

Removal of Cu (II) from industrial wastewaters through locally-produced adsorbent prepared from orange peel

Zainab A. Naser*, Hayder M. Abdul-Hameed

Department of Environmental Engineering, Collage of Engineering, University of Baghdad, Baghdad, Iraq

* Corresponding author's E-mail: zainabrazak6@gmail.com

ABSTRACT

Heavy metal ion contamination is a commonality in industries such as mining, electroplating, and metal processing. By increased discharge of copper contaminated water, toxic waste due to Cu (II) has become a severe ecological dilemma globally. Adsorption is considered one of the more practicable techniques suggested for heavy metal removal because of its sensitivity, ease of operation and reproducibility alongside economic benefits such as effectiveness. This study examined the adsorption characteristics of an adsorbent prepared from orange peel (OP) for copper (II) in solutions that were aqueous. In evaluating the useful benefit of the sorbent material, changes in solution pH, initial Cu (II) concentration, adsorbent dose, and contact time on the adsorption process were also examined. Additionally, adsorption equilibrium isotherm as well as adsorption kinetics were explored to understand adsorption processes.

Keywords: Agricultural wastes, Wastewater, Heavy metals, Adsorbent, Orange peel.

Article type: Research Article.

INTRODUCTION

Increased wastewater discharge from industries that is polluted with hazardous heavy metals as a result of rapid industrialization is a worldwide environmental matter (Razak *et al.* 2020; Rushdi *et al.* 2020). Toxic heavy metal ion contamination of water is commonly seen in mining, electroplating, and metal processing industries, as well as textile and battery manufacturing as heavy metals, such as copper, are released into water streams (Amarasinghe & Williams 2007; Al-Sharif *et al.* 2018). Unintended absorption of excessive Cu (II) through copper-contaminated water presents a multitude of health challenges in humans causing death in extreme cases or vomiting, nausea, diarrhea and epigastric pain in others. Technologies currently used for the elimination of Cu (II) from wastewaters are various and include conventional methods such as chemical precipitation (Hutchison *et al.* 2008; Al-Qaisi *et al.* 2018), adsorption (Skinner *et al.* 2007), biological treatment, and membrane filtration (Urgun-Demirtas *et al.* 2012; Al Jaaf *et al.* 2020). Adsorption is considered one of the more practicable techniques because of its reproducibility, ease of operation, and sensitivity alongside economic benefits such as effectiveness and economy. Comparatively, chemical precipitation necessitates a separation procedure, biological methods are either inefficient or too time-consuming to treat because of the heavy metals and microbial activity inhibition. Membrane filtration is also expensive. Underutilized resources such as natural materials in huge quantities or agricultural waste can potentially be employed as inexpensive adsorbents because they are available widely and are naturally beneficial (Deans & Dixon 1992). Several previous studies have examined the use of inexpensive natural adsorbents such as waste by-products, agricultural products and carbonaceous substances (Nguyen & Do 2001; Singh *et al.* 2005; Abdel-Ghani *et al.* 2007; Lahieb *et al.* 2020; Almhana *et al.* 2020; Gadhban *et al.* 2020) as possible alternatives to traditional techniques like precipitation. However, using raw wastes from plant as adsorbents can cause various issues and are becoming a growing concern worldwide (Gallouze *et al.* 2021). Increased COD, BOD and TOC levels from soluble organic components and their release often found in materials from plants such as cellulose, pectin, tannin and lignin (Nakajima & Sakaguchi 1990; Misran *et al.* 2021) can

