



REMOVAL OF HEXAVALENT CHROMIUM USING ACTIVATED CARBONS DERIVED FROM MARINE ALGAE GRACILARIA AND SARGASSUM SP.

Akbar Esmaeili

Department of Chemical Engineering, North Tehran Branch, Islamic Azad University, P.O. Box 1969633651, Tehran, Iran, akbaresmaeili@yahoo.com

Samira Ghasemi

Department of Chemical Engineering, North Tehran Branch, Islamic Azad University, P.O. Box 1969633651, Tehran, Iran

Abdolhossein Rustaiyan

Department of Chemistry, Science & Research Campus, Islamic Azad University, P.O. Box 14515-775, Tehran, Iran

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REMOVAL OF HEXAVALENT CHROMIUM USING ACTIVATED CARBONS DERIVED FROM MARINE ALGAE *GRACILARIA* AND *SARGASSUM* SP.

Akbar Esmaeili*, Samira Ghasemi*, and Abdolhossein Rustaiyan**

Key words: adsorption, *Gracilaria*, *Sargassum*, wastewater, activated carbon.

ABSTRACT

Industrial wastewater contain higher quantities of salts besides chromium (VI) ions, therefore, the effect of these salts on the adsorption of chromium (VI) should be studied. In this research, the batch removal of toxic hexavalent chromium ions from wastewater and aqueous solution using two different activated carbon marine algae *Gracilaria* (red algae) and *Sargassum* sp. (brown algae) were examined. Activated carbon prepared from *Gracilaria* and *Sargassum* sp. The chromium uptake was dependent on the equilibrium pH and chromium concentration, with pH = 2.0, being the optimum pH value. The experimental adsorption data were fitted to the Langmuir and Freundlich adsorption model. The maximum efficiencies of chromium removal were 91.53% and 91.98% for activated carbon prepared of *Gracilaria* and *Sargassum* sp., respectively. A significant fraction of the total chromium (VI) uptake was achieved within 60 min. The chromium (VI) uptake by the biosorbents was best described by pseudo-second order rate model. This investigation verifies the possibility of using activated carbon prepared from algae as valuable material for the removal of chromium from aqueous solution and wastewater.

I. INTRODUCTION

The presence of toxic heavy metals in water resulting from rapid industrialization and technological advances is a worldwide environmental problem. Removal of these pollutants from aqueous effluents has conventionally been accomplished through abiotic processes [24, 25, 30]. However, these processes can be expensive and not fully effective. Recently, increasing interest in the application of materials of biological

origin in heavy metals removal from diluted, large volume solutions has been observed [23, 28]. Biosorption, which uses the ability of biological materials to remove and accumulate heavy metals from aqueous solutions, has received considerable attention in recent years because of a few advantages compared to traditional methods [15].

Chromium is one of the key contaminants in the wastewaters of industrial dyes and pigments, film and photography, galvanometry and electric, metal cleaning, plating and electroplating, leather and mining. While hexavalent (CrO_2^{4-} and $\text{Cr}_2\text{O}_2^{7-}$) and trivalent (Cr^{3+} and CrOH^{2+}) species of chromium are prevalent in industrial waste solutions, the hexavalent form has been considered more hazardous to public health due to its mutagenic and carcinogenic properties [3, 4].

The adsorption process with activated carbon is attracted by many scientist because of the effectiveness for the removal of heavy metal ion at trace quantities. But the process has not been used extensively for its high cost. For that reason, the use of low cost materials as sorbent for metal removal from wastewater have been highlighted. More recently, great effort has been contributed to develop new adsorbents and improve existing adsorbents like granular activated carbon, other adsorbents such as iron oxide coated sand, porous cellulose carrier modified with polyethyleneimine etc. Biosorption technology can provide an alternative for the treatment of hexavalent chromium [2, 29]. It is reported that the process reaches to its optimum treatment level of hexavalent chromium at lower pH [19]. Activated carbon has indubitably been the most popular and widely used as adsorbent in wastewater treatment employed throughout the world. However, activated carbon remains a costly material since higher the quality of activated carbon; the greater will be its cost [11]. Thereby, searching a low cost activated carbon for the wastewater treatment is essential. The goal of present work is to test the ability of an activated carbon prepared from *Gracilaria* and *Sargassum* sp. to remove chromium (VI) ions from aqueous solution.

