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Title of manuscript

Comparison study of biosorption and coagulation/air flotation methods for chromium removal from wastewater: experiments and neural network modeling

Running title

Removal of chromium by biosorption vs. coagulation-DAF systems

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Comparison study of biosorption and coagulation/air flotation methods for chromium removal from wastewater: experiments and neural network modeling

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Abstract

This work aims to compare a biosorption method (BM) and a coagulation–dissolved air flotation method (C/DAFM) as inexpensive and effective means of eliminating hexavalent chromium (Cr[VI]) from industrial wastewater. Synthetic wastewater containing 1000 mg L⁻¹ chromium was used for the experiments. The effect of different parameters (initial pH, amount of adsorbent, and contact time) was investigated for the BM. At optimal conditions (initial pH 3, 7 g adsorbent, and contact time 120 min) maximum Cr(VI) removal was estimated to reach 68.1%. For the C/DAFM, poly aluminum chloride (PAC), and FeCl₃ were used as coagulants and the dose was determined by jar test. In optimal conditions of pH 7.5, pressure of 3 bars, and flotation time of 5 min, a maximum of 85% of chromium was extracted. In a comparison of C/DAFM and BM, C/DAFM showed higher ability and greater potential for Cr(VI) removal. Process optimization was carried out using an artificial neural network (ANN) to predict the most favorable conditions of operation for maximum percentage of Cr(VI) removal from wastewater.

Keywords: Removal, biosorption method, coagulation–dissolved air flotation method, optimization, artificial neural network.

1. Introduction
A toxic metal, Cr(VI) enters the environment through such industrial processes as electroplating, chromate preparation, and leather tanning. One approach to treatment of Cr(VI)-laden wastewater is reduction of Cr(VI) to Cr(III) to form a solid hydroxide. Ion exchange, solvent extraction, reduction, electrochemical precipitation, and adsorption may also be utilized for Cr(VI) reduction, but such treatment methods present disadvantages such as high cost involved with initial investment in equipment, as well as operational costs and costs for disposal of the residual metal sludge. The pursuit of efficacious yet cost-effective means of Cr(VI) removal is called for. Contemporary research has focused on biomasses derived from algae, fungi, or yeast, with a view toward the various advantages of each, including cost effectiveness, high selectivity, and eco-friendliness.

Brown algae, which form a diverse class of organisms found mainly in temperate to subpolar intertidal areas of the marine environment, have shown promise in a number of investigations. Sargassum bevanom, for example, is a species of brown algae that has a superior metal-adsorption capacity as compared to other biosorbents due to the powerful functional groups on its surface.

The technology offered by the C/DAFM has potential for removal of heavy metals and other particulate matter from wastewater and offers a simple and cost-effective method for Cr(VI) removal. Once adequate flocculation of the particles in the solution to be treated has been achieved, the C/DAFM makes use of solid/liquid separation by attaching microscopic air bubbles to flocs suspended in an aqueous solution and floating the particles to the surface, where they can be skimmed off. The bubbles are formed by a reduction of pressure in a water stream saturated with air at higher than 3 bars. Air introduced at the bottom of the container of water being treated forms small bubbles that collect on the suspended matter, and float it to the surface. The particulate matter may then be skimmed from the surface. Through this means particles having a higher density than the aqueous solution suspending them can be
made to float. The removed particles can be further processed, and any residuals in the
treated water may be filtered out\textsuperscript{17}. Process variables that control the C/DAFM, including the
saturation pressure, air-to-solids ratio, and the hydraulic surface-loading rate, depend on the
properties of the wastewater and can be adjusted to meet specific effluent quality standards\textsuperscript{18}.
The primary purpose of this study was to compare the effectiveness of two environmentally
friendly technologies characterized by their ability to remove heavy metals from solutions
without introducing pollution into the environment, as specifically applied to eliminating
Cr(VI) from wastewater. Removal of chromium from wastewater using C/DAFM in
comparison with BM has not been studied previously. In this study the BM was used to
evaluate the potential of the brown alga \textit{S. bevanom} as an adsorbent. Various factors were
varied for each method and their effects observed; based on this, optimal conditions for both
methods were established. The two methods were then compared to determine their relative
effectiveness under optimal conditions.

