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Effect of Process Variables, Adsorption Kinetics and Equilibrium Studies of Hexavalent Chromium Removal from Aqueous Solution by Date Seeds and its Activated Carbon by $ZnCl_2$

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Abstract

The adsorption of hexavalent chromium by preparing activated carbon from date seeds with zinc chloride as chemical activator and granular date seeds was studied in a batch system. The characteristics of date seeds and prepared activated carbon (ZAC) were determined and found to have a surface area 500.01 m^2/g and 1050.01 m^2/g , respectively and iodine number of 485.78 mg/g and 1012.91 mg/g, respectively. The effects of PH value (2-12), initial sorbate concentration(50-450mg/L), adsorbent weight (0.004-0.036g) and contact time (30-150 min) on the adsorption process were studied . For Cr(VI) adsorption on ZAC, at 120 min time contact, pH solution 2 and 0.02 adsorbent weight will achieve an amount of 35.6 mg/g adsorbed . While when use date seeds as adsorbent , conditions of 3 solution pH, 0.02 absorbent weight , and 120 contact time gave 26.49 mg/g adsorbed amount. Using both Langmuir, Freundlich and Sips models were explain the dsorption isotherms. It declare that the Sips model fits well with the experimental data with a maximum Cr(VI) adsorption capacity for (ZAC) and granular date stone 233.493 and 208.055 mg/g, respectively . The kinetics data which obtained at different initial Cr(VI) concentrations were examined by using pseudo-first-order, pseudo-second-order, and intra-particle diffusion models . The result gained showed that the second-order model was only describing well the empirical kinetics data of both (ZAC) and granular date seeds. It was noticed that the granular date seeds has adsorption performance lower than the (ZAC).

Keywords: Activated carbon, chemical activation, zinc chloride, date Seeds, Chromium Ion, Kinetics, Isotherm, Adsorption.

1- Introduction

The heavy metal ions presence in natural by the activity of volcanic and weathering of rocks and it can be found in a variety of industries, their potential impact is a major concern for a many research in environmental science due to their toxicity to many life forms [1].

Chromium ion is one of the contaminants which exist in hexavalent and trivalent forms [2]. Trivalent chromium is an essential element in humans and is much less toxic than the hexavalent one [3]. Overexposure to Cr(VI) causes cancer in the digestive tract and irritation and corrosion of the skin and, probably, to lung carcinoma and may cause epigastric pain, nausea, vomiting, severe diarrhea and hemorrhage [4]. Before wastewater transport and cycling into the natural environment, it is therefore fundamental to abstraction Cr(VI) from wastewater.

Chemical and physical processes such like electro-chemical precipitation, ion exchange, chemical precipitation, adsorption, ultra-filtration, electro-dialysis and reverse osmosis are available for the reduction of Cr(VI) concentration in wastewater [5-9] . Toxic sludge generation, incomplete metal elimination and high cost are the main defects of these processes.

The minimal cost treatment option had selected to the activated carbon adsorption, especially in processing low concentration of the wastewater streams and in meeting rigid level of treatment [1].

The black solid activated carbon substance similar to granular or powder charcoal and are carbonaceous material that have highly developed porosity, internal surface area and relatively high mechanical strength [10]. Removing a large variety of the toxic pollutants due to high removal efficiency in the activated carbon based system promoted the activated carbon adsorption to be widely attractive chosen in the treatment of chromium containing wastewaters [11-13].

Commercial activated carbons are very expensive, therefore it can be developed from low cost materials such as agricultural waste products, biomass and various solid substances [14-17]. Agricultural biomasses mainly consist of lignin, cellulose, hemi-cellulose and some proteins. Various low cost adsorbent from agricultural wastes were used in multi research workers like as coconut coirpith, saw dust, rice husk, banana pith, cottonseed hulls, apple wastes, sugarcane bagasse pith, peanut hull [18].

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The production of activated carbon from dates' seeds were concentrated mostly on the development of the high quality activated carbon [19] . Activated carbon were prepared from two processes; Physical activation processes, this process is usually carried out in two stages. The first stage is the carbonization stage to eliminate the bulk of the volatile matter and this achieved by using steam, nitrogen or carbon dioxide which used for mild oxidation followed by an activation stage to develop the porosity and surface area[20 , 21] . Chemical activation processes in which the raw material is impregnated with activating agents such as H_2SO_4 , H_3PO4 , $ZnCl_2$, and KCl; then heated at different temperatures [22-26] .Chemical activation by zinc chloride improves the pore development in the carbon structure, and because of the effect of chemicals, the yields of carbon are usually high [27].

The main objective of this research is to uptake toxic Chromium ion from different solutions concentration by developing a new type of activated carbon from date palm seed activated with zinc chloride and date seeds itself.

To estimate the maximum adsorption capacity of the toxic Chromium ion, it has been used a Batch adsorption process, and study the effect of process variables. Six mathematical models were also tested: the Langmuir's, Freundlich's and Sips's models for sorption isotherms, and first and second-order rate and intra-particle equations for description of kinetic.

2- Experimental Work

2.1. Materials

In the preparation of activated carbon, date seeds were applied as a biomass. To get rid of impurities of the date seeds, the seeds were first washed with water, dried at 110 ˚C for 24 h, for the preparation of activated carbon it has been selected fraction of particle sizes between 1 and 3 mm for this purpose the date seeds crushed using disk mill, and sieved. As dehydrating chemical reagent for activation of date seeds we used Zinc chloride (purchased from Didactic company) of purity 99.9%. A stock solution of Cr(VI) was prepared (1000 mg/L) by dissolving desired amount of potassium dichromate ($K_2Cr_2O_7$) in distilled water and to obtain total chromium concentration levels ranging from 50 to 450 mg/L the stock solution was diluted with distilled water . Hydrochloric acid, sodium thiosulfate, and iodine all these chemical were used for analytical grades.

2.2. Experimental Procedure Of Activated Carbon

The dried raw material weighting 10 g was well mixed with 100 ml of $ZnCl_2$ solution of impregnation ratio (weight of activating agent/weight of dried seeds) 1.25 g/g at room temperature for 24 h. For completely dried the impregnated samples were dried at 110 ˚C and after that the samples were stocks in a desiccator.