II. MATERIAL AND METHODS

1. Preparation of Biomass

Gracilaria and *Sargassum* sp. were collected from the Per-

Paper submitted 12/19/08; revised 07/17/09; accepted 08/25/09. Author for correspondence: Akbar Esmaeili (e-mail: akbaresmaeili@yahoo.com).

*Department of Chemical Engineering, North Tehran Branch, Islamic Azad University, P.O. Box 1969633651, Tehran, Iran.

**Department of Chemistry, Science & Research Campus, Islamic Azad University, P.O. Box 14515-775, Tehran, Iran.

sian Gulf on Qeshm Island, Iran. Before use, these were washed several times with tap water to remove the sand particles and salts.

They were then sun dried for 6 days. Dry biomass was chopped, milled (size fraction of 0.5-1 mm) and then used for adsorption experiments [22].

2. Activated Carbon

The dried red alga *Gracilaria* and brown alga *Sargassum* 120 g were added in small portion to 96 mL of 97% H₂SO₄. The resulting mixture was kept for 24 h at room temperature followed by refluxing in fume hood for 4 h. After cooling, reaction mixture was washed repeatedly with deionized water and soaked in 2% NaHCO₃ solution to remove any remaining acid, and pH of the activated carbon reached 7, dried in an oven at 150°C for 46 h.

3. Method Column Experiments

Continuous-flow sorption experiments were conducted in-glass column with an internal diameter of 2 Cm and 35°Cm in length. At the bottom of the column a 10 mm stainless was attached followed by cotton.

4. Preparation of Cr (VI) Solution

The stock solution of Cr⁶⁺ (1000 mg L⁻¹) was prepared in deionized water with potassium dichromate (K₂Cr₂O₇). All working concentrations were obtained by diluting the stock solution with deionized water, and pH was adjusted to the desired values according to the following experimental design with 1 M HCl and 1 M NaOH solutions.

5. Adsorption Studies

The effect of pH on the equilibrium adsorption was investigated by using different initial concentration of Cr⁶⁺ (10 mg l⁻¹) and different activated carbon dosages. The pH values were adjusted with diluted HCl and NaOH solutions. All the adsorption experiments were carried out at room temperature (23 ± 2°C), using agitation speed (400 rpm) for the minimum contact time required to reach the equilibrium. Adsorption of Cr⁶⁺ was studied using different weights of activated carbon prepared of *Gracilaria* and *Sargassum* and different concentration of Cr⁶⁺ and initial pH 2.0.

6. Determination of the Cr (VI) Contents

Concentration of Cr⁶⁺ in the solution before and after the equilibrium was determined by Atomic Absorption Spectrometry (AAS, GBC-932) using a Perkin –Elmer Analyst 300 atomic absorption spectrometer equipped with deuterium as background corrector, an air-acetylene burner, and controlled by IBM personal computer. The hollow cathode lamp was operated at 15 mA and the analytical wavelength was set at 324.8 nm.

7. Experimental Procedure

A series of flasks containing dichromate solutions of vary-

Table 1. Langmuir and Freundlich constants for adsorption of Cr⁶⁺ using activated carbon prepared of *Gracilaria* and *Sargassum* sp. activated carbon.

		Activated carbon prepared of <i>Gracilaria</i>			Activated carbon prepared of <i>Sargassum</i> sp.		
		0.4 g	0.6 g	0.8 g	0.4 g	0.6 g	0.8 g
Two-parameter models							
Langmuir	$q_{\max}(\text{mg/g})$	6.770	4.739	2.890	6.877	2.809	2.761
	$b(\text{L/mg})$	0.095	0.125	0.353	0.088	0.243	0.393
	R^2	1.000	0.999	0.988	0.999	0.995	0.985
Freundlich	$K_f(\text{L/g})$	0.612	0.520	0.687	0.579	0.527	0.699
	n	1.309	1.302	1.822	1.290	1.544	1.954
	R^2	0.996	0.999	0.904	0.998	0.999	0.882

