution increased from 3.11 to 11.98. These both decreasing and increasing of BOD and pH respectively occurs due to that, during the precipitation of $Cr(OH)_3$, may be some of suspended materials (organic in nature) are settled and also due to removal of Cr(III). The total content of chromium ions is not detected in the wastewater after treatment, this refer to that, the CKD can be used as a good efficient adsorbent for the complete removal of trivalent chromium from tanning wastewater.

CONCLUSIONS:

From the present study, it can be conclude that the Cement Kiln Dust (CKD) has a great potential to remove chromium (III) from the tannery wastewater. The percentage removal of Cr(III) depends on adsorbent dose, initial metal concentration, agitation rate, contact time and pH value. CKD adsorbed Cr(III) ions best at lower Cr(III) concentrations in the range (264-1728) ppm but the removal efficiency dropped to 11% when the metal concentration was increased to 3910 mg/l. Generally, the optimum conditions for chromium tanning removal operation wastewater investigated were 60 minutes and 1200 rpm, contact time and agitation rate respectively, addition 16 g of adsorbent per one liter of chromium tanning wastewater and pH value equals to 0.5 at the temperature 25 ± 2 °C.

The adsorption data for Cr(III) fitted well to the Langmuir and Freundlich isotherm models. So, the present study shows the feasibility of the practical use of Cement Kiln Dust as a low cost adsorbent for the completely removal of Cr(III) from tanning wastewater.

ACKNOWLEDGMENT

The authors gratefully acknowledge the financial support of the present work by the Al-Mergheb University.

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تحقيق لعملية إزالة الكروم ثلاثي التكافؤ من مخلفات مياه مدابغ الجلود باستخدام غبار قمائن الأسمنت

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الملخص:

النفايات السائلة للمدابغ لا تتميز بمحتوياتها العالية من المواد الثقيلة والعضوية الغليظة القوام ولكن أيضا بمحتواها العالي من المعادن الثقيلة السامة وبشكل خاص ايونات الكروم. الكروم يعتبر مصدرا هاما للتلوث بسبب الكمية الكبيرة من السوائل المطرودة والخارجة من المدبغة وهي تحتوي هذه المخلفات. قد بذل جهد في هذه الدراسة لتقييم قدرة مخلفات غبار قمائن الاسمنت (Cement Kiln Dust, CKD) على إزالة الكروم ثلاثي التكافؤ من مخلفات المياه المستخدمة في المدابغ باستخدام تقنية الأمتزاز. تأثير عدة متغيرات مثل جرعة المادة المازّة، التركيز الابتدائي لأيونات الكروم الثلاثي، معدل التقليب، زمن التلامس وتركيز الأسس الهيدروجيني للمحلول على كفاءة عملية الأمتزاز تم دراستها من خلال إجراء التجارب بطريقة الدفعات(Batch Process) عند درجة حرارة الغرفة. النتائج تُشير إلى أن عملية الأمتزاز تكون فعالة عندما يكون تركيز ايونات الكروم الثلاثي صغير وقيم تركيز ايون الهيدروجين منخفضة. والظروف المثلى التي تم تحقيقها لأجراء عملية إزالة ايونات الكروم من مجرى مخلفات مياه المدابغ كانت زمن التلامس 60 دقيقة ومعدل التقليب 1200 دورة بالدقيقة وقيمة تركيز ايون الهيدروجين 0.5 عند درجة حرارة 25±2 درجة مئوية. وجرعة المادة المازّة 16 جرام/لتر كانت كافية لعملية إزالة عالية الكفاءة للكروم الثلاثي التكافؤ من مخلفات مياه المدابغ حيث كانت قيم كفاءة الإزالة هي 95.11% و 93.72% لكلا العينتين التي تم دراستهما من مخلفات مياه المدابغ. البيانات التجريبية التي تم الحصول عليها أثناء الدراسة كانت متلائمة جيدا مع معادلات لانجموير و فريوندليش بثبوت درجة الحرارة. أقصى سعة امتزاز لأيونات الكروم الثلاثي التكافؤ لكل جرام من غبار قمائن الأسمنت كانت 303 مجم/جم. وتبين النتائج المستخلصة من الدراسة بأن (CKD) يمكن أن تستخدم كمادة مازة رخيصة الثمن لإزالة الكروم الثلاثي التكافؤ بشكل كامل من مياه المدابغ المستهلكة.