Biological Forum – An International Journal ***(Special Issue 2015)*** **7**(2): 39-46(2015)

ISSN No. (Print): 0975-1130
ISSN No. (Online): 2249-3239

Optimization of chromium adsorption from aqueous solutions by synthesis hybrid Nano- adsorbent from graphene

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(Received 28 May, 2015, Accepted 25 July, 2015)
(Published by Research Trend, Website: www.researchtrend.net)

ABSTRACT: Removal of hexavalent chromium from aqueous solutions by functionalized hybrid Nano porous Graphene with carboxyl as absorbent was performed. It has magnetic with iron deposition on the adsorbent by Fe_3O_4 and Fe_2O_3 for remove of absorbent from water. After adsorption of chromium from water absorbent was removed with using a strong magnet. The effect of various parameters such as PH solution, contact time, adsorbent dose, temperature and initial concentration of chromium in the process of reducing the concentration of pollutants was assessed. Adsorption isotherms with using Langmayr and Freundlich models evaluated. The results indicate that the adsorption isotherms are consistent. The efficiency of functionalized hybrid Nano porous graphene with carboxyl at pH = 3, the optimal dosage = 0.2 gr / lit, the optimum time of 60 minutes and most adsorption in 25 mg / lit of pollutants concentration which percentage adsorption was 68%. Adsorption isotherm of chromium based on Freundlich using hybrid functionalized Nano-porous graphene adsorbent from aqueous solutions n value equal to 4.34 and the value of k equal to 147.23 and chromium adsorption Langmayr isotherm model q amount equal to 80.64 and the amount of b equal to 400 was calculated. High levels of R2 shows adsorbent follow both of Freundlich and Langmayr models and compatible with both models.

Keywords: Nano porous Graphene ?functionalized, magnetic, isotherm, adsorption.

INTRODUCTION

Chromium and its compounds are toxic pollutants in the wastewater produced in many industrial processes such as plating, leather, cement, dyed crust is found in abundance (Kobya, 2004). Chromium (VI) is highly toxic form for the health and human health. The high toxicity of the metal, even at low levels causing serious problems for the ecosystem (Luo *et al*., 2012). Different ways to remove it from aqueous solutions, such as sedimentation (Mukhopadhyay *et al*., 2007) reduction (Farrell and Breslin, 2004) ultra-filtration (Korus and Loska, 2009) adsorption (Chen *et al*., 2010) and etc. in this study adsorption on the hybrid functionalized Nano porous Graphene will used. Many organic and inorganic such as activated carbon (Daifullah *et al*., 2007, Ahn *et al*., 2009) Graphene (Wu *et al*., 2011, Xu *et al*,. 2012), purolite (Balan *et al*., 2013) chitosan (Hu *et al*,. 2011) Waste biological (Aliabadi *et al*., 2012) Dolomite (Albadarin *et al*., 2012) and etc for the removal of metal ions and organic materials have been used. Nano porous Graphene has been synthesized by CVD and is functionalized with carboxyl is used as an adsorbent for chromium adsorption. To facilitate the separation of the adsorbent from the aqueous solution used magnetic Fe_3O_4 (Bhaumik *et al*., 2011 and Bhaumik *et al*., 2010, Dos *et al*., 2008). As used magnetic adsorbent for separating the aqueous solution was needed and useful also has a negative effect on the absorption efficiency (Li *et al*., 2012). It may be filled active site on the surface of the absorbent to absorb pollutants by magnetic nanoparticles and reduce absorption efficiency

MATERIALS AND METHODS

A. Synthesis of Nano-porous Graphene

Precipitation chemical vapor (CVD) for the synthesis of Nano porous Graphene in large volumes and low cost is an effective method (Mattevi *et al*., 2011, Munoz *et al*., 2013). In this method for direct synthesis of porous Graphene 5 gr naphthalene as a carbon source put in quartz reactor tube for the synthesis of Graphene. Then by the rock wool separated carbon resource and catalyst. Catalyst consists of a copper foil in the reactor. Graphene is formed on the Metals such as copper (Li *et al*., 2009), nickel (Reina *et al*., 2009) and etc reported.