A stainless steel reactor (2.5 cm diameter x 10 cm length) which it had a removable cover with 2 mm and to allow the escape of the pyrolysis gases for this purpose the reactor has a hole at the center of that cover, the reactor other end was closed. The reactor was placed in a furnace and heated to carbonization the dried impregnated samples at constant rate of 10 ˚C /min and held at carbonized temperature 500˚C for carbonization time 2 h. To allow the carbonized samples to be cool after the end of activation time they were withdrawn from the furnace. Following that, the samples were placed with 0.1 M HCl solution, the liquid to solid ratio will kept 10 ml/g.

The mixtures were left overnight at room temperature, and then filtered and washed the samples repeatedly with distilled water until the pH of filtrate reached 6.5-7 [28].Following that , the samples were dried at 110 ˚C for 24 h, to calculate the yield of the activated carbon that produce it will be weighted . at last the samples were stocked in hermetic bottles.

To determine the yield of activated carbon, it was used the following equation:

$$\text{Yield}\,(\%) = \frac{W_f}{W_o}\ \text{x}100 \qquad (1)$$

Where W_f and W_o are the final dry weight activated carbon samples (g) and the dry weight of raw material (g), respectively.

2.3. Adsorbents Characteristics

Selected physical, chemical and adsorption properties were used to characterize the adsorbents such these properties like bulk density, surface area, ash content, pH, conductivity, iodine number and maximum Cr uptake.

The specifics of the characterization methods were illustrated as follows: Bulk density is a measurement of the weight of substance that can be included in a given volume under fixed conditions, it was calculated according to procedure that reported by Ahmedna et al. [29].

Moisture content was calculated for the adsorbent samples using oven drying method as has been illustrated by Adekola and Adegoke [30]. Ash content of the adsorbent samples is the remnant that remains when the carbonaceous portion is burned off [31].

Iodine number which is defined as the one gram of adsorbent samples adsorbed milligrams of iodine , the micropore content(0 to 20 Å) of adsorbent samples were measured by adsorption of iodine from solution,it was calculate by standard methods[32] . Surface area was estimated by applying of BET equation to the adsorption–desorption isotherm of N_2 at 77 K [33]. NOVAWin2 data analysis software was used to perform these calculations [34]. The morphology of prepared activated and raw material was examined by scanning electron microscopy (SEM) (300 K Pixel CMOS).

The concentrations of Cr(VI) ion was estimated calorimetrically. The maximum wavelength for absorbance at which the absorbance values of Cr(VI) will be measured was (λ max = 540 nm) by making the color of Cr(VI) solution a purple-violet color with 1,5-diphenylcarbazide in the acidic condition.

This was performed by Dissolving 0.25 g 1,5-diphenylcarbazide in 50 ml acetone stock in a brown bottle discard when the solution becomes discolored ,transfer 95 ml of the Cr(VI) solution to be to a 100 ml volumetric flask , add 2 ml diphenylcarbazide solution and mix , add H_2SO_4 solution to give a pH of 2 ± 0.5 , dilute to 100 ml with reagent water , and let stand 5 to 10 min for full color development and measured its absorbance [35].

2.4. Adsorption Isotherms

The adsorption experiments of Hexavalent chromium were executed in order to calculate the maximum adsorptive capacity of the prepared activated carbon and date seeds and this done by using 100 ml Erlenmeyer flasks. In each flask a volume of 20 ml of Cr(VI) solution with initial concentrations of 50–450 mg/l for Cr(VI) where put in the flask. By adding 0.1 M HCl solution the pH of the solutions was achieved to 2 . In the each flask ,adding 0.02 g of the prepared activated carbon and date seeds , with average particle size of 250 µm, and hold in a shaker (Type TR-1, Germany) at 120 rpm for 4.5 h for Cr(VI) solution to reach the equilibrium.

After that samples were filtered before the concentrations of Cr(VI) were analyzed by using UV - Visible Spectrophotometer (Shimadzu UV-160A) at maximum wave length 450 nm for Cr(VI) . The Cr(VI) final concentration at equilibrium, q_e (mg/g), was determent by the following equation:

$$q_e = \frac{(C_o - C_e)\,V}{W} \qquad (2)$$

Where C_o and C_e are initial and equilibrium concentrations of the Cr(VI) (mg/l), respectively, V is the volume of the aqueous Cr(VI) solution (l), and W is the weight of adsorbents used (g).

Equilibrium adsorption of Cr(VI) on the prepared activated carbon and date seeds was applied be fitting the experimental adsorption data by using the most widely isotherm equations , were these equation named the Langmuir[36], Freundlich[37] and Sips [38]. These isotherm equations can be written as:

Langmuir isotherm $$q_e = \frac{q_L K_L C_e}{1 + K_L C_e} \qquad (3)$$

Freundlich isotherm $$qe = kFCe1/n \qquad (4)$$

Sips isotherm $$q_e = \frac{q_s\, k_S C_e^{1/m}}{1 + k_S C_e^{1/m}} \qquad (5)$$

For the Langmuir isotherm, an equilibrium parameter, R_L, is the essential characteristics of this isotherm, which can expressed as follows [39]:

$$R_L = \frac{1}{1 + K_L C_o} \qquad (6)$$

where q_L (mg/g) is the maximum adsorption capacity of ZAC and date seeds , The Langmuir constant related to rate of adsorption is represent by K_L (L/mg) ,The Freundlich constants are K_F((mg/g) (L/mg)1/n) which give an idea about the adsorption capacity and n which represent the adsorption intensity, the Sips maximum uptake of Cr(VI) per unit mass of date stone and ZAC is represent by q_s (mg/g) is, Sips constant K_s((L/mg)1/m) which related to energy of adsorption , and constant m could be considered as the Sips constant characterizing the heterogeneous of system.

C_o is the highest initial solute concentration and where R_L is the type of isothermal, if when R_L equal to zero that mean the isotherm is reversible ,and when R_L greater than zero and less than 1,the isotherm is preferable ,and the isotherm is linear R_L equal to 1 , or not preferable when R_L greater than 1. To analysis experimental data, it has been used the least-squares retreat program according to Hooke-Jeevesand Gauss-Newton method. The parameters in each equation and harmonization between the experimental and determent data can be obtained by using this type of program in terms of correlation coefficient R^2.

