

## SEEDS-BASED ACTIVATED CARBON FOR COPPER REMOVAL FROM GROUNDWATER

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### Abstract

The current study investigates the possibility of preparing of cost-effective activated carbon from seeds that collected from local agricultural wastes. In this investigation, date palm seeds (DPCAC), peach seeds (PSAC), and apricot seeds (ASAC) were used to prepare activated carbon, which was used as a permeable reactive barrier (PRB) to remove copper ( $\text{Cu}^{+2}$ ) from contaminated groundwater. Activated carbon has been prepared from these seeds using chemical activation method (using drenching agent, such as phosphoric acid ( $\text{H}_3\text{PO}_4$ )). The effect of different parameters such as the contact time, initial pH of the solution, agitation speed, initial copper concentration, and sorbent dosage was studied in batch experiments. The best removal of copper (95.54% for DPSAC, 88.64% for PSAC and 94.0% for ASAC) was obtained at contact time of 100 min for DPSAC and 80 min for both PSAC and ASAC, pH of 6, agitation speed 250 rpm, and sorbent dosage of 3 g/100 ml. The sorption data for  $\text{Cu}^{+2}$  ions, obtained by batch experiments, have been subjected to the Langmuir and Freundlich isotherm models. The results showed that Langmuir model provided the best description of sorption of  $\text{Cu}^{+2}$  onto DPSAC, PSAC and ASAC. COMSOL Multiphysics 3.5a software, based on finite element method, was used for solving of partial differential equations that describe the transformation of copper in the one-dimensional (1D) under equilibrium conditions. The predicted results (COMSOL solution) and experimental results showed that the PRB restricts the movement of ions of copper. However, predicted and experimental results proved that DPSAC has high affinity for copper ions in comparison with both PSAC and ASAC. Finally, a good agreement between the predicted and experimental results has been noticed because the root-mean squared error (RMSE) less than 0.1%, which proves the effectiveness of these tools in description of copper transportation.

Keywords: Activated carbon, Apricot seeds, Copper, Date palm seeds, Groundwater, Migration, Peach seeds, Permeable reactive barrier.

## 1. Introduction

Groundwater pollution represents a big challenge for many countries, especially for those countries who use it as a source for drinking or irrigation purposes [1]. Groundwater pollution is usually occurred because of the polluted seepage from the polluted areas [1]. Although the planet of Earth faces serious pollution problems, such as the huge production of solid wastes and soil pollution [2-9], water pollution is one of the most challenges problems due to the limited amount of this element in the ecosystem of this planet [10-12]. Although there are  $1.4 \times 10^9$  km<sup>3</sup> of water in the ecosystem of the earth planet [13], very minor amount ( $\leq 2.5\%$ ) of this vast amount is fresh water [14-18]. Unfortunately, this minor amount of fresh water is not completely available for humankind as the majority of fresh water is captured in glaciers, snow cover, and groundwater leaving about 1% of the fresh water available for drinking purposes [19-24].

In addition, the available amount of fresh water, nowadays, is subjected to an increasing pollution process due to the increasing global population and industrial growth [25]. Therefore, some forecasting studies confirmed that within the next few decades more than 50% of the world's population would not have enough drinking water [26-30]. Heavy metals is one of the most problematic forms of water pollution, which could be resulted from various industrial activities, such as mining operation, electroplating, oil processing plants and petrochemical industries [22, 31]. Phenomenon of heavy metal pollution is a predominant problem in the groundwater due to the abundant occurrence of heavy metals in the earth's crust.

Groundwater is defined as water found beneath the surface of the ground and seeped down from the surface by migrating through the spaces in geologic formations [32]. To control water pollution, different treatment methods, monitoring and sensing technologies, and management plans were developed [33-38].

According to the literature, the pump-and-treat technique was the most commonly used technology for treatment of polluted groundwater [39]. However, this technique is expensive, difficult to be operated and maintained, and time-consuming. In addition, trapping of contaminant mass in the pumping system results in the failure of pumps. Thus, permeable reactive barrier (PRB) technology has recently brought a big deal of attention as an effective alternative for treatment of groundwater [39].

For example, Faisal and Ahmed [40] developed a permeable reactive barrier, made from Kerbala's sand (KS) and waste foundry sand (WFS) (in equal ratio), to remove copper from shallow aquifers water in batch experiments. The obtained results indicated that this PRB has achieved a complete copper removal. Additionally, it has been found that the Langmuir model well represents the sorption process of copper on the PRB.

