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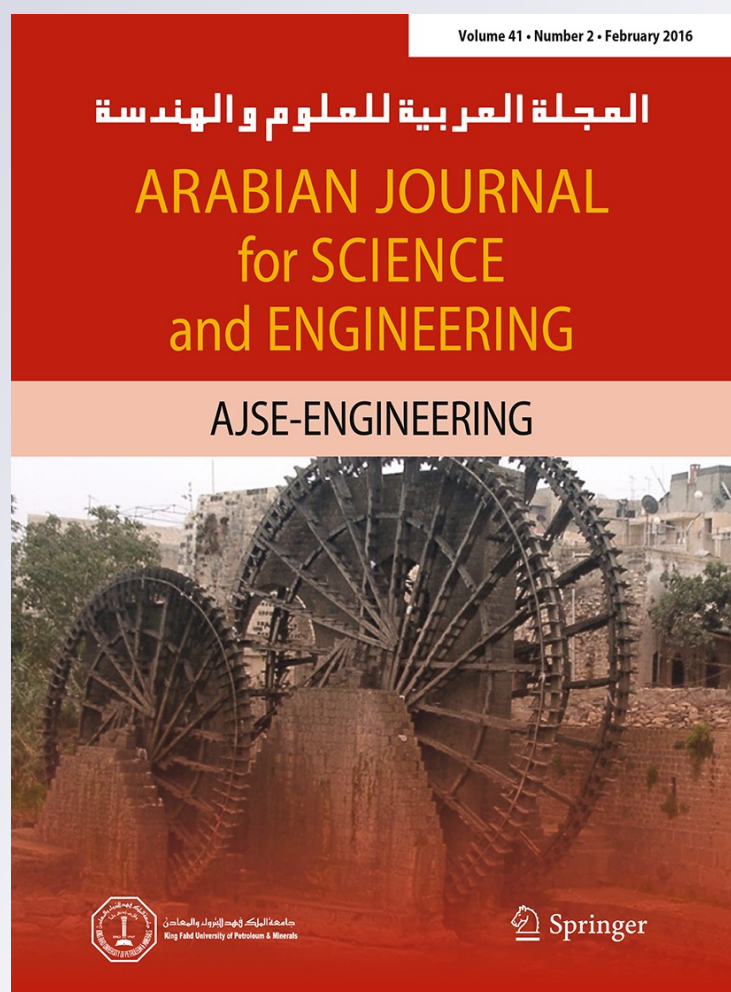
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# Remediation of Cu(II) from Well Water of Iraq by Using Cortex of Fruits and Agricultural Waste

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**Abstract** Copper ion Cu(II) is among the main pollutants present in excessive amounts in underground water in many locations in Iraq. The present work studies adsorptive removal of Cu(II) from well water using waste materials such as watermelon shell, lemon, and banana peels. Influences of many parameters like initial concentration, dosing, contact time, pH, and particle sizing were examined. The elimination efficiency of the watermelon shell was 90 % compared with that of lemon Peel was 78 %, while for banana Peel was observed to be 65 %. Meanwhile, the highest possible adsorption capacity was 9.54 mg/g for watermelon shell, 8.24 mg/g for lemon peel, and 7.65 mg/g for banana peel. The column test was applied to the real well water, confirmed that reloading of the column could enhance its own removing efficiencies by 15–30 %. However, characterization of adsorption of the watermelon shell, measured from FTIR and SEM micrograph, verified that watermelon shell is a powerful adsorbent due to its own functional groups as well as cavities on its surface texture. In the same context, comparison between adsorption capability of watermelon shell and both Langmuir and even Freundlich models points out that Langmuir is much better fit the experimental data. Overall results confirm that watermelon shell is an excellent and also alternative adsorbent material for eliminating copper ions. Moreover, it is natural, environmentally friendly, and cost-effective for treating contaminated well water in Iraq.

**Keywords** Well water · Fruit cortex · Adsorption · Heavy metals · Removal efficiency

## 1 Introduction

It is seriously noted that dissolving of heavy metals resulting from industrial activities could pollute underground water, which threatens the environment and public health due to their toxicity, accumulation in food chain, and persistence in nature [1]. The pollution of well water is one of the major environmental concerns in Iraq today. This problem is considered enormous, especially in the remote areas of Iraq. In those locations, there is no standard of drinking water to protect against both naturally occurring and man-made contaminants that may be found in the drinking water. Most distant locations in Iraqi are not covered with the pipeline system for the clean water, as in many developed countries. Nonetheless, there is no easy way to supply these areas with disinfectant water. Therefore, most of the people live in these locations depend totally on the well water.

Heavy-metal contamination exists in aqueous wastes of many industries (e.g., metal plating, mining operations, tanneries, radiator manufacturing, smelting, alloy industries, and storage battery industries) [2]. According to the healthy drinking water standard, one of the wool contaminants which should be removed from wastewater is copper Cu(II), merely because of its toxicity to human beings at levels of 100–500 mg/day [3,4]. Copper is one of the main contaminants issuing coming from electrical, electroplating, and metal finishing industries. It is predominately based in high concentrations close to mines, landfills, and water disposal sites [5]. The World Health Organization in 2006 recommended 2.0 mg/L as the maximum passable concentrations of copper in drinking water [6], while the standard specification of

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drinking water in Iraq states that 1.0 mg/L is the maximum allowable copper ion concentration. For human being, copper toxicity would cause diseases, cancers, and many health consequences [7]. Hence, the removal of copper from wastewater before its discharge into the aquatic system is extremely important and deserves immediate attention.