Copper is a better choice for the synthesis of Graphene because of low cost and flexibility. The next step put the tube in the reactor and heated until temperature of 1000 °C for 1 hour and by passing hydrogen gas activated copper catalyst and Nano porous Graphene is formed on the copper surface (Munoz *et al*., 2013).

B. functionalization of Nano porous Graphene

To functionalize Nano porous Graphene with carboxyl, 1 gr of Graphene in 25 ml of nitric acid and 75 ml of sulfuric acid for 3 hours at 60°C put in the Ultrasonic then with the distilled water washed until a neutral pH and dry the adsorbent at 60°C for 8 hours in the oven.

C. Synthesis of Magnetic Nano- porous Graphene functionalized

First 0.9 gr of Graphene with 250 ml of distilled water for 1 hour at 60ºC put in ultrasonic bath. 10 gr Fecl3 and 4 gr $FeCl_2$ dissolved in 25 ml of distilled water then under gas N_2 for 5 to 10 minutes with the mixer up added to the solution of Graphene and after 10 to 15 minutes at 80°C to the mixing up 5 ml of 28% ammonia are added drop by drop. With keeping the magnet at the bottom of the container, 3 or 4 times solution washed with ethanol and distilled water then dry absorbent in the oven at temperature of 80 ° C for 4 hours (Chandra *et al*., 2010, Li *et al* 2011).

D. Characterization

By scanning electron microscopy (SEM, edxS360, Mv2300) surface morphology, size and distribution of synthesized composite were analyzed. Characterization The XRD pattern was recorded on an X-ray diffract meter (XRD, Quantachrome,NOVA2000) with Cu-Ka radiation . Hybrid functionalized Nano porous Graphene surface area using N_2 adsorption at liquid nitrogen temperature (-196 °C) by transmission electron microscopy (TEM, PHILIPS, EM 208 S) with 100 keV. FT-IR spectrum of synthesized composite was measured on a spectrophotometer (Varian 3100, USA).

E. Experiments Adsorption

For preparation solution of Chrome 0.283 gr of Kr_2O_7 powder was dissolved in 1 liter of distillation water and 1,000 milligrams per liter of chromium stock solution was prepared. The PH solution was adjusted with a concentration of 1.0 M NaOH and HCL. Of this solution were used to provide standard solutions and removal tests. Chromium in water by Hybrid Functionalized Nano porous Graphene in different conditions of pH, contact time, adsorbent dose, the amount of pollutants and temperature was absorbed to achieve optimal value for each of the factors listed.

Determining optimum pH method. To determine the optimum pH of the 1000 mg/ lit Cr Stoke solution and each 100 mg chromium (VI) soluble added in 9 containers. The value of 0.035 gr of absorbent at laboratory was added at each of the nine containers in various quantities of pH (2, 3,4, 5, 6, 7, 8,9,10). The Shaker machine at 200 rpm per minute for an hour, and solids was removed with the help of a strong magnet and the amount of remaining chromium (VI) were measured by atomic absorption.

Determining optimum time method. The value of 0.035 gr from absorbent in laboratory temperature in optimum pH is added to six of 100 mil solution of chromium (VI) at 5, 15, 30, 60, 90, 120 minutes and put them on Shaker with 200 rpm then remove absorbent with used magnet then atomic absorption spectrophotometer was used to measure the amount of residual chromium (VI)

Determining adsorbent dose method. To obtain the optimal amount of NPG /Fe3O4+CooH for effective removal of chromium, different values adsorbent (0.02, 0.05, 0.1, 0.15, 0.2 gr) added to six dishes within a concentration of 100 mgr/lit of dissolved chromium in optimal pH and temperature laboratory. Dishes put on Shaker at the optimum time and 200rpm around were separated with magnet; the amount of residual chromium (VI) was measured by atomic absorption.