2. Materials and methods

2.1. Materials

Potassium dichromate (K$_2$Cr$_2$O$_7$) was dissolved in distilled water to make a
stock solution of Cr(VI) (1000 mg L$^{-1}$). Distilled water was added to dilute the solution as
needed for each experiment. HCl (1 M) and NaOH (1 M) were used to adjust the pH as called
for.

2.2. BM study

2.2.1. Biomass preparation

In the BM study, \textit{S. bevanom} collected along the Persian Gulf, Bushehr, Iran, was used as the
biosorbent in all experiments. To obtain a clean biomass, the alga was initially washed three
times with deionized water. Next, the biosorbent was placed in an oven to dry for 24 h at 70
°C. The dried biosorbent was ground and then sifted to obtain a mesh size of 200–300.
2.2.2. Batch experiment

All experiments were performed using 250-mL Erlenmeyer flasks kept at room temperature and agitated occasionally at 300 rpm. Various parameters, including initial pH, adsorbent dosage, and contact time, were evaluated in the batch experiments. Also, in this study, the changing of initial pH, adsorbent dosage and contact time is from 2 to 8, from 1 to 10 g and from 15 to 150 min, respectively.

2.2.3. Continuous experiments

A glass column with an ID of 2 cm and 25 cm in length was used to carry out the continuous-flow sorption experiments. The column was fitted with an adjustable plunger and 0.5-mm stainless sieves at the base and head. The bottom 2 cm of the column were filled with a layer of 1.5-mm glass spheres in order to achieve a uniform intake of solution. *S. bevanom* was placed on top of the glass beads at the selected sorbent bed height. The flow rate of the Cr(VI) solution was regulated by a Miclins PP 40 peristaltic pump and forced up through the column at 5 mL/min. Sampling was done from the outlet flow at designated intervals of time and analyzed for presence of Cr(VI). After elution, the bed was rinsed using distilled water until the wash-effluent pH had stabilized near 7. It should be noted that, at each stage, 200 ml samples were taken. A diagram of the BM system used is shown in Figure 1a.

2.3. C/DAFM study

A diagram of the C/DAFM system used is shown in Figure 1b. The C/DAFM was carried out in six stages: (1) a sample solution at the desired pH was placed in the flotation tank, (2) PAC was added as a coagulant, (3) the chromium solution and PAC were mixed rapidly, (4) compressed air was injected into the flotation tank using a compressor, (5) flotation was continued for 30 min, and (6) samples were collected from the sampling point.

2.4. Analytical methods
Analysis of the concentration of Cr(VI) in the samples was carried out using a Unicam 929 atomic absorption spectrometer (AAS). The following equation was used to calculate the removal percentage:

\[ \text{Removal efficiency} \% = \frac{C_0 - C_f}{C_0} \times 100 \]  

(1)

where \( C_0 \) is the initial chromium concentration (mg/L) and \( C_f \) is the final chromium concentration (mg/L).

The sorption capacity was computed as follows:

\[ q(t) = (C_i - C_t)(mg/L) \times \frac{V(L)}{m(g)} \]  

(2)

where \( q \) is the amount of metal adsorbed at time \( t \), \( C_i \) was the liquid-phase concentration of the solute at the initial time, \( C_t \) was the liquid-phase concentration of the solute at a given time, \( V \) was the solution volume, and \( m \) was the mass of the adsorbent.

The amount of adsorption at equilibrium was computed as follows:

\[ q_e = (C_i - C_e)(mg/L) \times \frac{V(L)}{m(g)} \]  

(3)

where \( q_e \) is the amount of adsorption at equilibrium and \( C_e \) is the ion concentration (mg/L) at equilibrium.