2.5. Adsorption Kinetics

Adsorption kinetics provides valuable information about the reaction pathways and mechanism of the reaction. The kinetic experimental was done by employing a batch technique to study the effect of contact time and the uptake concentration of Cr(VI) adsorption. The aqueous samples were taken at preset time intervals, and the concentrations of Cr(VI) were similarly measured.

The diffusion steps that normally controlled any adsorption process are : (i) the solute will transfer to the film surrounding the adsorbent from the bulk solution , (ii) then the solute will transfer to the adsorbent from the film , (iii) at last the solute will transport to the internal sites from the surface followed by attaching of the metal ions to the active sites. The overall rate of the adsorption process will be determined by the slowest steps and usually it is considered that the step (ii) leads to the adsorption of the surface and the step (iii) leads to the adsorption in the intra-particle [2]. A q_t (mg/g) is the amount of Cr(VI) that adsorbed at time t , was estimated depending on the concentration of Cr(VI) at time t using

Eq. (1). Pseudo first-order model [40], pseudo-second-order model [41], and intraparticle diffusion model [42] were used to analyze the kinetic data. These models can be shown by the following equations:

Pseudo-first-order model

$$\ln(qe - qt) = \ln(qe) - K1t \qquad (7)$$

Pseudo-second-order model

$$\frac{t}{q_t} = \frac{1}{k_2 q_e} + \frac{t}{q_e} \qquad (8)$$

Intraparticle diffusion model

$$qt = K3t1/2 + C \qquad (9)$$

where q_e and q_t (mg/g) are the amount adsorbed of Cr(VI) at equilibrium and at time t (min), respectively, K_1 (1/min) is the adsorption rate constant, the rate constant of second-order equation is K_2 that has a unit (g/mg min) , the intraparticle diffusion rate constant is K_3 that has a unit (mg/g $min^{1/2}$) , constant that gives the idea about boundary layer thickness presented by C that has a unit (mg/g) . The application of kinetics models was shown by adopting the value of correlation coefficient R^2.

3- Results And Discussion

3.1. Yield and Characteristics

In Table (1) the yield and characteristics of ZAC and date seeds were determined and summarized. This Table shows 43.23% yield of ZAC which was higher than that reported by K. Suresh et al. [43] who found that 14.8 % yield could be obtained from date palm seed by CO_2 activation.

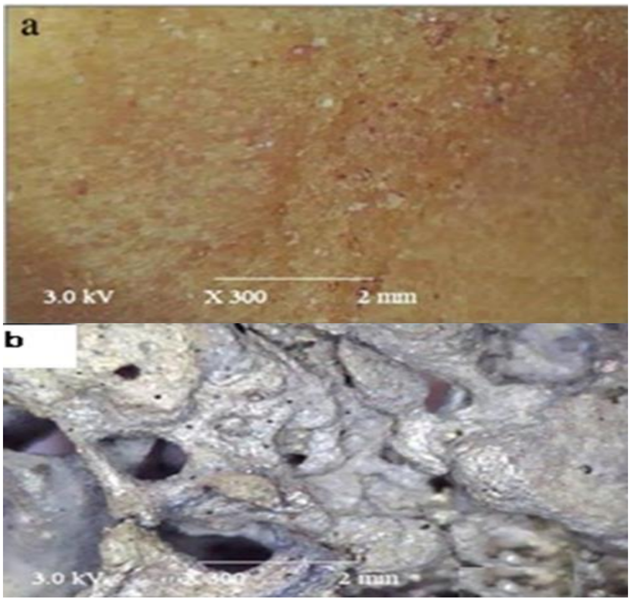

Fig. 1. SEM micrographs (300_x) of date seeds(a) and ZAC(b)

Table1. Characteristics of ZAC and dates seeds

Characteristic	ZAC	Date seeds
Yield(%)	43.23	
Bulk density (g/ml)	0.342	0.340
Surface area (m^2/g)	1050.01	500.03
Ash content (%)	2.04	1.66
Moisture content (%)	13.86	7.80
PH	6.1	4
Conductivity (μs/m)	320	
Iodine number (mg/g)	1012.91	485.78

The surface area and iodine numbers are consider the most important characteristics, the surface area of ZAC is 1050.01 m^2/g and for date seeds is 500.03 m^2 /g as has been shown in Table (1). Attia A. A. et al [5] showed that the prepared activated carbon that activated with sulphuric acid onto olive seeds has a surface area 448 m^2 /g. Because of higher activity of zinc chloride which make an activated carbon with high pore , it can be noted from Table (1) that iodine number of activated carbon was higher than that of date seeds, which mean ZAC has a micropore content greater than date seeds. the high surface area for an agricultural waste biomass enables date seeds to be used successively for removal of Cr from aqueous solutions.

Haimour and Emeish [44] showed that The iodine numbers activated carbon prepared by chemical activation of date seeds using phosphoric acid , was 485.78 mg/g and this result is less than that obtained in this study for ZAC.

The capability of date seeds to remove most of chromium ion and that because of the high iodine number of date seeds which is a measure of micropore content (0 to 20 Å), where the Cr(VI) ion have molecular sizes in the range of micropores content.

The SEM images for the prepared activated carbon and date seeds were shown in Fig. (1) .it can be notice from Fig. (1a), that the date seeds have a very smooth surface and have very few pores on its surface. Fig.(1b) shown The surface of the prepared activated carbon which activate with $ZnCl_2$, it can been seen become rough and many pores appear on the surface , and that become After $ZnCl_2$ impregnation and activation at activation temperature 500 ◦C, impregnation ratio 1.25 g/g and activation time 2 h ,the reason for that the $ZnCl_2$ impregnation had important effect on the increase in specific surface area and expansion of micropores .

3.2. Effect of Process Variables

In order to study the effect of solution pH (2-12), contact time (30-150 min.), and adsorbent weight (0.004-0.036 g) on adsorbed amount and removal percentage of Cr(VI) onto ZAC and date seeds, a set of experiments was carried out.

a. Effect of Solution pH

The pH of the solution has an effect on the adsorbent's charge density and the metallic species, so that the solution pH is consider one of the most parameters that have an influence on the metals ion adsorption [2].

