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USAGE OF WASTE ACTIVE SLUDGE OBTAINED FROM A MEMBRANE BIOREACTOR SYSTEM AS A LOW-COST BIOSORBENT FOR REMOVAL OF CU (II) IONS

Ülküye Dudu GÜL[1]*, Cağlayan AÇIKGÖZ[2], Hülya SİLAH[3]

[1]Vocational School of Health Services, Biotechnology Application and Research Center, Bilecik Seyh Edebali University, 11210, Bilecik, Turkey
[2]Faculty of Engineering, Department of Chemical &Process Engineering Bilecik Seyh Edebali University, Turkey
[3]Department of Chemistry, Faculty of Art & Science, Bilecik Seyh Edebali University, 11210 Bilecik, Turkey

*Corresponding author
E-mail: ulkuyedudugul@gmail.com

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Abstract

This study aims to suggest an inexpensive adsorbent. It is also planned to reuse the waste sludge of the membrane bioreactor (MBR) system. For this reason, copper biosorption capacity of waste sludge compost of fungal biomass from an MBR unit was investigated. The effects of pH (2, 4, 6, and 8), contact time (1- 24h), and Cu (II) concentration (9.74 to 98.21 mg/L) were tested at batch scale level experiments. To describe the equilibrium isotherms, the Langmuir and Freundlich models were used. Also, kinetic properties were determined as pseudo-first-order and pseudo-second-order models. To determine the functional groups of adsorbent, Fourier Transform Infrared Spectrophotometer (FTIR) analysis was done. The maximum Cu (II) removal was 89.65 % at pH 4. The maximum Cu (II) uptake capacity of adsorbent was found as 14.20 mg/g. The results of this study showed that the dried active sludge, a waste of MBR system, was an efficient and low-cost adsorbent for removal of Cu (II) from industrial effluents.

Key words: adsorption, Cu (II) removal, waste active sludge, wastewater treatment

INTRODUCTION

Heavy metal pollution represents an important problem for the aqueous environment. In addition to this, heavy metals have toxic effects on living organisms [1]. Most of the industrial activities like metal plating and mining consume large volumes of heavy metal contaminated water and these heavy metals such as copper Cu(II) are hazardous. For instance, Cu(II) does not degrade easily in the environment and can be harmful to human health. The levels of Cu(II) ions above 1.3 mg/L cause stomach and intestinal problems in humans. Unfortunately, waste streams from industries contaminated with Cu(II) may contain up to 500 mg/L Cu(II) [2]. So the effluents containing heavy metals like Cu(II) need to be treated before discharging to the aqueous environment. The removal of heavy metals is very important due to their destructive effects on the environment and human health. A variety of conventional methods such as chemical precipitation, ion exchange, filtration, solvent extraction, reverse osmosis, and membrane technologies are recommended to remove heavy metals from aqueous solutions [3]. While the concentration of heavy metals is ranged from 1 to 100 mg/L, the conventional methods are not sufficient for removal of the heavy metals. Also, these methods have some disadvantages such as producing toxic chemical sludge and not being eco-friendly. Adsorption is reported as an effective technology for the removal of pollutants like heavy metals from aqueous solutions [3, 4]. The advantage of adsorption is described as effective in removing heavy metals via the utilization of cheap adsorbent [5]. Most of the researches is still focusing on searching cheap and effective adsorbents for the removal of heavy metals [6, 7]. A variety of adsorbents such as microorganisms, plant by-products, and waste materials have been used to remove heavy metals from aqueous medium [8, 9]. Fungal biosorbents have been proved efficient and economical for the removal of metal ions from aqueous solutions [10, 11]. The structure of the fungal cell wall has excellent metal-binding properties. However, there isn't enough information about the usage of waste fungal biomass for the removal of heavy metal. The membrane bioreactor (MBR) systems present effective pollutant removal and excellent effluent quality for the treatment of industrial wastewater [12, 13]. Although active sludge technology is widely used all over the world, the waste sludge of a bioreactor unit is considered as an environmental problem because of causing solid waste pollution. For environmental and ecological reasons, the disposal of this sludge has become immensely important. Samples of active sludge were collected from the MBR system as solid waste and reused as an adsorbent in this study. The aim of this work is to reuse the waste sludge compost of fungal biomass obtained from the MBR and to examine the potential usage of this dried waste as a low-cost adsorbent. This study is intended to demonstrate the technical feasibility of simple and low-cost procedure to remove toxic heavy metals such as copper from aqueous solutions. To our knowledge, this is the first report correlating with copper removal by using waste sludge of an MBR.

