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Using Activated Carbon developed from Iraqi Date Palm Seeds as Permeable Reactive Barrier for Remediation of Groundwater Contaminated with Copper

Ziad T. Abd Ali
Department of Environmental Engineering / College of Engineering/ University of Baghdad
E-mail: z.teach2000@yahoo.com

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Abstract

The possibility of using activated carbon developed from date palm seeds wastes as a permeable reactive barrier (PRB) to remove copper from polluted shallow groundwater was investigated. The activated carbon has been developed from date palm seeds by dehydrating methods using concentrated sulfuric acid. Batch tests were performed to characterize the equilibrium sorption properties of new activated carbon in copper-containing aqueous solutions, while the sandy soil (aquifer) was assumed to be inert. Under the studied conditions, the Langmuir isotherm model gives a better fit for the sorption data of copper by activated carbon than other models. At a pilot scale, One-dimensional column experiments were performed, and an integrated model based on the solution of an advection-reaction-dispersion mass balance equation, using COMSOL Multiphysics 3.5a software which is based on finite element method, was developed to study the space and time concentration of copper within groundwater. Experimental and numerical results proved that the PRB represents a potential role in the restriction of the copper plume migration. Also, these results showed that the greater thickness of PRB results in a better treatment of copper and that the barrier starts to saturate with contaminant as a function of the travel time. However, a good agreement between the predicted (theoretical) and experimental results with RMSE not exceeded the 0.08 proved these methods are effective and efficient tools in description of copper transport phenomena adopted here**.**

Keywords: *date palm seeds, activated carbon, Copper, Permeable reactive barrier, Groundwater, Transport.*

1. Introduction

The pollution of groundwater by pollutants has been considered since the industrial revolution [1]. Copper, as well as other heavy metals, is released into the environment in a number of different ways and it finds the way to get into the water-streams and groundwater thus make environmental pollution that presents threat to plants, animals, and humans, therefore serious of complex problems can be happen [2]. "Groundwater, which is water found beneath the surface of the ground and seeped down from the surface by migrating through the soil matrix and spaces in geologic formations, is generally more reliable for use than surface water" [3]. Historically, the pump-and-treat technique was the most common technology used for remediation of groundwater. This technique is costly, difficult, and ineffective most of the time in removing enough pollution to restore the groundwater to drinking water standards in acceptable time frames. The inability to extract pollutants from the subsurface due to hydrogeologic factors and trapped residual contaminant mass was the primary reason for the failure of pump and treat. Accordingly, the other method used to remediate groundwater contaminated with different types of contaminants was permeable reactive barrier (PRB) technology. It is found to be more cost-effective than pump and treat and has demonstrated the potential to diminish the spread of contaminants [4]. "Activated carbon has versatility and wide range of applications and it has been proven to be an effective adsorbent for the removal of a wide variety of organic and inorganic pollutants from different media. Therefore, production of low-cost activated carbon becomes the aim of many researchers since the commercial activated carbon is still very expensive" [5]. The worthy aspects of this study are the development of new activated carbon from date palm seeds as an inexpensive and efficient reactive material and the ability of using of this material in PRBs for the removal of copper from polluted groundwater.

2. Description of Sorption Data

For the description of sorption data five isotherm models were used. A summary of these models was presented by "Hamdaoui and Naffrechoux" [6] as follows:

- The Langmuir model can be expressed as in equation (1):

$$q_e = \frac{q_m \, b C_e}{1 + b C_e} \qquad \dots(1)$$

where q_m is the maximum adsorption capacity (mg/g), b (L/mg) is the constant related to the free energy of adsorption, C_e (mg/l) is the equilibrium concentration of the solute in the bulk solution, and q_e (mg/g) is the amount of solute adsorbed per unit weight of adsorbent at equilibrium.

- The Freundlich model is given by equation (2):

$$q_e = K_F C_e^{1/n} \qquad \dots(2)$$

where n is an empirical coefficient indicative of the intensity of the adsorption and K_F is the Freundlich sorption coefficient.

- The Elovich model is based on a kinetic principle assuming that the adsorption process increase with sites of adsorption. It can be written as in equation (3):

$$\frac{q_e}{q_m} = K_E C_e \exp\left(-\frac{q_e}{q_m}\right) \qquad \dots(3)$$

where K_E is the Elovich equilibrium constant (L/mg), and q_m is the Elovich maximum adsorption capacity (mg/g)

- The Temkin model assumes that with coverage the heat of adsorption of all the molecules in the layer decreases linearly because of the interactions of adsorbent–adsorbate. This model is represented by equation (4):

$$\theta = \frac{RT}{\Delta Q} \ln K_o C_e \qquad \dots(4)$$

where K_o is the Temkin equilibrium constant (L/mg), ΔQ is the variation of adsorption energy (kJ mol^{-1}), T is the temperature (K), R is the universal gas constant (kJ mol^{-1} K^{-1}), and θ ($=q_e/q_m$) is the fractional coverage.

