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HEXAVALENT CHROMIUM REMOVAL FROM AQUEOUS SOLUTION VIA ADSORPTION ON GRANULAR ACTIVATED CARBON: ADSORPTION, DESORPTION, MODELING AND SIMULATION STUDIES

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ABSTRACT

Hexavalent chromium is one of the contaminants recognized as a carcinogenic and mutagenic agent. Therefore, it is essential to remove it from wastewater before disposal. In this study, removal of hexavalent chromium via adsorption on commercial granular activated carbon (GAC) was investigated as a function of adsorbent dosage, initial solution pH, initial Cr (VI) concentration, contact time and temperature. The batch experiments were conducted at three temperatures (17, 27 and 37°C) and the results showed that Cr (VI) removal kinetics obeys pseudo second order rate equation. Equilibrium studies showed that the experimental data fitted well with Longmuir isotherm adsorption model. Thermodynamic parameters were also determined and results suggest that the adsorption process is a spontaneous and endothermic. Reversibility of Cr (VI) adsorption and repeated availability performance of the adsorbent, was investigated by desorption process. In addition, artificial neural network (ANN) was utilized to simulate the experimental data. The results showed that the training step of the network was successful and therefore the simulation could be applied to predict hexavalent chromium removal with high accuracy.

Keywords: hexavalent chromium, adsorption, granular activated carbon, adsorption kinetics, adsorption isotherm, desorption, ANN.

1. INTRODUCTION

Chromium is one of the contaminants which exist in hexavalent and trivalent forms. Trivalent chromium is an essential element in human nutrition and is much less toxic than the hexavalent one, which is recognized as a carcinogenic and mutagenic agent [1]. Chromium compounds are widely used by different industries such as metal plating, paints and pigments, leather tanning, textile dyeing, printing inks and wood preservation, so huge quantity of wastewater containing chromium is discharged into the environment [2]. Therefore, removal of Cr (VI) from wastewater is essential before disposal.

Conventional methods for removing dissolved heavy metal ions include chemical precipitation, chemical oxidation or reduction, filtration, ion exchange, electrochemical treatment and membrane technology. These processes have considerable disadvantages such as incomplete metal removal, requirements for expensive equipment and monitoring system, high reagent and energy requirements or generation of toxic sludge or other waste products that require disposal [3]. Adsorption can be an effective and versatile method for removing chromium particularly when combined with appropriate regeneration steps. This solves the problems of sludge disposal and renders the system more economically viable, especially if low cost adsorbents are used [4]. Activated carbon (AC) seems to be an attractive choice for chromium removal both for its removal efficiency and for the rapid adaptation to pollutant loading alteration [5].

Although AC possesses large sorption capacity of heavy metal, the very high unit cost when compared to the other commercially available adsorbents restricts their potential use for environmental protection applications. Thus, testing the reversibility of heavy metal ion adsorption with AC to diminish their replacement cost is needed before practical use of AC in water treatment [6].
For proper selection and design of a removal system, the knowledge of the adsorb ability in different conditions is desirable. The experimental data of adsorb ability in batch systems evaluate only suitability of the adsorption in the interest conditions [7]. Currently artificial neural networks (ANN) are found to be excellent options for simulation these types of complex processes. In the adsorption process chemical engineering, the ANN was also found to be successfully applied to predict the adsorption equilibrium of solid-liquid systems [8].

In several previous reports, various authors have documented the use of organic and inorganic substances and compounds as adsorbents [7-12]. Also several studies of heavy metal removal were carried out using adsorbents developed from natural materials [13, 14]. It is rare to find comprehensive work investigated about optimization, modeling and simulation of an adsorption process together.

The present investigation deals with the application of granular activated carbon (GAC) in the removal of chromium (VI) from aqueous solutions. The removal amount of Cr (VI) was measured in different effective conditions such as: adsorbent dosage, initial Cr (VI) concentration, solution pH, contact time and temperature. Adsorption kinetics, equilibrium isotherm and thermodynamic parameters of the process were also investigated. The reversibility of Cr (VI) adsorption by GAC and effects of different parameters on the amount of

Cr (VI) desorbed, was investigated by desorption process to evaluate repeated availability performance of the adsorbent in water treatment. In addition, the adsorption process was simulated using an artificial neural network to predict the adsorption equilibrium of Cr (VI) onto a GAC at different operating conditions.

2. MATERIALS AND METHODS

The granular activated carbon used in this study was purchased from Norit Americas Inc. (Norit Nederland BV) with surface area 1100 m^2/g and the bulk density around 1 gr/cm^3. The stock solution of Cr (VI) was made by dissolving a known amount of $K_2Cr_2O_7$ (analytical grade) in a pre-determined volume of double distilled water.

Different dosages of the adsorbents were added to the solutions. The adsorption process was taken place in a shaking flask (at 190 rpm) at 3 different temperatures (17, 27 and 37 °C), so that adequate contact time between adsorbent and the metal ion was obtained. The suspension was filtered after a regular interval of time through Whatman No. 42 filter paper and the filtrate was analyzed to evaluate the concentration of Cr (VI) metal in the solution. Metal analysis was carried out by using a Cary50 model UV-visible spectrophotometer.