Determining the concentration of Cr (VI) method. Chromium solution pH adjusted based on the optimal pH then to the five different concentrations of hexavalent chrome solutions ranging from 25, 50, 100, 150, and 200 mgr/lit added at the laboratory and the optimum adsorbent then putting on the Shaker and by magnet separation took place and measuring the amount of Cr(VI) retained by atomic adsorption.

Determining optimum temperature method. First optimal pH of 1000 mg Stoke chromium is adjusted then this solution added to four 100 mil containers through the addition of optimize adsorbent dose in four temperature at 10, 20, 30 and 50 ° C for 60 minutes with around 200 rpm and then magnet remove adsorbent and measuring the amount of chromium retained by atomic adsorption.

F. Determine adsorption isotherms

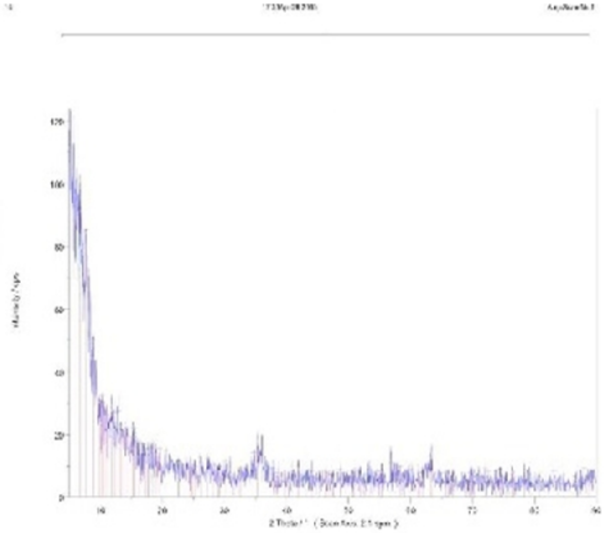
To determine the adsorption isotherm, Into five dishes100 ml of different concentrations of chromium solution (25, 50, 100, 150 and 200 mg/lit) under the optimal Condition (pH = 3, t = 60 min and 200 rpm) with the help of magnets separating solids, by atomic absorption spectrophotometer to measure the amount of residual Cr(VI). Adsorption isotherms were determined by using Excel software.

RESULTS AND DISCUSSION

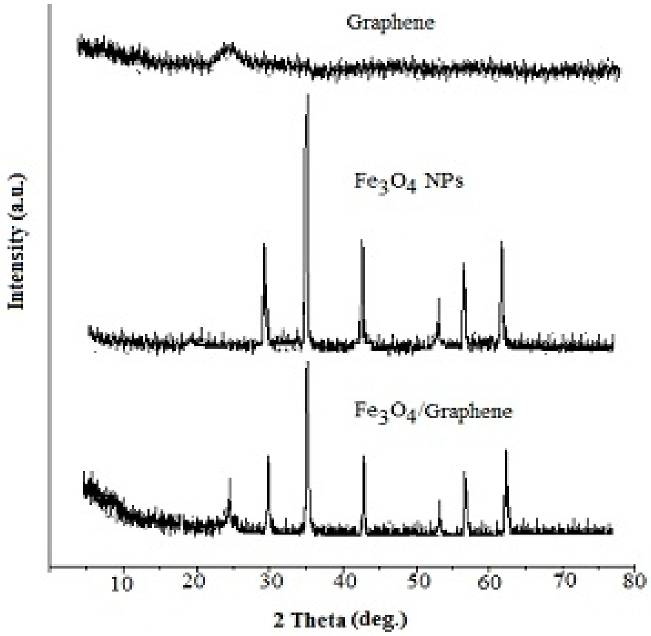
A. Characterization of the synthesized adsorbent

The XRD pattern of porous graphene, Fe_3O_4 nanoparticles and porous magnetic igraphene is shown in Fig. 1(a) and hybrid functionalized Nano porous Graphene is shown in Fig.1 (b). The angle of the adsorbent synthesized in the range of 2? = 5-90 °, with the use of radiation Cu k? (? =1.5 0A) shown. In this model, the carbon peak in the range of 24.7 marked.