ing concentrations from 10 to 60 mg/L prepared from the stock solution. Adjustment of pH was carried out using 1N NaOH and 1N H₂SO₄. Batch equilibrium sorption experiments were carried out in Erlenmeyer flasks for 120 min. (0.4, 0.6 and 0.8 g of biomass, 30 mL of metal solution) in a rotary shaker. These experiments were done of aqueous Cr⁶⁺ solutions of initial concentrations ranged from 10 to 50 mg l⁻¹ for activated carbons using initial pH 2.0. The initial and equilibrium chromium concentrations in each flask were determined by AAS. To maximize Cr removal by the adsorbent, batch experiments were conducted at constant temperature using the best conditions of all suitable such as dose, pH, initial concentration and contact time. Next adsorption experiments were carried out with only limits.

8. Equilibrium Isotherm

1) Chromium Uptake Capacities and Sorption Isotherm

The amount of metal adsorbed by activated carbon was calculated from the difference between metal quantity added to the biomass and metal content of the supernatant using the following equation:

$$q_e = (C_0 - C_e) V/M \quad (1)$$

where q_e is the metal uptake (mg metal adsorbed per g adsorbent), C_0 and C_e are the initial and equilibrium metal concentration in solution (mg L⁻¹), V is the volume of the solution (mL) and M is the weight of activated carbon (g). To examine between sorbed and aqueous concentration at equilibrium, sorption isotherm models, and Langmuir and Freundlich adsorption models were used for the data. These isotherm constants for Cr⁶⁺ are presented in Table 1.

A. Freundlich Adsorption Model

The Freundlich model [8] habitually gives a better fit for adsorption from liquids and can be expressed as:

$$q_e = K_f \cdot C_e^{1/n} \quad (2)$$

In this model, the rate of adsorption is of the constants $1/n$ and K_f ($L g^{-1}$). For a good adsorbent, $0.2 < 1/n < 0.8$, and a smaller value of $1/n$ shows better adsorption and formation of rather strong bond between the adsorbate and adsorbent. Many researchers have used this model to interpret this sorption data for various systems [9, 14, 17, 26].

B. Langmuir Adsorption Model

The most widely used isotherm equation for modeling equilibrium is the Langmuir equation. It is assumed that there is a finite number of binding sites that are homogeneously shared over the adsorbent surface. These binding sites have the same adsorption of a single molecular layer and there is no interaction between adsorbed molecules. The saturated monolayer isotherm can be represented as [18]:

$$q_e = b \cdot q_{max} \cdot C_e / (1 + b \cdot C_e) \quad (3)$$

where q_e is metal ion adsorbed ($mg g^{-1}$), C_e is the equilibrium concentration ($mg L^{-1}$), q_{max} is the maximum adsorption capacity and b is an affinity constant, whereas, q_{max} represents a practical limiting adsorption capacity when the surface is fully covered with metal ions and assists in the comparison of adsorption performance, in cases where the sorbent did not reach its full saturation in experiments and b is constantly related to the affinity of the binding sites [10].

2) Adsorption Kinetic Studies

The kinetics of adsorption describes the rate of chromium ions uptake on activated carbon prepared of *Gracilaria* and *Sargassum*, which controls the equilibrium time. These kinetic models included the first-order and second-order equations.

A. Pseudo First –Order Model

The pseudo first–order rate expression is described by the following equation [13].

$$dq_t/dt = k_1 (q_e - q_t) \quad (4)$$

where, q_e is copper sorbed at equilibrium per unit weigh of orbent (mg/g); q_t is copper sorbed (mg/g); and k_1 is the rate constant (min^{-1}). The integrated form of (4) becomes:

$$\log (q_e - q_t) = \log (q_e) - (k_1/2.303) t \quad (5)$$

A plot of $\log (q_e - q_t)$ versus (t) indicates a straight line of slope $(k_1/2.303)$ and an intercept of $\log (q_e)$.