2.5. ANN process

An ANN is a system that employs numerous elements to form a computational model. The elements, or neurons, are interconnected by weight coefficients that can be used for training the ANN to solve a particular problem in a set. In most ANN systems the neurons are separated into layers, with each layer performing a specific function. The network architecture of a given ANN consists of the configuration of the layers and the patterns
among them. In general, the neurons within a given layer will either be connected or not. In a feedforward neural network architecture, neurons connect to an output layer through an input signal. The weighted connections among neurons are adjusted using backpropagation, the error between the input data and the predicted values is minimized using Widrow-Hoff learning \(^{19}\). As ANN architecture is founded on neurons, transfer functions, layers, and their connections, designing a specific ANN consists of configuring the number of neurons, their arrangement layers, the number of layers, how the layers interconnect, the input weights, and the transfer functions. Trial and error is used to establish the number of layers and the number of neurons in each layer. This investigation focused on pH, amount of adsorbent, and contact time as the input variables for the first network, whereas pressure, flotation time, and bed height were the input variables for the second network. The experimental values for percentages of chromium removal with BM and C/DAFM were introduced as network output variables (network response). MATLAB software was used \(^{20}\).

### 3. Results and discussion

#### 3.1. ANN

Network training was used to determine the ANN weights. In much the same way as a mathematical model is calibrated, 75% of the data were used for data-collection training to establish the percentage of chromium eliminated from wastewater. For both the BM and the C/DAFM, 20 data sets comprised the training data. Figures 2a and 2b contrast the experimental data against the values predicted by the models. The \(R^2\) values obtained indicate the models to be acceptable. The optimal number of neurons in the hidden layer was found to be 9 neurons for both the BM and the C/DAFM (Tables 1 and 2). The root-mean-square errors for the training algorithms tested are presented in Table 3. The Levenberg-Marquardt (LM) algorithm was found to work best, and the LM-trained network chosen as the best predictor of process effectiveness.
In this study, typology of chromium based on reduction of Cr\textsuperscript{6+} to Cr\textsuperscript{3+} in acidic media with low pH was occurred. \(2\text{HCrO}_4^- + 6\text{I}^- + 14\text{H}^+ \rightarrow 2\text{Cr}^{3+} + 3\text{I}_2 + 8\text{H}_2\text{O}\).

Iodide ion was used as a reducing agent to react with chromium (VI) and produce \(\text{I}^3^-\) and Cr(III) ions. Due to low concentration of chromium in the sample, cloud point method was used for pre-concentration to \(\text{I}^-\) which will react with cetyl trimethyl ammonium bromide and forms a negatively charged complex. Ammonium cerium sulfate (IV) \([\text{Ce(NH}_4)_4(\text{SO}_4)_4]\) are used to oxidization of all Cr(III) to Cr(VI) and following reaction occurred: \(3\text{Ce}^{4+} + \text{Cr}^{3+} \rightarrow \text{Cr}^{6+} + \text{Ce}^{3+}\).

Cr\textsuperscript{6+} sample was passed through the column that coated with strong acidic resin and samples from its bottom collected and then preconcentration and extraction with UV-Vis device carried out and finally, absorption of Cr(VI) occurred.

3.2. BM result

3.2.1. Initial pH

Usually pH is the most important factor in effectiveness of adsorption processes. The effect of initial pH was observed when varied from 2 to 8 at the initial chromium concentration of 1000 mg L\textsuperscript{-1}, amount of adsorbent 7 g and amount of adsorbent 10 g, and contact time of 150 min (Figure 2c). Above pH 3, chromium removal decreased when pH was increased. The maximum removal obtained was 70% at pH 3. Therefore, \textit{S. bevanom} was the most stable in acidic or alkaline pH conditions. The greater sorption capacity at lower pH is possibly due to the breaking up of functional sites on the sorbent. In the higher pH ranges, where the chromium primarily takes the form of HCrO\textsubscript{4}\textsuperscript{-}, removal decreased exponentially as pH increased, due to the greater attraction between the HCrO\textsubscript{4}\textsuperscript{-} ions and the H\textsuperscript{+} ions on the algal surface. Equilibrium for various chromium ions is shown below\textsuperscript{14, 22}:

\[
\text{H}_2\text{CrO}_4 \leftrightarrow \text{H}^+ + \text{HCrO}_4^- \tag{4}
\]
For discussion at low pH,