deplete oxygen levels in the water, endangering aquatic life. So far, remedies such as treatments with chemicals of solid adsorbents have been utilized as an approach for enhancing their chemical as well as physical characteristics alongside boosting their capacity for adsorption to solve such difficulties (Reddy *et al.* 1997; Feng *et al.* 2009; Mohammadi & Fataei 2019; Rzaij *et al.* 2020; Olasehinde *et al.* 2020; Amer Alshamri *et al.* 2021; Aljeboori *et al.* 2021; Abdouni *et al.* 2021). Orange peel (OP) is an inexpensive, readily and abundantly available agricultural byproduct of the orange juicing business. OP's chemical makeup consists of limonene, chlorophyll pigments, lignin, pectin, hemicellulose and cellulose. Numerous studies have examined the use of orange waste in the preparation of an adsorbent as a precursor material by chemical treatment (Khormaei *et al.* 2007; Biswas *et al.* 2007; Lahieb *et al.* 2020). This paper discusses and examines the results of an experimental work done on preparing an adsorbent from OP while elaborating on its adsorption characteristics for copper (II) in aqueous solutions. In evaluating the benefits of the sorbent material, changes in pH of solution, initial Cu (II) concentrations, adsorbent doses as well as contact times on the process of adsorption were also explored.

MATERIALS AND METHODS

2.1 Adsorbent preparation

All the compounds utilized in this study were analytically pure. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ was used to make a stock solution of Cu (II) in 0.5 g L^{-1} concentration, which was subsequently diluted to the required concentrations for each test. The solution pH was modified with 0.1 mol L^{-1} NaOH and 0.1 mol L^{-1} HCl. OP waste was gathered from a local juice market, and then rinsed with distilled water. After drying the OP at room temperature, it was pulverized and crushed. Later, the OP waste was carbonized at $250 \text{ }^\circ\text{C}$ for 1 hour at 0.5 L min^{-1} using carbon dioxide gas. After reaching the ultimate temperature, the gas flow was changed to nitrogen, and the activation temperature ($350 \text{ }^\circ\text{C}$) was altered by 0.4 L min^{-1} for 1 hour (abbreviated as OPAC). Physical and material qualities for OP and OPAC were estimated in the labs (Petroleum R&D Center) and are reported in Table 1 based on information from the provider.

Table 1. Physical and concoction successes for orange peel.

Properties	OP	OPAC
Experimental area, $\text{m}^2 \text{ g}^{-1}$	0.3974	2.0909
Bulk density (g cm^{-3})	0.3215	0.2359
Appearance density (g cm^{-3})	0.2851	0.2097
real density (g cm^{-3})	1.5888	1.4494

2.2 Adsorption isotherm and kinetics order

In the understanding of an adsorption process, adsorption equilibrium is the key. For designing an efficient adsorption system, a good understanding of adsorption isotherms is critical (Lin & Wang 2009). Langmuir and Freundlich isotherms are two-parameter ones. The Langmuir adsorption equation, first developed to portray the adsorption of a gas-solid phase, is utilized to compare as well as measure different adsorptive capacities (Elmorsi 2011). To account for surface coverages, the Langmuir isotherm balances the relative adsorption and desorption rates. The Langmuir equation can be stated linearly as follows (Dąbrowski 2001):

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

where the quantity of adsorbed adsorbate per unit mass of activated carbon is denoted by q_e (mg g^{-1}) while the adsorbate's equilibrium concentration is abbreviated as C_e (mg L^{-1}) and q_e is the quantity of adsorbate adsorbed per unit mass of activated carbon (mg g^{-1}). The Langmuir constant and maximum uptake of Cu^{+2} per unit mass of adsorbent b (mg g^{-1}), and the Langmuir constant are both linked to the adsorption rate K_L (L mg^{-1}) and are derived using (C_e/q_e) vs C_e plots. The equilibrium parameter RL given by equation 2 is one more feature of the Langmuir isotherm, defining the type of adsorption process that can be either irreversible ($RL = 0$), favorable ($0 < RL < 1$), linear ($RL = 1$) or unfavorable ($RL > 1$).