- The Kiselev model can be written as in equation (5):

$$k_1 C_e = \frac{\theta}{(1-\theta)(1+k_n\theta)} \qquad \dots(5)$$

where k_1 is the Kiselev equilibrium constant (L/mg), θ ($=q_e/q_m$) is the fractional coverage, and k_n is the constant of complex formation between adsorbed molecules.

3. Experimental Work

3.1. Materials

Iraqi date seeds were used to prepare the activated carbon; it washed with hot distilled water to remove the dust and other foreign materials, dried in oven at 200◦C for 24 h. "The dried seeds were milled, sieved to the particle size of 1–0.6 mm and stored in airtight container for carbonization. 2 kg of dried date palm seeds were added in small portion to H_2SO_4 (98%, 2 L) during 5 h followed by boiling for 20 h in a fume hood. Cool in ice bath and the reaction mixture was poured onto cold water (5 L) and filtered. The obtained carbon was dried in an open oven at 120 ◦C for 24 h followed by immersed in 5% $NaHCO_3$ (4 L) to remove any remaining acid and then filter" [7]. The produced carbon was then washed with distilled water until pH of the activated carbon reached 6, dried at 105 ◦C for 24 h and sieved to the particle size 1-0.6 mm (porosity = 0.45) and kept in a airtight container until used.

The sandy soil was used as aquifer in the conducted experiments. This soil had a particle size distribution ranged from 63 µm to 0.71 mm (porosity = 0.41) with an effective grain size, d_{10}, of 110 µm, a median grain size, d_{50}, of 180µm and a uniformity coefficient, $C_u = d_{60}/d_{10}$, of 1.73.

The salt of $Cu(N0_3)_2$ (manufactured by BDH, England) was used in the preparation of the copper stock solution by dissolving it at a known concentration in distilled water. The solutions used for the study were obtained by dilution of the stock solution to the required concentration. The initial pH of each of the solutions was adjusted by the addition of HNO_3 or NaOH solution. The initial concentrations of the copper, and the corresponding concentrations after fixed the time periods, were measured by an atomic adsorption spectrophotometer (AAS) (Shimadzu, Japan).

3.2. Batch Experiments

These tests are carried out to specify the best conditions of contact time, initial pH of the solution, initial copper concentration, dosage of activated carbon and agitation speed. Series of 250 ml flasks are employed and each flask is filled with 50 ml of copper solution which has initial concentration of 50 mg/L. About 0.5 g/50 ml of activated carbon was added into different flasks and these flasks were kept stirred in the high-speed orbital shaker at 200 rpm for 90 min. A fixed volume (20 ml) of the solution was withdrawn from each flask. This withdrawn solution was filtered to separate the activated carbon and a fixed volume (10 ml) of the clear solution was pipetted out for the concentration determination of copper still present in solution. The measurements were carried out using atomic adsorption spectrophotometer (AAS) (Shimadzu, Japan). However, the adsorbed concentration of copper on the activated carbon was obtained by a mass balance. Kinetic studies were investigated with different values of pH (3, 4, 5, 6 and 7), different values of initial concentration of copper (50, 60, 70, 80 and 90 mg/L), amounts of activated carbon dosage (0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8 and 1 g/50 ml) and finally values of agitation speed (0, 50, 100, 150, 200 and 250 rpm).

From the best experimental results, the amount of copper retained in the activated carbon phase, q_e, was calculated using equation (6) [8]:

$$q_e = (C_o - C_e)\frac{V}{m} \qquad \ldots(6)$$

where C_o and C_e are the initial and equilibrium concentrations of copper in the solution (mg/L), V is the volume of solution in the flask (L), and m is the mass of adsorbent material (activated carbon) in the flask (g).

3.3. Continuous Experiments

The reactor setup used in the present study was constructed of Perspex column having height and diameter equal to 50 and 5 cm, respectively. This reactor was schematically shown in Fig.1. The column is equipped with four sampling ports at a distance of 10 cm (port 1), 20 cm (port 2), 25 cm (port 3), 35 cm (port 4), from the bottom. These ports along the length of the column were assembled of stainless steel fittings which blocked with Viton stoppers (DOIT, Dongguan Doit Rubber Products, Guangdong, China). Sampling was carried out at a specified periods using a syringe which was inserted into the center axis of the column. The column was packed with sandy soil as aquifer and activated carbon developed from date palm seeds wastes (reactive media) as barrier in the configuration and alignment illustrated in Fig. 1. This column was then filled with distilled water that was fed slowly into the bottom of the column and forced upward through the medium in order to expel the air from the medium (soil and activated carbon). The contaminated solution with copper, which simulated the contaminated groundwater, was introduced into the column from storage tank controlled by gravity (constant head flow), flow-meter and two valves.