MATERIALS AND METHODS

Preparation of Heavy Metal Solution

The stock Cu (II) solution was prepared by dilution of $CuSO_4$ (Merck) having a final concentration of 1 g/L of Cu (II). Appropriate volumes of the stock solutions were added into the aqueous solutions in the experimental series.

Preparation of Adsorbent

Fungal strains Aspergillus versicolor and Rhizopus arrhizus were obtained from Ankara University Biology Department Biotechnology Laboratory Culture Collection. The mixed cultures were inoculated into the MBR System in order to decolorize the textile dyes in another study. The active sludge (containing Aspergillus versicolor and Rhizopus arrhizus), used in this study, was used for the decolorization of simulated textile wastewater in MBR in our previous study [14]. After decolorization process, the active waste sludge containing R. arrhizus and A. versicolor was drained from the bioreactor system, and washed with distilled water, then dried at 80 ºC. The dried waste sludge was smashed and, a known amount of powdered waste sludge was used as an adsorbent for adsorption studies. The dried adsorbent was sieved by using an 80 µm sieve. All experiment series performed with the solutions contained 0.5 g/L of the adsorbent.

Adsorption Studies

Adsorption studies were carried out by the batch technique in 250 mL flasks containing 200 mL of Cu(II) including synthetic solutions at the desired level of each component at the beginning of the adsorption. The flasks were continuously agitated on a shaker at a constant shaking rate of 1.118 × 10 g for 24 hours to ensure that equilibrium was reached. To determine the contact time on adsorption, the waste fungal biomass (Dry weight: 0.5 g/L) was added into flasks contained 200 mL distilled water with 49.40 mg/L Cu(II) at pH 2 for 0 to 1440 minutes. The effect of pH was determined in flasks contained distilled water with 49.40 mg/L Cu(II) at pH 2, 4, 6, and 8. To examine the effect of Cu(II) concentrations on adsorption the adsorbent was added into flasks contained distilled water with 9.74, 45.96, 76.49, and 98.21 mg/L Cu(II). The 200 mL distilled water flask containing only Cu(II) without adsorbent was used as a control. Samples (3 mL) were taken every 2 hours and centrifuged at 5.59 × 10-2 g for 5 min (Hettich EBA12 model centrifuge).

Analytical Methods

A 3 mL of sample was taken from each flask, every 2 hours and the concentration of Cu (II) in the supernatant was determined by atomic absorption spectrometry (GBC933AA). The 200 mL of distilled water flask containing only Cu(II) without adsorbent was used as a control. Experiments were conducted in triplicate and the results are the average of triplicate measurements. Finally, the removal percentage (Cu (II) Removal %) and uptake capacity (qm, mg/g) were calculated by Equations (1) and (2), respectively.

$$\%Cu(II)Removal = \frac{C_0 \quad C_f}{C_0} x100\% \qquad \text{Eq. (1)}$$

$$q_m = \frac{(C_0 - C_f)V}{W} \qquad \text{Eq. (2)}$$

In this equation; C0 (mg/L): initial concentration of Cu (II) ion, Cf (mg/L): the residual concentration of Cu (II) ion, V (L): the solution volume, W (g): the dosage of biosorbent

Statistical analysis

The experimental analyzes were done triplicate. SPSS 17 was applied to analyze the data and ANOVA (one-way) was used to compare average values. Correlation coefficients were calculated by using the Microsoft Excel package.

Fourier Transform Infrared Spectrophotometer (FTIR) Analysis

The functional groups on the surface of waste active sludge in the absence and presence of Cu (II) were identified by using the FTIR technique. FTIR spectra were recorded by the Perkin Elmer (Spectrum 100) spectrophotometer in the region of 650- 4000 cm^{-1}. Dry powdered samples of waste fungal biomass un-loaded and loaded with Cu (II) ions were prepared and used in FTIR analysis.

