

## ADSORPTIVE REMOVAL OF CHROMIUM BY DRIED BROWN ALGA *SARGASSUM HORNSCHUCHII*

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### **Abstract**

Adsorption of hexavalent chromium using dried macro-alga *Sargassum hornschurchii* was studied under controlled laboratory conditions. Experimental parameters such as contact time, biomass dosage and pH were studied in a metal concentration of 25 mg L<sup>-1</sup>. The adsorption of chromium was fast at the initial phase of study and gradually with time became slowly. The maximum removal percentage of chromium was statistically significant and it was found to be 99.36% at 36 hrs and pH 3 using 8 g L<sup>-1</sup> algal dosage. Five consecutive adsorption / desorption cycles were operated on metal solution using 0.1 M NaOH and 0.1 M HCl. It was found that chromium adsorption decreased following each adsorption / desorption cycle. Results indicated the superiority of HCl for the recovery of chromium from algal biomass. The present investigation shows that the locally biosorbent *Sargassum hornschurchii* possesses high adsorption performance with regard to the removal of chromium from its aqueous solution.

**Keywords:** Adsorption; Chromium; Removal percentage; *Sargassum hornschurchii*.

### **Introduction**

The discharge of heavy metals into aquatic ecosystems has become a matter of concern in the world. Heavy metals are among the conservative pollutants that are not subject to bacterial attack or other breakdown or degradation. As a result, their concentrations often exceed the permissible levels normally found in soil, waterways and sediments. Actually, when heavy metals accumulate in the environment and in food chains, they can profoundly disrupt biological processes (El-Nady and Atta, 1996).

The need for economical, effective and safe methods for removing heavy metals from wastewaters has resulted in the search for unconventional materials that may be useful in reducing the levels of heavy metals in the environment. Consequently, biological materials have emerged as an ecofriendly and economic option (Davis *et al.*, 2000 and Gupta *et al.*, 2000).

Among the biological materials investigated for heavy metal removal, the biomass of marine algae, otherwise known as seaweeds, has been reported to have high uptake capacities for a number of heavy metal ions (Senthilkumar *et al.*, 2006, Brinza *et al.*, 2009 and Esmacili *et al.*, 2011). Several experiments were

conducted to compare the ability of many macro-algae for removing heavy metals (**Romera *et al.*, 2007** and **Onyancha *et al.*, 2008**). They have been found to be very effective in removing heavy metal from water because of their large surface area, high binding affinity and their cheap availability (**Tsui *et al.*, 2006**, **Murphy *et al.*, 2008** and **Liu *et al.*, 2009**).

Brown algae among the various groups of algae received the most attention owing to their high uptake capacity (**Romera *et al.*, 2006** and **Brinza *et al.*, 2007**). Non-viable algal biomass frequently exhibits a higher affinity for metal ions compared with viable biomass. This is probably due to the absence of competing protons produced during metabolism. Moreover, drying and grinding biomasses reveal the sites where metal ions could be sequestered and so increase the probability of encountering metal ions. Besides, the metal removal system using dried biomass is not subject to toxicity limitations (**Rocha *et al.*, 2006**, **Parameswari *et al.*, 2009** and **Mane *et al.*, 2010**).

Many studies have indicated that a number of factors may affect the biosorption process, such as pH (**Rezaee *et al.*, 2005** and **Lia *et al.*, 2006**), biomass concentration (**Gong *et al.*, 2005**), heavy metal concentration (**Kiran *et al.*, 2005**) and contact time (**Devi *et al.*, 2010**).

The application of biosorption as a viable commercial technique implies the recovery of bound metals and the subsequent recycling of the biosorbents (**Schiewer and Volesky, 2000**). Reusability of sorbent can be evaluated by comparing the sorption potential of regenerated biomass with the original biomass (**Zhao *et al.*, 1999**). There are several different types of eluent agents (**Vijayaraghavan *et al.*, 2004** and **Deng *et al.*, 2007**) such as organic and inorganic acids solutions and complexing agents (**Hashim *et al.*, 2000** and **Gong *et al.*, 2005**). One of the best desorbent agents for metal desorption are the acids (**Lodeiro *et al.*, 2006**).