The charge of the surface might be positive or negative. A large number of surface functional groups were in the pore wall of activated carbon. The of Cr(VI) adsorption depend on the solution pH can largely be related to the functional groups type and its ionic state and also on the chemistry of adsorbate in the solution[11].

The effect of pH on Cr(VI) adsorption was studied over a pH range of 2–12 on ZAC and date seeds samples at 50 mg/L initial concentration ,adsorbent weight 0.02 g , solution volume 20 ml and contact time 120 min as shown in Fig. 2.Where fig. 2 represent the prepared activated carbons and date seeds data ,it can been seen that the effectiveness of the adsorbents occur in the acidic range and maximum uptake occurred at pH 2 for ZAC and pH 3 for date seeds . The adsorption increases from 6.25 mg/g to 26.49 mg/g for date seeds and 9.5 mg/g to 35.6 mg/g for ZAC of pH from 12 to 2.When the pH was raised from 2.0 to 7.0 the capacity of the adsorption has a sharp decrease and in basic range the effect became negligible, due to high electrostatic force of attraction the higher adsorption became in the lower pH ranges. The most ionic forms that Cr(VI) may be exist in aqueous solutions are ($HCrO_4^-$, $Cr_2O_7^{-2}$, CrO_4^{-2}) and the pH has in effect on the stability of these ions in aqueous systems [45]. The diffusion of chromate ions increase into the bulk of the adsorbent depend on the lowering the solution pH which increase number of H+ ions ,and thereby the negative charge on adsorbent surface will be neutralize [46]. The mostly form of Cr(VI) adsorbed on the surface of activated carbon was $HCrO_4^-$ ions, it is well known that the pH between 1.0 and 4.0,the $HCrO_4^-$ ion was the stabile form of chromate ions [45].

The pH increase will be decrease adsorption amount and that because of increase the number of OH^- ions in the bulk which obstruct the diffusion of Cr(VI) ions. And that agree with other investigators have also been reported that [47-51] they found that the optimum pH value was in the range 2-3 for the removal of Cr(VI) from aqueous solutions.

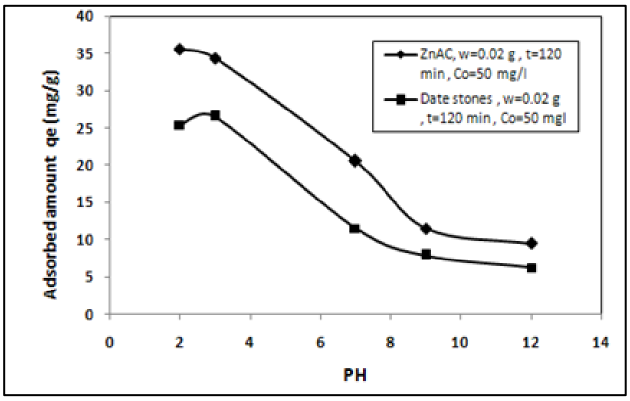

Fig. 2. Effect of solution pH on adsorbed amount

b. Effect of adsorbent dose

As shown in Fig.(3) the effect of sorbent dosages on the adsorption amount of chromium ion by both ZAC and date seeds on the pH 2 and 3 for ZAC and date seeds ,respectively , time 120 min , the ZAC and date seeds mass were varied from 0.004 to 0.036 g/ 20 ml, initial concentration of chromium ion 50 mg/l . As has been shown from Fig.(3) that the date seeds and the prepared activated carbons followed the predicted pattern of decreasing adsorption amount as the dosage was increased and reaches a saturation level at high doses for both date seeds and prepared activated carbon this is probably because of the resistance to mass transfer of Cr(VI) from bulk liquid to the surface of the solid .The adsorption decreasing from 52 mg/g to 14.22 mg/g for date seeds and from 75 to 16.99 mg/g for ZAC of adsorbent dosages 0.004 to 0.036 g/20 ml. Increasing adsorption with dose can be attributed also to the increase in surface area and the availability of more adsorption sites and which was noticed by A. A. Attia et al.[5].

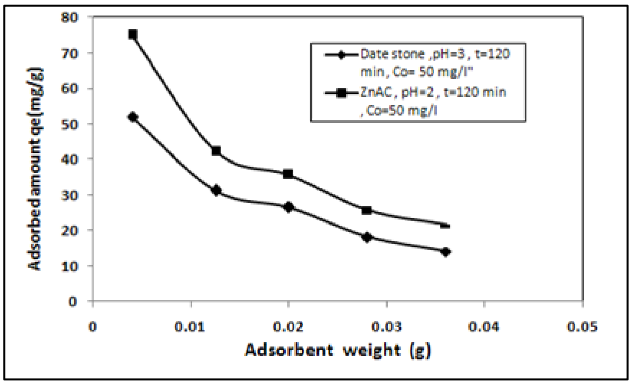

Fig. 3. Effect of adsorbent weight on adsorbed amount

c. *Effect of Contact Time*

The amount of Cr(VI) adsorbed on date seeds and ZAC was studied as a function of the mixing time at initial concentration of 50 mg/L of Cr(VI) at 0.02 g of adsorbent and the desired pH . The results are given in Fig. (4). It is clear from this figure that the adsorption of Cr(VI) increased with increase in contact time from 30 to 150 min ,for date seeds the adsorption amount increase from 15.778 mg/g to 27.216 mg/g and for ZAC 24.692 mg/g to 36.442 mg/g , then became slow up to 150 min and no change in the adsorption amount was observed with further increase the contact time up to 24 h in the case of both date seeds and ZAC samples. The equilibrium time by the nature and compactness of the adsorbents.