Two values of flow rate (7 and 17 mL/min) were selected for the column test experiments. The selection of these values was to satisfy the laminar flow, i.e., Reynolds number (R) < 1 − 10 [9], which is the predominant situation for groundwater flow in the porous medium. Monitoring of copper concentration along the length of the column in the effluent from sampling ports was conducted for a period of 7 days, then samples of water were taken regularly (after 1, 2, 4, and 7 days) from these ports and immediately introduced in glass vials and then analyzed by atomic adsorption spectrophotometer. The filling material in the column was assumed to be homogeneous and incompressible, and constant over time for water-filled porosity. The volumetric water discharge through the column cross section was constant over time and set as the experimental values. The copper inlet concentration was set constant. All tubing and fittings for the influent and effluent lines should be composed of an inert material. A solution of 1 g/L NaCl in deionized water as a tracer was continuously fed into the column at four values of flow rate (5, 10, 15, and 20 mL/min) for a tracer experiment, adopting the same method of Ujfaludi [10], to determine the longitudinal dispersion coefficient (D_L) for the reactive medium (activated carbon) and sandy soil (aquifer).

4. Results and Discussion

4.1. Fourier Transform Infrared Analysis

The Fourier transfer infrared spectroscopy (FTIR) analysis has been considered as a kind of direct mean for identifying the characteristic functional groups on the surface of the activated carbon, which are responsible for copper binding [11]. The FTIR spectrums of activated carbon samples after and before copper sorption were examined using a Shimadzu FTIR, 8000 series spectrophotometer. The displacement in the infrared frequencies support that carboxylic acid, Alcohols, and Sulfonates are the functional groups causing the sorption of copper onto activated carbon as shown in Fig. 2, Table 1,[12]. Accordingly, the mechanisms that controlled the sorption of copper by using activated carbon may be the adsorption process supported by the presence of the functional groups described previously.

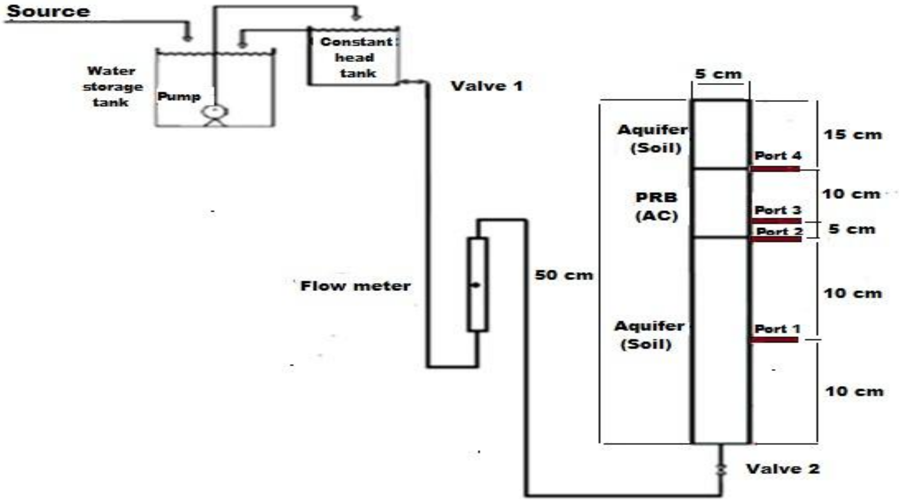


Fig. 1. Schematic representation of column test.

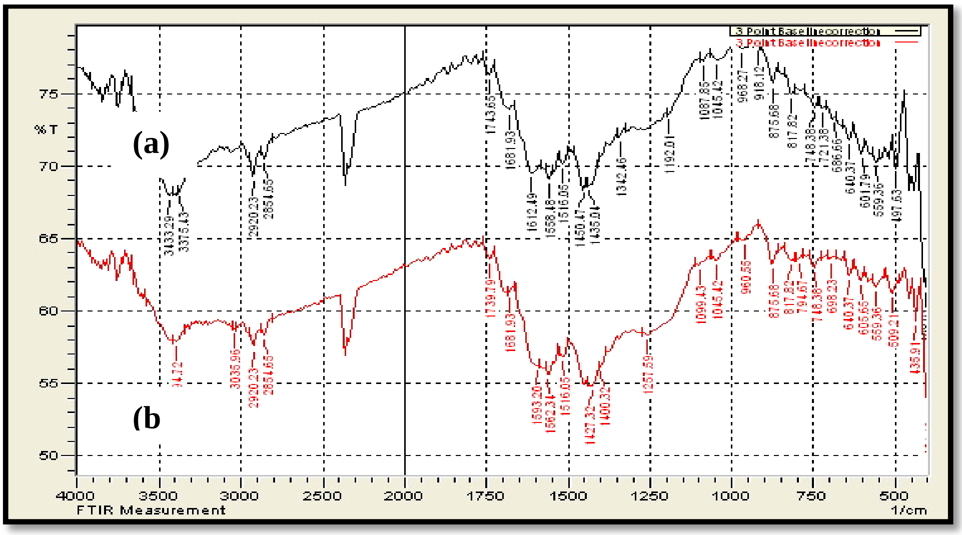


Fig. 2. FTIR spectrum of activated carbon (a) before and (b) after, loaded of Cu (II)

Table 1,
Functional groups responsible for copper sorption onto activated carbon.