Peaks aspects of nano particles on the absorbent structure in the range of 45.5 ° and 55.6 ° are shown (Hou *et al*., 2011, Hou *et al* 2011). A FT-IR spectrum of two composite of functionalized hybrid Nano porous Graphene before and after absorption of chromium (VI) is provided (c). In Part (d) FT-IR spectra of Nano porous graphene before and after functionalize are shown (d). The FTIR spectrum in the form of a 1586 wave of radiation vibrations is C = O (Bai *et al*., 2012, Gong *et al*., 2011). View of magnetic nanoparticles at a wavelength of 582cm^{-1} showing the Fe-O bonds between groups are in the form of Tetrahedron (Badruddoza *et al*., 2013) And groups that have emerged in the wavelength 1421.86 cm^{-1} represents aromatic C = C bonds and bond alkoxy CO limited at 1029 cm^{-1} appeared (Ji *et al*., 2011). The morphology, size and surface area of the nano porous graphene were analyzed by SEM and TEM. Image of porous graphene of SEM in Fig. 1 (f) is shown.TEM image of synthesis adsorbent is shown in part (g) of figure1, that's how nanoparticles of Fe_3O_4 sited on the synthesis adsorbent is shown. Morphology of the adsorbent in the TEM shows. Fe_3O_4 high density on absorbent and absorbent layer and Nano particle magnetic interface determined. The average size of Fe_3O_4 nano particles is about 30 nm (Fig. 1).



(a)



(b)