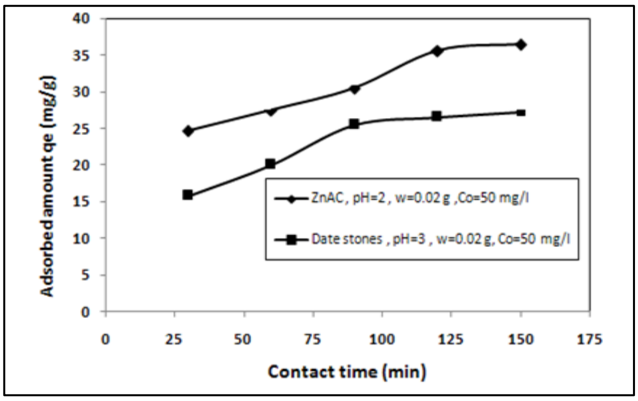

Fig. 4. Effect of contact time on adsorbed amount

3.3. Adsorption isotherms

Langmuir, Freundlich and Sips isotherms, Eqs. (3,4)–(5) are fitted with the experimental equilibrium data for Cr(VI) adsorption on ZAC and date seeds that calculated from Eq. (2). Table (2) presented the calculated constants of the three isotherm equations along with R^2 values. This Table shows that the Sips isotherm had the best correlation coefficient with R^2 values 0.9999 and 0.999 for adsorption of Cr(VI) on ZAC and Date seeds , respectively that because the Sips isotherm has ability to predict wide ranges of adsorbate concentration.

When correlate experimental adsorption data at was found that the sips isotherm as compared with Langmuir and Freundlich isotherms has a successful application with the three parameters[52,53]. In Fig.(5) Sips isotherm was using to correlate the experimental data . It can been seen from this figure that when using ZAC the adsorbed amount of Cr(VI) increases.

The Table (2) , showed the Langmuir isotherm, the values of R_L are 0.0297 and 0.1387 of ZAC and the date seeds , respectively, If the average of the R_L values for each of the different initial concentrations used is between 0 and 1 [54] it indicates favorable adsorption of Cr(VI) adsorption on ZAC and Date seeds. It can been seen from Table (2) that the fitting results of Freundlich model has a predict to favorability of adsorption when the value of (1/n) less than one, that will be represent a favorable adsorption condition [55,56] and that agree with the results obtained where the magnitude of the exponent, (1/n) 0.266 and 0.435 of ZAC and date seeds, respectively. The results of Table(2) show that the data which fitted to the and isotherms showed the correlated coefficient values R^2 for Langmuir higher than that of Freundlich isotherms, which approve the monolayer adsorption. In this study, Table (2) shows the Maximum Cr(VI) adsorption capacity of 233.493 and 208.055 for ZAC and date seeds , respectively , as has been calculated from Sips isotherm fitting.

Table (3) compared the maximum adsorption of Cr(VI) that obtained in this study to those obtained in the literature for activated carbons prepared from various agricultural wastes. It can be seen from this Table that the prepared activated carbon can be classified as one of the effective adsorbents for this purpose.

Table 2. Adsorption isotherm parameters for Cr(VI) on ZAC and Date seeds

Sample	Freundlich Isotherm			
	K_s((mg/g)	$(L/mg)^{1/n}$ 1/n	R^2	
ZAC	52.021	0.266	0.968	
GDS	15.456	0-435	0.989	
Sample	Sipes Isotherm			
	q_s(mg/g)	K_s((l/mg)$^{1/m}$)	m	R^2
ZAC	233.493	0.1008	1.2178	0.9999
GDS	208.055	0.1818	0.9462	0.9998
	Langmuir Isotherm			
Sample	q_m(mg/g)	K_L(l/mg)	R_L	R^2
ZAC	218.77	0.0726	0.0297	0.997
GDS	215.43	0.0138	0.1387	0.9998

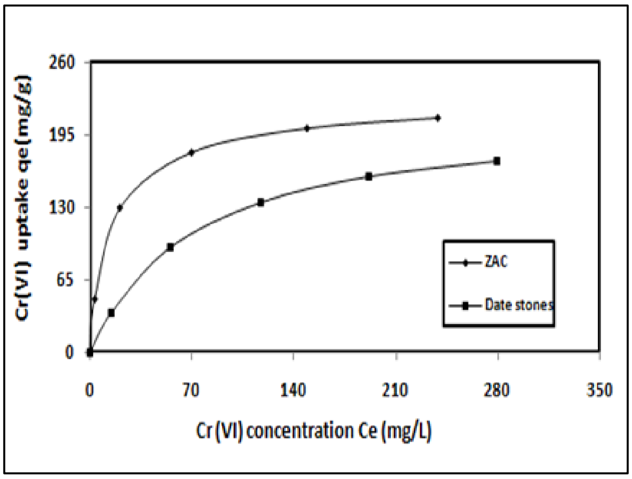

Fig. 5. Equilibrium isotherm of Cr(VI) adsorption on ZAC and Date seeds samples correlated with Sips equation (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 2 and 3for ZAC and date steeds respectively ; contact time, 5.5 h)

Table 3. Comparison of Cr(IV) maximum adsorption capacity onto activated carbon prepared from various precursors

Activated carbon	Activator	Maximum capacity (mg/g)	Ref.
olive bagasse-carbon	physical activation of steam	88.59	[2]
olive stones-carbon	sulfuric acid	71	[5]
Wood-carbon	KOH	180.3	[57]
Hazelnut shell -carbon	H2S04	52.2	[58]
Date stones-carbon	sulfuric acid	120.48	[59]
Tamarind wood -carbon	$ZnCl_2$	28.019	[10]
Date seeds-carbon	$ZnCl_2$	233.493	Present study

3.4. Kinetics of Adsorption

The influence of contact time on adsorption capacity of ZAC and date seeds for different initial concentrations of Cr(VI) illustrated in Fig.(6) and Fig.(7) .When time increase the adsorption capacity for Cr(VI) increase and that shown in these figures , and about 4.5 h the adsorption attained equilibrium . An adsorption capacity for ZAC and date stone, respectively of 210,65 and 170,03 mg/g is obtained at 4.5 h contact time, 450 mg/L initial Cr concentration, 2 pH value and 0.02/20 ml adsorbent dose. These figures also show that when increasing initial concentration the amount of Cr(VI) adsorbed, q_e (mg/g), increase . Moreover the adsorption was quick at the initial stages and dropped gradually after the equilibrium point till it reach almost constant. The removal of Cr(VI) will become independent of initial concentrations and that because of the available surface to initial Cr(VI) concentration ratio was larger at low concentrations , but the adsorption capacity will depend upon the initial concentration in the case of higher concentrations, because this ratio was low.