Wave No. (cm^{-1})	**Type of bond**	**Functional group**	**Displacement after sorption**
3375.43	O-H	Carboxylic acid	19.29
1743.65	C=O	Carboxylic acid	3.86
1192.01	C-O	Carboxylic acid	65.58
1087.85	C-O-C	Alcohols	11.58
968.27	S-O	Sulfonates	7.72

4.2. Influence of Batch Operating Parameters

4.2.1. Effect of Contact Time and Initial pH of Solution

The effect of initial pH of the solution and the contact time on copper sorption is illustrated in Fig. 3, where 0.5 g of activated carbon was added to a 50 ml of Cu(II) solution at 25 ºC. As the Fig.3 shows, at the beginning, the increasing rate of adsorption was fast as the contact time increased till reaching the equilibrium time (60 min). This could be the result of the availability of many adsorbent sites for the Cu(II) adsorption. The adsorption rate was reduced because of the decreasing of the remaining vacant surfaces as result of formation of replusive forces between the Cu(II) on the solid surfaces and in the liquid phase [13]. As well as the reduction in the competition between Cu(II) and proton for the surface sites causes the increase in the removal of copper on increasing pH, this results in a reduce in columbic repulsion of the sorbing copper. However, the removal efficiency was decreased on increasing the pH value. Fig.3 shows that at a pH of 6 the removal efficiency of Cu(II) was at its maximum value.

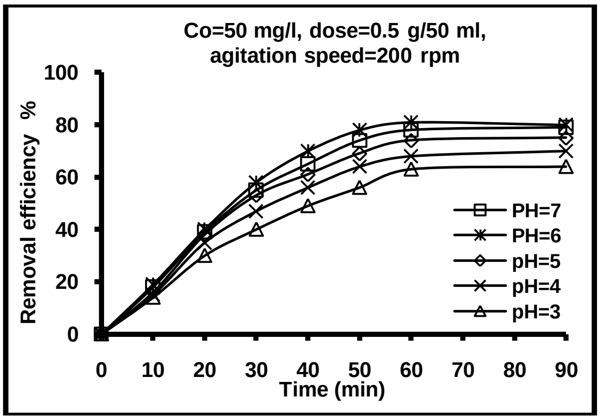


Fig. 3. Removal efficiency of copper on activated carbon as a function of contact time and initial pH

4.2.2. Effect of Activated Carbon Dosage

The effect of activated carbon dosage on the removal efficiency of the copper was examined at different dosages of activated carbon. These dosages were ranging from 0.2 to 1 g and added to 50 mL of copper solution as present in Fig.4.This figure showed that the increasing of the activated carbon dosage from 0.2 to 0.6 g at fixed initial copper concentration will lead to improve the removal efficiency, this was because of a higher dosage of activated carbon in the solution leads to greater availability of sorption sites, it's also clear that after a dosage of activated carbon (0.6 g/50 mL), the maximum adsorption sets in; hence, even with further addition of activated carbon, the amount of copper bond to the activated carbon and its amount in solution remain constant.

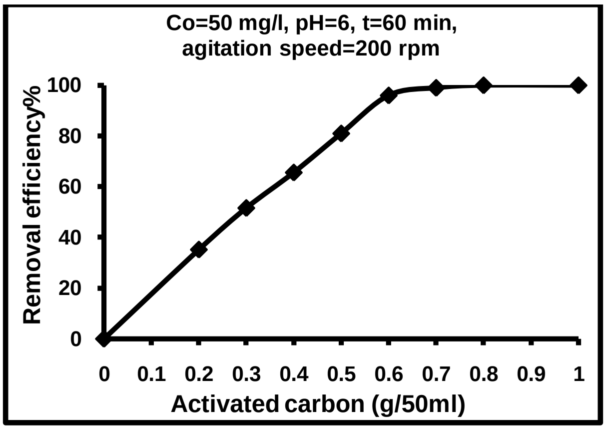


Fig. 4. Effect of activated carbon dosage onremoval efficiencies of Cu(II)

4.2.3. Effect of Initial copper Concentration

Fig. 5 presents the effect of Initial Concentration of copper on its removal efficiency. Its shows that when increasing the initial concentration of copper from 50 to 90 mg/L, the removal efficiency reduces from 96 to 74.4%, this represents the saturation of the active sites available on the activated carbon to interact with pollutant, so with increasing concentration the less favorable sites became involved in the process[14].