Fronczyk, et al. [41] investigated the ability of natural and engineered limestone PRB for the removal of copper from groundwater. Batch experiments were carried out at sorbent mass of 0.5 g, and 50 mL of contaminated solutions at different pH values. The results of this study indicated that the removal of copper using this type of PRB is very sensitive to the pH level, and the best removal efficiency, 94%, could be achieved at alkaline media (pH > 6.3). Gholami, et al. [42] investigated the possibility of using encapsulated magnesium peroxide (MgO<sub>2</sub>) nanoparticles as PRB for removal of the hydrocarbon compounds, toluene and naphthalene, from

groundwater in the continuous flow model. The obtained results indicated that toluene was metabolized slower than naphthalene by 10 days. Additionally, the results of this study indicated that 100% removal efficiency could be achieved in biotic conditions, while the presence of abiotic conditions decreased the removal of toluene to 36%.

In this context, the present study aims to prepare cost-effective activated carbon from agricultural wastes (seeds); namely date palm seeds (DPSAC), peach seeds (PSAC) and apricot seeds (ASAC). The prepared activated carbon will be used as PRB to remediate copper from polluted groundwater. It is noteworthy to highlight that the activated carbon has been used as a media for PRB, in the current study, because it has been demonstrated to be a powerful adsorbent for the elimination of extensive diversity of organic and inorganic contaminants from water and wastewater [43]. Additionally, activated carbon could be prepared through low-cost and simple processes [43].

## 2. Experimental Work

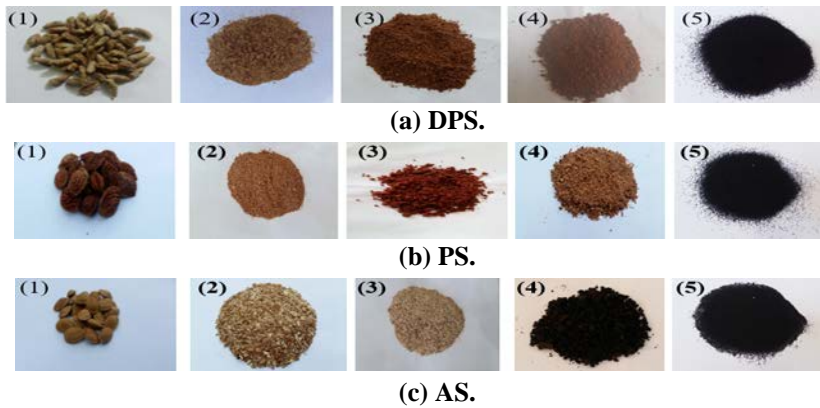
### 2.1. Materials and methods

Plant seeds, which have been collected from agricultural wastes, were initially cleaned and cut into small pieces (about 2 mm in both length and width). Then, these pieces were dried using an electrical oven at temperature 105 °C for two hours, and grinded using a household blender. The production of activated carbon from the mentioned seeds was carried out in the following stages: drenching, and carbonization and activation. Initially, the sliced seeds were placed in 1 L glass containers, and then phosphoric acid (1M) was decanted carefully into the containers at ratio of 4 ml of acid/2 g of seeds [44].

The solution was left for 24 hours at temperature of 25 °C. it is must be highlighted that both zinc chloride and phosphoric acids are used in the preparation of activated carbon, but the phosphoric acid is relatively safer than zinc chloride [45]. At the end of the 24 hours period, the drenched sliced of seeds were left in air for 24 hours, then it was dried using an oven at temperature 120 °C for two hour [44].

In the second step, the sliced seeds was carbonized by placing it on a metallic oven tray and heated at temperature of 500 °C for 60 min [46]. Then, the sliced seeds were washed with distilled water until pH of 7 was reached; then dried, using an electric oven, at temperature 120 °C for 60 min to remove moisture. The sliced seeds were grinded and sieved through 75 µm – 600 µm to get powdered activated carbon that will be used latter in the treatment of groundwater [44, 46]. Figure 1 shows plant seeds, used in this study, and the produced activated carbon.