Industries such as leather tanning, petroleum refining, and pulp production generate large quantities of chromium in the environment. Chromium is present in the industrial effluents as trivalent and hexavalent. Trivalent chromium is relatively less toxic and less mobile (**Anderson, 1997**), while hexavalent chromium is toxic, carcinogenic and mutagenic to animals as well as humans (**Costa, 2003**). Different governments regulated the concentration of Cr in discharges to be less than 0.5 mg L<sup>-1</sup> irrespective of its oxidation state (**Bishnoi *et al.*, 2007**). This has led to removal of chromium from wastewater before discharge. However, the current methods being employed such as chemical precipitation are not feasible to reduce the concentration to 0.5 mg L<sup>-1</sup> meanwhile, various biosorbents offers a potential alternative (**Han *et al.*, 2007**). Chromium possesses a high oxidation state and high solubility. These characteristics make the remediation process of chromium difficult (**Smith and Gadd, 2000**). Only the Cr (VI) ion is capable of passing through cell membranes, hence the oxidation of

Cr (III) to Cr (VI) facilitates the bioavailability of chromium. Cr (VI) is the form of chromium that is mostly found at contaminated sites (**Dirilgen, 1998**).

The goal of the present investigation is to assess the potential of dried marine brown alga, namely *Sargassum hornschurchii* to adsorb Cr (VI) from aqueous solution. The effect of contact time, pH and algal dosage were studied. This study plays a role in understanding the adsorption phenomenon and help in the development of algal adsorption efficiency.

### ***Materials and Methods***

#### **Adsorbent:**

*Sargassum hornschurchii* (Plate 1) is a brown macroalga, harvested from the coast of Abu-Qir, Alexandria, Egypt. It is best known for its brown or dark green color. Generally, it consists of a holdfast, a stipe, and a frond. Oogonia and antheridia occur in conceptacles embedded in receptacles on special branches. Some species have berry like gas-filled bladders, which help keep the fronds afloat to promote photosynthesis. The samples of algal biomass were washed with distilled water 2-3 times to remove dirties, sand and epiphytes. The samples were subsequently kept on filter papers, then sun dried for 6 hours followed by drying in an oven at 40 °C till constant weight. The dried samples were then ground using a mortar, stored in powdered form for following analysis.



**Plate (1): *Sargassum hornschurchii* C. Agardh.**

### **Metal solution:**

Metal solution of chromium was prepared by dissolving 25 mg of potassium dichromate [K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>] in 1 liter deionized distilled water. The adsorption capacities have been estimated using Perkin Elmer 2380 atomic absorption spectrophotometer. The samples were diluted, whenever necessary. All experiments were conducted in duplicate at constant temperature of 25±1°C to be representative in environmentally relevant condition.

### **Effect of contact time:**

The experiment was performed by mixing 0.4 g of dried alga in 100 ml of metal solution. pH of solution was kept without treatment. The mixture was periodically agitated for pre-defined time in the range of 6 to 48 hrs with 6 hrs interval in between. The solid biomass was separated from the liquid phase and the later was analyzed for the concentration of chromium ions.

### **Effect of algal dosage:**

Different dry algal dosages in range, 1 to 10 g L<sup>-1</sup> were contacted with a series of 100 ml chromium solutions in a concentration of 25 mg L<sup>-1</sup>. The samples were shaken for the desired time and finally were collected from the aqueous phase. Then, solutions were analyzed for residual metal concentration.

### **Effect of pH:**

The effect of pH on chromium removal was examined by varying the pH of the solution from 1 to 9. The pH was adjusted by 0.1M HCl and 0.1M NaOH solutions using a pH meter (model WPA LINTON CAMBRIDGE UK. CD 620). Dried biomass samples were added. Flasks were continuously shaken and samples were taken after the desired time. Thereafter, the dried algal materials were removed through filtration. The concentration of chromium was determined in filtrate.

### **Desorption and reuse of biosorbents:**

For adsorption/desorption study, dried biomass was contacted with 100 ml Cr (VI) solution (25 mg L<sup>-1</sup>) with periodically agitation for 36 hours. After that, algal biomass was separated by filtration, washed with distilled water for three times to remove residual Cr (VI) on the surface and dried via filter paper. Subsequently, it was transferred to 25 ml of desorption solutions: 0.1 M NaOH and 0.1 M HCl (Jalali *et al.*, 2002, Bishnoi *et al.*, 2007 and Brinza *et al.*, 2007) and mixtures were shaken for 1 hour. The adsorption/desorption study was conducted again by removing the algal biomass from desorbing medium, washing with distilled water and reconditioning for adsorption in the succeeding cycle and this cycle was carried out for five times. After each cycle, sample of metal solution was analyzed by atomic adsorption spectrophotometer to evaluate the adsorption capacity of alga.