RESULTS AND DISCUSSION

The Effect of Contact Time

The effect of treatment time on the concentration of Cu (II) in aqueous solution is given in Fig. 1. The Cu (II) uptake process was reached the plateau values within 480 min. After 480 minutes, the Cu (II) concentration wasn't considerable change in the aqueous solution so it was considered as the optimum contact time.

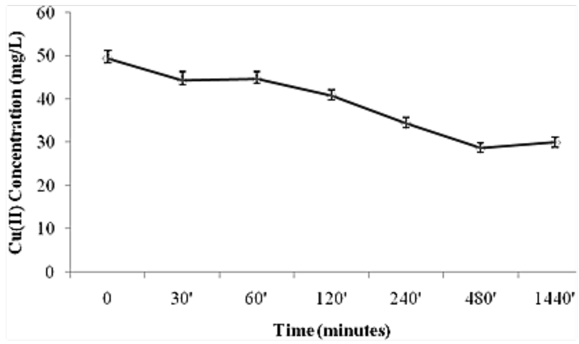

Figure 1. Effect of contact time on the concentration of Cu (II) in aqueous solution (Co:49.40 mg/L; pH:2; adsorbent dosage: 0.5 g/L; p= .05, df = 3). Error bars represent the standard error of the mean.

The Effect of pH

pH is an important factor affecting heavy metal adsorption. The pH value affects the biosorption process by both protonation or deprotonation of the functional groups on the adsorbent surface and the ionization potential of the heavy metal in solution [15]. Fig. 2 indicates the effect of initial solution pH on the removal of Cu (II) from aqueous solution at 49.40 mg/L initial Cu (II) concentration and 25 oC. The highest Cu (II) removal percentage was obtained at pH 4 (Fig. 2). Recently, Xie et al. (2017) showed that the functional groups on the adsorbent had an important role in copper uptake [16] . It was verified that the surface of waste active sludge (contained fungal biomass) had negatively charged sites like carboxylate groups having weaker but acidic groups dominantly. The Cu (II) ions were mainly adsorbed on these acid sites. At low pH, the surface of waste active sludge would also be surrounded by hydronium ions which decrease the Cu (II) interaction with binding sites of the dried waste active sludge by greater repulsive forces. As a result, the optimal pH value for Cu (II) biosorption was chosen as 4.0 and the continuous experiments were performed at this pH value. Gochev et al. (2012) recently showed that the dead biomass of Trametes versicolor removed Cu (II) ions maximally at pH 4 [17]. The results of pH experiments were fitted with the literature. The pH value 4 was selected for continuing experiments.

The Effect of Initial Cu(II) Concentration

The Cu (II) removal values at different initial metal ion concentrations are given in Figure 3. It was clear that the removal capacity of dried active sludge for Cu (II) decreased with increasing initial metal concentration. The removal rate of Cu (II) decreased from 89.65 to 6.22% with increasing initial Cu (II) concentration from 9.74 to 98.21 mg/L (Fig. 3). The augmentation of initial Cu (II) concentrations resulted in reducing the metal removal percentage. The initial Cu (II) concentration studies showed that a finite number of surface binding sites on the active sludge surface were saturated by higher Cu (II) concentrations.

At higher concentrations, the removal of metals is related to the initial metal concentration because available sites for metal biosorption become fewer. According to these results, the treatment efficiency can be increased by diluting the wastewater containing high metal ion concentrations in wastewater treatment systems. Recently, the researchers about heavy metal removal by low-cost adsorbents gain importance. The Cu (II) removal by inexpensive adsorbents such as olive mill solid residue, tree fern, wheat shell, and grape stalks wastes from aqueous solutions were investigated previously. Copper uptake capacities of olive mill solid residue, tree fern, wheat shell, and grape stalk wastes were shown as 13.5 [18], 10.6 [19], 10.8 [20] and 10.1 [21] mg/g, respectively. Recently, Abdel-Galil et al. (2016) showed that the maximum sorption capacity (qm) for the Leucaena waste biomass

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sorbent toward Cu (II) was 7.89 mg/g [22]. In this study, maximum Cu (II) uptake capacity of dried waste active sludge compost of fungal biomass obtained from MBR was calculated as 14.20 mg/g. The effect of initial Cu (II) concentration experiments showed that the waste active sludge used in this study performed the best copper uptake capacity while comparing other reported ones.