B. Pseudo Second–Order Model

The sorption data also analyzed a pseudo second-order [12], given by

$$dq_t/dt = K_2 (q_e - q_t)^2 \quad (6)$$

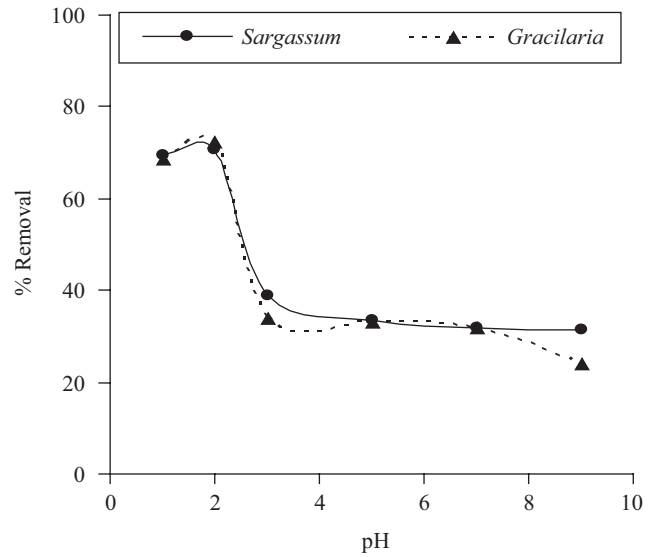


Fig 1. Effect of pH on sorption of $10 mg L^{-1} Cr^{6+}$ prepared of *Gracilaria* and *Sargassum* sp. by activated carbon after 60 min.

where K_2 is the equilibrium rate constant ($g/mg, min$). Q_e and q_t are the sorption capacities at equilibrium at time t .

The integrated form of (5) becomes:

$$1/(q_e - q_t) = 1/(q_e) + K_2 t \quad (7)$$

Which has linear form:

$$t/q_t = 1/K_2 \cdot q_e^2 + (1/q_e)t \quad (8)$$

A plot t/q_t versus t shows a straight line of slope $(1/q_e)$ and an intercept of $(1/K_2 \cdot q_e^2)$ [16].

III. RESULTS AND DISCUSSION

The present study shows that activated carbon prepared of red algae *Gracilaria* and brown algae *Sargassum* have an ability to remove Cr^{6+} from the contaminated water. The data obtained from this work supports the view that the activated carbon is an effective and low cost adsorbent for the removal of Cr^{6+} from aqueous solution. The adsorption of metal ions is dependent on activated carbon, concentration of metal ion, retention time and pH of the metal solution.

1. Effect of pH on Adsorption

The initial pH of the metal solution is an important parameter affecting adsorption of metal ions [21]. The effect of initial pH on the removal of Cr^{6+} using activated carbon prepared of *Gracilaria* and *Sargassum* was studied (Fig. 1). It is clear from the removal of Cr^{6+} decreased with an increase in pH from 1.0 to 9.0, and an optimum pH of about 2.0 was observed.

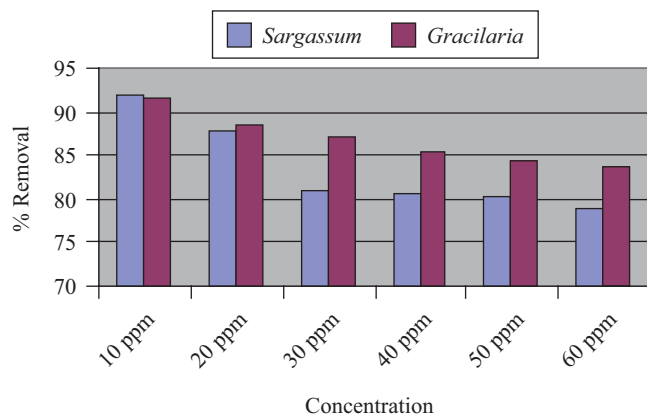


Fig 2. The effect of initial concentration of metal ion on the percentage of removal of Cr^{6+} at pH 2.0 and room temperature using 0.6 g marine algae *Gracilaria* (red algae) and *Sargassum* sp. (brown algae) and activated carbon doses.

In previous literature, similar has been reported by the nature of the biosorption at different pH [27].

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 $\text{pH} < 3$. While at $\text{pH} > 3$ the sorbent cell wall possesses more group carrying net negative charge which repulses the metal anions.

However, there is a removal at $\text{pH} > 3.0$ but the rate of removal was reduced, which could be considered the presence of physical adsorption on the surface of the sorbent [20]. At low pH, on the other hand, Cr (VI) had a higher redox potential and favored Cr (VI) bioreduction [16].