To study the effect of adsorbent dosage, initial concentration of Cr (VI) and solution pH, the experiments were conducted in different GAC dosage from 0.025 to 0.3 g, initial concentration of Cr (VI) from 30 to 350 mg/l and solution pH from 2 to 10. The adsorption isotherm experiments were conducted at 17, 27 and 37 °C for the adsorbent dosage varying from 0.04 to 0.24 g with initial concentration of Cr(VI) 70 mg/l and pH about 2. The kinetics study of the process was done at 17, 27 and 37 °C with 0.2 g for the adsorbent dosage and 70 mg/l for initial concentration of Cr (VI). The samples were taken and analyzed at every 50 minutes up to 250 minutes.

To evaluate the reversibility of Cr(VI) adsorption and adsorbent regeneration, 200 mg of adsorbent were added into 100 ml of Cr (VI) solution with initial concentration of 80 (mg/l). As the adsorption reached equilibrium, the amount of Cr (VI) adsorbed onto adsorbent (q) was measured and then the solution was filtered using a Whatman No. 42 filter paper to recover the adsorbent. The adsorbents were added into 100 ml of water, NaOH and HNO_3 solutions in order to determine the effect of pH of regeneration solution on desorption process. The regeneration solution strength, contact time and temperature were verified from 0.03 to 0.5 M, 2 to 48 hr and 15 to 35 °C respectively to study the effect of these parameters on the recovery and regeneration amount. The adsorption/desorption process was repeated for 10 cycles.

3. THEORETICAL STUDY

3.1. Modeling of adsorption kinetics

The study of adsorption kinetics is significant as it provides valuable insights into the pathways and the mechanism of the process.

Several kinetics models are used to explain the mechanism of the adsorption processes. A simple pseudo-first order equation is given by Lagergren equation [15]:

$$\log(q_e - q) = \log q_e - \frac{K_1}{2.303} t \quad (1)$$

Where q_e is the amount of solute adsorbed at equilibrium per unit weight of adsorbent (mg/g), q is the amount of solute adsorbed at any time per unit weight of adsorbent (mg/g) and k_1 is first order adsorption rate constant.

On the other hand, the pseudo-second order equation based on equilibrium adsorption is expressed as [15]:

$$\frac{t}{q} = \frac{1}{K_2 q_e^{\,2}} + \frac{1}{q_e} t \quad (2)$$

Where k_2 is the pseudo-second order rate constant (g/mg. min). The linear regression correlation coefficient value shows that which model can justify our data.

3.2. Modeling of adsorption isotherm

To examine the relationship between adsorbate concentrations in the solid and aqueous phases at equilibrium, sorption isotherm models are widely employed. The Langmuir and Freundlich models are most widely used to model the equilibrium data of adsorption.

According to Langmuir model, adsorption occurs uniformly on the active sites of the adsorbent, and once an adsorbate occupies a site, no further adsorption can take place at this site. Thus, the Langmuir model is given by the following equation:

$$\frac{C}{q_e} = \frac{1}{bQ_0} + \frac{C}{Q_0} \quad (3)$$

Where Q_0 and b are the Langmuir model parameters. C is the equilibrium solution concentration (mg/l) of Cr (VI) in aqueous phase and q_e is the equilibrium amount of Cr (VI) adsorbed into the adsorbent (mg/g) [16].

The Freundlich isotherm is an empirical model that is based on adsorption on heterogonous surface and is given by the following equation:

$$\log q_e = \log K + \frac{1}{n} \log C_e \quad (4)$$

Where k and n are Freundlich constants, which represent adsorption capacity and adsorption intensity, respectively [16].

3.3. Simulation with neural network

ANNs are used to correlate the complex relationship between the input and output of any process irrespective of the physical meaning of the system. It consists of an input layer, an output layer and at least one hidden layer which the layers are containing several interconnected nodes (neurons). Numbers of neurons in input and output layers are related to matrix of input and output data. Number of neurons in hidden layer is a fitting parameters and it can be calculated with trial and error technique but recently genetic algorithms (GA) is applied

to this calculation. In addition, a genetic algorithm (GAs) is used to determine the momentum and the learning rates for minimizing the time and effort required to find the optimal architecture.

ANN simulations are flexible and well trained and can perform perfectly, where empirical or experimental data are enough [17]. There are powerful built-in features in Neuro Solutions for Excel to find the optimum neural network for a problem. These include the ability to train a neural network multiple times, vary any neural network parameters across multiple runs, genetically optimize network parameters, and create your own custom batches in this study the input vector is temperature, initial concentration, PH, dose of adsorbent and time and the output vector is removal percent on the water pollutant.

4. RESULTS AND DISCUSSIONS

4.1. Effect of adsorbent dosage on removal efficiency

The removal of chromium by activated carbon at different adsorbent doses (0.025-0.3 g) for the chromium concentration 70 mg/l is investigated. The results are shown that the removal percentage of Cr (VI) increases rapidly with increasing in the GAC dosage due to the greater availability of the adsorbent (Figure-1). The increasing in adsorbent dosage resulted in an improvement of removal percentage from 21 to 92%.