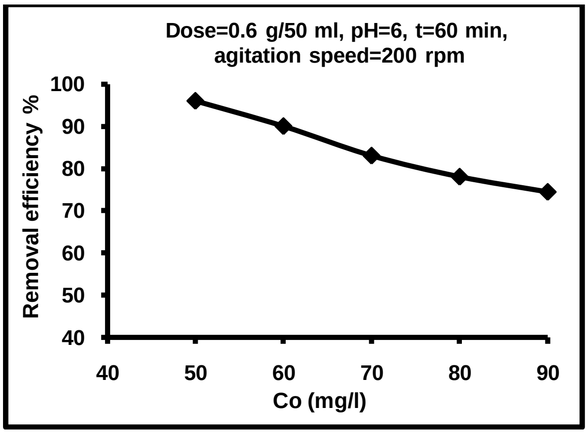


Fig. 5. Effect of initial copper concentration on removal efficiency of copper on activated carbon

4.2.4. Effect of Agitation Speed

Nearly 7% of the copper was removed before shaking (agitation speed = 0) and with the increase of the rate of shaking, the uptake increases, as illustrated in Fig. 6 . On increasing agitation speed from 0 to 250 rpm, there is a gradual increase in copper uptake, at which about all of the copper (100%) is removed. This can be due to improving the diffusion of copper towards the surface of the reactive medium (activated carbon) and more contact between the binding sites and the copper in the solution.

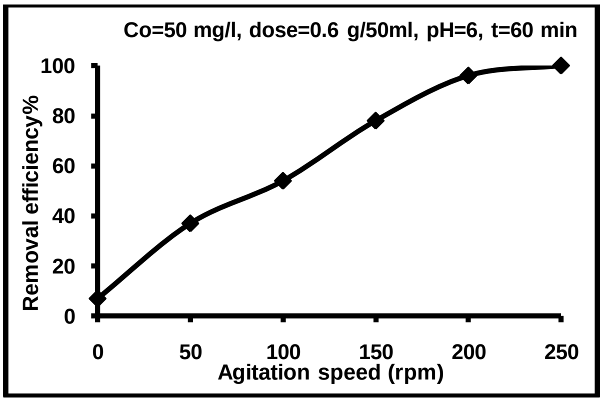


Fig. 6. Effect of agitation speed on removal efficiency of copper

4.3. Sorption Isotherms

The parameters that give the higher removal efficiency of copper at its initial concentration of 50 mg/L on activated carbon were pH of 6, agitation speed of 250 rpm, an equilibrium time of 60 min, and activated carbon dosage of 0.6g/50mL. The results of the sorption experiment were fitted with the previously described linearized form of five isotherm models. The fitted parameters and coefficient of determination (R^2) for each model are illustrated in Table 2. In comparison with the other models, the Langmuir isotherm model provided the higher correlation for copper sorption on activated carbon, therfore the Langmuir isotherm model was used to describe the copper sorption on these media in the partial differential equation (PDE) governing the transport of a copper in one dimension continuous mode. Considering the sandy soil (aquifer) as a non- reactive medium (inert) is one of the main aspects of the present study, so that just the adsorbate (copper) and adsorbent (activated carbon) are examined in batch experiments.

Table 2
Parameters of isotherm models for sorption of copper onto activated carbon.

Isotherm models	Parameter	
	b (L/mg)	4.874
Langmuir	q_m (mg/g)	4.374
	R^2	0.9986
	K_F (mg/g)(L/mg)$^{1/n}$	3.662
Freundlich	n	21.32
	R^2	0.9533
	q_m (mg/g)	10.033
Elovich	K_E (L/mg)	0.3709
	R^2	0.9497
	ΔQ (KJ/mole)	7.0729
Temkin	K_o (L/mg)	1.0007
	R^2	0.9221
	k_1 (L/mg)	0.5431
Kiselev	k_n	-0.8201
	R^2	0.9242

4.4. Longitudinal Dispersion Coefficient

Table 3 presents the results of experimental work representing the measurement of longitudinal dispersion coefficient (D_L) at different values of velocity (V) for soil and activated carbon had the following linear forms equations (7, and 8) [10]:

$D_L = 6.490\ V + 0.5325$ (R^2=0.9960)for soil …(7)

D_L=12.889 $V + 0.0325$ (R^2=0.9953)for AC …(8)

The longitudinal hydrodynamic dispersion of these equations has the general form of coefficient as in equation (9) [15]:

$$D_L = \alpha_L V + \tau D_o \quad \text{…(9)}$$

where D_o = molecular diffusion coefficient, and τ = tortuosity. As cited by Holzbecher, the τ value for theoretical work is equal to the porosity (n) [15]. In the present study the molecular diffusion coefficient of copper in the water (D_o) used was 9.7×10^{-6} cm^2/s [16]. Therefore the longitudinal dispersivity (α_L) is 6.49 and 12.889 cm for soil and activated carbon, respectively

Table 3,
measured values of the longitudinal dispersion coefficient and dispersivity for used mediums as a function of mean pore velocity.