Adsorption of Cr$^{6+}$ below pH 3.0 suggests that bind of the negatively charged chromium species occurred through electrostatic attraction to the positively charged functional groups on the surface of sorbent cell wall due to the presence of more functional groups carrying positive charge at pH < 3. While at pH > 3 the sorbent cell wall possesses more group carrying net negative charge which repulses the metal anions. However, there is a removal at pH > 3.0 but the rate of removal was reduced, which could be considered the presence of physical adsorption on the surface of the sorbent$^{23}$. At low pH, on the other hand, Cr (VI) had a higher redox potential and favored Cr (VI) bioreduction$^{24,25}$. Acidic pH biomass contains the positive oxidation state of chromium due to protonation and Chromium remains in the anionic form (i.e. HCrO$_4^-$ or Cr$_2$O$_7^{2-}$), hence sorbed optimally onto the active sites of the biomass.

### 3.2.2. Contact time

Effect of contact time on chromium sorption by *S. bevanom* (initial pH = 3, initial chromium concentration 1000 mg/L, 7 g adsorbent) is shown in Figure 2a. Chromium sorption rate reached a maximum of 65.97% with a contact time of 120 min, with little variation in the sorption rate thereafter. Based on this observation, a 120-min contact time was used in subsequent experiments.

### 3.2.3. Amount of adsorbent

The effects of *S. bevanom* doses between 1 g and 10 g (in 500 mL aqueous solution, pH = 3, contact time 120 min, initial chromium ion concentration 1000 mg/L) were compared. The maximum percentage of chromium ions adsorbed was yielded with an adsorbent dose of 7 g.
As the adsorbent dose increased from 1 to 7 g, the percentage of chromium ions adsorbed increased from 6.82% to 65.82%. Doses above 7 g did not result in any significant increase in removal of chromium. As can be seen in Figure 2b, increase in adsorbent concentration resulted in a marked increase in the adsorption percentage of chromium onto the biosorbent, which can be attributed to that fact that the increased amount of adsorbent provides greater surface area. Based on these observations, a concentration of 7 g of *S. bevanom* in the 500-mL solution was chosen as an optimal amount of adsorbent.

Globally, it would be of relevance increasing the knowledge on Cr(VI) retention processes when this pollutant interacts with sorbent materials. In this way, Fernández-Pazos et al. studied quantitative and kinetic aspects regarding Cr(VI) adsorption/desorption on various solid media (fine and coarse mussel shell, un-amended and mussel shell-amended forest and vineyard soils, slate processing fines and pyritic material). In addition to the kinetic characterization, it would be interesting to elucidate complementary aspects, such as the effects on Cr(VI) retention caused by changing pH, or the fractions where the retained Cr(VI) was bound, which can aid to estimate the degree of stability of that retention.