Sandy soil, with grain size of 75 µm - 1 mm, was used as an aquifer. While the required concentration of copper was achieved by adding the required amount of  $\text{Cu}(\text{NO}_3)_2$  salt, made by BDH-England, into the water samples.



**Fig. 1. (1) Seeds before cutting and drying, (2) After drying, (3) Powdered in organic state, (4) After soaking, (5) Powdered activated carbon of DPS, PS and AS.**

## 2.2. Batch experiments

The batch experiments, 3 sets for each experiment, were carried out at the conditions below. These experiments were carried out at different contact times (ranging from 20 to 40, 60, 80, 100 and 120 min). All experiments were carried out at the laboratory of environmental engineering at the University of Babylon. The following values were chosen according to the literature [39, 41, 47].

- The temperature was set to 25°C to perform sorption isotherms.
- Contact time of 20, 40, 60, 80, 100 and 120 min
- pH values of 2, 4, 6, and 8.
- Sorbent dosages of 0.1, 0.25, 0.50, 1, 2, 3, 4 and 5 g activated carbon per each 100 ml were.
- Initial concentrations of copper ranging from 50 to 100, 150, 200 and 250 mg/L.
- Agitation speeds of 50, 100, 150, 200, and 250 rpm.

Batch experiments were carried out using five 250 ml flasks containing 100 ml of polluted water and 3 g activated carbon for concentration of copper. The flasks were firmly closed and shaken, using shaker incubator, for different times (20, 40, 60, 80, 100 and 120 min) [48]. The removal of copper was measured by taking 20 ml of solution, at intervals of 5, 10, 15, 20 and 25 hours, and filtered on filter papers (Teknik No.1.). The residual copper, in the filtrate, was measured using atomic absorption spectrophotometer (AAS). While the amount of copper held in the activated carbon ( $q_e$ ) was calculated as follows [48]:

$$q_e = (C_o - C_e) \frac{V}{m} \quad (1)$$

where  $C_o$  and  $C_e$  are initial and balance contaminant concentration in solution (mg/L),  $V$  is the size of solution in the flask (L), and  $m$  is the mass of adsorbent (activated carbon) (g) [49]. Langmuir model (Eq. 2) and Freundlich model (Eq. 3) were used for the description of sorption data. Langmuir and Freundlich models were calculated by graphing the  $q_e$  against of the  $C_e$  at constant temperature. Langmuir and Freundlich models are expressed by the following equations [50]:

$$q_e = \frac{abc_e}{1+bc_e} \quad (2)$$

$$q_e = K_F c_e^{1/n} \quad (3)$$

where  $a$  is the maximum adsorption (mg/g),  $b$  is a constant (l/mg),  $K_F$  is the coefficient of Freundlich sorption (mg/g), and  $n$  is an empirical coefficient.

It is noteworthy to mention that Langmuir model and Freundlich isotherms models have been used in this study as they provide accurate description for the adsorption method [50].

### 2.3. Column tests

Figure 2 shows arrangement of the adsorption media that used in the present study. A Perspex column having height and diameter of 80 and 5 cm, respectively. The column is supplied with seven testing ports (valves) distributed at the 12 cm (port 1), 24 cm (port 2), 36 cm (port 3), 42 cm (port 4), 48 cm (port 5), 60 cm (port 6), 70 cm (port 7) from the bottom of the column. These valves are made from stainless-steel. Sampling process was carried out at the specified periods from these valves using syringe that inserted into the centre of the column.

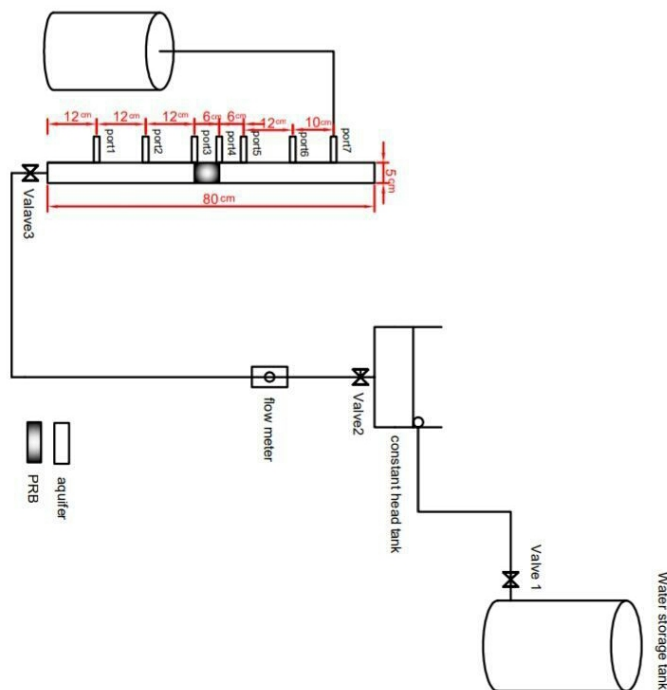


Fig. 2. Schematic diagram of the laboratory-scale column.