### Determination of the adsorption capacity:

The metal removal percentage was evaluated (Basha *et al.*, 2007, Saratale *et al.*, 2009 and Tan *et al.*, 2009) from the following equation:

$$\text{Removal percentage of metal \%} = \frac{C_i - C_f}{C_i} \times 100$$

Where,  $C_i$  is the initial metal ion concentration before adsorption process in  $\text{mg L}^{-1}$  and  $C_f$  is the equilibrium metal ion concentration after adsorption process in  $\text{mg L}^{-1}$ .

### Statistical analysis:

Statistical analysis employed SPSS version 10.0 for testing significance of differences between treatments at the 0.05 probability level ( $P \leq 0.05$ ). The experiments were tested with analysis of variance (ANOVA).

## Results and Discussion

### Effect of contact time:

Figure (1) illustrates the effect of contact time on the removal percentage of chromium by *S. hornschurchii*. It is clear that the concentration of metal in the solution was significantly reduced by increasing contact time till 36 hrs. This reducing was fast at initial stage of the contact period and became slower with increase in time. This finding suggested that the availability of a large number of vacant sites on the algal surface for adsorption during the initial stage of time. Over time, the remaining vacant surface sites were difficult to be occupied due to the repulsive forces between the solute molecules on the solid and bulk phase (Kumar *et al.*, 2010).

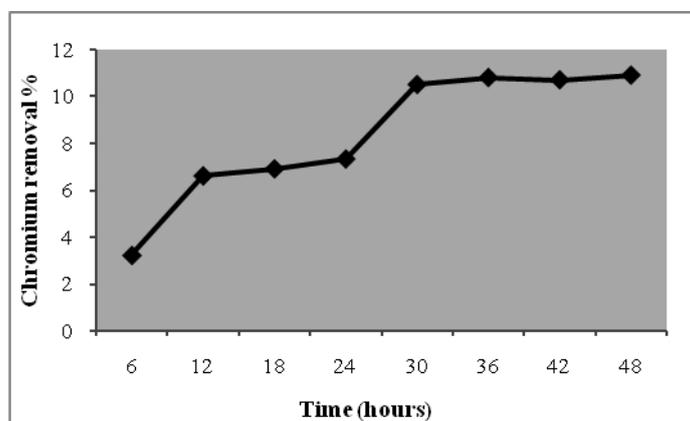
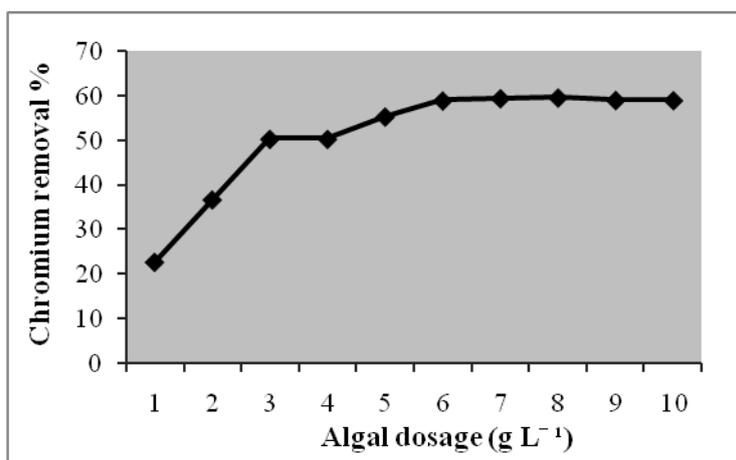


Figure (1): Removal percentage of chromium by *S. hornschurchii* dry biomass at different time periods.

**Effect of algal dosage:**

The adsorption efficiency of *S. horschuchii* for chromium ions as a function of biomass dosage was investigated. Fig. 2 shows a steeply increase in removal percentage of metal with the biomass loading up to 8 g L<sup>-1</sup>. At this point, the maximum removal percentage of chromium was statistically significant with a percent of 59.68% and it was almost the same at higher dosages. Actually, for a fixed initial metal concentration, increasing adsorbent dose provided a greater surface area, which could accommodate a higher quantity of metal. However, due to the equilibrium limitation, the quantity of metals being adsorbed for a certain unit surface area of adsorbent slightly decreased. Since near the equilibrium, the remaining vacant surface sites were difficult to be occupied as there is strong repulsion between adsorbate ions bound to active sites of adsorbent and other free ions in solution to occupy the vacant sites. Similar findings were obtained by **Sari and Tuzen (2008)** who used the dried red alga *Ceramium virgatum* for the first time for the removal of chromium and reported the maximum biosorption yield (90%) at about biomass dosage 10 g L<sup>-1</sup>. **Singh (2007)** observed increase in percentage removal of Ni (II) with increase in algal dose of *Oscillatoria* sp. or *Spirogyra* sp. from 1 to 10 g L<sup>-1</sup>. **Gupta and Rastogi (2009)** postulated that the adsorption of chromium ions by *Oedogonium hatei* increased with increasing the algal dosage and the adsorption was almost constant at dose higher than 5 g L<sup>-1</sup>.