(g
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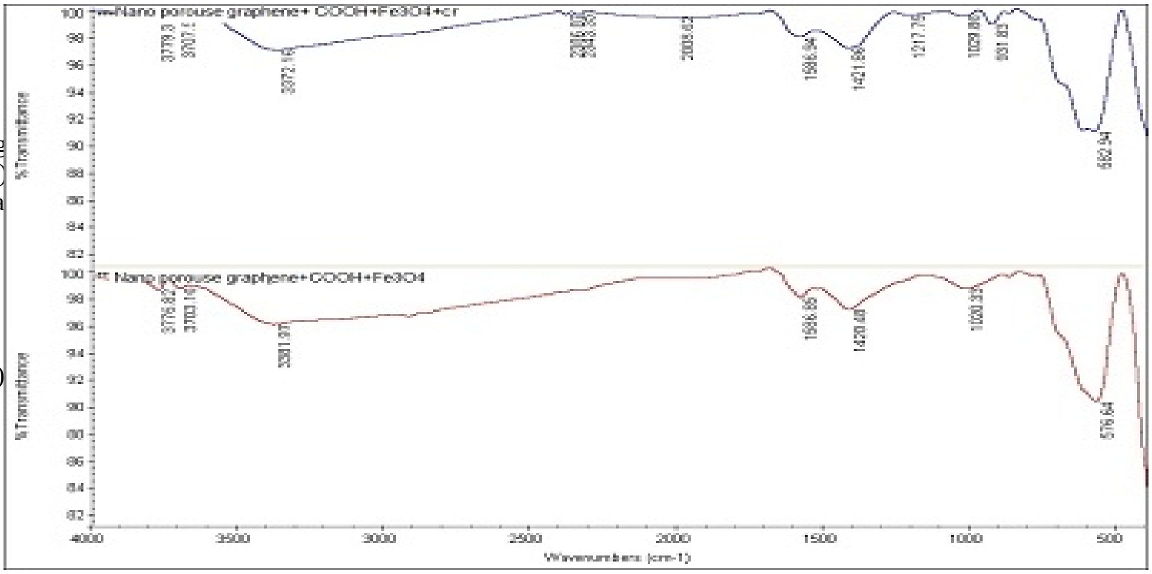
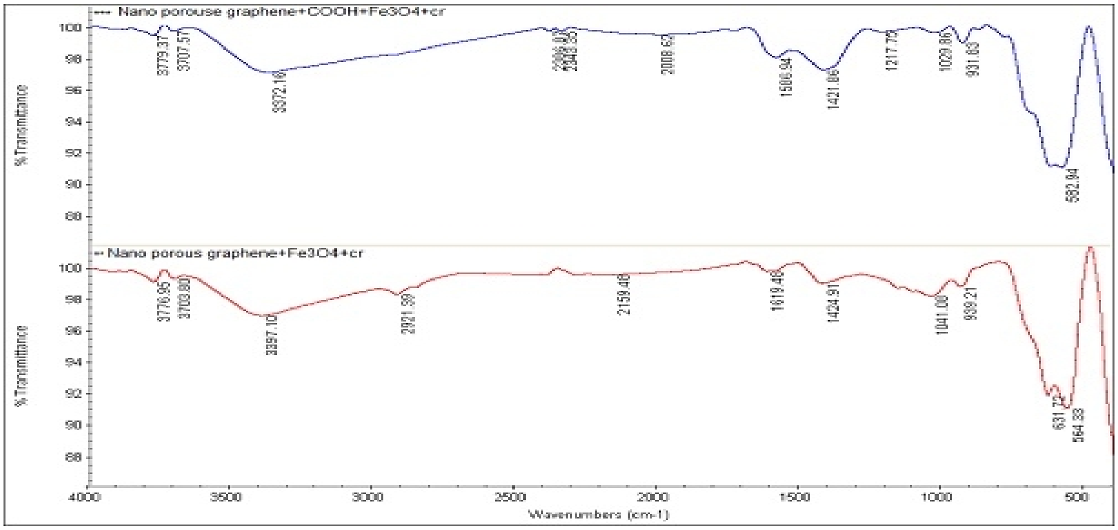
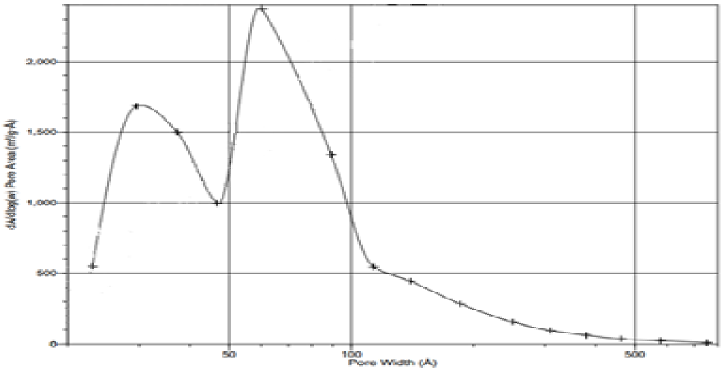


(c)