4.2. Effect of initial Cr (VI) concentration on removal efficiency

The initial concentration of Cr (VI) provides an important driving force to overcome all mass transfer resistances of metal ions between the aqueous and solid phases. Figure-2 shows that when the initial Cr (VI) ion concentration increased from 30 to 350 mg/l, Cr (VI) adsorption removal decreased from 98% to 58% and the uptake capacity of GAC increased. The decreasing in removal percent was due to the saturation of the sorption sites on adsorbents. In addition, the increasing in uptake capacity of activated carbon with the increasing of Cr (VI) ion concentration is due to higher availability of Cr (VI) ions in the solution, for the adsorption. At Figure-2 is also shown that the curve after 350 mg/l became flatten. The adsorbent surface has determined and constant capacity that at the initial concentrations more than 350 mg/l this surface is saturated so that the removal percentage curve is become flatted.

4.3. Effect of initial pH on removal efficiency

The pH value of the solution is an important factor that controls the sorption of Cr (VI). Figure-3 shows the extent of removal of Cr (VI) as a function of pH. It shows that at lower pH, the Cr (VI) removal efficiency was higher and at higher pH the removal reduced considerably. With decreasing in pH, the amount of Cr (VI) removal increased with the optimum achieved at pH range 1-2. The chromium ion exists in different forms in aqueous solution such as $HCrO_4^-$, $Cr_2O_7^{2-}$, CrO_4^- and the stability of these forms is mainly dependent on the pH of the solution. The behavior for better adsorption at low pH by GAC may be attributed to the large number of H^+ ions present at low pH values which in turn neutralize the negatively charged adsorbent surface, thereby reducing hindrance to the diffusion of chromate ions.

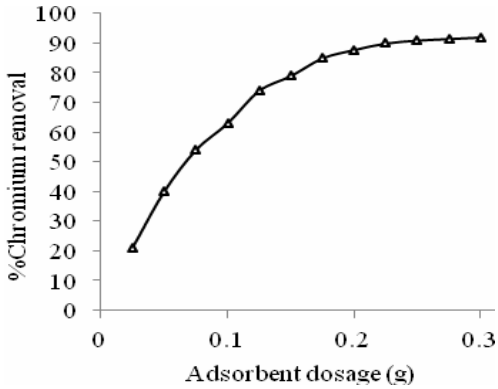

Figure-1. Effect of adsorbent dosage on Cr (VI) removal efficiency at pH=2, 70 mg/l initial Cr (VI) concentration and temperature 300 K.

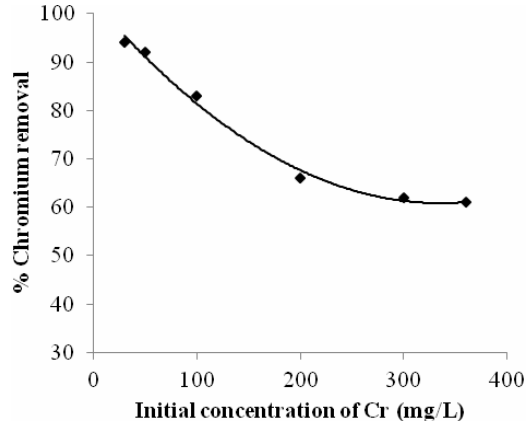

Figure-2. Effect of initial Cr (VI) concentration on Cr (VI) removal efficiency at pH=2, 2 g/l GAC dosage and temperature 300 K.

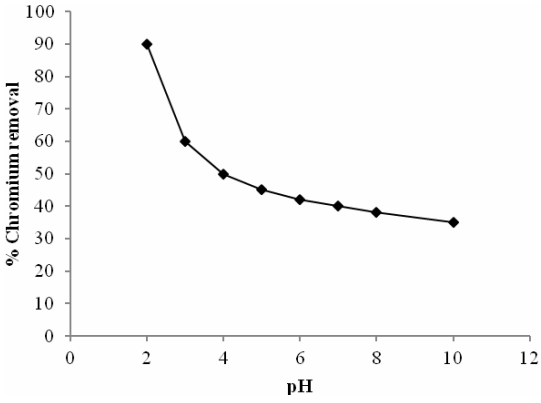

Figure-3. Effect of initial pH on Cr (VI) removal efficiency at 70 mg/l initial Cr (VI) concentration, 2 g/l GAC dosage and temperature 300 K.

4.4. Effect of contact time and temperature on removal efficiency

Figure-4 Shows the effect of contact time and temperature on the adsorption of Cr (VI) at 17, 27 and 37 °C on GAC. The temperature has two main effects on the adsorption process. An increasing in temperature is known to increase the diffusion rate of the adsorbate molecules across the external boundary layer and within the pores. Furthermore, changing the temperature will modify the equilibrium capacity of the adsorbent for a particular adsorbate. Results of effect of contact time on the process show that removal efficiency increased with an increasing in contact time before equilibrium is reached and after equilibrium removal efficiency would be constant. Results shows that the amount of Cr (VI) adsorbed increased with increasing in contact time and temperature.