In the literature review, several different types of sorbents were investigated to be used as adsorptive elimination of Cu(II) from its aquatic solutions. For example, activated carbon is the most-employed sorbent for removal, but its usage is restricted because of elevated cost for activation and imperfect regeneration [8]. In comparison with activated carbon, agricultural waste and industrial by-product may be cost-effective and are preferable sorbents. In the same context, many agricultural wastes have been reported as potential and low-cost adsorbents for the removal of heavy metals. For instance, fruit peels have been actively used for the elimination of heavy metals from aqueous solution, such as: banana peel [9], mango peel [10], orange peel [11], citrus peel [12], and jackfruit peel [13]. However, watermelon could be involved to eliminate metal ions from an aqueous solution because of carboxylic groups and lignocellulose materials that are found in the peels [14].

The present work will investigate using watermelon shell as a remover of the copper ions from underground well water of Iraq. However, the results will be compared with those of lemon and banana peels. Watermelon shells are specifically used because Iraqi's soil is the best location for growing watermelon, and considering the size of the watermelons, they would produce more waste than other fruit peels. The present study will consider the effect of many variables on the adsorption process in batch experiments, such as: initial concentration of Cu(II), dose, contact time, pH, particle size, and its application in fixed bed column to treat the actual contaminated well water. However, analyzing the adsorption capacity with Langmuir and Freundlich adsorption isotherms and characterizing the adsorbent material with FTIR and SEM are made to show the effect of copper ion binding on the functional groups and surface morphology.

## 2 Materials and Methods

Adsorbent preparation is the first step in this study. There are great numbers of adsorbent structures which differ slightly in stability and in the existence of a foreign molecule pockets. The adsorbent is pictured as automatically forming pockets that fit closely enough to the foreign molecule to hold it by van der Waals' forces, hydrogen bonds, interionic attractions, and other types of intermolecular interaction [15]. In the present work, the preparation process starts with collecting from the local markets the following fruits: watermelon shells (WSs), lemon peels (LPs), and banana peels (BPs). After that, they

were washed with tap water followed with distilled water. After washing, the cortices of fruits were cut into little divisions and dried under sunlight for 7 days. The dried pieces were washed with distilled water and then kept in the oven at 70 °C for 6 h to guarantee drying. Lastly, the dried materials were ground to powder using grinder and sieved to get two particle sizes (1 and 2 mm).

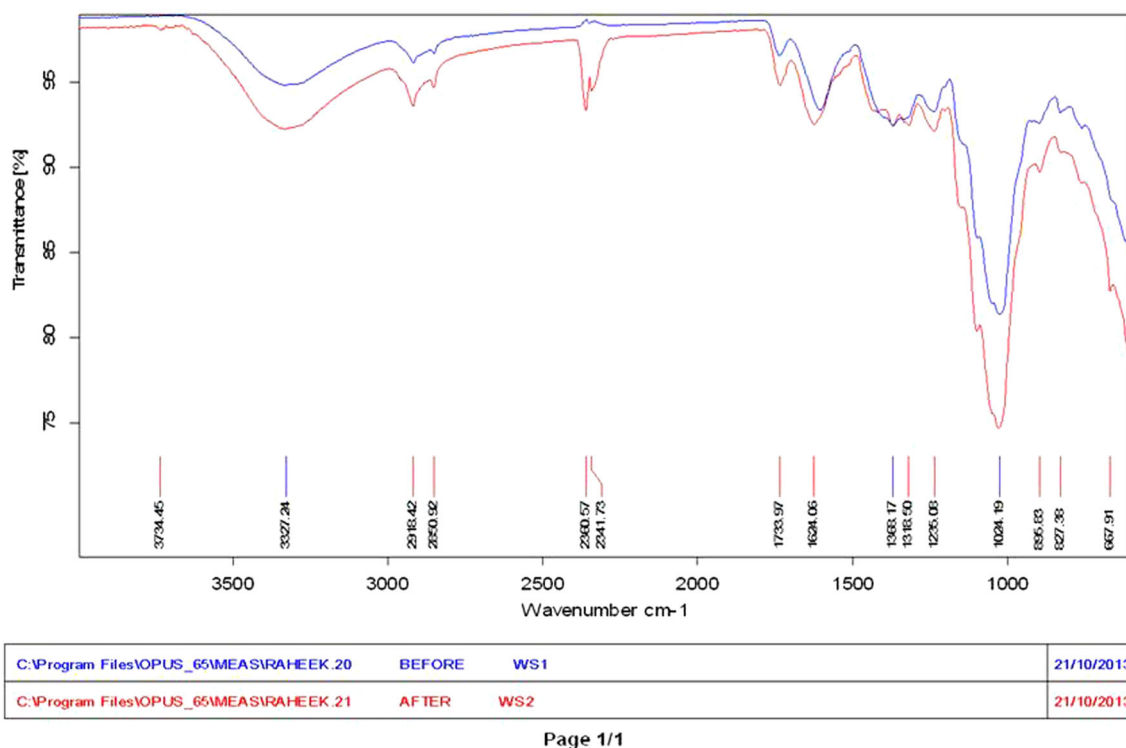
The second step was synthetic solution preparation. Such kinds of solutions emulate the major ion compositions of natural waters which are useful in most experiments that aim to understand a biogeochemical process. The synthetic freshwaters of any desired composition should ensure that the ionic components of each concentrated stock solution cannot form an insoluble material [16]. In the present work, the stock solution of Cu(II) was prepared by dissolving salts of nitrates in deionized water and diluted to the desired metal concentrations. The pH adjustments were established using 0.1 M HCl and 0.1 M NaOH solutions. All chemical components of analytical grade were obtained from Sigma-Aldrich Company, and deionized water was used for dilution.

The third step in the methodology was the actual treatment of polluted well water. Column method was used to eliminate the heavy metal from water. However, a process used for the purpose of generating high retention yields with various characteristics, such as keeping the adsorbent separated from the contaminated liquid to avoid supplementary separation method. In addition, the column size can be scaled to industrial-level handling higher volume of contaminated water at low cost accounting that raw materials can be gained from wastes rejected from fruit processing corporations [17]. In this method, 5 g of adsorbent powder was weighed and fixed in 10 cm high, 2.5 cm i.d. column using glass wool support. However, a 10 ml of actual underground well water was collected from remote area of Hilla city, which is located in the south of Iraq, and 10 mg/l of Cu(II) was added keeping the column closed until total adsorbent humidification. After that the column was opened to allow water to flow, and pressure was applied to the top of the column keeping constant flow rate of 1 ml/min. From the effluent, a sample of 1.0 ml was collected every 1 min, labeled, and analyzed. To study the effect of reloading, the well water was reloaded into the column in order to test the amount of metal adsorbed after more than one loading.