Flow rate (ml/min)		**5**	**10**	**15**	**20**
Sandy Soil (aquifer)	***V* (cm/s)**	0.01	0.02	0.03	0.04
	Re	0.0179	0.0359	0.0538	0.0718
	D_L (cm^2/s)	0.593	0.670	0.725	0.791
	α_L (cm)	6.490			
Activated carbon (PRB)	***V* (cm/s)**	0.009	0.018	0.027	0.036
	Re	0.0696	0.1392	0.2088	0.2784
	D_L (cm^2/s)	0.158	0.250	0.381	0.501
	α_L (cm)	12.889			

4.5. Modeling Application

Advection dispersion processes is the cause of copper transport in a porous medium, so that the one-dimensional system of copper transport in a porous media can be represented by the following equation (10) [17]:

$$D_z \frac{\partial^2 C_{Cu}}{\partial z^2} - V_z \frac{\partial C_{Cu}}{\partial z} = \frac{\partial C_{Cu}}{\partial t} + \frac{\rho_b}{n}\frac{\partial q}{\partial t} \qquad \ldots(10)$$

where C_{Cu} = copper mass concentration in water, V_z = velocity of flow, D_z = longitudinal dispersion coefficient in the z direction, C_{Cu} = copper mass concentration in water, ρ_b = dry adsorbing material bulk density, and q = copper concentration on solid,.

, the second term (q) on the right side of equation (10) can be changed by the Langmuir model equation (1) under isothermal conditions. The initial copper concentrations are assumed to be zero throughout the entire flow domain and the boundary conditions used in COMSOL Multiphysics 3.5a are reported in equation (11):

Lower boundary (at $z = 0$): C_{Cu} = 50 mg/L

Upper boundary (at z=50 cm): advective flux as in equation (11):

$$\frac{\partial C_{Cu}}{\partial z} = 0 \qquad \ldots(11)$$

Interior boundaries in the barrier zone (i.e., at z=20 and 35 cm): continuity.

With and without the presence of PRB the concentration lines of copper in the aquifer were calculated by the COMSOL Multiphysics 3.5a software at two different values of flow rates (7 and 14 mL/min) and at different time intervals as shown in Fig.7. This figure shows that the propagation of the copper plume was restricted by the barrier and this propagation will be increased as increasing flow rate.

The PRB thickness effect varied from 0 to 25 cm as shown in Fig. 8, where the copper treatment in the down gradient of the barrier (z = 35 cm) at a rate of flow of 20 mL/min. It is clear that the copper treatment will be better with the increment of the PRB thickness. This is due to the greater retention time of the copper solution that leads to better sorption process. However, on increasing the travel time, the barrier starts to saturate; so that the retardation factor of the copper was decreased, this means that the percentage functionality of activated carbon for containing pollutant was reduced.