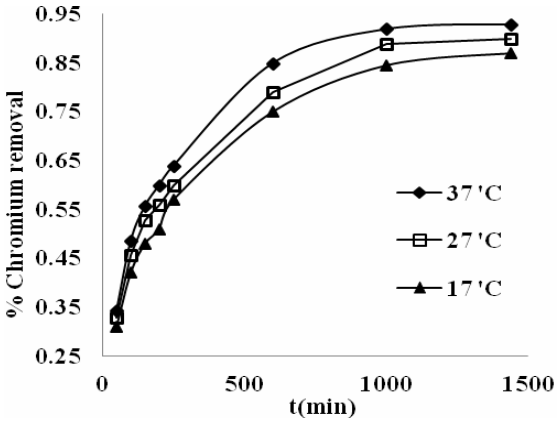

Figure-4. Effect of contact time and temperature on Cr (VI) removal efficiency at pH=2, 70 mg/l initial Cr (VI) concentration, and 2 g/l GAC dosage.

4.5. Kinetics of adsorption

The kinetics of Cr (VI) adsorption on the GAC was analyzed using pseudo first-order and pseudo second-order kinetics models (Figures 5, 6). The conformity between experimental data and the model predicted values was expressed by the correlation coefficients (R^2, values close or equal to 1). As seen in Table-1, the value of R^2 calculated from pseudo-second order kinetics is almost higher. These results indicate that the adsorption of Cr (VI) on the GAC follows pseudo-second order kinetics. The values of rate constant (K) and correlation regression coefficient (R^2) are calculated reported in Table-1 for two models.

Now with known values of rate constant and temperature, we can determine the adsorption activation energy by using the Arrhenius equation [18]:

$$K_2 = K_0 e^{\frac{-E}{RT}} \quad (5)$$

Where K_0 is the temperature-independent factor (g/mg. min), E is the sorption activation energy (kJ/mol), R is the gas constant (8.314 J/mol K) and T is the solution temperature (K). Arrhenius plot of the adsorption rate constants (K) versus temperature was shown in Figure-7. From the slope and intercept of the line k_0 is 0.04 g/mg. min and E is equal to 10.525 KJ/mol. Hence, the relationship between K and T can be represented in an Arrhenius form as

$$K_2 = 0.04 e^{\frac{-10.525}{8.314T}} \quad (6)$$

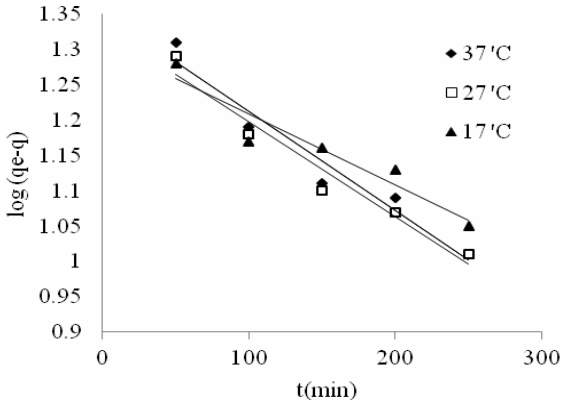

Figure-5. Pseudo-first order kinetics plots for the adsorption of Cr (VI) on GAC.

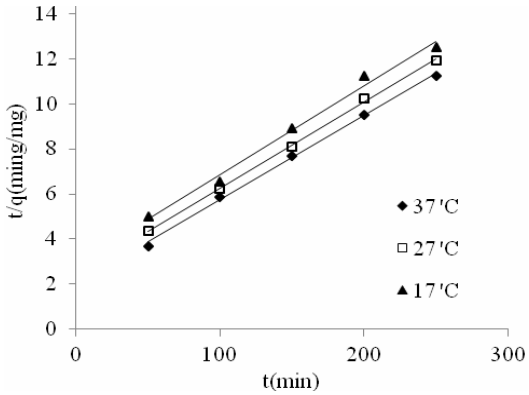

Figure-6. Pseudo-second order kinetics plots for the adsorption of Cr (VI) on GAC.

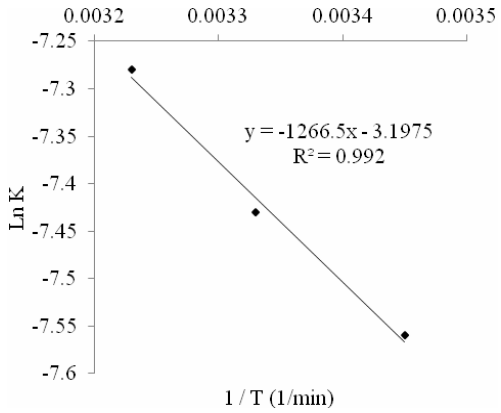

Figure-7. Determination of the adsorption activation energy, E.

Table-1. The kinetics model constants and correlation coefficients for adsorption of Cr (VI) on GAC.