The fourth step in methodology was results analysis. Herein, batch process was used to investigate the elimination of Cu(II) from artificial wastewater. Adsorption tests were performed involving 25 ml of copper solution of desired concentrations (5, 10, 15, 20 mg/L), adsorbent dosage of 0.5, 1, 1.5, 2 g/L in 250-ml flasks at room temperature, and pH of solution was 2, 4, 6, 8, and 10 for several intervals as time of contact of 1, 2, and 4 h. Adsorption quantity of a Cu(II) per weight of WS, BP, LP, and the percentage of removed copper were estimated using Eqs. 1 and 2, respectively [17].







**Fig. 1** FTIR analysis of watermelon shell after and before absorption

$$q_e = \frac{(C_i - C_f) \times V}{m} \quad (1)$$

$$Cu_{\text{Removal}} = \frac{(C_i - C_f)}{C_i} \times 100 \quad (2)$$

where  $q_e$  (mg/g) is adsorption capacity;  $C_i$  (mg/l) and  $C_f$  (mg/l) are initial and final concentrations, respectively, while  $m$  (g) and  $V$  (l) are mass of adsorbent and volume of solution, respectively.

The tests were performed under various cases of pH, initial concentration, time, quantities of adsorbent. After undergoing experimental procedures, the liquid was filtered through standard laboratory filtration using Whatman filter paper no. 40 which has ashless filter paper with medium speed and retention (8  $\mu$ m). The amount of copper adsorbed was measured using AA-6300 double-beam atomic absorption spectrophotometer which has a wavelength range from 185 to 900 nm. The adsorbent after using was dried to identify the functional groups by characterization using Fourier transform infrared spectrometer (FTIR) model FTIR-8400S. Lastly, scanning electron microscope (TESCAN) was used to identify the surface morphology.

### 3 Results and Discussion

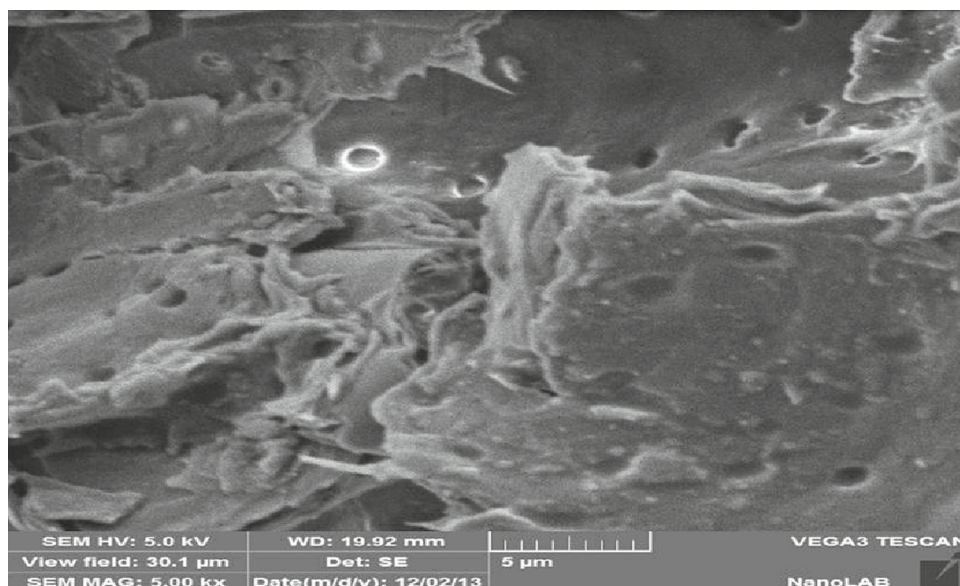
In the present work, the adsorption capacities to take off Cu(II) from the underground water of well in Iraq were

assessed. Different fruit cortex waste materials such as watermelon shell, lemon peels were used. The cortexes were applied in batch tests and also in the form of a package in conventional column. Adsorption capacity and removal efficiency as a percentage removal of Cu(II) ions were calculated according to Eqs. 1 and 2, respectively. However, batch experiments and column test were done, and the operating variables affecting the removal efficiency were studied. The sensitivity analyses of the most important factors which influence removing capability were investigated. The results are as presented in the subsections below.

#### 3.1 Characterization of Adsorbent

The FTIR technique is a significant instrument to distinguish several main functional groups, which can adsorb pollutant ions. The FTIR spectrum of watermelon before and after Cu(II) adsorption is offered in Fig. 1. The FTIR spectra were used to determine the vibration frequency changes in the functional groups of the adsorbent. The spectra were traced on a same scale of the transmittance axis for WS before and after adsorption. FTIR spectra show a number of adsorption peaks, denoting a complicated nature of the examined adsorbent. The spectrum displays the following bands: 3327 (O–H stretching vibrations), 2918 (C–H stretching vibrations), 1734 (C=O stretching vibrations of carboxylic acid), 1624 (C=C stretching vibrations), 1368 (salts of carboxylic

**Fig. 2** Typical SEM micrograph of watermelon shell before adsorption of copper ions



acid), 1235, 1024 (C–O–H), 896, and  $668\text{ cm}^{-1}$ . The spectra at 2361 and  $2342\text{ cm}^{-1}$  appeared which would have resulted from the complication of copper ion with the functional groups from watermelon shell. The comparisons of FTIR spectra of native and metal-loaded WS show the shift in peaks may be attributed to changes in counter ions associated with carboxyl ate and hydroxyl anions, suggesting that acidic groups and carboxyl and hydroxyl groups are predominant contributors to metal uptake. Strong shift in the hydroxyl peak confirms the binding of metal ions.