In addition, reductions on the biomass such as carbohydrate and protein could supply electrons for Cr (VI) bioreduction, with partial release of soluble organics or final oxidized product [27].

2. Effect of Metal Ion Concentration on Adsorption

The initial concentration of metal ion provides an important driving force to overcome all mass transfer resistances of metal ions between the aqueous and solid phases [20].

Figure 2 represented the effect of different initial concentrations of Cr^{6+} (10-60 mg L^{-1}) on adsorption Cr^{6+} using marine algae *Gracilaria* (red algae) and *Sargassum* sp. (brown algae) and its activated carbon.

The results show the increase in initial concentration of Cr^{6+} led to decrease in the percentage of removal for both adsorbents due to saturation of the sorption sites on adsorbents, the maximum uptake for chromium with initial concentration, 10, 30, 50 mg L^{-1} at pH 2.0 and 0.6 g activated carbon prepared. *Gracilaria* and *Sargassum* were obtained as 91.53%, 87.05%, 84.51% and 91.98%, 86.24%, 83.58%, respectively.

3. Effect of Alga Mass

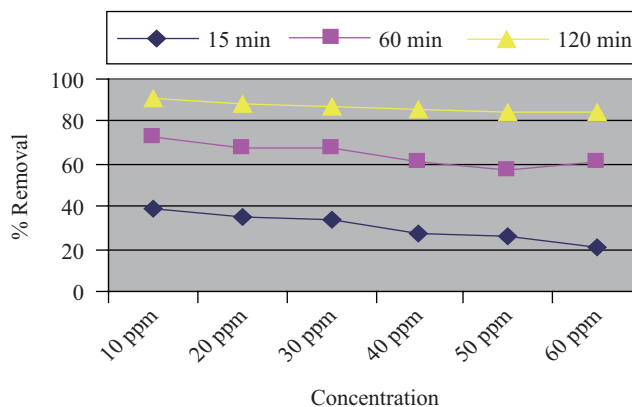


Fig 3. The effect of initial concentration of metal ion on the percentage of removal of Cr^{6+} at pH 2.0 and room temperature with different retention time *Gracilaria* (red algae) and activated carbon doses.

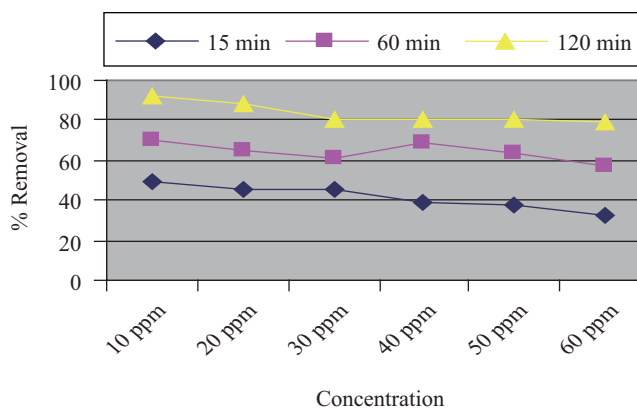


Fig 4. The effect of initial concentration of metal ion on the percentage of removal of Cr^{6+} at pH 2.0 and room temperature with different retention time *Sargassum* sp. (brown algae) and activated carbon doses.

Different dosages of biosorbents did not have any effect on the results, but 0.6 and 0.8 g of marine algae *Gracilaria* (red algae) and *Sargassum* sp. (brown algae) showed higher uptake. The kinetic experiments show the adsorption on all the sorbents was rapid and maximum sorption capacities were achieved in 2 h.

4. Effect of Retention Time

Figures 3 and 4 indicate that removal ability increased with increasing contact time and concentrated heavy metals before equilibrium reached. Other limits such as dose of adsorbent, pH of solution and, initial concentration was kept optimum. While temperature was kept at $(23 \pm 2^\circ\text{C})$ it can be seen from Figs. 3 and 4 that Cr removal efficiency increased when contact time was increased from 15 to 120 min. Optimum contact time for both adsorbents was found to be 120 min. In previous literature, similar has been reported by the evaluation of the marine alga *Gracilaria corticata* for the adsorption of Cu (II) from wastewater in a packed column reported [5].