Model		290 K	300 K	310 K
Pseudo first order	R^2	0.909	0.948	0.955
	K_1(1/min)	0.00069	0.00059	0.0005
Pseudo Second order	R^2	0.992	0.998	0.999
	K_2(g/mg.min)	0.00052	0.0006	0.00069

4.6. Adsorption isotherms

In this study, Langmuir isotherm has a better fitting model than Freundlich as the former have a higher correlation regression coefficient (Figures 8 and 9). This indicates the applicability of monolayer coverage of the Cr (VI) on the surface of adsorbent. This can be explained by the high surface area of the GAC. Therefore, only monolayer adsorption occurred on its surface, in spite of any surface modification. Constant parameters and correlation coefficients calculated for different adsorption models at different temperatures for Cr (VI) adsorption were reported in Table-2.

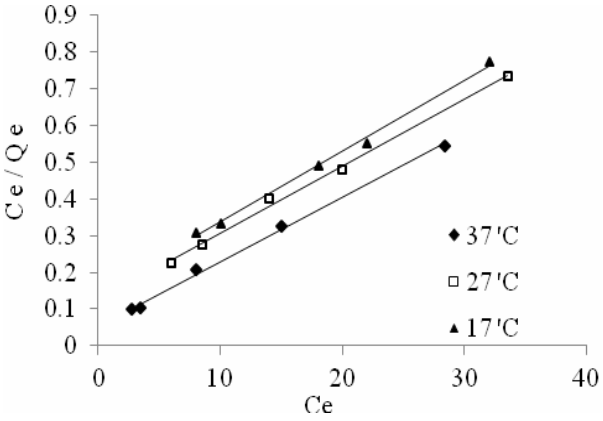

Figure-8. Langmuir plots for adsorption of Cr (VI) at different temperatures.

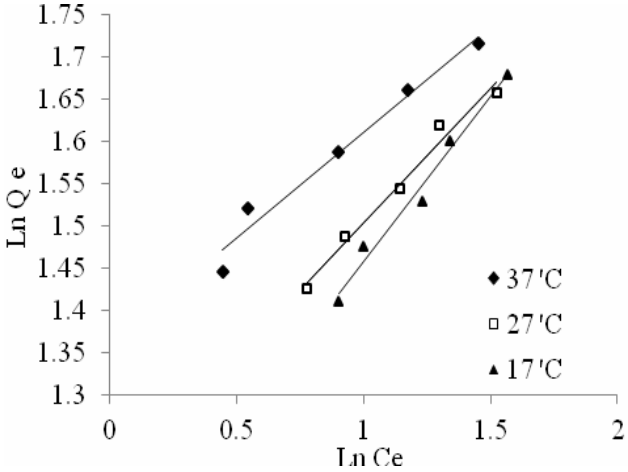

Figure-9. Freundlich plots for adsorption of Cr (VI) at different temperatures.

4.7. Thermodynamic parameters

Thermodynamic parameters such as Gibbs free energy (ΔG^0), enthalpy (ΔH^0) and entropy (ΔS^0) were calculated using the following equations [8]:

$$K_c = \frac{q_e}{C_e} \quad (7)$$

$$\Delta G^0 = -RT\ln K_c \quad (8)$$

$$\ln K_c = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (9)$$

Table-2. Isotherm model constants and correlation coefficients for adsorption of Cr (VI) on GAC.

Model		290 K	300 K	310 K
Langmuir	R^2	0.996	0.997	0.997
	Q_0 (mg/g)	51.81	54.64	57.14
	b (L/mg)	0.135	0.148	0.322
Freundlich	R^2	0.991	0.982	0.970
	k	11.77	15.37	22.95
	n	2.55	3.15	4

Where K_c is the equilibrium constant, q_e is the solid phase concentration at equilibrium (mg/l) and C_e is the equilibrium concentration in solution (mg/l). ΔH^0 and ΔS^0 were obtained from the slope and intercept of the Arrhenius plot of K_c versus T (Figure-10) and the standard Gibbs ΔG^0 values (kJ/mol) were calculated from the Eq. 8. Table-3 shows the calculated values of the thermodynamic parameters for the adsorption of Cr (VI) on GAC. The positive value of enthalpy change ΔH^0 for the process confirms the endothermic nature of the process, the positive entropy of adsorption ΔS^0 reflects the affinity of the adsorbent material toward Cr (VI) and the negative free energy values ΔG^0 indicate the feasibility of the process and its spontaneous nature. The amount of ΔG^0 (<10 Kcal) suggest that the adsorption is physical an also The increase in ΔG^0 with increasing temperature shows that the adsorption is more favorable at high temperature.

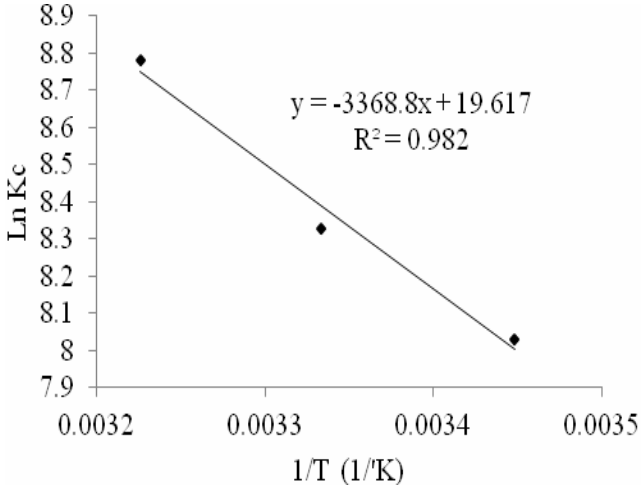

Figure-10. Graphical determination of ΔH^0 and ΔS^0.