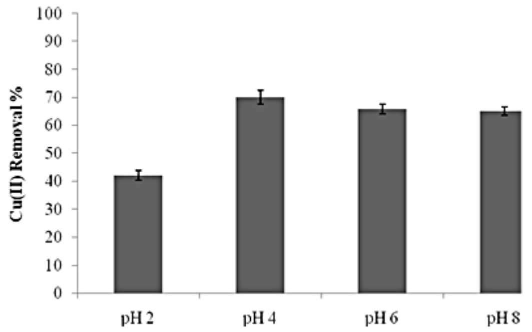

Figure 2. Effect of pH on the removal of Cu (II) by waste fungal biomass (Co: 49.40 mg/L; pH:2; contact time:1440; adsorbent dosage: 0.5 g/L; p = .05, df = 3). Error bars represent the standard error of the mean.

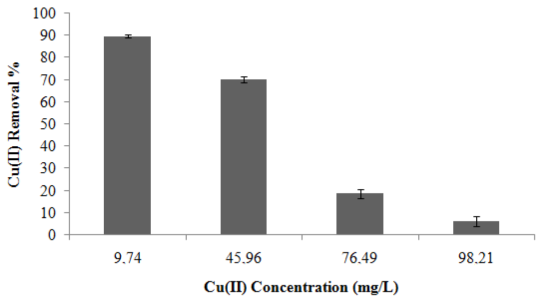

Figure 3. Effect of initial Cu (II) concentration on the removal of Cu (II) by waste fungal biomass (pH:2; contact time:1440; adsorbent dosage: 0.5 g/L; p = .05, df = 3). Error bars represent the standard error of the mean.

Biosorption Isotherms

The value of adsorption isotherms obtained for Cu (II) uptake was plotted using Langmuir and Freundlich equations. The widely used Langmuir isotherm assumes monolayer adsorption onto a solid surface [23] which is given as;

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m K_L} \qquad \text{Eq. (3)}$$

In this equation; q_e (mg/g): the amount of Cu (II) adsorbed per unit weight of adsorbent at equilibrium, q_m (mg/g): the maximum Cu (II) uptake per unit mass of adsorbent, K_L (L mg/g): the Langmuir constant related to the energy of sorption which quantitatively reflects the affinity between the adsorbent and adsorbate, Ce (mg/L): the equilibrium concentration of adsorbate.

The values of q_m and K_L were calculated from the slope and intercept of the graph using Eq.(3).

The Freundlich equation [24] is used for heterogeneous surface energy systems and given as;

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \qquad \text{Eq. (4)}$$

where K_F is the Freundlich isotherm constant (L/g) and 1/n is the heterogeneity factor. The values of n and K_F are calculated for waste fungal biomass from the intercept and slope of the Freundlich linear plot of log q_e versus log Ce, respectively.

The comparison of the R^2 values showed that the Langmuir model fits better with the experimental data than the Freundlich one (Fig. 4; Table 1) for waste active sludge. The Langmuir equation is used for homogeneous surfaces. Similarly, Subbaiah et al. (2011) reported that the Langmuir isotherm was fitted with the Cu (II) biosorption by dead Trametes versicolor biomass [25].