The microporous structure of watermelon shell with particle size of ( $5\text{ }\mu\text{m}$ ) was observed at a resolution of  $500\times$  (Figs. 2, 3). The micrograph of adsorbent shows some cavities in the surface structure capable of uptaking copper ions as well as irregular and porous microstructure of the adsorbent. It is clearly seen that there is a considerable change in morphology of WS before and after copper binding.

### 3.2 Effect of Initial Copper Concentration

Adsorption experiments were conducted to study the effect of the initial concentration of Cu(II) in the solutions on the rate of metal adsorption on WS, LP, and BP. The experiments were carried out at a fixed adsorbent dose of ( $1\text{ gm}$ ) and at various initial Cu(II) concentrations (5, 10, 15, and 20 ppm) for 2-h contact time as shown in Fig. 4. As the initial concentration increases from 5, 10, 15, and 20 ppm, the equilibrium removal of Cu(II) decreases. This may be due to the fact that at lower concentrations, almost all the copper molecules are adsorbed very quickly on the outer surface, but further increases in initial Cu concentrations lead to fast saturation of adsorbent and thus most of the Cu adsorption took place slowly inside the pores. The results agree with those of Sağlam [18].

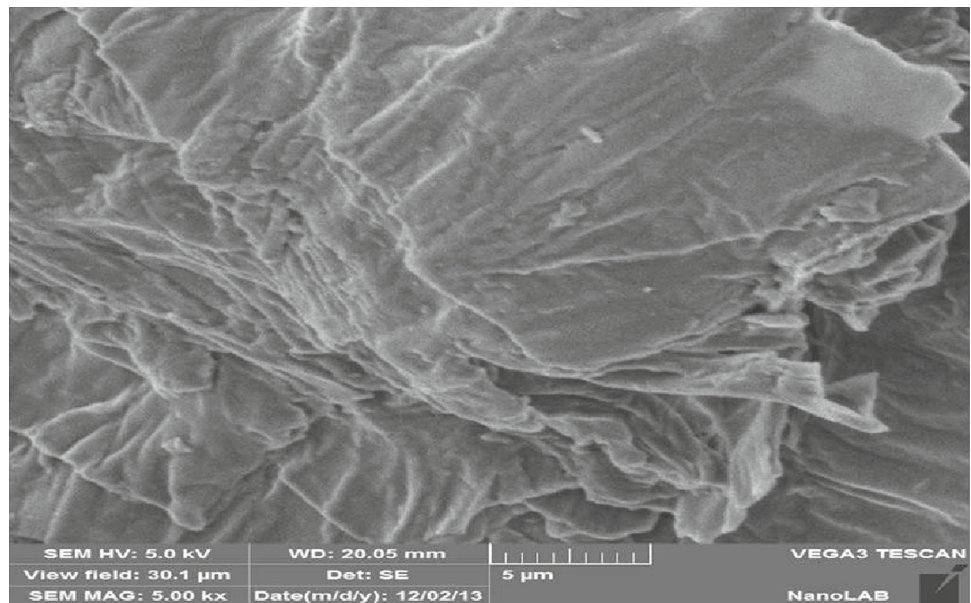
Also the adsorption capacity increases with increase in initial concentration, since concentration gradient between bulk and surface of the adsorbent raises with increase in concentration, and this results in the increase in driving force responsible for high uptake of metal ions. The maximum adsorption capacities of Cu(II) were 9.54, 8.24, and  $7.65\text{ mg/g}$  using WS, LP, and BP, respectively. Figure 5 shows the residual concentrations of copper ions in the effluent. From the results, it can be clearly seen there is correspondence with the WHO which states that  $1\text{ mg/L}$  is safe for drinking water. Therefore, watermelon shell is considered as efficient adsorbent to remove copper ions from wastewater in Iraq.

### 3.3 Impact of Adsorbent Dosage

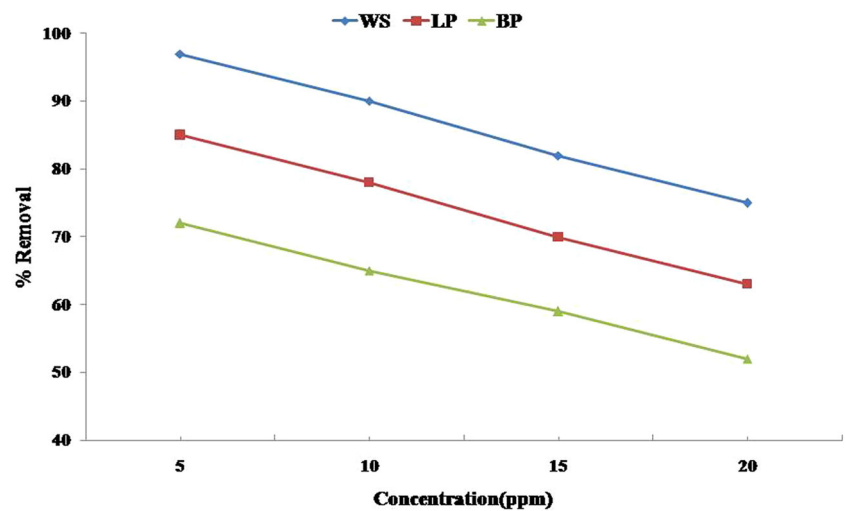
Adsorption of Cu(II) among WS, LP, and BP was examined by varying the amount of adsorbent in the experiment solution while regarding the first Cu(II) concentration fixed at ( $10\text{ mg/L}$ ). Raising in adsorbent dose boosted the elimination percentage of Cu(II), as displayed in Fig. 6, that is consequent raise the adsorption surface area of adsorbent. However, in accordance with certain dosage, the variation in concentration is quite low. This is attributed to elevated adsorbent concentration; thither a so rapid surface adsorption upon adsorption face that it achieves less solute concentration in the solution while the adsorbent dosage is minimal. So, through an increment in adsorbent dosage, the quantity of Cu(II) adsorbed per unit mass of undermost residue is lowered, thence causing a reduction in equilibrium taking up amount. The farthest elimination was detected as 90 % for WS, 78 % for LP, and 65 % for BP at  $1\text{ g/L}$  dosing. The results show good agreement with those of Banerjee et al. [17].