Table-3. Thermodynamic parameters for the adsorption of Cr (VI) on GAC.

Temp. (°K)	ΔG° (KJ/mol)	ΔH°(KJ/mol)	ΔS°(KJ/mol.K)
290 K	- 19.36	28.008	0.163
300 K	- 20.78		
310 K	- 22.63		

4.8. Desorption results

4.8.1. Effect of regeneration solution pH on desorption

Figure-11 shows the recovery of Cr (VI) under 3 different pH of regeneration solution. It shows that at higher pH, the recovery of Cr (VI) and regeneration of GAC was higher. The main compound lead the Cr (VI) being recovered is OH^-. At low and neutral pH, the concentration of OH^- is lower than alkaline solutions, so the Cr (VI) recovery percentage is lower. Therefore NaOH Solution was selected as regeneration solution at desorption tests.

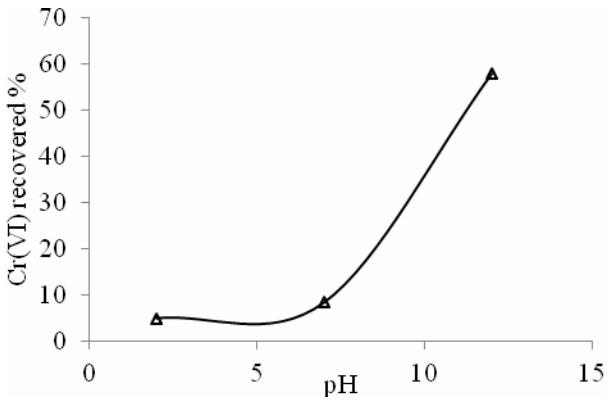

Figure-11. Effect of regeneration solution pH on desorption.

4.8.2. Effect of regeneration solution strength on desorption

The effect of strength of regeneration solution (NaOH) on the recovery of Cr (VI) and regeneration of GAC is shown in Figure-12. This Figure shows that when the strength of regeneration solution increased from 0.03 to 0.5 M, Cr (VI) recovered percentage increased from 16% to 61%. That's because of the higher OH^- concentration at higher NaOH solution concentration.

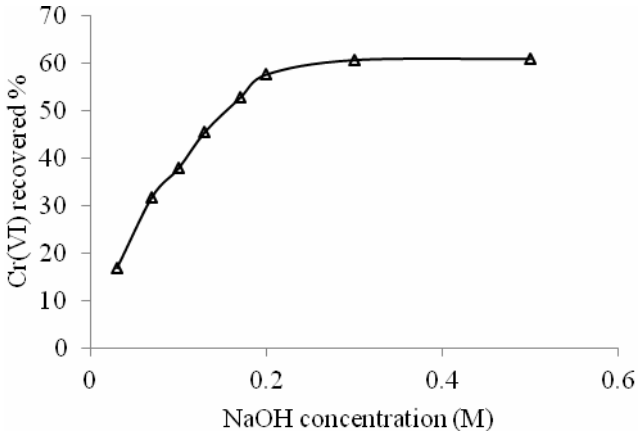

Figure-12. Effect of NaOH concentration on desorption.

4.8.3 Effect of contact time on desorption

Figure-13 shows the effect of contact time on percentage of recovered Cr (VI). Results of effect of contact time on the process show that desorption efficiency increased with an increasing in contact time before equilibrium is reached and after equilibrium Cr (VI) recovered percentage would be constant.

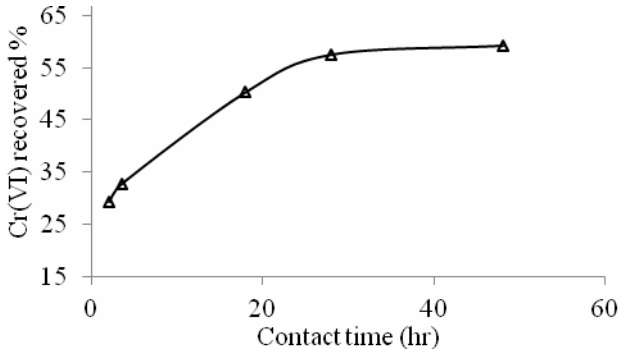

Figure-13. Effect of contact time on desorption.

4.8.4. Effect of temperature on desorption

The amount of recovered Cr (VI) is shown in Figure-14. Its obvious from this Figure that increasing in Temperature brings about increasing in recovery of Cr (VI) and regeneration of GAC. An increasing in temperature results in increasing of particle movements and this phenomenon facilitate the detachment of Cr (VI) from the GAC surface. Also this increasing shows that the desorption process is exothermic.