$$R = 1 / (1 + b C_0) \quad (2)$$

The Freundlich isotherm, that is related to the process of adsorption which occurs on surfaces that are heterogenous (Gallouze *et al.* 2021), is an isotherm that describes heterogeneity on a surface and the exponential

distribution of active sites with their energies (Ayawei *et al.* 2015). The Freundlich isotherm in its non-linear form is as follow (Fost & Aly 1981; Ruthven 1984):

$$q_e = K_f [C_e]^{1/n} \quad (3)$$

where Freundlich constants K_f and n are found from the intercept and slope of the $\ln q_e$ vs $\ln C_{eq}$ plots, with K_f and n suggesting the adsorption capacity of the adsorbent and the favorableness ($n > 1$) of the adsorption process, respectively.

RESULTS AND DISCUSSION

3.1. Adsorbent characterization:

To determine the functional groups responsible for the adsorption process, shown in Fig. 1a-b, FTIR spectra of OP in the region of 800–3800 cm^{-1} was conducted. When compared to the activated orange peel, the spectrum pattern of OP showed variations in peak absorption between OP and OPAC. The OP FTIR spectrum helps identify the major bands for aliphatic structures (2938 cm^{-1}), lignin-like compounds (1435.79, 1100.73, and 1013.71 cm^{-1}), and polysaccharide markers (2938.72 and 581.93 cm^{-1} ; Abu-Ilaiwi *et al.* 2004). It is perceived that $\text{C}=\text{O}$ stretching in carboxylic acids ($-\text{COOH}$) and/or alkyl esters ($-\text{COOR}$) is the reason for the 1814.48 cm^{-1} peak. As discussed in literature (Zaccheo *et al.* 2002), this is typical of pectin. The peak at 1435.79 cm^{-1} is characteristic of lignin. Such outcomes seem to be in agreement with the existence of pectin, lignin and cellulose in OP. Moreover, it is determined that aliphatic structures CH_2 and their $\text{C}-\text{H}$ stretching vibrations result in the peak identified at 2938.72 cm^{-1} . On the other hand, carboxylate (COO^-) and its $\text{C}=\text{C}$ and / or asymmetric $\text{C}=\text{O}$ stretching results in the peak at 1614.48 cm^{-1} (Zaccheo *et al.* 2002). Lastly, it appears that symmetric $\text{C}-\text{O}$ stretching of COO^- could lead to the peak at 1435 cm^{-1} . A wide band is visible at 1049.13 cm^{-1} . This could be due to polysaccharide structures and their $\text{O}-\text{H}$ deformation along with $\text{C}-\text{O}$ stretching (Deepashree *et al.* 2012). In this broad band, some minor peaks can be discerned, though they are not well-defined. These minor peaks, together with the (631.34-591.93) cm^{-1} peak attributed to out of phase ring stretching, are typical of cellulose. The major discrepancies between the OP and OPAC spectra were noted in around 2942.65, 2026.65 and 873.57 cm^{-1} . Scanning electron microscopy (SEM) characterization for OP before and after carbonization and activation were examined. Significant adjustments in the morphology of the biosorbents surfaces were exposed. Surface morphologies of OP and OPAC are not comparable as OP surface is less uneven as compared to the activated carbonated OP. This indicates the chemical binding of AC which means that it readily attaches itself to the OP surface (Figs. 1 and 2).

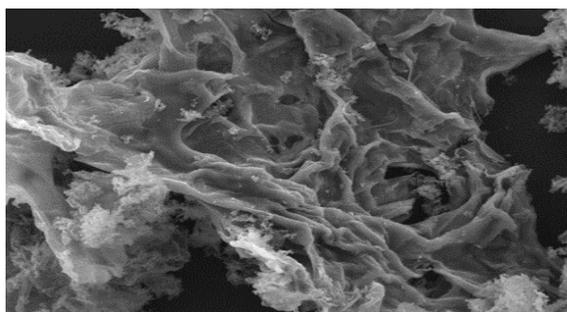


Fig. 1. SEM of orange peel before activation.