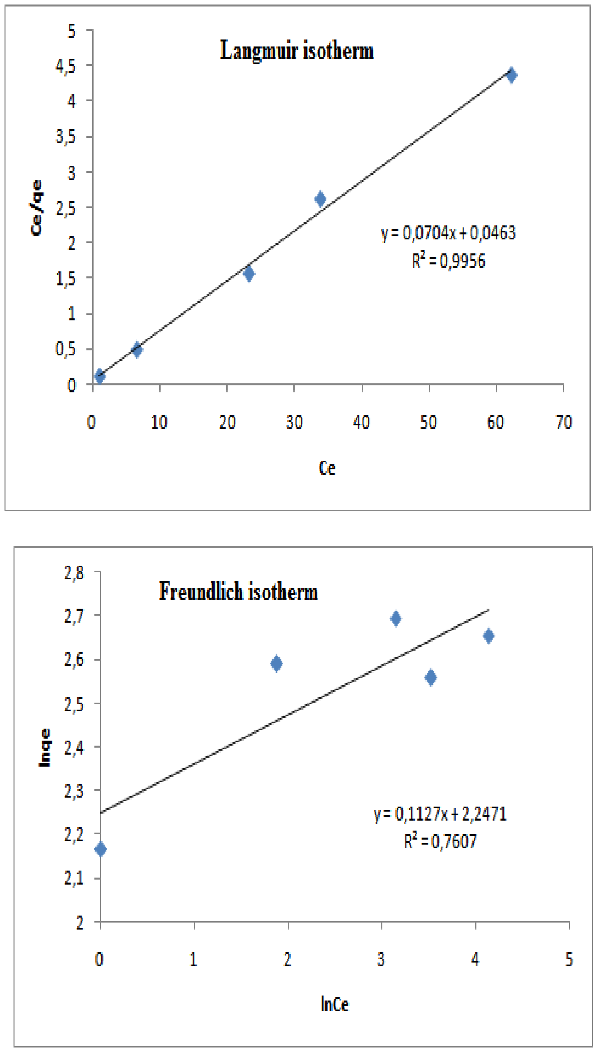

Figure 4. Adsorption isotherm graphics

Recently, Kan et al. (2015) showed that Cu (II) adsorption on mushroom biomass equilibrium is best described by the Langmuir model [26]. The adsorption on the waste active sludge contained fungal biomass was fitted with the Langmuir model in this study. The adsorption capacity, q_m, estimated from the Langmuir model for Cu (II) is 14.20 mg/g. When the adsorption capacity of the MBR is compared to the removal of Cu (II) ions with other biosorbents, it is seen that the MBR system is a good alternative method for copper removal. Liang et al. isolated an indigenous Cu-resistant bacteria strain from heavy metal-contaminated soil and used this bacterial strain as a biosorbent to remove Cu (II) ions from aqueous solution. They found that the maximum sorption capacity of this biosorbent was 12.6 mg/g [5].

Langmuir			Freundlich		
q_m (mg/g)	K_L (Lmg/g)	R^2	N	K_F (L/g)	R^2
14.20	1.52	0.9956	8.87	9.46	0.7607

Table 1 Langmuir and Freundlich Constants for Cu (II)

Biosorption Kinetics

The kinetic rates in the present study were modeled using pseudo-first and second-order models. The pseudo-first-order model was defined as follows;

$$\log(q_e - q_t) = \frac{-k_1}{2.303\,t} + \log q_e \qquad \text{Eq. (5)}$$

In this equation; q_t (mg/g): the amounts of Cu (II) adsorbed at time t, qe(mg/g): the amounts of Cu(II) adsorbed at equilibrium, k_1 (1/min): the rate constant of pseudo-first-order equation. The slope and intercept of the graph of log (q_e - q_t) versus t correspond to the value of constants k_1 and q_e respectively.

The pseudo-second-order model can be expressed by the following equation;

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad \text{Eq. (6)}$$

In this equation, k_2 is the rate constant of the pseudo-second-order equation.

The applicability of two kinetic models; pseudo-first order model and pseudo-second-order model were used to investigate the kinetics of Cu (II) adsorption onto waste active sludge compost of fungal biomass. The graphics for adsorption kinetics are given in Fig. 5.