A 36 cm soil layer, in a dry condition, was placed in the bottom of the column. Then, activated carbon layer, 6 cm in height, was placed on the top of the soil layer followed by a 180 cm soil layer. The copper solution was pumped into the column from the storage reservoir at constant head flow. The pumping process was also controlled using a flowmeter and three controlling valves. The influence of flow

rate was investigated at two values, namely 5 and 10 ml/min. Removal of copper was monitored, along the column length, by collecting samples of the testing ports at intervals of 5, 10, 15, 20 and 25 hours.

Additionally, a tracer test was performed to calculate the coefficient of longitudinal dispersion ( $D_L$ ) for both sandy soil and activated carbon. The tracer test was performed by pumping saline solution, 1 g of NaCl dissolved in 1.0 L distilled water, continuously into the column at different flow rates (5, 10 and 15 mL/min). Electrical conductivity was measured with time, as an indicator of salt concentration, using a conductivity meter installed at port 7. Value of  $D_L$  was calculated using the following formula [51]:

$$D_L = \frac{1}{8} \left[ \frac{(z_0 - Vt_{0.16})}{(t_{0.16})^{0.5}} - \frac{(z_0 - Vt_{0.84})}{(t_{0.84})^{0.5}} \right]^2 \quad (4)$$

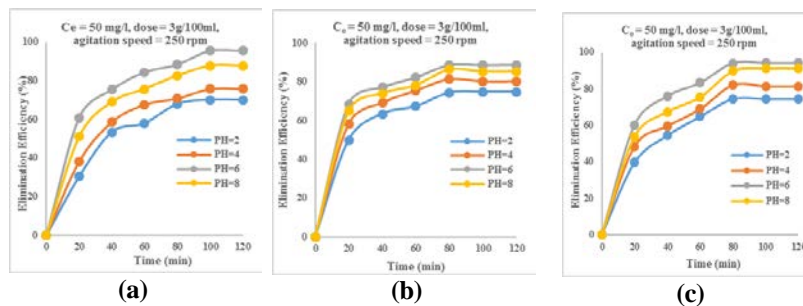
where  $V$  is the average pore velocity of seepage,  $t_{0.16}$  and  $t_{0.84}$  are the required time to reach  $C/C_0 = 0.16$  and  $0.8$ , respectively.

### 3. Results and Discussion

#### 3.1. Batch experiments

##### 3.1.1. Influence of contact time and pH value

The influence of contact time of the removal of copper has been investigated at different periods. Figure 3 shows the influence of contact time and pH value on the removal of copper. These experiments were carried out using 3g of DPSAC, PSAC and ASAC (per each 100 ml of copper solution at 25°C). This figure indicated that the adsorption rate has significantly increased with the increase of the contact time, until it reaches the equilibrium time (= 100 min for DPSAC and 80 min for PSAC and ASAC). This could be attributed to the fact that the majority of adsorption sites on the surface of the sorbent are occupied by the copper ions during the early stage of treatment, which in turn decreases the adsorption rate due to formation of repulsive forces between the metals on the solid surfaces and in the liquid phase [52]. Additionally, it can be seen from Fig. 3 that neutral level of pH is favourable for the removal of copper. The reduction the removal of copper in the acidic and alkaline environments could be explained by the competition between copper ions and protons,  $OH^-$ , and  $H_2$  for the available adsorption sites. It is obvious from Fig. 3 that the higher removal efficiency of copper was attained at pH of 6.