Table 2. Comparison of the first and second order adsorption rate constants and calculated and experimental q_e values for different initial Cr^{6+} concentration and activated carbon weight.

Parameter		First-order kinetic model			Second-order Kinetic model		
Activated carbon	Initial Cr concentration (mg L^{-1})	K_1	q_e (calculated)	R^2	K_2	q_e (calculated)	R^2
0.6 g <i>Gracilaria</i>	20	0.010	0.174	0.854	0.475	1.141	0.993
	40	0.002	0.041	0.234	0.547	1.256	0.993
	60	0.001	0.023	0.698	0.491	1.436	0.997
0.6 g <i>Sargassum</i>	20	0.006	0.119	0.963	0.726	1.026	0.969
	40	0.001	0.029	0.973	2.010	0.943	0.999
	60	0.004	0.016	0.916	1.701	0.998	0.981

5. Adsorption Isotherm

Adsorption isotherms are important for describing how adsorbate will interact with an adsorbent and are critical in the use of adsorbent. Equilibrium studies in adsorption isotherm are characterized by certain constants whose values express the surface properties and affinity of the adsorbent. Equilibrium between adsorbent and adsorbate was described by adsorption isotherms, usually the ratio between the quantity adsorbed and the remaining in the solution at a fixed temperature ($23 \pm 2^\circ\text{C}$), at equilibrium. To study the adsorption isotherm, two models were analyzed.

6. Langmuir and Freundlich Isotherms

The Langmuir adsorption isotherm is the most widely applied adsorption isotherm. A basic assumption of the Langmuir theory is that adsorption takes place at specific homogeneous sites within the adsorbent.

The applicability of the empirical Freundlich isotherm was also analyzed based on the sorption on heterogeneous surface, using the same set of experimental data of dried red and brown algae and its activated carbon. The isotherm experimental results showed the data could be well modeled according to the Langmuir and Freundlich adsorption isotherm. The Langmuir constant (q_{max}) is dependent on experimental conditions such as solution pH. Another importance in evaluating sorbent performance is the initial gradient of the adsorption isotherm, since it indicates the sorbent affinity at low metal concentrations. In the Langmuir equation, this initial gradient corresponds to the affinity constant (b). As we can see from Table 1 data could be well modeled according to the Langmuir, Freundlich adsorption isotherm. In previous investigation, similar method with algae marine *Gracilaria* for the biosorption of Ni (II) has been reported [6].

7. Kinetic Modeling

The Lagergren first-order rate constant ($k_{1,\text{ads}}$) and q_e determined from the model indicated that this model failed to estimate q_e since the experimental values of q_e differed from those estimated in Table 2. Subsequently, the rate of uptake of Cr^{6+} onto the biomass increased quickly to 120 min and no

further adsorption was observed beyond this period. The Cr^{6+} uptake by the biosorbents was best described by pseudo second-order rate model.

The pseudo second-order model is based on the supposition that adsorption follows a second-order which means the rate of occupation of adsorption sites is proportional to the squares of number of unoccupied sites [1, 15].

Previous literature for the evaluation of the activated carbon prepared from the algae *Gracilaria* for the adsorption of Cu (II) the adsorption follows second order kinetic reported [7].

IV. CONCLUSION

1. The study showed that the batch removal of toxic hexavalent chromium ions from wastewater and aqueous solution using two different activated carbon marine algae *Gracilaria* (red algae) and *Sargassum* sp. (brown algae) was investigated.
2. The chromium uptake was dependent on the equilibrium pH and chromium concentration, with pH = 2.0, being the optimum pH value.
3. Batch equilibrium sorption experiments were carried out in Erlenmeyer flasks for 120 min. (0.4, 0.6 and 0.8 g of biomass, 30 mL of metal solution) in a rotary shaker. These experiments were done from aqueous Cr^{6+} solutions of initial concentrations ranged from 10 to 50 mg L^{-1} for activated carbons using of pH 2.0.
4. The maximum efficiencies of chromium removal were 91.53% and 91.98% for activated carbon prepared of *Gracilaria* and *Sargassum* sp., respectively.
5. The experimental adsorption data observed agreed with the Langmuir and Freundlich adsorption model.
6. The chromium (VI) uptake by the biosorbents followed pseudo second-order rate model.

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