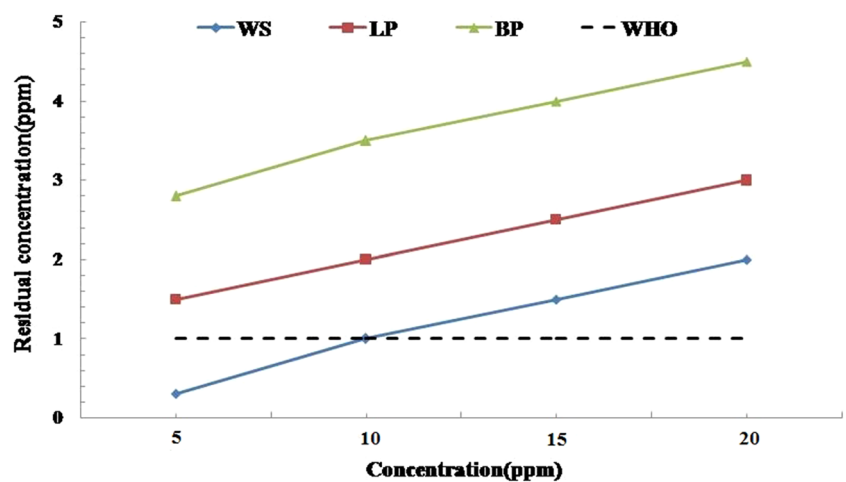
**Fig. 3** Typical SEM micrograph of watermelon shell after adsorption of copper ions



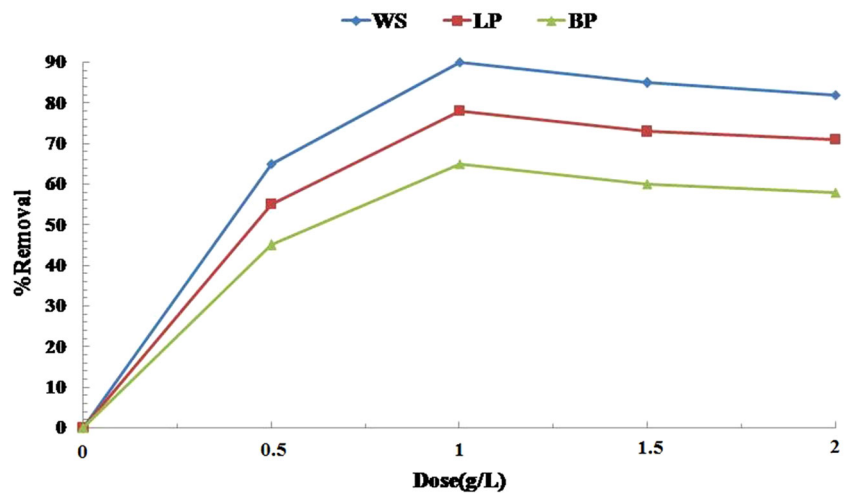
**Fig. 4** Effect of initial concentrations on removal percentage (dose = 1 g/l, contact time = 2 h, pH = 8, 1 mm particle size)



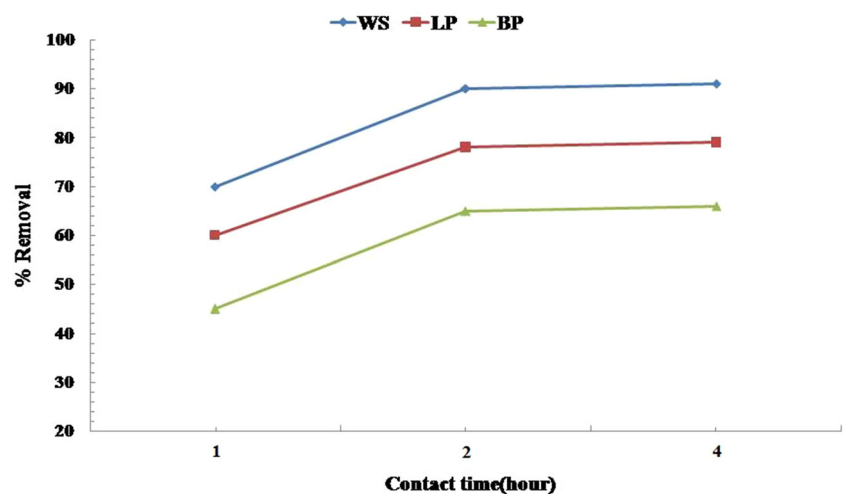
**Fig. 5** Effect of initial concentrations on residual concentration in comparison with WHO (dose = 1 g/l, contact time = 2 h, pH = 8, 1 mm particle size)



**Fig. 6** Effect of dose on removal percentage (initial concentration = 10 mg/l, pH = 8, contact time = 2 h, 1 mm particle size)



**Fig. 7** Effect of contact time on removal percentage (initial concentration = 10 mg/l, dose = 1 g/l, pH = 8, 1 mm particle size)



### 3.4 Impact of Contact Time

Contact time influences the scope of adsorption for Cu(II) ions. Figure 4 presents the difference in the range of adsorption percentages of Cu(II) on WS, LP, and BP with time for constant metal concentration. As it can be seen from Fig. 7, the amount of the adsorbed Cu(II) on the adsorbent increases rapidly with time in the initial stage (1–2 h range), and at the same time (2 h), it attains a fixed value beyond which no more is removed from solution, i.e., further increase in contact time has no significant effect on uptake amount. At this point, the amount of the Cu(II) desorbed from the adsorbent is in a state of dynamic equilibrium with the amount of the metal being adsorbed onto the adsorbents (WS, LP, and BP). Similar results were obtained by Bello et al. [19].