**Fig. 3. Removal of copper by (a) DPSAC (b) PSAC (c) ASAC as a function of contact time and pH.**

### 3.1.2. Influence of sorbent dosage

The influence of sorbent dosage on the removal of copper was investigated by changing the added quantity of activated carbon from 0.1 to 5 g per 100 mL of solution. Figure 4 shows that increasing the activated carbon dosage from 0.1 to 3 g, at initial copper concentration of 50 mg/L, has significantly enhanced the removal efficiency. This enhancement is attributed to the increase of the number of adsorption sites that are readily available for copper removal [52].

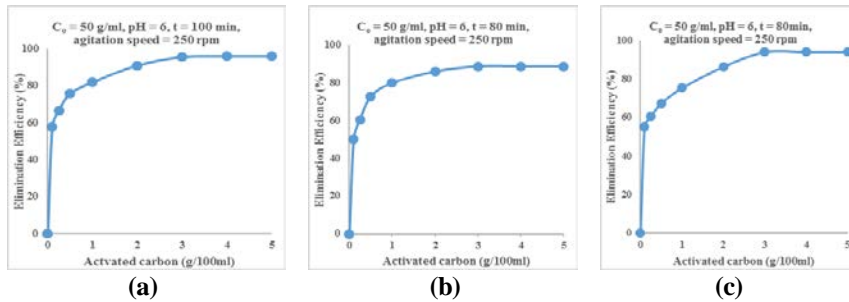


Fig. 4. The influence of (a) DPSAC, (b) PSAC and ASAC dosages on elimination efficiencies of Copper.

### 3.1.3. Influence of initial concentration of copper

Figure 5 proves that the removal efficiency of copper is inversely proportional to the initial concentration of copper. It shows that when the initial concentration of copper increased from 50 to 250 mg/L, the removal efficiency decreased from 95.54% to 82.84% for DPSAC 88.64% to 75.71% for PSAC, and 94.0% to 56.01% for ASAC, respectively. This could be attributed to the fact that, at low initial concentration, the number of adsorption sites was enough to remove the majority of copper ions. While at higher concentration, the adsorption sites will not be enough to remove the high number of copper ions, which decreases the removal efficiency [53].

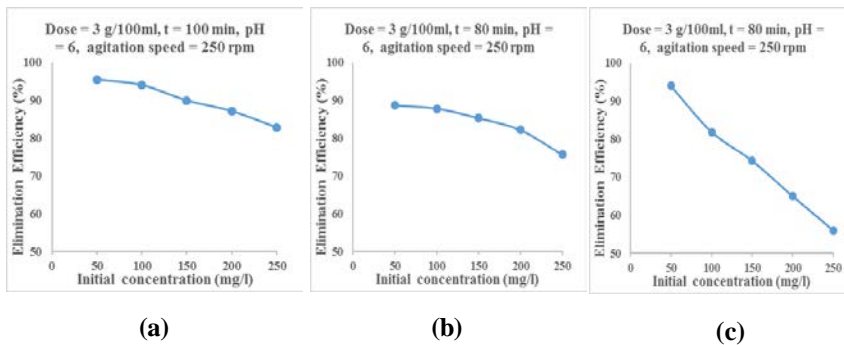
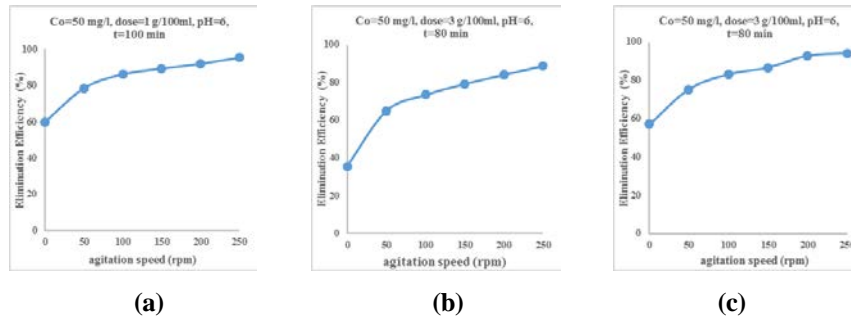


Fig. 5. Influence of copper concentration on the removal efficiency by (a) DPSAC, (b) PSAC and (c) ASAC as adsorbents.