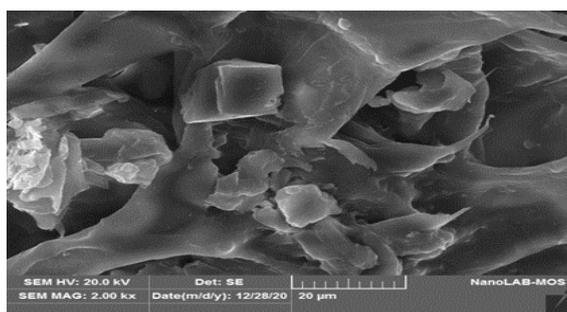


Fig. 2. SEM of orange peel after activation.

3.2 Effects of pH

The reaction mixture's initial pH affects the efficiency of metal ion removal (Abia *et al.* 2003). The effects of initial pH were evaluated using pH values of 4, 6.5, and 11 for the removal of copper (II) from a solution of 75 mg L⁻¹. For cations sorption, the increased pH brought about an elevated copper (II) uptake. The optimum removal rate of 99.6% was obtained for a pH value of 6.5, thereafter, the removal efficiency declined as a result of the competing OH⁻ and copper (II) ions.

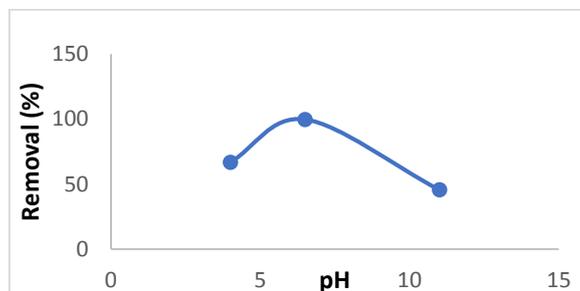


Fig. 3. Effect of pH on copper (II) removal, dose = 0.5 g L⁻¹, time = 180 min, Cu = 1 mg L⁻¹.

3.3 Effects of adsorbent dose

Effects of adsorbent doses on the removal procedure were examined using varying values of OPAC (0.5, 1, 1.5 and 2 g L⁻¹). It turns out that an elevation in the adsorbent dose, upraises the amount of copper removed. This elevation can be linked to the large amount of active sites for OPAC that are available as dosage is increased. The best removal value was obtained when the dose value for OPAC was 1 g L⁻¹. Additional dosage did not lead to improvement which can be attributed to the overlapping of particles.

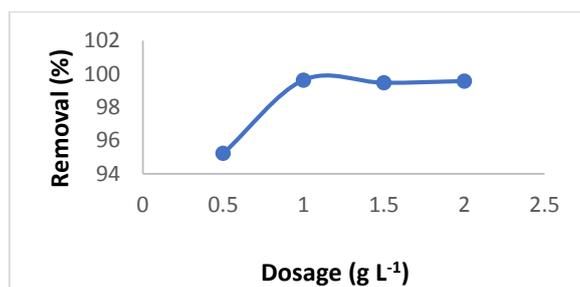


Fig. 4. Effects of doses on copper (II) removal, pH = 6.5, time = 180 min, Cu = 1 mg L⁻¹.

3.4 Effect of Agitation Speed (RPM)

Agitation speed has an important effect on pollutant uptake. To study the effect of RPM, several values were tested (100, 150, 200, 250, and 300). The removal percent was found to increase as agitation speed was raised up to 200 RPM, reaching a removal rate of 99.63% at its peak, afterward it started to decline (Fig. 5). The rate of shaking used must be suitable to stir all the available binding sites for metal uptake. In low speed the adsorbent accumulates at bottom. However, higher speeds lower the removal of metal as sufficient time is not allowed for the metal ions to be adsorbed (Anwar *et al.* 2010). A rise in the agitation speed (RPM), increases the molecular diffusion rate from the bulk liquid to the liquid boundary layer containing the OPAC particles. This is due to an increased turbulence as well as a reduction in the liquid boundary layer thickness (Kuśmirek & Wia,tkowski 2015).