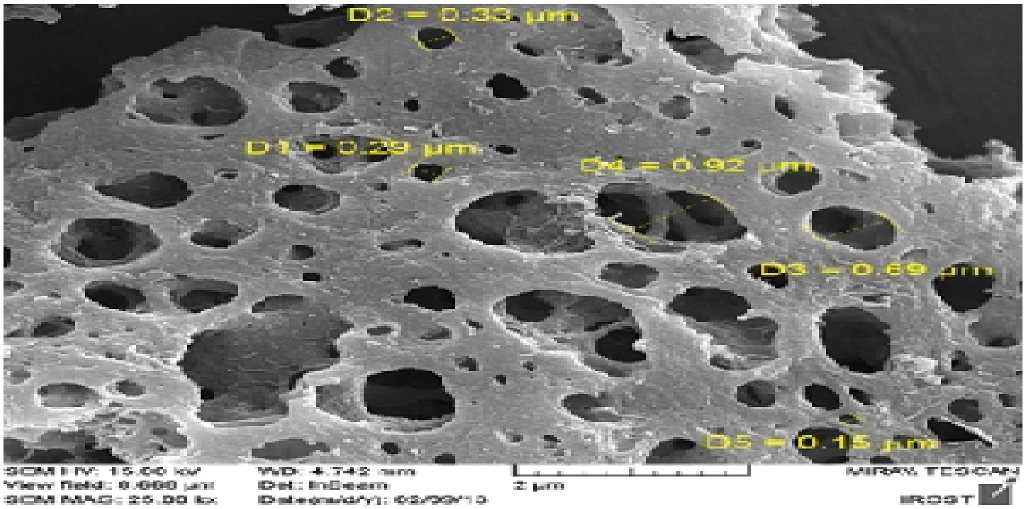


(d)

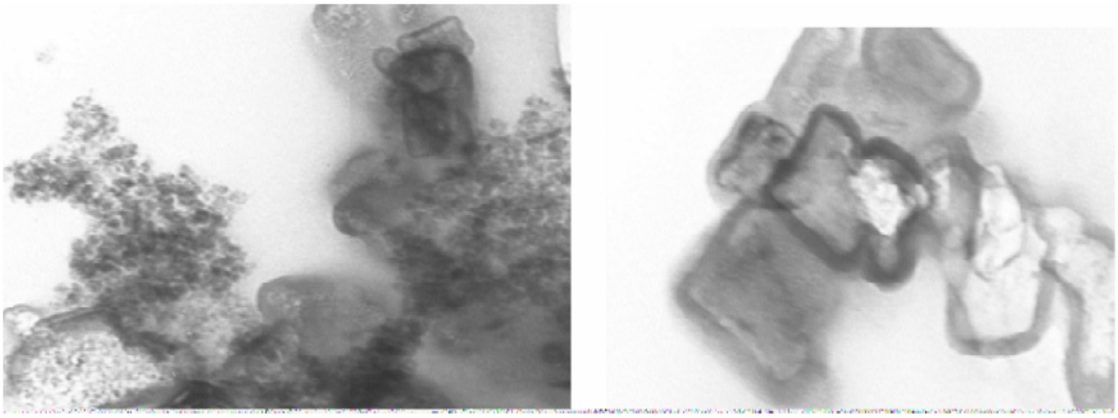
Sample	Surface area m^2g^{-1}	Pore size A°	Pore volume cm^3g^{-1}
G	850	99.5	2.11



(e)



(f)



(g)

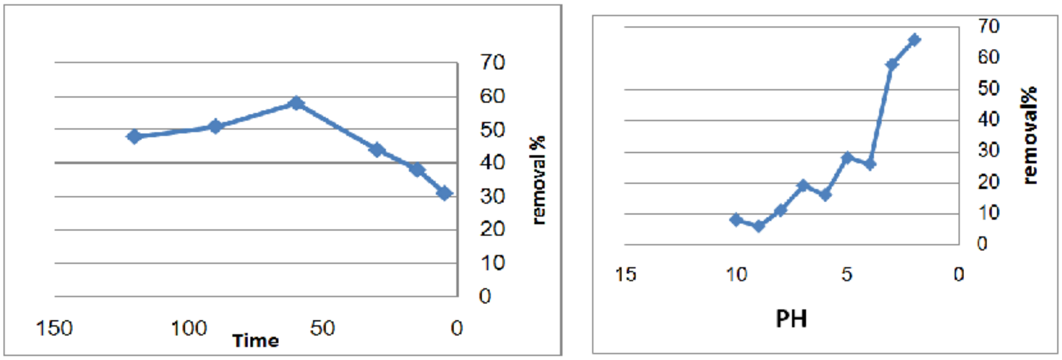
Fig.1. Characterization of synthesis adsorbent, Xrd analysisof graphene , Fe_3O_4 NPs, and Fe_3O_4 /Graphene(a), Xrd analysis of Hybrid functionalized Nano porous graphene(b) FT-IR spectra of synthesis adsorbent before and after Cr(VI) adsorption(c), FT-IR spectra of synthesis adsorbent before and after functionalize(d), BET analysis of Nano porous graphene (e), SEM image of Nano porous graphene(f), TEM image of Nano porous graphene(g).