### 3.1.4. Influence of agitation speed

The influence of agitation speed on the removal of copper from water has been investigated by carrying out several tests at different agitation speed, ranging from 0 to 50, 100, 150, 200, and 250 rpm. The obtained results, Fig. 6, indicated that when the agitation speed increased from 0 to 250 rpm, the removal of copper has been increased from 60.15 %, 35.69% and 57.05% to 95.52%, 88.86% and 94% for DPSAC, PSAC and ASAC, respectively. This could be explained by the fact that increasing the agitation speed improves the diffusion of copper towards the surface of the absorption medium (activated carbon) that increases the contact between the binding sites and the copper ions, which enhances the removal efficiency [54].



**Fig. 6. Influence of agitation speed on the removal of copper by (a) DPSAC, (b) PSAC and (c) ASAC as adsorbents.**

### 3.2. Sorption isotherms

The parameters that give the higher removal efficiency of copper by activated carbon were pH of 6, agitation speed of 250 rpm, activated carbon dosage of 3 g/100ml, and an contact time of 100 min for DPSAC and 60 min for both PSAC and ASAC (the best contact time obtained from the experimental work). The results of the sorption experiment were fitted with the previously described linearized forms of Langmuir and freundlich models. The fitted limits and coefficient of determination ( $R^2$ ) for Langmuir and freundlich models are represented in Table 1. In comparison with the other models, Langmuir model provided the highest correction for sorption of copper on activated carbon; it also showed a good ability to describe the process of copper sorption on activated carbon (governs the migration of copper in one-dimensional column).

**Table 1. Parameters of Langmuir and Freundlich models for sorption of copper onto activated carbon.**

Adsorbent	Langmuir constants			Freundlich constants		
	a(mg/g)	b(l/mg)	$R^2$	a(mg/g)	b(l/mg)	$R^2$
PDPAC	8.475	0.108	0.9935	1.264	0.496	0.9305
PPAC	9.524	0.036	0.9904	0.569	0.613	0.9597
APAC	4.950	0.087	0.9933	0.402	0.402	0.9665



### 3.3. Sorption isotherms

Table 2 presents the results of the measurement of  $D_L$  at different flow rates ( $V$ ), which was calculated as follows:

$$D_L = 35.046 V + 0.0262 \quad \text{for soil} \quad R^2 = 0.9998 \quad (4)$$

$$D_L = 7.8397 V + 0.0125 \quad \text{for DPSAC} \quad R^2 = 0.9119 \quad (5)$$

$$D_L = 5.198 V + 0.0064 \quad \text{for PSAC} \quad R^2 = 0.9121 \quad (6)$$

$$D_L = 10.467 V + 0.0128 \quad \text{for ASAC} \quad R^2 = 0.997 \quad (7)$$

These equations could be summarized by the following general form:

$$D_L = D_{mesh} + \tau D_o \quad (8)$$

Where  $D_{mesh}$  is the coefficient of mechanical dispersion and  $D_o$  is the coefficient of effective molecular diffusion.

**Table 2. Measured values of the longitudinal dispersion coefficient  $D_L$  as a function of flow rate.**

Flowrate (ml/min)		5	10	15
<b>PDPAC</b>	$V$ (cm/s)	0.0131	0.0262	0.0393
	$D_L$ (cm <sup>2</sup> /s)	0.1336	0.181	0.339
	$\alpha_L$ (cm)		7.8397	
<b>PPAC</b>	$V$ (cm/s)	0.013	0.0262	0.0393
	$D_L$ (cm <sup>2</sup> /s)	0.0862	0.1181	0.223
	$\alpha_L$ (cm)		5.198	
<b>APAC</b>	$V$ (cm/s)	0.0122	0.0244	0.0367
	$D_L$ (cm <sup>2</sup> /s)	0.1446	0.26	0.401
	$\alpha_L$ (cm)		10.467	
<b>Sandy soil</b>	$V$ (cm/s)	0.0119	0.0238	0.0357
	$D_L$ (cm <sup>2</sup> /s)	0.4399	0.867	1.274
	$\alpha_L$ (cm)		35.046	

### 3.4. Modeling application

Advection dispersion process is the cause of copper migration in a porous medium, so that the one-dimensional system of copper migration in the porous media can be represented by the following equations.

$$D_z \frac{\partial^2 C_{Cu}}{\partial z^2} - V_z \frac{\partial C_{Cu}}{\partial z} = \frac{\partial C_{Cu}}{\partial t} + \frac{\rho_b}{n} \frac{\partial(q)}{\partial t} \quad (9)$$

where  $C_{Cu}$  = copper mass concentration in water,  $V_z$  = speed of flow,  $D_z$  = coefficient of longitudinal dispersion in the  $z$  direction,  $\rho_b$  = bulk density of dry adsorbing material, and  $q$  = copper concentration on solid, the subsequent term ( $q$ ).