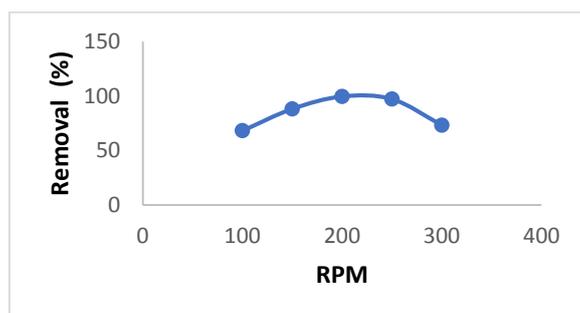


Fig. 5. Effect of RPM on copper (II) removal, pH = 6.5, dose = 0.5 g L⁻¹, time = 180min, Cu = 1 mg L⁻¹.

3.5 Effect of contact time

Contact duration was found to be critical in influencing adsorption efficiency. Uptake of metal ions grew from 71.81% to 99.48% as contact time increased from 15 to 90 min. The best contact time is defined as the time that gives the best removal, and not the highest removal in the shortest time. The removal percent after 60 min becomes nearly constant due to occupation of all binding groups by metal ions, so this point is considered as equilibrium time (Hamzezadeh *et al.* 2020).

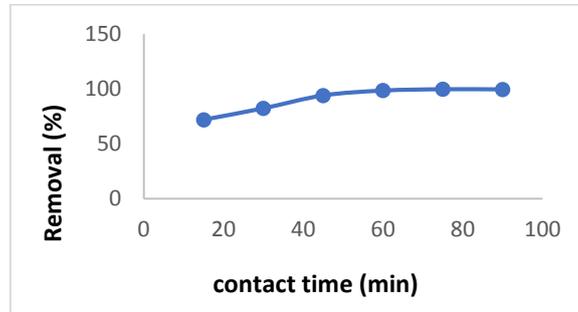


Fig. 6. Effects of contact time on copper (II) removal, pH = 6.5, dose = 0.5 g L⁻¹, Cu = 1 mg L⁻¹.

3.6 Effects of initial concentration

Fig. 7 illustrates the effects of initial metal concentration on efficiency of adsorption. Tests for adsorption took place in a mixed metal ion solution at various initial Cu²⁺ concentrations. These were in the 0.5 to 2 mg L⁻¹ range. An elevated metal ion removal (%) took place with a reduction in the initial ion concentration. This can be linked to the rise in the percentage of surface-active sites to the total metal ions existing in the solution as ion concentrations are reduced. As a result, the adsorbent may have an interaction with all metal ions which might lead to the removal of these ions from the mixture. The adsorbent's per unit weight can determine the quantity of metal adsorbent (Amarasinghe & Williams 2007).

3.7 Adsorption isotherm experiments

Experiments for equilibrium isotherm took place through the mix up of 0.5 g OPAC with 50 mL metal ion solution for 3 hours. Different initial metal concentrations (0.5, 1, 1.5, and 2 mg L⁻¹) were tested. The Langmuir model has a better fit for Cu⁺² adsorption on OPAC (R₂ > 0.964) than Freundlich one. This suggests that the process of adsorption is a monolayer adsorption. The equilibrium parameter RL suggests that the nature of adsorption is favorable given 0 < RL < 1 (Mohammed *et al.* 2021).

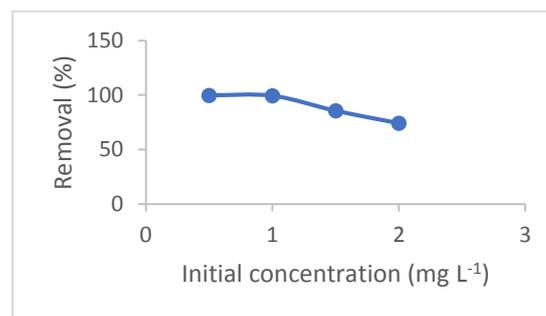


Fig. 7. Effects of initial concentration on removal of copper (II), dose = 0.5 g L⁻¹, time = 180 min, pH = 6.5.