The possible of monolayer coverage for Cr(VI) on the surface of adsorbent because the curves showed that the adsorption led to saturation [11].

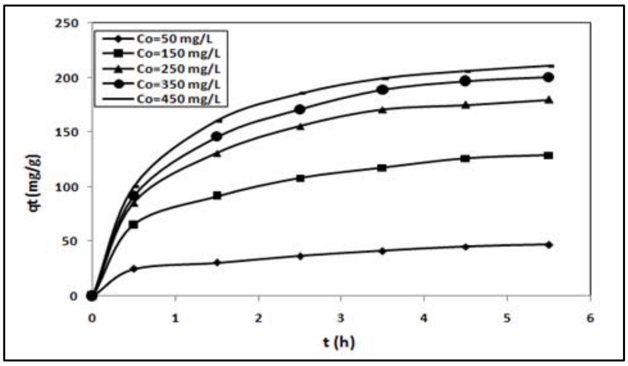

Fig. 6. Effect of contact time on adsorbed amount of Cr(VI) on ZAC at different initial concentration (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 2 ; contact time, 5.5 h)

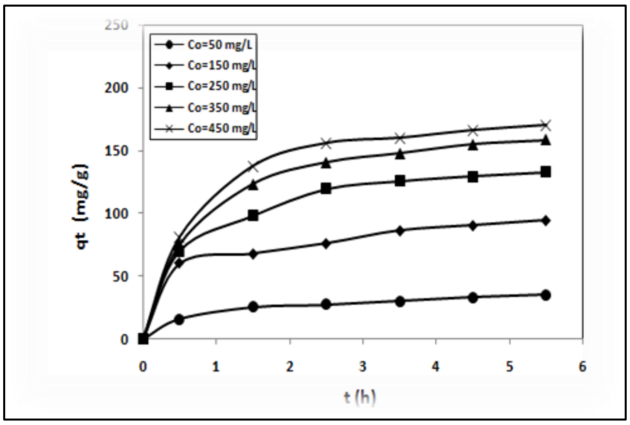

Fig. 7. Effect of contact time on adsorbed amount of Cr(VI) on date seeds at different initial concentration (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 3 ; contact time, 0.5-5.5 h)

The three kinetic models: pseudo-first order pseudo-second order and intraparticle diffusion models, Eqs. (7)–(8) ,where applied to the experimental kinetic data of Cr(VI), which determined from Eq. (2). Table (4) and (5) for ZAC and date seeds as adsorbents, respectively, showed the values of R2 and calculated constants of the three kinetic equations at different initial Cr concentrations.

The linear plot of ln(qe – qt) versus t (Fig.6) and (Fig. 7) for pseudo-first order equation , as shown in Table (4) and Table (5), the value of R2 was low and that representing a poor pseudo-first order fit to the experimental data. (Fig. 8) and (Fig. 9) with linear plot of t/qt versus t for pseudo-second order equation, High R2 values are obtained as shown in Table(4) and Table(5).

It clear that the adsorption kinetics better represent by pseudo-second order kinetic model, and that mean that the adsorption of Cr (VI) on ZAC and date seeds fitting with second-order kinetics.

Therefore, it may be concluded that Cr(VI) adsorption onto produced carbon consist of chemical adsorption due to the fact that pseudo-second-order kinetic model suggests that adsorption process involves chemisorption mechanism .And that agreed with the result obtained by K. J. Cronje et al. [12] for adsorption of Cr(VI) using activated carbon prepared onto sugarcane bagasse activated with zinc chloride.

Table 4. Kinetic parameters for Cr adsorption on date seeds

C_o(mg/L)	Pseudo-first order model			
	qe, exp (mg/g)	qe, cal (mg/g)	K1 (1/min)	R2
50	35.203	3.998	0.4926	0.9544
150	94.532	5.659	0.53007	0.9550
	132.954	7.1527	0.7257	0.9946
	154.355	8.0156	0.7634	0.9839
250	170.033	7.744	0.7428	0.9790
350				
450				
C_o (mg/L)	**Pseudo-Second order model**			
	qe, exp (mg/g)	qe, cal (mg/g)	K2 (g/mg min)	R2
50	35.203	37.335	1.3235	0.992
150	94.532	98.483	1.9004	0.987
250	132.95	147.51	1.5986	0.997
350	154.35	177.18	1.5139	0.999
450	170.03	189.35	1.6484	0.998
C_o (mg/L)	**Intraparticle diffusion model**			
	qe, exp (mg/g)	C (mg/g)	K3 (mg/g min$^{1/2}$	R2
50	35.203	9.0943	11.433	0.9527
150	94.532	42.813	22.161	0.979
250	132.95	43.155	43.504	0.953
350	154.35	45.539	55.184	0.926
450	170.03	52.177	58.593	0.881

Table 5. Kinetic parameters for Cr adsorption on ZAC

C_o(mg/L)	Pseudo-first order model			
	qe, exp (mg/g)	qe, cal (mg/g)	K1 (1/min)	R^2
50	46.95	4.89	0.620082	0.925
150	128.0	7.70	0.734123	0.9542
	179.69	8.76	0.771996	0.9948
	200.30	9.80	0.83976	0.9810
250	210.65	9.20	0.7747	0.9983
350				
450				
C_o (mg/L)	**Pseudo- Second order model**			
	qe, exp (mg/g)	qe, cal (mg/g)	K2 (g/mg min)	R^2
50	46.95	51.54	1.224	0.9804
150	128.90	144.21	1.338	0.9948
250	179.69	205.21	1.316	0.9979
350	200.30	232.55	1.204	0.9984
450	210.65	238.09	1.458	0.9998
C_o (mg/L)	**Intraparticle diffusion model**			
	qe, exp (mg/g)	C (mg/g)	K3 (mg/g min$^{1/2}$)	R^2
50	46.95	13.42	14.72	0.9902
150	128.90	36.81	43.16	0.9901
250	179.69	45.88	65.04	0.9619
350	200.30	46.05	75.04	0.9836
450	210.65	57.68	74.89	0.9417