### 3.5 Impact of pH

The effect of pH on the removal of Cu(II) is shown Fig. 8. It is vividly shown that Cu adsorption by WS increases with

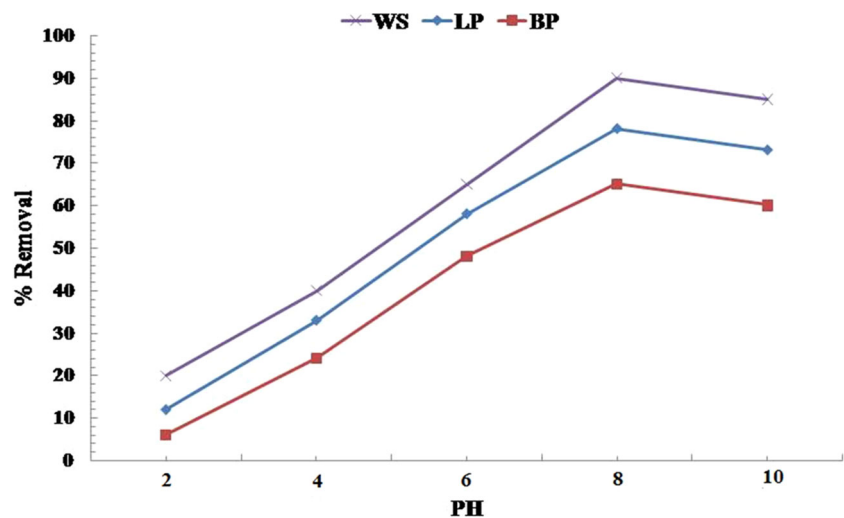
increasing pH from 2 to 10. The optimum pH for Cu(II) adsorption with watermelon shells was predicated to be 8. Less metal elimination at elevated acidic pH could be owing to opposition of metal binding locations among positively charged Cu(II) ions and hydrogen ions. As well, at lower pH, the adsorbent face turned into further positively charged, thus reducing attraction between WS and metal ions. Further increase in pH (beyond pH 8) can be referred to a deposition of Cu(II) as  $\text{Cu}(\text{OH})_2$ . That minimizes the concentration of Cu(II) in the solution and consequently reduces elimination capability of WS. Comparable effects have been obtained for LP and BP in good agreement with the results of Bharathi and Ramesh [17].

### 3.6 Impact of Particle Size

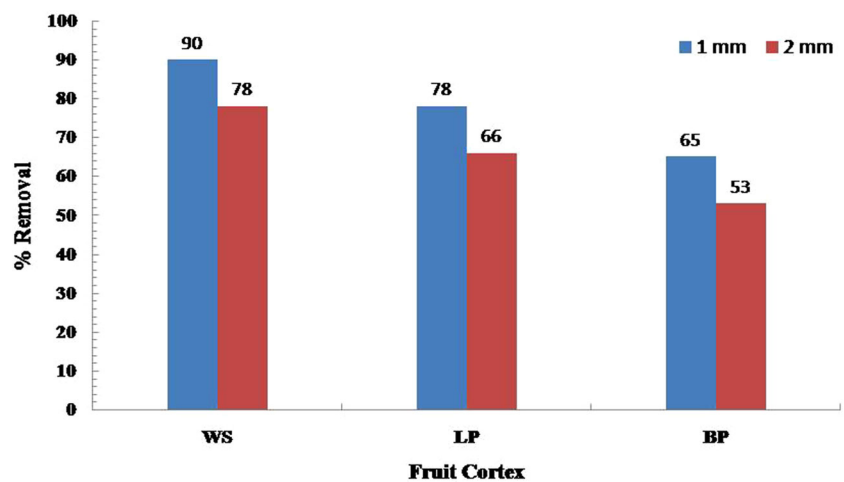
The consequences of change in particle sizes on Cu(II) adsorption rate are presented in Fig. 9. The impact of changing the adsorbents particle size (1, 2 mm) on elimination competence displays a further prevailing elimination of



**Fig. 8** Effect of pH on removal percentage (initial concentration = 10 mg/l, dose = 1 g/l, contact time = 2 h)



**Fig. 9** Effect of particle size on removal percentage (initial concentration = 10 mg/l, dose = 1 g/l, contact time = 2 h, pH = 8)



Cu(II) by minimal particles. This is attributed to an increment in overall surface area that supplies additional adsorption locations for metal ions. With regard to a greater particles, the propagation impedance to mass transfer is elevated, and almost the inside face of the particle does not involved in adsorption and thus, the magnitude of Cu(II) adsorbed is little. Also, smaller particle size produces lower flow inside the column and also increases the capillarity effect producing biggest liquid detention in the column.

### 3.7 Impact of Reloading

When reloading the Cu(II) solution through the column, an improvement was observed on metal retention obtained as shown in Fig. 10. For WS, the removal efficiency percentage was improved by 16 % when reloading for three times, and for LP, it was improved by 22 % with three times loading, while for BP, the improvement in removal efficiency was 27 %. These results are in good agreement with results of

Kelly et al. [20] who reported an improvement in removal efficiencies by 10–50 % when reloading for three times.