For verification of model, parameters and constants related to the soil and activated carbon were evaluated either through laboratory tests and through approximation from relevant literature data [39, 47] (Table 3).

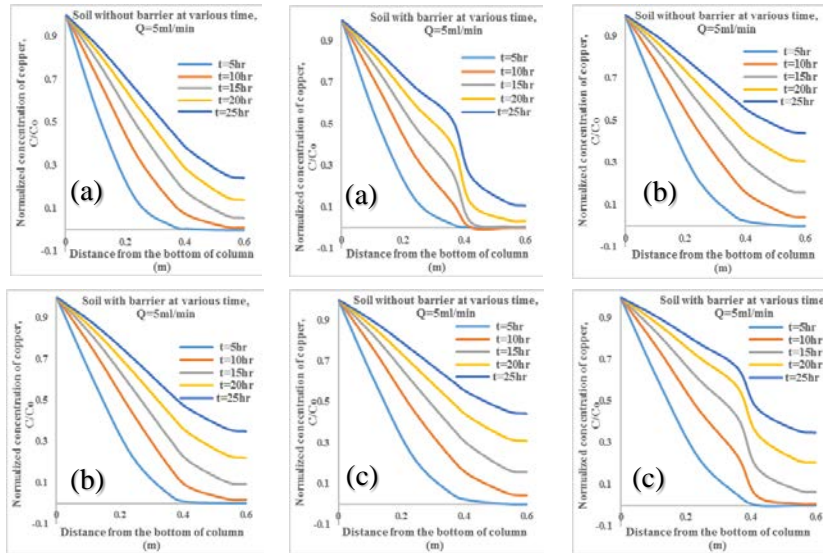
**Table 3. Parameters, constants, boundary and initial conditions used in the transport modeling of copper in pilot plant column.**

Item	Location	Type/Value
<b>Aquifer characteristics</b>	Sandy soil	Porosity (nA) = 0.44
		Sandy soil depth before PRB(cm) = 36 Sandy soil depth after PRB (cm) = 32 Longitudinal dispersivity ( $\alpha_L$ , cm) = 35.046 Bulk density (g/cm <sup>3</sup> ) = 1.47415
<b>Aquifer characteristics</b>	DPSAC, PSAC, and ASAC	Porosity (nB) = 0.41, 0.40 and 0.40
		Barrier bed depth (cm) = 6
		Longitudinal dispersivity ( $\alpha_L$ , cm) = 7.8397, 5.198 and 10.467 Bulk density (g/cm <sup>3</sup> ) = 0.55988, 0.67688, 0.64513
<b>Initial condition</b>	The initial concentration of Cu <sup>+2</sup> (mg/L) = 0	
<b>Boundary conditions</b>	Concentration of Cu <sup>+2</sup> at distance =0 (mg/L) = 50	
	Advection flux ( $\frac{\partial C}{\partial z}$ ) at distance =60 cm = 0	

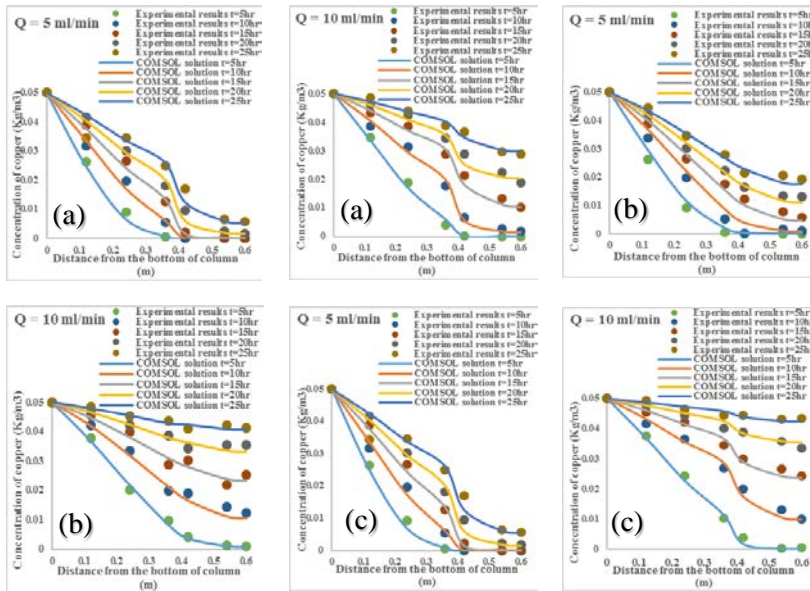
Figure 7 shows the normalized concentration lines of copper in the soil, which were determined using the COMSOL software. These values were calculated with and without the presence of permeable reactive barrier at a flow rate of 5 mL/min after 5, 10, 15, 20 and 25 hours. This figure demonstrates the potential role of the permeable reactive barrier in hindering the spreading of copper ions, where it can be seen that the change in copper concentration after 0.4 m from the bottom of the column became very slow.