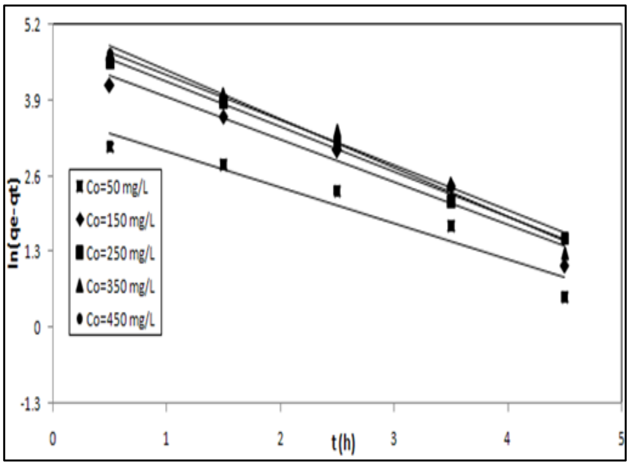

Fig. 8. Pseudo-first order kinetic for Cr(VI) adssorption on ZAC at different concentrations (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 2 ; contact time, 0.5-5.5 h)

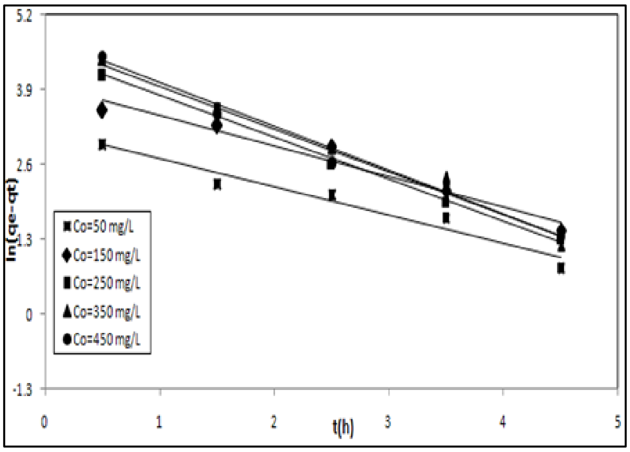

Fig. 9. Pseudo-first order kinetic for Cr(VI) adsorption on date seeds at different concentrations (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 3 ; contact time, 0.5-5.5 h)

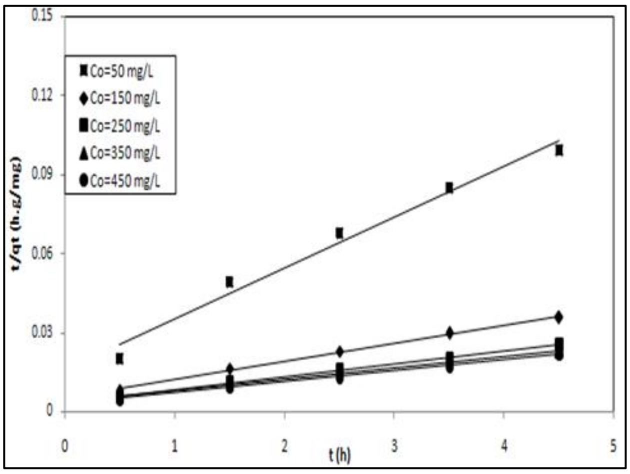

Fig.10. Pseudo-second order kinetic for Cr(VI) adsorption on ZAC at different initial concentrations (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 2 ; contact time, 0.5-5.5 h)

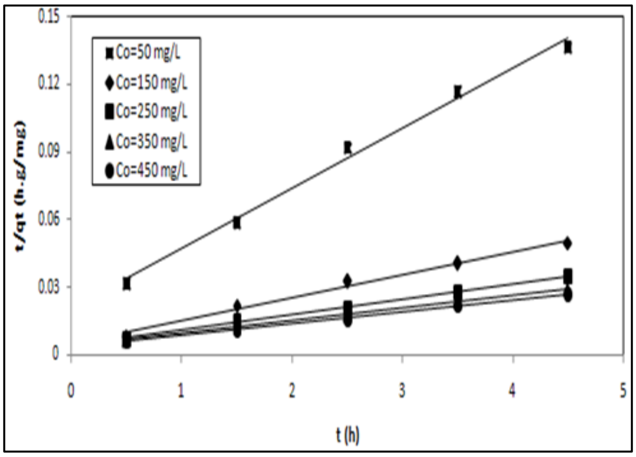

Fig. 11. Pseudo-second order kinetic for Cr(VI) adsorption on date seeds at different initial concentrations (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 3 ; contact time, 0.5-5.5 h)

3.5. The Intraparticle Diffusion Model

The intraparticle diffusion mode was determined from Eq.(9) . The linear plot of qt versus $t^{1/2}$ and from the slopes of the plots the values of K_3 were determined.

The C Values gave a thought about the boundary layer thickness, when the intercept was larger, that made surface sorption was the rate controlling step.

If the intraparticle diffusion is involved in the adsorption process, then the plot of *q*t versus *t*1/2 would result in a linear relationship, and the intraparticle diffusion would be the controlling step if this line passed through the origin [60] .

Fig.(12) and Fig. (13) represent the data for the adsorption of Cr(VI) on to ZAC and date seeds applied to intraparticle diffusion model and the results are given in Table (4) for ZAC and Table (5) for date seeds Consistent with Eq. (9).

It can be seen from the shape of Fig. (12) And Fig.(13) shows strait lines not passed through the origin for most studied initial concentrations of Cr(VI) which indicate that the intraparticle diffusion is not only the rate controlling step for the adsorption of the Cr(VI) on ZAC and date seeds.

The values of intercept *C* in Table (4) and Table (5) provide an information about the thickness of the boundary layer, i.e. the resistance to the external mass transfer.

The larger the intercept is the higher the external resistance from these Tables it can be seen that the *C* value increase with increase of initial Cr(VI) concentration, which indicated increase of the thickness of the boundary layer and hence increase of the chance of internal mass transfer and decrease of the chance of the external mass transfer.