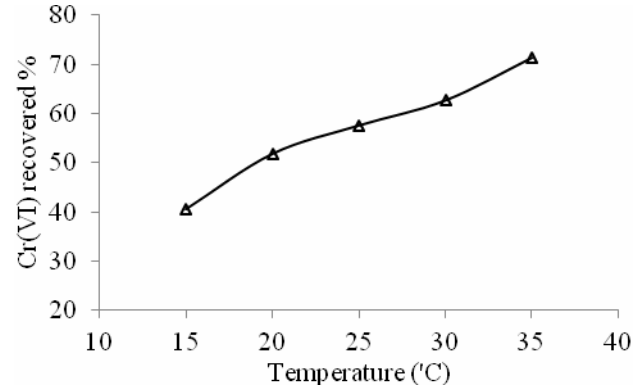

Figure-14. Effect of temperature on desorption.

4.8.5. Number of acceptable successive adsorption/ desorption processes

The Cr (VI) recovery and GAC regeneration under 10 cycles of adsorption and desorption (n) is presented in Figure-8. As the n increased the percentage of Cr (VI) recovered and adsorbent regeneration and also Cr (VI) removal, until 5 of these cycles slightly decreased and after that, until 10, sharply decreased. It is shown that GAC can be reused for 5 adsorption/desorption processes but after that it looses its adsorption/desorption ability and its performance drops down.

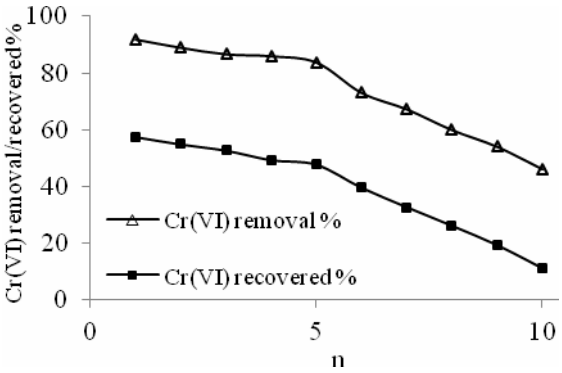

Figure-15. Number of adsorption/desorption processes.

4.9. Simulation results

In this study, the process of the networks designing is managed by NeuroSolutions for Excel Release 4.2 software, produced by NeuroDimension, Inc., which incorporates ANN and GA. It is used to obtain the optimal network size and parameters in the ANN adsorption amount of Cr (VI). The input vectors, 86 set of experimental data contain adsorbent dosage, initial concentration of Cr (VI), time and pH. There is just one neuron in output layer which is the amount of adsorption. The ANNs contain three layers and feed forward back propagation is used for training the input data. The number of neurons in the hidden layer is nine which is obtained by GA. The Figure-16 shows the plot of amount of adsorption predicted by ANN versus amount of adsorption obtained from experiments, during the training process. The slope line=1.01 and amount R^2=0.975 show good training of the network. The ANN-GA is tested with experimental data set.

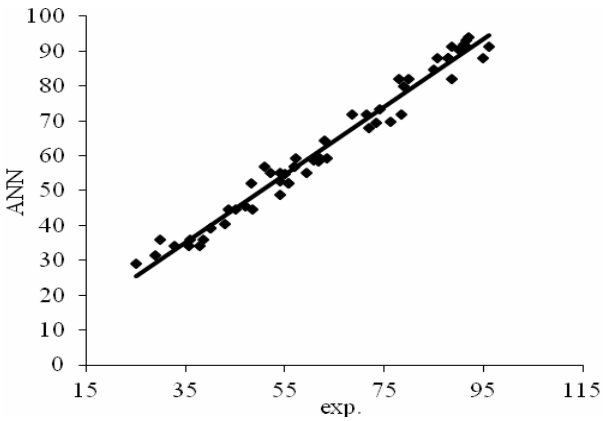

Figure-16. Comparison of experimental data and the simulation results in the training step (R^2=0.975)

ANN is a tool that can simulate the experimental data and can predict the result of conditions that have not been experimented. Furthermore, some models can predict the results of different conditions such as isotherm models. However, these models have some restrictions. In the equation of these models, some parameters such as pH is considered as constant parameter so that cannot predict the effect of parameters such as pH. ANN prediction is

extensive and includes the effect of parameters such as pH (Figure-17). Figures 18 and 19 show successful prediction of the modeling and simulation in the other conditions compared with experimental data. However it's obvious from Figure-20 that unlike the successful prediction of the simulation, modeling's prediction is not precise and acceptable.

To quantification of the comparison between experimental data and simulation results, the mean standard error (MSE), Normal mean standard error (NMSE), mean average error (MAE), minimum and maximum absolute error (AE_{min} and AE_{max}) and Regression fitting index (R^2) are calculated in prediction of amount of adsorption and reported in Table-4.

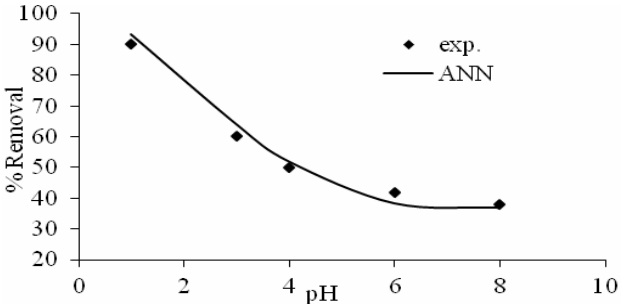

Figure-17. Comparison of experimental data and simulation results in the prediction step (pH effect) R^2=0.98.