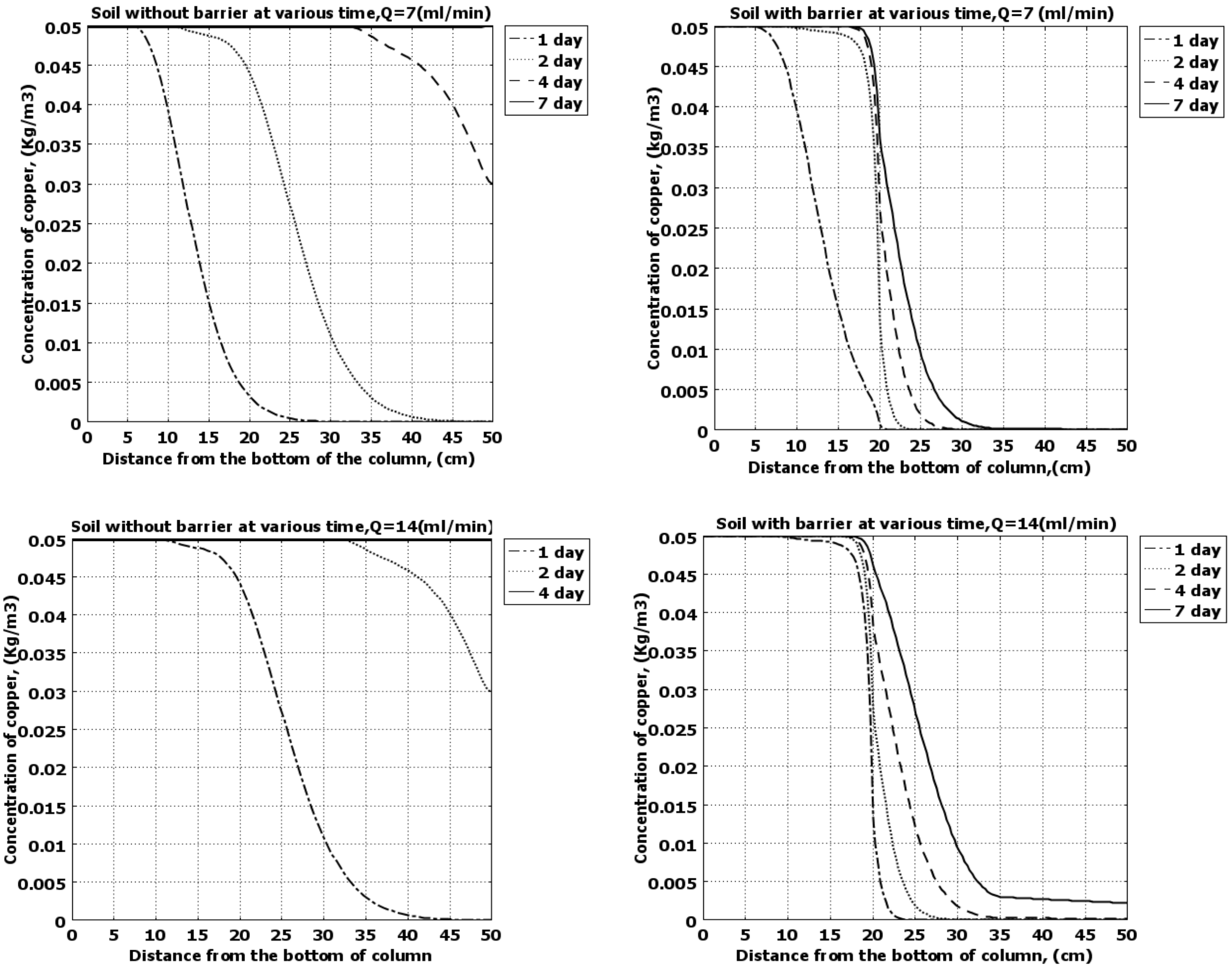


Fig. 7. Representation of copper concentration along the length of the column with and without presence of barrier at two values of flow rate using COMSOL software.

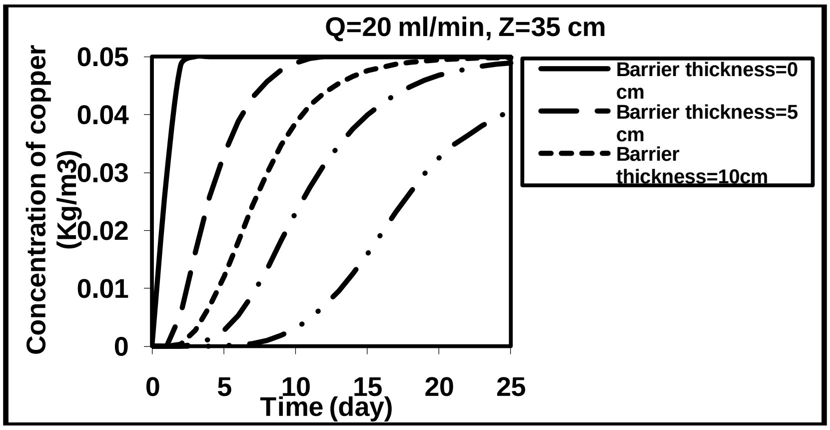


Fig. 8. Concentration of copper versus time of various barrier thickness.

In addition, to investigate the relation between PRB thickness and its longevity, COMSOL Multiphysics 3.5a was used as valid tool. The time required for maintaining the concentration of contaminant down gradient of the barrier to less than the quality limit prescribed for drinking water is the definition of the "longevity". Under two different values of flow rate the longevity of the barrier increased in a linear trend with increasing the thickness of the barrier for copper as illustrated in Fig. 9 .

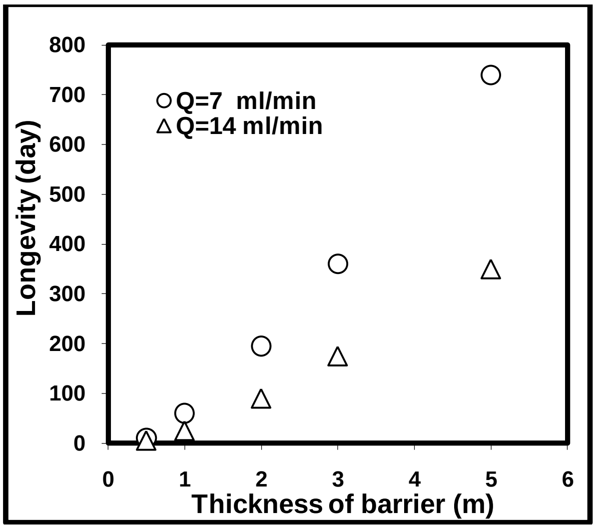


Fig. 9. Relationship between the thicknesses of the barrier and its longevity as predicted by COMSOL software.