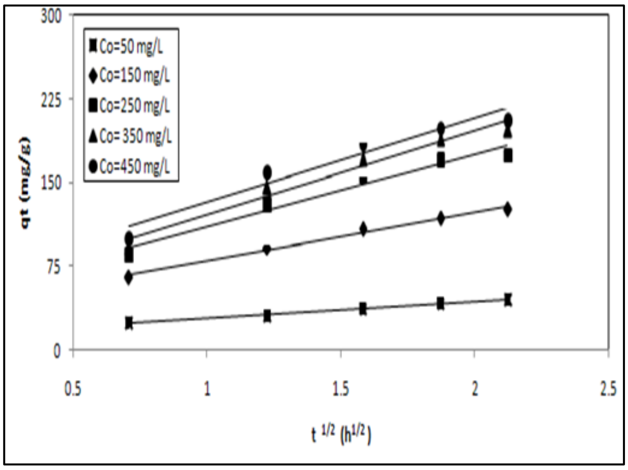

Fig. 12. Intraparticle diffusion plot for Cr(VI) adsorption on ZAC at different initial concentration (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 2 ; contact time, 0.5-5.5 h)

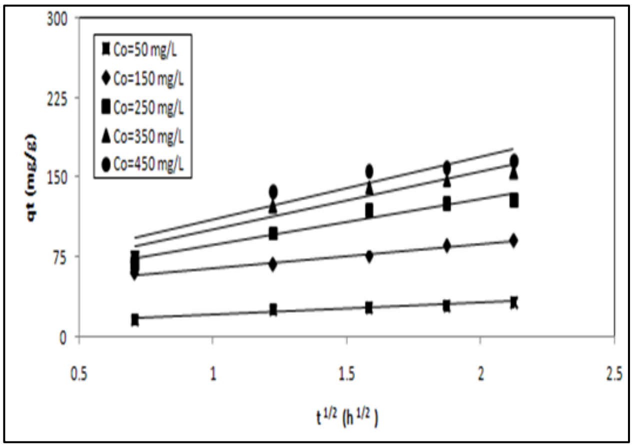

Fig. 13. Intraparticle diffusion plot for Cr(VI) adsorption on date seeds at different initial concentration (Cr(VI) conc., 50–450 mg/L; volume of Cr(VI) solution, 20 mL; amount of activated carbon, 0.02 g; pH, 3 ; contact time, 0.5-5.5 h)

4- Conclusions

The main conclusions that can be drawn from this study are:

- Chemical activation of date seeds with zinc chloride with corresponding yield of 43 % , at impregnation ratio 1.25 g/g and carbonization temperature 500 ˚C for carbonization time 2 h , produced activated carbon with 1012.91 mg/g iodine number and 1050.01 m^2/g surface area.
- A Cr(VI) adsorbed amount 35.6 mg/g were obtained at 2 solution pH, ZAC Adsorbent weight 0.02 g , and 120 min contact time.
- Solution pH of 3, date seeds adsorbent weight 0.02 g , and 120 min contact time gave maximum 26.49 mg/g adsorbed amount.
- Maximum Cr(VI) adsorption capacity of activated carbons prepared by zinc chloride and date seeds were 210.651 and 170.033 mg/g, respectively was obtained at operating conditions of 2 pH value for ZAC and 3 pH for date seeds as adsorbent , 0.02g/20ml adsorbent dose and 4.5 h contact time.
- The maximum uptake of activated carbons prepared by zinc chloride and date seeds, as calculated from Sips isotherm model, were 233.493 and 208.055 mg/g , with R^2 value of 0.9999 and 0.9998, respectively .
- The adsorption kinetic data were well described by the pseudo -second order model for both ZAC and date seeds which provides the higher correlation coefficient and calculated *q*e agree well with the experimental data.
- The intraparticle diffusion played a significant role, but it was not the main rate-determining step during adsorption of chromium on ZAC and date seeds.

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Nomenclature

C_o : Initial concentration of Cr(VI) (mg/l).
C_e : Equilibrium concentration of Cr(VI) (mg/l).
W : Mass of activated carbon used (g).
q_m : Maximum amount of Cr(VI) adsorbed per unit mass of activated carbon (mg/g).
R^2 : Correlation coefficient .
V : Volume of aqueous Cr(VI) solution (l).
W_f : Weight of dried date seeds (g).
w_o : Weight of final activated carbon (g).

Abbreviations

ZAC : Activated carbon by zinc chloride .
Cr(IV) : Hexavalent Chromium ion .
GDS : granular date seeds.

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الخلاصة

يهدف البحث إلى ازالة ايون الكروميوم باستخدام نوى التمر المطحون و الكاربون المنشط المحضر من نوى التمر بطريقه التنشيط الكيميائي وباستعمال كلوريد الزنك كعامل منشط. ايضا بينت النتائج الكاربون المنشط الناتج باستخدام كلوريد الزنك كعامل منشط كان ذو مسحه سطحيه (1050,01 متر^2 /غرام) ونسبه امتزاز لليود (1012,91 ملغرام / غرام). بينما تم تحديد المساحة السطحية و نسبة امتزاز اليود500,03 متر^2/غرام و485,78 ملغرام / غرام . تم دراسة تأثير قيمة pH (2-12) , التركيز الاولي ل ايون الكروميوم (50 -450) ملغرام/ لتر , وكمية المادة المازة (0,004-0,036غرام) و زمن امتزاز (30-150 دقيقة) . تم الحصول سعه امتزاز لأيون الكروميوم عند استخدام الكاربون المنشط بالزنك كلورايد (35,6 ملغم/غرام) عند الظروف التشغيليه : pH (2), وزن المادة المازه الى(0,02غرام) , وزمن امتزاز (120 دقيقه). في حين عند استخدام نوى التمر كمادة مازة , تم الحصول على سعه امتزاز (26,49 ملغم/غرام) عند الظروف التشغيليه : pH (3), وزن ماده مازه (0,02 غرتم) , وزمن امتزاز (120دقيقه). تم استخدام معادله سيبس بشكل ناجح لتمثيل نتائج امتزاز ايون الكروميوم على الكاربون المحضر ونوى التمر , حيث اعطى اعلى سعه امتزاز لايون الكروميوم على الكاربون المحضر ونوى التمر (233,493ملغم/غرام) و (208,055 ملغم/غرام) على التوالي.ايضا تم دراسة حركية عملية الامتزاز و بينت النتائج ان معادلة الدرجة الثانية مثلت النتائج التجريبية لعملية الامتزاز بشكل جيد ل ايون الكروميوم على الكربون المحضر ونوى التمر .