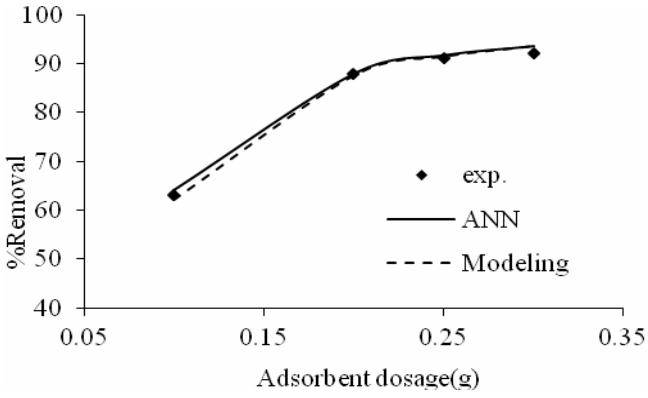

Figure-18. Comparison of experimental data, modeling and simulation results in the prediction step (Adsorbent dosage effect).

Table-4. Performance of GA-ANN simulation for test data set for network.

Performance	N
MSE	15.54898262
NMSE	0.047134634
MAE	2.967981341
AE_{min}	0.204873915
AE_{max}	11.94086174
R^2	0.979571558

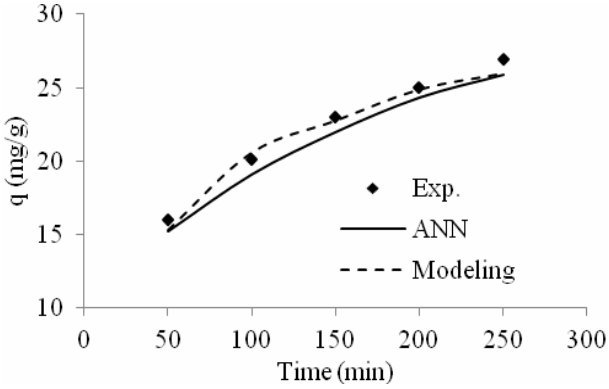

Figure-19. Comparison of experimental data and simulation results in the prediction step (Kinetics and Pseudo Second order equation).

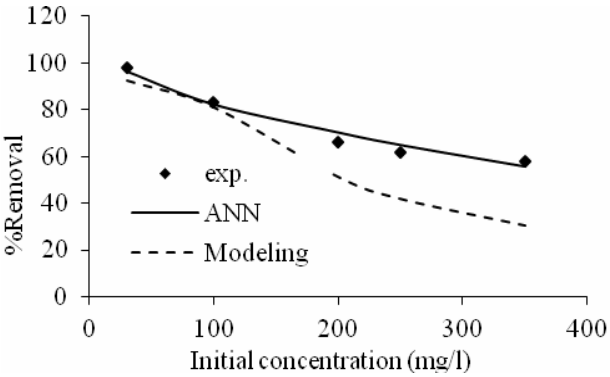

Figure-20. Comparison of experimental data, modeling and simulation results in the prediction step (Initial concentration affect.

5. CONCLUSIONS

The following major conclusions can be drawn based on the above study:

- The removal of Cr (VI) from aqueous solutions strongly depends on the pH of the solution, adsorbent dosage, initial Cr (VI) concentration, temperature and contact time. The maximum adsorption capacity was obtained at pH 2. Increase in the dose of the adsorbent and decrease in the initial concentration of Cr (VI) leads to increase in Cr (VI) adsorption and increase in temperature and contact time increases the Cr (VI) removal;
- The adsorption kinetics data were modeled using the pseudo-first order and pseudo-second order kinetics equations. It was shown that the pseudo-second order kinetics described best the sorption kinetics;
- The experimental results have been analyzed by Langmuir and Freundlich adsorption isotherms. Langmuir isotherm has a better fitting model than Freundlich as the former has a higher correlation regression coefficient. Thus, indicating the applicability of monolayer coverage of the Cr (VI) on the surface of adsorbent;

- The thermodynamic parameters ΔH^0, ΔS^0 and ΔG^0 values of Cr (VI) adsorption onto GAC show that the adsorption process is a spontaneous and endothermic;
- Desorption experiments showed that the percentage of Cr (VI) recovered and adsorbent regenerated, increase with increasing in pH and strength of regeneration solution, contact time and temperature. Results of Cr (VI) recycling and adsorbent regeneration showed that after 5 cycles of adsorption/desorption process GAC can be regenerated and reused well without any high decreasing in its performance; and
- Artificial neural network (ANN) was utilized for simulation of experimental results. A comparison between the simulated results and the experimental data gave high correlation coefficient (R^2 = 0.975) and simulation with the neural networks based on genetic algorithm could be applied to predict hexavalent chromium uptake purposes with high correlation coefficient (R^2 = 0.98).

Thus, the results show that the GAC can be effectively applied for the removal of Cr (VI) from wastewater.

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