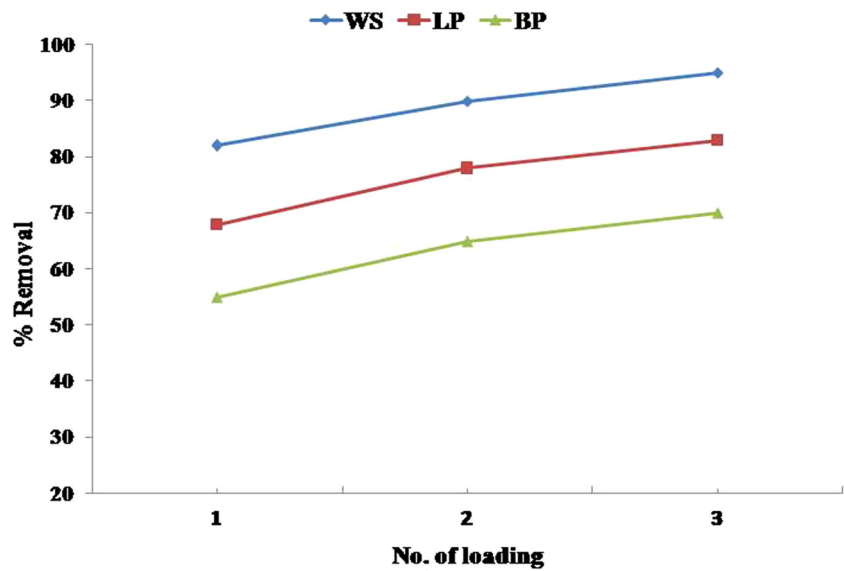
### 3.8 Breakthrough Curve of Fixed Bed Column

The experimental column operated until the copper ions in the effluent stream started appearing. For practical purposes, the working life of the column is above breakthrough point and this is important in process design because it directly affects the feasibility and economics of the process. The breakthrough capacity of Cu(II) is shown in Fig. 11. It is clear from the figure that during the first 5 min of operation, the well water containing 10 mg/l of copper ions can be passed through the column without being detected Cu(II) in the effluent.

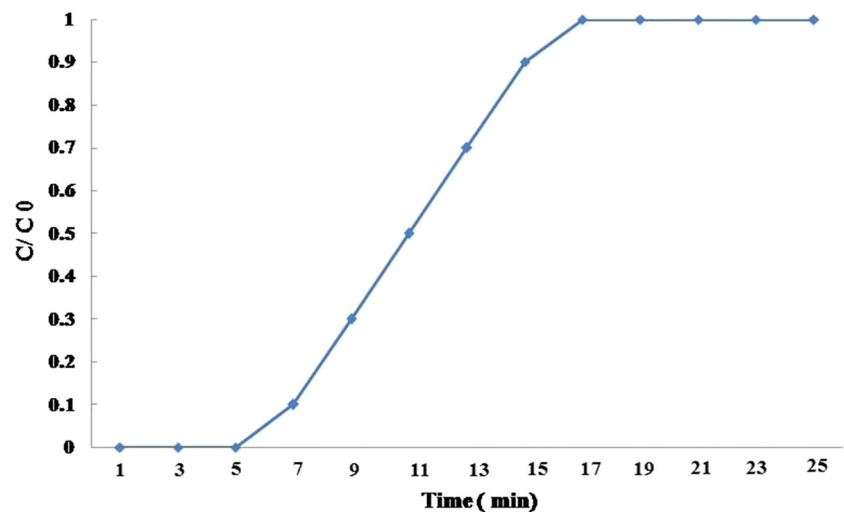
### 3.9 Adsorption Models

Results of WS adsorption capacity were analyzed using Freundlich and Lambert equation at different temperatures.

**Fig. 10** Effect of number of loadings on removal percentage in column test (initial concentration = 10 mg/l, pH = 8, particle size = 1 mm)



**Fig. 11** Breakthrough curve of Cu(II) for underground well water in column test (metal concentration = 10 mg/l, pH = 8, WS adsorbent particle size = 1 mm)



Meanwhile, the characteristic parameters of each adsorption isothermal were estimated. The linear form of Langmuir isotherm is given by the following equation [17].

$$1/q_e = (1/q_m) \times (1/b) \times (1/C_e) + (1/q_m) \quad (3)$$

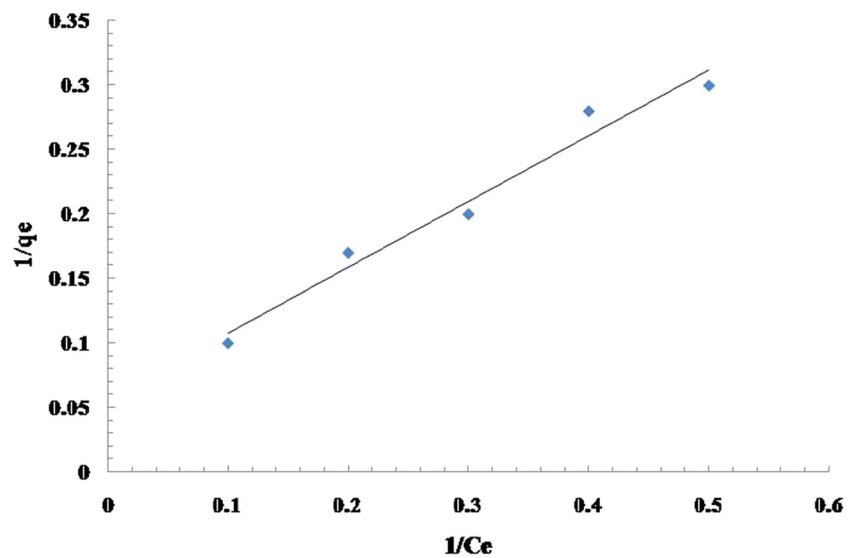
where  $q_e$  represents the amount of metal which is adsorbed per unit weight of adsorbent;  $q_m$  is the maximum adsorption capacity (mg/g) determined by the number of reactive surface sites in an ideal monolayer system;  $C_e$  is the concentration of metal ion (mg/l) at equilibrium; and  $b$  is a constant related to bonding energy associated with pH-dependent equilibrium constant. Through analysis, it was vividly shown that the plot of  $1/q_e$  versus  $1/C_e$  exhibits straight line as in Fig. 12, and the values of  $b$  and  $q_m$  parameters for this relation are documented in Table 1. However, it is found that  $q_e$  estimated from the model as  $q_e(\text{cal.})$  is very close to  $q_e$  determined experi-

mentally as  $q_e(\text{exp.})$  showing that Langmuir isotherm fits the experimental data. The linear form of Freundlich isotherm is represented by the following equation [17].