### 3.2.4. Cr(VI) adsorption/desorption as a function of pH

To study adsorption, triplicate samples (1 g) of mussel shell were added with 10 mL of solutions containing 5 mg L\(^{-1}\) Cr(VI) and different concentrations of HNO\(_3\) (0.0025 M, 0.0038 M, 0.005 M, 0.0075 M) or NaOH (0.0025 M, 0.0038 M, 0.005 M, 0.0075 M), also including NaNO\(_3\) 0.01M. Control samples were constituted by each of the solid materials added with 10 mL of solutions containing NaNO\(_3\) 0.01M and 5 mg L\(^{-1}\) Cr(VI), but without HNO\(_3\) or NaOH. After shaking for 24 h, all samples were centrifuged for 15 min at 4000 rpm, and filtered through acid-washed paper. The resulting liquid was analyzed for pH and Cr(VI). Adsorbed Cr(VI) was calculated as the difference between added Cr(VI) concentration and that remaining in the equilibrium solution. Desorption was studied using triplicate samples (1
g each) of the same solid materials than in the adsorption trials, that were added with 10 mL of solutions containing 100 mg L\(^{-1}\) Cr(VI), also including NaNO\(_3\) 0.01M as background electrolyte. After shaking for 24 h, all samples were centrifuged for 15 min at 4000 rpm, and filtered through acid-washed paper, discarding the liquid phase. The remaining solid phase was then subjected to a desorption procedure, adding 30 mL of solutions containing NaNO\(_3\) 0.01 M and diverse HNO\(_3\) or NaOH concentrations aiming to provide a wide pH range, then being different for the various solid samples. After shaking for 24 h, all samples were centrifuged for 15 min at 4000 rpm, and filtered through acid-washed paper. The resulting liquid was analyzed for pH and Cr(VI). Desorbed Cr(VI) was calculated as the difference between the amount retained in the adsorption phase and that released to the equilibrium solution in the desorption phase, and it was expressed as percentage of the total amount adsorbed\(^{27}\).

### 3.2.5. Bed height

Figure 3a shows how bed heights of 10 cm, 15 cm, and 20 cm with initial Cr(VI) concentration of 1000 mg/L and flow rate of 5 mL/min affected chromium removal. When bed height was increased from 10 cm to 20 cm, the volume of the treated solution increased due to increasing diffusion of the chromium ions into the sorbent, correlated to decreasing axial dispersion in the mass transfer. This allowed sufficient time for the solute to diffuse into the biosorbent, remaining in the column for a longer period of time and treating a greater quantity of effluent. The increased sorption capacity achieved with increased bed height is attributable to the greater adsorbent concentration in larger beds. With more sorption sites available, the uptake of chromium ions in the column increased. The greatest Cr(VI) removal (85.09%) was achieved with the maximum bed height of 20 cm. It should be noted that as the
bed height increased from 10 to 20 cm the breakthrough time also increased. Also, it should be noted that, the range of 10 to 20 cm is range for sampling.

3.3. C/DAFM system

In C/DAFM systems, the liquid to be treated is first placed in a container and pressurized by dissolving air in it at approximately 1.5–3 bars. The supersaturated solution is conducted into a flotation tank, allowing the pressure to be released. Air precipitates out of the solution, forming small 30–120 mm gas bubbles, which cling to the particulate matter in the solution and raise it to the surface, allowing it to be eliminated. In this investigation, the effects of flotation time, pressure, and bed height on the C/DAFM system were investigated.

3.3.1. Flotation time and pressure

The effect of varying pressures from 1.5 to 3 bars on the chromium removal percentage at a constant flotation time of 1–6 min and bed height of 1 m in the presence of PAC as a coagulant were measured (Figure 4b). The removal of Cr(VI) increased over the first 5 min, gradually leveling off until it reached equilibrium. The chromium removal reached 85% at a constant pressure of 3 bars.

The effects of pressure are dependent on the solubility of gases in liquids. At higher pressures, more gas is soluble in any given liquid than at lower pressures; the extra gas dissolved at higher pressure evaporates from the solution when the pressure is reduced. In this study maximum chromium removal was obtained at a pressure of 3 bars, and therefore this pressure was selected as the optimal value in the following experiments.

3.3.2. Bed height

Two different bed heights, 1 m and 2 m, at a constant pressure of 3 bars and flotation time 5 min, were tested. The amount of chromium removal decreased when bed height increased from 1 m to 2 m. The maximum chromium removal obtained at a bed height of 1 m was 85%. This bed height was thus selected as the optimal value for chromium removal.
3.4. Comparison between C/DAFM and BM

In this study a higher percentage of chromium removal was achieved using the C/DAFM than with the BM. The maximum chromium removal obtained with the BM was about 65% when carried out using the optimal conditions of initial pH 3, contact time 120 min, and adsorbent dose of 7g at the initial chromium concentration of 1000 mg/L, whereas with the C/DAFM, maximum chromium removal reached 85% at a pressure of 3 bars, bed height of 1 m, and flotation time 5 min.