A comparison between the theoretical (COMSOL solution) and experimental results for copper concentrations at 5, 10, 15, 20 and 25 hours and flow rate of 5 ml/min along the test column are shown in Fig. 8. It can be obviously noticed, from this figure, that the predicted and experimental results are highly agreed. Additionally, the root-mean squared error (RMSE) was used as a statistical tool to discover the grade of contract between these results [55]. The calculated RMSE was less than 0.1%, which confirms the agreement between the predicted and experimental results.

Finally, it is noteworthy to mention that the obtained results from the current study are in a good agreement with the previous studies, such as the study of Chamanchi, et al. [56] and El-Said, et al. [57].



**Fig. 7. The normalized concentrations of copper as a function of the distance from the bottom of the column in (a) DPSAC, (b) PSAC and (c) ASAC.**



**Fig. 8. Comparison between predicted (COMSOL solution) and experimental results for copper concentrations on (a) DPSAC, (b) PSAC and (c) ASAC at two values of flow rates.**

#### 4. Conclusions

The current study attempted to investigate the possibility of preparing of cost-effective activated carbon from seeds, namely date palm seeds, peach seeds, and apricot seeds. The results of the experiments evidenced the following facts:

- Removal of copper using activated carbon is positively influenced by increasing the contact time, agitation speed and dosage of activated carbon. However, it is negatively influenced by increasing the copper concentration.
- Neutral pH level is very favourable for copper removal by activated carbon.
- Sorption process of copper on the DPSAC, PSAC and ASAC could be efficiently described by Langmuir isotherm model.
- Generally, the experimental results proved that the activated carbon that prepared from wasted seeds is a cost-effective and efficient media for the removal of copper from contaminated groundwater.

#### Nomenclatures

$a$	the capability of maximum adsorption (mg/g)
$b$	the constant (l/mg)
$C/C_o$	normalized concentration
$C_e$	balance concentration, mg/L
$C_o$	initial concentration of $\text{Cu}^{+2}$ , mg/L
$D_L$	the Coefficient of longitudinal dispersion $t$ , $\text{m}^2/\text{sec}$
$D_{mesh}$	the coefficient of mechanical dispersion, $\text{m}^2/\text{sec}$
$D_o$	the coefficient of effective molecular diffusion, $\text{m}^2/\text{sec}$
$D_z$	dispersion coefficient in the direction $z$ , $\text{m}^2/\text{sec}$
$K_F$	the coefficient of Freundlich sorption (mg/g)
$m$	mass of activated carbon in the flask, g
$n$	porosity
$q_e$	quantity of solute eliminated from solution, mg/kg
$R^2$	constant of determination
$V$	size of solution in the flask, L
$V_z$	speed of flow in the direction $z$ , m/sec
$D_{mesh}$	The coefficient of mechanical dispersion.
$D_o$	Coefficient of effective molecular diffusion

#### Greek Symbols

$\tau$	Tortuosity factor
$\alpha_L$	longitudinal dispersivity, cm
$\rho_b$	bulk density, $\text{g}/\text{cm}^3$

#### Abbreviations

RMSE	Root-mean squared error
AS	apricot seeds
ASAC	activated carbon from apricot seeds
$\text{Cu}^{+2}$	copper
1D	One-dimensional
DPS	date palm seeds
DPSAC	activated carbon from date palm seeds
PRB	Permeable reactive barrier

PS	peach seeds
PSAC	activated carbon from peach seeds

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