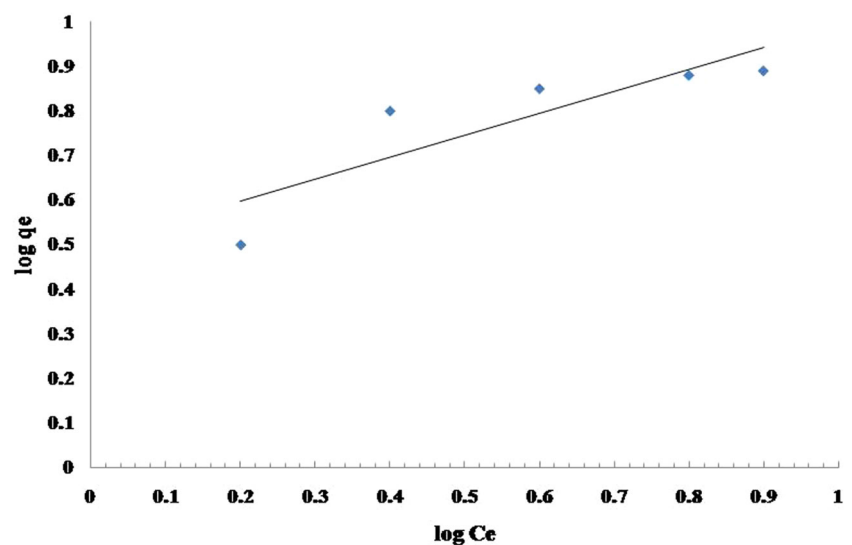
$$\log q_e = \log K + (1/n) \log C_e \quad (4)$$

Herein,  $K$  is Freundlich constant and  $n$  is another constant that informs about the heterogeneity degree of the surface sites, when  $n$  approaches zero, the surface site heterogeneity increases. It is also clear that the  $\log q_e$  versus  $\log C_e$  exhibits straight line as shown in Fig. 13. The values of Freundlich constants are reported in Table 1, and it is found that Freundlich isotherm is also obeyed since  $q_e(\text{exp.})$  values are close to  $q_e(\text{cal.})$ . In the same context, the higher correlation coefficient ( $R^2$ ) for Cu(II) adsorption by WS indicates that the system obeys both Langmuir and Freundlich models, but Langmuir model is a better fit.

**Fig. 12** Langmuir isotherm for Cu(II) removal by WS



**Fig. 13** Freundlich isotherm for Cu(II) removal by WS



**Table 1** Isotherms constant for Cu(II) removal by WS

Langmuir coefficients			Freundlich coefficients		
q max. (mg/g)	b (l/mg)	R <sup>2</sup>	K (mg/g)	n	R <sup>2</sup>
9.54	0.2	0.981	3.16	1.3	0.975

From Table 1, the  $n$  value of Cu(II) is noticed to be greater than 1, so denoting that adsorption is appropriate to eliminate Cu(II) by watermelon shell.

### 3.10 Differentiation of Adsorption Capability

Adsorption capacities of WS, LP, and BP for Cu(II) elimination are calculated from Eq. 1 and compared as shown in

Table 2. Such comparison of various adsorbents is to provide useful data for exhibiting the performance of different adsorbents. Table 2 indicates that the adsorption capacity of watermelon shell, lemon peel, and banana peel is better than that of common agriculture waste, such as orange peel, sawdust, tea fungal biomass, soya waste, *Saccharomyces cerevisiae*, and *Datura innoxia*. It was also observed that watermelon shell has the highest adsorption capacity of 9.54 mg/g. At the same time, as an agricultural waste, watermelon shell can be simply found and accumulated in great amounts in Iraq, which could offer formidable features in comparison with apple waste and tree fern in economical conditions. As well as, employing WS as an adsorbent might not request a complex alteration, appointing that this adsorption process is cost-effective and user-friendly for treating polluted water from Iraqi underground wells.



**Table 2** Comparisons of adsorption capacity of various adsorbents for Cu(II) removal

References	Adsorbent	Adsorption capacity (mg/g)
Yu et al. [21]	Sawdust	1.79
Annadurai et al. [22]	Orange peel	3.65
Razmovski and Šćiban [23]	Tea fungal biomass	4.65
Ho [24]	Tree fern	11.7
Lee and Yang [25]	Apple waste	10.8
Khan et al. [26]	Sago waste	2.5
Huang et al. [27]	<i>Saccharomyces cerevisiae</i>	1.9
Lujan et al. [28]	<i>Datura innoxia</i>	7.20
Present study	Watermelon shell	9.54
Present study	Lemon peel	8.24
Present study	Banana peel	7.65

## 4 Conclusions

The present work clearly suggests that using fruit cortexes as adsorbent is much economic, effectual, and more viable. The current work shows that cortexes of WS, LP, and BP can be efficiently used to remove Cu(II) from wastewater solution without even posing any threat to the water quality. The different operation parameters observed in batch experiments during the process of investigations reveal that the pH of the solution, dosage, particle size, initial copper ion concentrations, and contact times govern the overall process of adsorption. The potential of watermelon shell for adsorbing copper ions is indicated through its physical and chemical characterization, showing chemical structures (such as COOH, carboxyl, N–H, among others) of this material as well as morphological aspects. The experiment at the equilibrium fits well the Langmuir isotherm model with maximum adsorption capacity of 9.54 mg/g for watermelon shell. Reloading metal solution over the test column results in perfection on metal retention (removal percentage increases by 16, 22, and 27 % for WS, LP, and BP, respectively). Hence, watermelon shell has proved to be an effective and alternative adsorbent for the removal of copper ions from polluted water because of its considerably high adsorption capacity, being naturally renewable and thus cost-effective adsorbent for treating of underground well water in Iraq.

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