4. Conclusion

The comparative effectiveness of the C/DAFM vs. the BM for removal of Cr(VI) from a solution was studied. With the BM, using dried S. bevanom as the biomass, 65% removal was achieved under optimal conditions including initial pH 3, contact time 120 min, and amount of adsorbent 7 g in 500 mL solution. In the C/DAFM system, maximum chromium removal achieved was 85% at optimal conditions including pressure of 3 bars and flotation time of 5 min. Comparison of the two methods demonstrated that for an initial chromium concentration of 1000 mg L\(^{-1}\), the C/DAFM is more effective than the BM.

References


Figure captions

**Fig. 1.** Schematic diagram of experimental setup for (a) Schematics of experimental bioreactor designed for removal of MEG from gas-refinery wastewater: (a) air pump, (b) wastewater pump, (c) feed tank, (d) packed bed, and (b) dissolved air flotation method

**Fig. 2.** (a) Actual measured values vs. those predicted by ANN for Cr(VI) removal, biosorption method; (b) Actual measured values vs. those predicted by ANN for Cr(VI) removal, coagulation–dissolved air flotation method; (c) Effect of initial pH on Cr(VI) removal, constant contact time 150 min, amount of adsorbent 10 g, and Cr (VI) concentration 1000 mg L$^{-1}$

**Fig. 3.** (a) Effect of contact time on Cr(VI) removal, initial pH 3, amount of adsorbent 10 g, and Cr(VI) concentration 1000 mg L$^{-1}$. (b) Effect of amount of adsorbent on Cr(VI) removal, initial pH 3, contact time 120 min, initial Cr(VI) concentration 1000 mg L$^{-1}$

**Fig. 4.** (a) Breakthrough curves for Cr(VI) sorption onto *S. bevanom* at different bed heights. (b) Effect of flotation time on the C/DAFM at constant pressure of 3 bars and height 20 cm
Tables and figures

Table 1: Effect of number of neurons in hidden layer on performance of neural network for Cr(VI) removal by biosorption method

<table>
<thead>
<tr>
<th>Number of neurons in hidden layer</th>
<th>RMSE</th>
<th>$R^2$</th>
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<tr>
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<tr>
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Table 2: Effect of number of neurons in hidden layer on performance of neural network for Cr(VI) by C/DAFM

<table>
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<th>Number of neurons in hidden layer</th>
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<tr>
<td>10</td>
<td>9.23</td>
<td>0.654</td>
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</tbody>
</table>
Table 3: Root-mean-square errors for training algorithms for 9 neurons in hidden layer of ANN for Cr(VI) removal by BM and C/DAFM

<table>
<thead>
<tr>
<th>Algorithm No.</th>
<th>Algorithm</th>
<th>Transfer Function</th>
<th>Hidden Layer</th>
<th>Output Layer</th>
<th>RMSE (BM)</th>
<th>RMSE (C/DAFM)</th>
<th>R² (BM)</th>
<th>R² (C/DAFM)</th>
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<tr>
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<td>9</td>
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<td>Tansig</td>
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<td>Purelin</td>
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<td>0.995</td>
<td>0.771</td>
</tr>
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Figure 2a

\[ R^2 = 0.998 \]
\[ \text{RMSE} = 0.884 \]
\[ Y = 0.98X + 0.53 \]
Figure 2b

The graph shows a linear relationship between the percentage of Cr (VI) removal (predicted) and the percentage of Cr (VI) removal (experimental). The equation of the line is $Y = 0.95X + 3.34$, with $R^2 = 0.971$ and $RMSE = 0.823$. The data points are scattered around the line, indicating a good fit of the model to the experimental data.
Figure 2c

[Graph showing Cr(VI) removal (%) vs pH with two lines: Experimental and ANN.]
Figure 3a
Figure 3b
Figure 4a