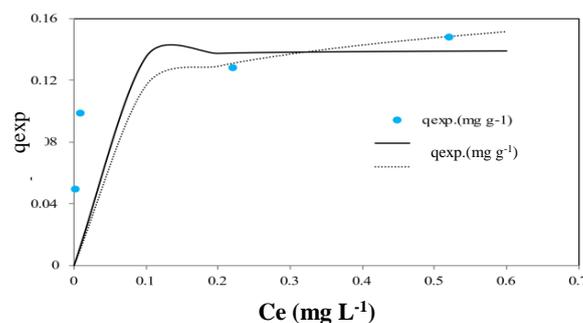


Fig. 8. Adsorption isotherms for Cu onto OP waste at 25 °C.

Table 2. Freundlich and Langmuir isotherm constants for Cu onto OP waste at 25 °C.

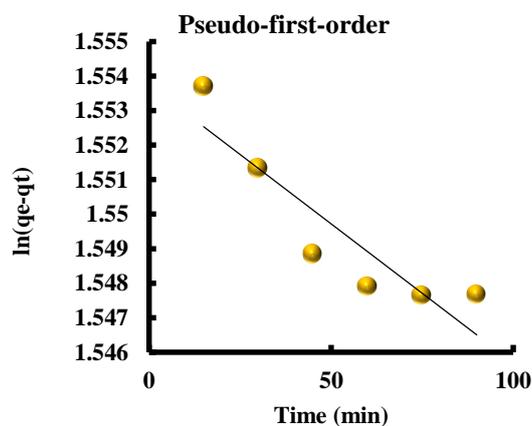
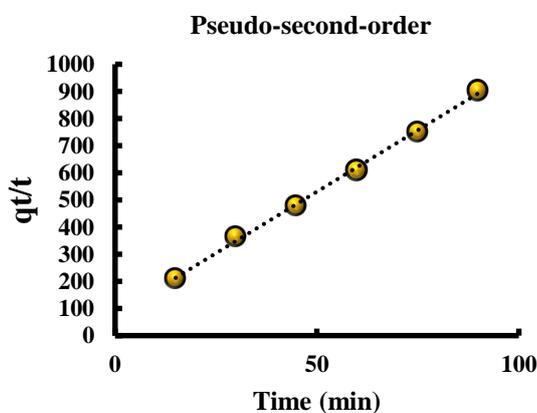
Freundlich			Langmuir		
k_f	1/n	R	q	b	R
0.163	6.87	0.895	0.1397	297.2	0.964

3.8 Adsorption kinetics

Studying adsorption kinetics is substantial as it offers great importance to the reaction pathway and the appropriate adsorption mechanism. There are several kinetic models: first, second, pseudo first, and pseudo-second order models. The parameters of each individual kinetic model is illustrated in Table 3 where K_1 is the constant of first order kinetics (min^{-1}), K_2 is the constant of second order kinetics [$(\text{mg L}^{-1})^{-1} \text{min}^{-1}$], and R^2 is the correlation coefficient. According to the regression correlation coefficients (R^2), Cu^{+2} adsorption following the 2nd order kinetic model, that is linked to the assumption of chemisorption, is the rate-limiting step (Snyder *et al.* 2003; Lin & Wang 2009).

Table 3. Kinetic parameters to remove the Cu^{+2} in optimum conditions.