B. The effect of PH

The pH of the solution on the Cr(VI) removal is one of the main parameters that plays an important role in controlling the absorption process (Cho *et al* 2009). The amount of chromium removal in various value of pH from 2 to 10 in Fig. 2(a) are shown. It can be seen that the reduction efficiency of Cr (VI) increasing by increasing the acidity of the solution. The maximum rate adsorption in pH 2-3, the research is consistent with the results (Suksabye *et al*., 2007 and Bhattacharya *et al*., 2008). Also in similar studies pH=3 is optimum pH for the removal of chromium (VI) by nZVI- Fe_3O_4 Nano composite, activated carbon and saw dust (Selvi *et al*., 2001, Lv *et al* ., 2012) in the acidic pH, chromium (VI) various forms (Cr_2O_{72}-, $HCrO_{4-}$, Cr_3O_{10} 2-V2- Cr_4O_{13}), all of which are bare negative charge(Lv *et al*., 2011) it is concluded that the reduction process (i.e., the reduction of Cr(VI) to Cr(III)) at acidic condition promotes the efficiency of Cr(VI) removal, which was also suggested by other reports in the literature (Wu *et al*., 2013).

C. The effect of time

As shown in Figure 2 (b) is evident increasing adsorption of chromium in the optimal pH =3 and 0.035 gr of absorbent to increase contact time to 60 minutes, but then it reached equilibrium state, raising the contact time the availability of Cr(VI) ions to the active sites on the adsorbent surface is limited, which makes the adsorption efficiency reduce (Kakavandi *et al*., 2014).



(a) (b)

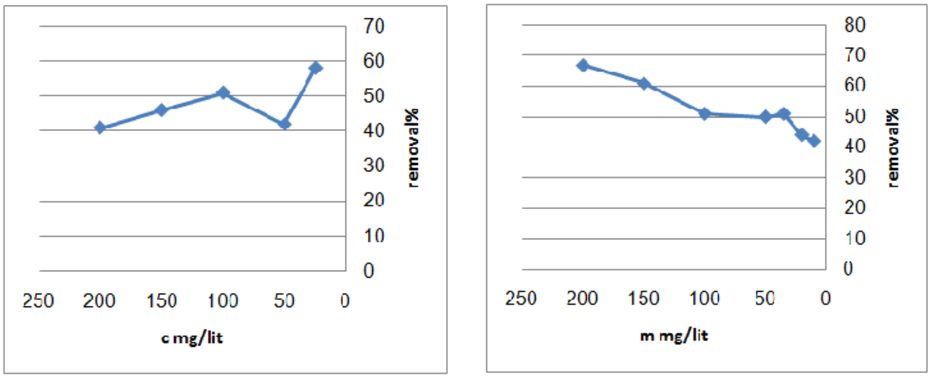
Fig. 2. The effect of pH (a) and the effect of time (b) on the adsorption efficiency.

D. The effect of adsorbent dosage

Effect of The absorbent optimal concentration on the Cr(VI) removal in optimal condition (pH=3, t=60 minutes, 200rpm) (a) is shown in Fig. 3(a). It is clear by increasing the absorbent, Cr(VI) removal from aqueous solution has increased .in similar studies obtained similar results (Kakavandi *et al*., 2014).

