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Biosorption of chromium using factorial experimental design

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Abstract

An experimental design technique has been used to investigate the biosorption of chromium from water solutions, simulating typical tanning effluents. The United States Environmental Protection Agency (EPA) usually allows solutions containing heavy metals to be discharged if the concentration is less than 5.0 mg/L. The removal of Cr^{3+} and Cr^{6+} was studied, separately, using the factorial design 2^3. The three factors considered were pH, temperature, and metal concentration at two markedly different levels: Cr^{3+}, pH (2.0 and 6.0), *T* (29 and 55 °C), and metal concentration (10 and 1200 mg/L); Cr^{6+}, pH (1.0 and 3.0), *T* (29 and 55 °C), and metal concentration (10 and 1200 mg/L). Experiments were carried out in a batch type reactor system with 0.2 g of biosorbent (*Sargassum* sp.), and 50 mL of Cr^{3+} or Cr^{6+} solutions. The efficiency of chromium removal during an exposition time of 6 h was then evaluated. The results were analyzed statistically using the Student's *t*-test, analysis of variance, *F*-test, and lack of fit to define the most important process variables affecting the metal removal efficiency.

The most significant effect regarding Cr^{3+} uptake was ascribed to interaction between metal concentration and pH. For Cr^{6+}, the most significant effect was ascribed to metal concentration.
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1. Introduction

Heavy metals are widely employed in the textile, tanneries, and metal plating industries, among others. Use of these metals generates liquid wastes, which are usually continuously disposed of without any treatment. In some cases, physical and physicochemical treatments are employed, such as ion exchange, electrodialysis, ultrafiltration, precipitation, and flash evaporation, and, less frequently, biological processes.

The major disadvantages of conventional treatments are the costs of the final disposal of residual sewage, the energy consumed, and the chemicals involved.

The biological process of biosorption is favored because of an abundance of certain biomasses, generated as a byproduct in industrial fermentation processes, and seaweed, which is found in large quantities in seas and lakes. It is considered a potentially viable method on both technical and economic grounds, because of its low operating costs [1], reduced volume of residual sewage, and the decontamination efficiency for highly diluted effluents. Additionally, metal can be recovered from the biosorbent and reused. These advantages have stimulated the development and scaling up of biosorption processes [2].

Biosorption is the first stage in metal accumulation. It involves adsorption of metal onto the cell walls of microorganisms, independently of metabolism [3]. Many microorganisms can be employed in this process, including the seaweed *Sargassum* sp.

Factorial design was employed avoiding the traditional one-factor-at-a-time experiments. Common statistical tools, such as analysis of variance, *F*-test, the Student's *t*-test, and lack of fit, were used to define the most important process variables affecting the metal removal efficiency [4].

The factorial experimental design methodology involves changing all variables from one experiment to the next. The reason for this is that variables can influence each other, and the ideal value for one of them can depend on the values of

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the others. This interaction between variables is a frequent phenomenon [4].

Many studies concerning uptake of heavy metals have indicated that temperature [5–11] and pH [2,5–8,10–25] influence removal efficiency. However, few employ the factorial design method for evaluating the influence of the operation variables on these processes [23,24].

Barros Jr. et al. [23] studied the effects of pH (4.0 and 5.5), heavy metal concentration (5.0 and 10 g/L) and biomass concentration (0.4 and 0.7 g/L) on biosorption of cadmium from oil field waters using *Aspergillus niger*. The biosorption process studied was modeled based on 2^3 factorial design. The most important factor was the biomass concentration. An increase in the removal efficiency occurred with an increase in biomass concentration and pH. However, the removal efficiency decreased with an increase in initial concentration of cadmium.