Model	Equation	Parameters		
		C_0 (mg L^{-1})	K	R^2
First-order kinetic	$C(t) = C_0 e^{-K_1 t}$	1	0.0634	0.9651
Second-order kinetic	$\frac{1}{C(t)} = \frac{1}{C_0} + K_2 t$	1	2.6527	0.7218
Pseudo-first-order	$\ln(q_e - qt) = q_e - K_1 t$	1	0.00018	0.639
Pseudo-second-order	$\frac{t}{qt} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e}$	1	1.063	0.99825

**Fig. 9a.** Experimental data and the fitted linear equations of pseudo 1st order for Cu^{+2} adsorption on OPAC.**Fig. 9b.** Experimental data and the fitted linear equations of pseudo 2nd order for Cu^{+2} adsorption on OPAC.

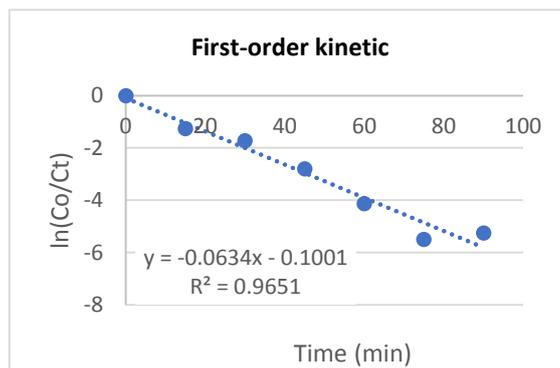


Fig. 9c. Experimental data and the fitted linear equations of 1st order kinetic for Cu⁺² adsorption on OPAC.

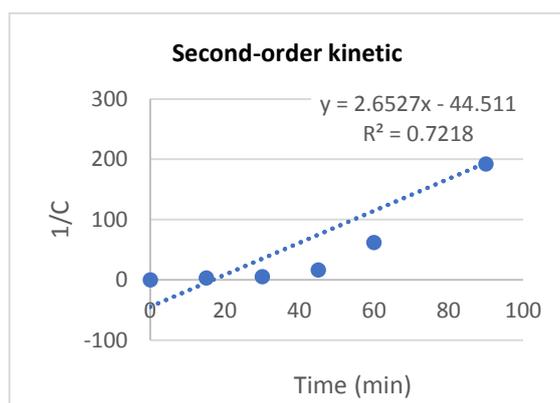


Fig. 9d. Experimental data and the fitted linear equations of 2nd order kinetics for Cu⁺² adsorption on OPAC.

CONCLUSION

OP and chemically modified OP are both effective copper (II) biosorbents as compared to other bio-sorbents discussed in earlier studies. This work's primary conclusions are as follows:

The uptake of copper (II) increased with a rise in the solution pH. Optimum removal rate of 99.6% was obtained when pH value was 6.5.

The best removal value is obtained when the adsorbent dose concentration is 1g L⁻¹.

Metal ion removal percent elevates as agitation speed is upraised. Removal rate peaks at 99.63% at 200 RPM, afterward the removal rate starts to decline.

Metal ions uptake grows from 71.81% to 99.48% as contact time increases from 15 to 90 min. The best contact time was 60 min.

Adsorption tests were performed at varying initial Cu²⁺ concentrations in the ranges of 0.5 to 2 mg L⁻¹. The ratio of ion removal elevates by a decreased initial ion concentration. The highest removal rate achieved was 99.63% at 1 mg L⁻¹.

Linked to the assumption of chemisorption, the 2nd order model is the rate limiting step. The Langmuir model has a better fit for Cu⁺² adsorption on OPAC ($R^2 > 0.964$) than the Freundlich one. This suggests that adsorption processes are monolayer adsorption ones. The equilibrium parameter RL suggests that the nature of adsorption is favorable given $0 < RL < 1$.

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Bibliographic information of this paper for citing:

Naser, ZA, Abdul-Hameed, HM 2022, Removal of Cu (II) from industrial wastewaters through locally-produced adsorbent prepared from orange peel. *Caspian Journal of Environmental Sciences*, 20: 45-53.