E. The effect of Cr(VI) concentration

The Effect of chromium different concentrations including 25, 50, 100 and 200 mg/lit in optimum conditions (pH=3, time=60, 200rpm, m0=0.2 gr) is shown in Figure 3(b). By increasing the concentration of pollutants reduced absorption percentage, main reason for this result seems limited active site on the absorbent surface to absorb chromium (Lv *et al*., 2012, Jung *et al*., 2013).



(b) (a)

Fig. 3. (a) Adsorbent dosage effect on Cr (VI) removal and (b) Cr (VI) concentration effect on adsorption.

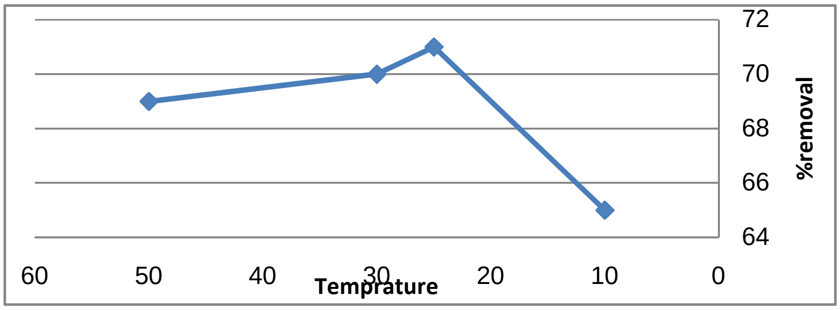


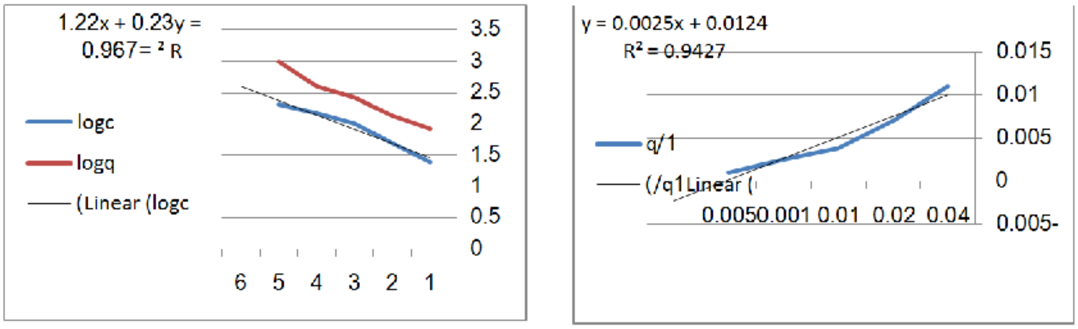
Fig. 4. The effect of temperature on Cr(VI) removal.

F. The effect of temperature

In this experiment, increasing the temperature to 20°C increases the absorption efficiency, but thereafter no effect on the absorption process. Results show that temperature increases up to a certain amount leading to increasing of absorption.

G. Adsorption isotherms

Figure 5 shown chromium (VI) adsorption isotherms. The Langmuir and Freundlich isotherm models were used to evaluate Cr(VI) adsorption on the absorbent in optimal condition (25 to 200 mg/lit metal concentrations, pH 3, the contact time of 60 minutes, adsorbent dose 0.03 gr/lit) With respect to Figure 5 (a) to the equation y = 0.0025x + 0.0124 respectively. The amount of 1/q equal to 0/0124 and thus the amount of q equal to 80/64 was calculated. The amount of 1/qbc equal to 0/0025 in result of b equal to 400 was calculated. Figure 5 (b) is shown Freundlich isotherm model in optimal condition the equation y = 0 / 23x + 1.22 was calculated. The amount of 1/n logC0 equal to 0.23 in result of n value equal to 4/34 calculated, also value of k equal to 147.23 was calculated.



(a) (b)

Fig. 5. (a) Langmayr model of Cr (VI) adsorption isotherm using hybrid Nano- porous graphene functionalized (b) Freundlich model of Cr (VI) adsorption isotherm using hybrid Nano- porous graphene functionalized.

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