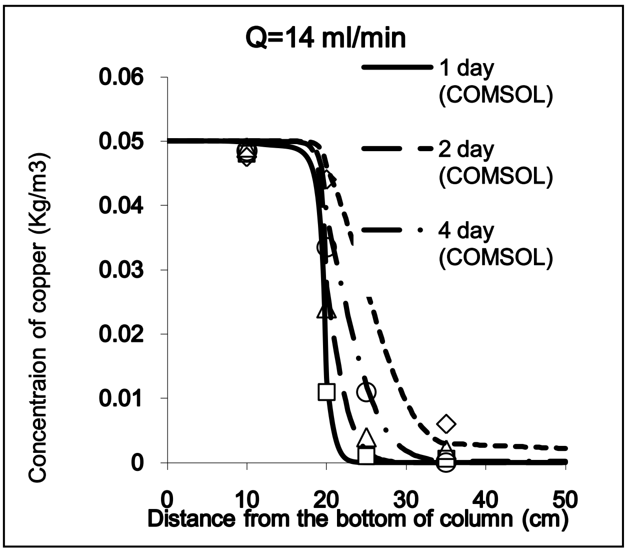
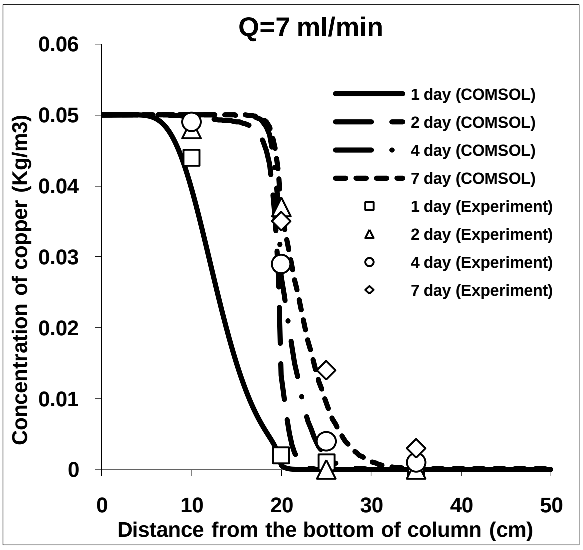


Fig. 10. Comparison between experimental results and COMSOL solution for copper concentrations at two values of flow rate.

Fig. 10 illustrate a comparison between the COMSOL solution (theoretical) experimental results for copper concentrations during the transport the copper plume at two different flow rates in a various periods of times along the tested column. It's obvious there is nearly an agreement between the theoretical and experimental results. In addition, to find the degree of agreement between these results a statistical tool was used (RMSE) [18]. The root-mean squared error values don't exceed 0.08, confirming a good agreement.



5. Conclusions

1. The batch results indicated that there are several parameters such as Contact time, initial pH of the solution; initial copper concentration, activated carbon dosage, and agitation speed affect the sorption process the sorption process between copper and activated carbon. The best values of these parameters that will achieve the maximum removal efficiency of copper (100%) were 60 min, 6, 50 mg/L, 0.6 g/50 mL, and 250 rpm, respectively.
2. Copper sorption data on activated carbon was correlated reasonably well by the Langmuir sorption model with coefficient of determination (R^2) of 0.9986.
3. The Fourier transfer infrared spectroscopy (FTIR) analysis proved that carboxylic acid, Alcohols, and Sulfonates are the functional groups responsible for the sorption of copper onto activated carbon
4. The numerical solutions that are solved by COMSOL Multiphysics 3.5a to describe the one dimension equilibrium transport of copper along the length of aquifer and PRB proved that the activated carbon barrier is an efficient technique to restrict the contaminant plume. These results showed that the greater thickness of PRB results in a better treatment of copper and that the barrier starts to saturate with contaminant as a function of the travel time. However, a good agreement between the predicted (theoretical) and experimental results with RMSE not exceeded the 0.08

proved these methods are effective and efficient tools in description of pollutant transport phenomena adopted here

5. Experimental results proved that the activated carbon developed from date palm seeds wastes was a good choice as an inexpensive and efficient reactive material for permeable reactive barrier to remove the copper from contaminated groundwater

6. References

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استخدام الكاربون المنشط المطور من بذور تمر النخيل العراقى كجدار تفاعلى نفاذ لمعالجة المياه الجوفيه الملوثه بالنحاس

زياد طارق عبدعلي
قسم الهندسه البئيي / كلية الهندسة/ جامعه بغداد
البريد الالكتروني:z.teach2000@yahoo.com

الخلاصة

في هذا البحث يتم التحقق من امكانية استخدام الكاربون المنشط المطور من بذور تمر النخيل كجدار تفاعلي نفاذ لازاله النحاس من المياه الجوفيه الضحله. الكاربون المنشط صنع من بذور تمر النخيل عن طريق عملية (dehydration method) باستخدام حامض الكبريتيك المركز. عمليات الدفع (batch) تم استخدامها لايجاد خواص التوازن في عملية امتزاز النحاس على سطح الكاربون المنشط بينما التربة الرملية تم فرضها على انها خاملة. النتائج العمليه اشارت بان الموديل لانكمير هو الافضل تطابقا لوصف عملية الامتزاز بالمقارنه بالموديلات الاخرى بنفس ظروف الدراسه. تم استخدام عمود باتجاه واحد (1D) لمحاكاة جريان المياه الجوفيه باتجاه واحد معتمدا على معادله (adv. - disp. equation) والتى تم حلها باستخدام برنامج الكومسول. النتائج العدديه والعمليه اثبتت ان الجدار التفاعلى النفاذ يؤدي دورا مهما في اعاقه الملوث وكذلك ان عملية الازالة تزداد بزيادة سمك الحاجز وقد ظهر توافق جيد مابين نتائج الكومسول والعملى