Peternele et al. [24] studied the uptake of Cd and Pb, using a 2^3 factorial design. Three operation factors were analyzed: temperature (30 and 50 °C), ionic strength (0.1 and 1.0 mol dm^{-3}), and pH (5 and 6). The fixed parameters were time of exposition (8 h) and initial adsorbent concentration (0.2 g/L). The authors concluded that temperature was the most important factor in the single system (Pb^{2+}), while ionic strength was the most important variable for the binary system (Pb^{2+} and Cd^{2+}) for the range of concentrations studied. In the single system the adsorption increases with increasing temperature and in the binary one the adsorption diminishes with increasing ionic strength.

For heavy metal uptake, such as cadmium onto a seaweed, it was verified that temperature influenced adsorption [8].

Published biosorption studies [2,5–8,10–25] show that pH influences removal efficiency of heavy metals. Various authors have evaluated removal efficiency as a function of pH variation, or have obtained isotherms at different pH values. Results have revealed that metal uptake by biosorbent gives better efficiencies in acid environments.

Chromium is a toxic element present in many industrial processes. Hexavalent chromium is known to be much more dangerous than Cr^{3+}. Extensive use of chromium, e.g., in electroplating, tanning, and as a biocide in cooling water of power plants, invariably results in discharges of chromium-containing effluents [5].

The objective of this work was to establish how pH, temperature, and initial concentration of chromium interacted and ultimately affected chromium removal efficiency from synthetic effluents by means of *Sargassum* sp. A factorial design 2^3 scheme was used to study the removal of Cr^{3+} and Cr^{6+}, separately, from aqueous solutions.

2. Materials and methods

A residue of *Sargassum* sp. seaweed obtained after extraction of biological cosmetics components was washed with potable water for removal of sand and other solids, and again washed with deionized water until no color was visualized in the rinse water. Finally, it was dried at 60 °C for 24 h and the resulting material was screened to a particle size between 28 and 35 mesh.

The chromium salts employed were $CrK(SO_4)_2{\cdot}12H_2O$ and $K_2Cr_2O_7$ (MERCK), for the preparation of solutions of trivalent and hexavalent chromium, respectively.

Batch experiments were carried out under the following conditions: 0.2 g of *Sargassum* sp., 50 mL of chromium solution, and an agitation speed of 200 rpm. The pH, temperature, and initial chromium concentration employed are shown in Table 1. Samples were collected after 6 h. Control samples were made in absence of any metal. Aliquots for analysis were filtered, and the residual chromium concentration was measured by atomic absorption spectrometry (Perkin Elmer, model AAS-1100B).

Table 1
High and low levels of factors

Factor	Species			
	Cr^{3+}		Cr^{6+}	
	Low level	High level	Low level	High level
pH	2.0	6.0	1.0	3.0
Temperature (°C)	29	55	29	55
Initial concentration (mg/L)	10	1200	10	1200

Sixteen duplicate experiments were carried out: eight for Cr^{3+} and eight for Cr^{6+}. All possible combinations of variables, called factors in the jargon, were used, and a matrix was established according to their high and low levels, represented by +1 and −1, respectively.

3. Results and discussion

Results for Cr^{3+} and Cr^{6+} uptake are shown in Table 2.

Table 2
Experimental factorial design results for Cr^{3+} and Cr^{6+} uptake

Factor			Species					
T	*C*	pH	Cr^{3+}			Cr^{6+}		
			Removal efficiency (%)[a]		Average (%)	Removal efficiency (%)[a]		Average (%)
1	1	1	75.2	74.8	75.0	26.1	21.6	23.9
1	1	−1	11.4	5.4	8.4	16.2	27.0	21.6
1	−1	1	62.7	56.4	59.6	81.3	80.9	81.1
1	−1	−1	82.9	83.5	83.2	67.3	65.1	66.2
−1	1	1	99.5	99.5	99.5	7.7	13.1	10.4
−1	1	−1	2.5	17.3	9.9	11.7	15.8	13.7
−1	−1	1	62.3	68.9	65.6	84.6	83.7	84.1
−1	−1	−1	73.4	74.2	73.8	65.1	66.1	65.6

[a] Experiments in duplicate.

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