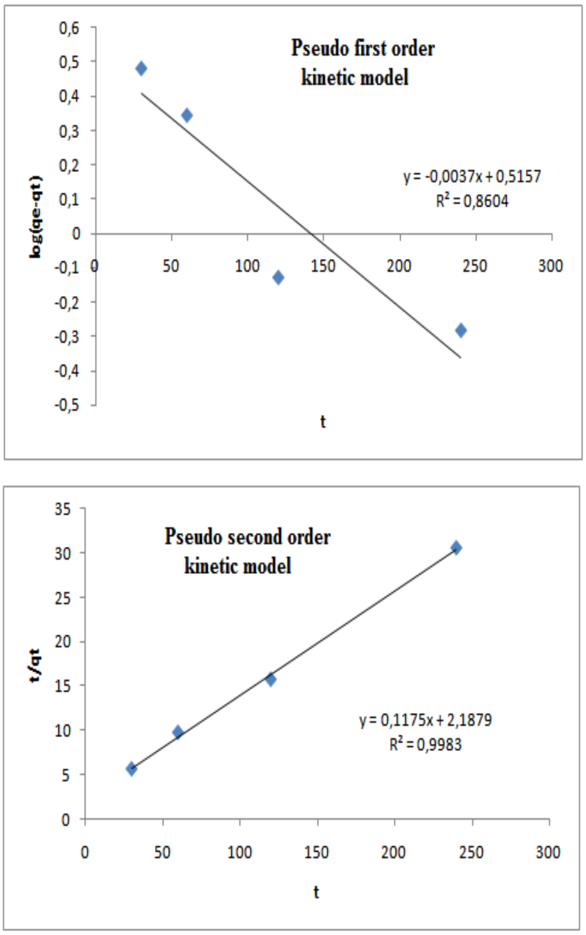

Figure 5. Adsorption kinetic graphics

As shown in Table 2, the experimental qeexp value did not agree with the calculated qecal from the pseudo-first-order model for waste fungal biomass. The qecal value of the pseudo-second-order model was near with the qeexp value and the correlation constant of the pseudo-second-order model was higher than the pseudo-first-order model. According to these results, the biosorption of Cu (II) by the waste active sludge was compatible with the pseudo-second-order model. This result was similar to the results of the study of Subbaiah et al. (2011) showing the Cu (II) biosorption properties of T. versicolor [25].

Table 2 Kinetic Parameters for The Adsorption of Cu (II) onto Waste Fungal Biomass

Pseudo-first-order model				Pseudo-second-order model			
qe_{cal}	qe_{exp}	k_1	R^2	qe_{cal}	qe_{exp}	k_2	R^2
3.278	8.37	8.52×10^{-3}	0.8604	8.51	8.37	6.52×10^{-3}	0.9983

FTIR Analysis

To explain the adsorption mechanism; it is important to identify the functional groups of adsorbents that are responsible for the biosorption process of metal ions. FTIR spectroscopy gives essential information related to the changes of fungal biomass surface before and after the adsorption process. FTIR spectrums of nature and copper loaded waste active sludge are given in Fig. 6. The wideband approximately between 3500-3000 cm^{-1} presents to bonds of -OH and -NH groups. As seen in Fig. 6, these bands were shown at 3274 cm-1 before adsorption but after biosorption, it was shifted to 3280 cm^{-1}. Also, the band at 1538 cm^{-1}, was related to amide bond, was shifted to 1529 cm^{-1} after adsorption and the intensity of this band was increased. As seen in Fig. 6, the bands were shown at 1016.5 cm^{-1} before adsorption but after biosorption, this band was shifted to 1021 cm-^{1} and the intensity of the peak was increased. Meanwhile, the change in wavenumber of functional groups and absorption intensity is related to the interaction of copper ions with active sites of waste active sludge containing fungal biomass. It is considered that the bonds between copper and active sites of waste active sludge are formed due to electrostatic interaction mechanisms [27, 28].

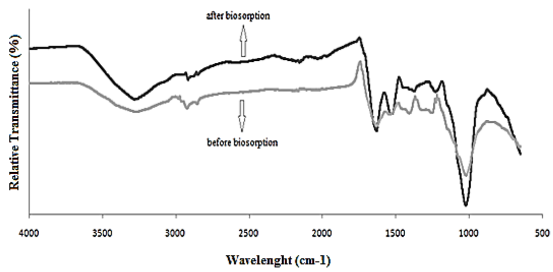

Figure 6. FTIR spectra of waste fungal biomass before and after biosorption

CONCLUSIONS

Recent studies focus on the requirement of low-cost biosorbents and, waste management is also a concerning issue today. The purpose of this study is to recommend an alternative way the elimination of the waste of MBR unit and re-use this waste as a low-cost biosorbent. The results of the current study show that the Cu (II) removal was reached 89.65%, maximally. The waste active sludge containing fungal biomass has been introduced as a successful adsorbent for the removal of Cu (II) ions from aqueous solutions in this study. Since the active sludge compost of fungal biomass was a solid waste of MBR which was used for decolorization of textile dyes, used in this study, it was freely, abundantly, and locally available and this adsorbent is expected to be economically viable for the treatment of metal-containing wastewater.

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