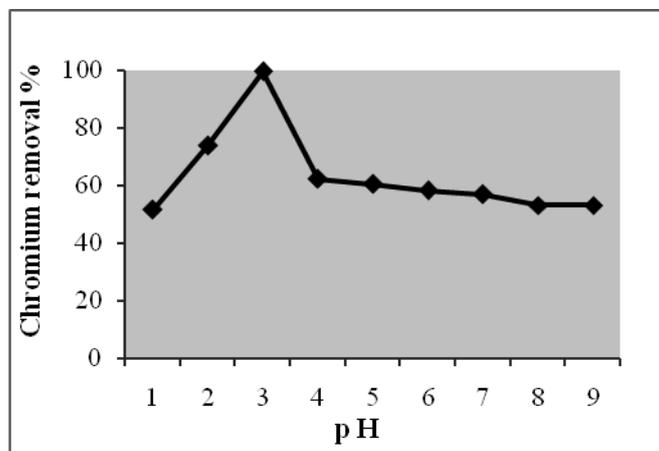
**Figure (2):** Removal percentage of chromium using different dry biomass dosage of *S. horschuchii*.

In contrast, **Mehta and Gaur (2001)** reported that at given equilibrium concentration, the biomass takes up more metal ions at lower than at higher algal

doses. The decrease in biosorption process with increase algal dose could be explained as a consequence of partial aggregation of the algal mass at higher concentration, which results in the decrease in effective surface area for the adsorption (Karthikeyan *et al.*, 2007). Merrin *et al.* (1998) also suggested that the higher biomass concentration caused screen effect of dense outer layer, blocking the binding sites from metal ions, resulting in lower metal removal per unit biomass.

#### Effect of pH:

The pH of the solution influence significantly both metal binding sites on the cell surface and the chemistry of metal in solution (Dursun, 2006). Heavy metals are taken up from water predominantly by ion exchange. Carboxyl and sulphate groups have been identified as the main metal-sequestering sites in seaweed and, as these groups are acids, its availability is pH dependent (Cossich *et al.*, 2002). *Sargassum* sp. presents a high content of ionizable groups (carboxyl groups from mannuronic and guluronic acids) on the cell wall polysaccharides, which makes it, at least in theory, very liable to the influence of the pH (Antunes *et al.*, 2003). From Fig. 3, a trend of increasing chromium adsorption by the studied adsorbent with increasing pH could be observed for pH values in range of 1 to 3, where the removal percentage of metal increased from 51.48 to 99.36%. Consequently, the maximum removal efficiency for chromium was observed at pH 3, while it decreased when the pH exceeded this value. This may be due to that increased positive charge ( $H^+$ ) density on the sites of biomass surface at low pH values (pH 1 to 3) restricted the approach of metal cations as a result of repulsive force.



**Figure (3): Removal percentage of chromium by *Sargassum hornschurchii* dry biomasses at different pH values.**

In contrast, when the pH value increased, biomass surface was more negatively charged and the adsorption of the metal ions with positive charge ( $\text{Cr}^{6+}$ ) was reached maximum around pH 3. Similar interpretations were also reported in many literatures for metal biosorption (**Rezaee *et al.*, 2006; Rakesh *et al.*, 2010**). As the pH increased more than 3, anionic hydroxide complexes of the metal ions were formed and competed with the active sites leading to decrease in adsorption rate. This reasoning is in agreement with that of **Antunes *et al.* (2003)** and **Kumar *et al.* (2006 and 2007)**.

#### **Desorption and reuse of biosorbents:**

Biotechnological exploitation of biosorption technology for removal of heavy metal(s) depends on the efficiency of the regeneration of biosorbent after metal desorption (**Gupta *et al.*, 2000**), where this regeneration is crucially important for keeping the process cost down. Therefore effective, mild, non-polluting and cheap desorbing agents should be used for regeneration of biomass to guarantee its restoring close to the original state for use in multiple cycles with undiminished metal uptake, whenever it is possible.

Figure (4) showed the adsorption curves profiles of chromium for five times adsorption / desorption cycles using 0.1 M NaOH and 0.1 M HCl as desorption agents. It has been observed that throughout all adsorption / desorption cycles using any of the studied desorption agents, chromium adsorption significantly decreased following each cycle. After the first adsorption / desorption cycle using NaOH, the examined dried biomass achieved chromium removal percentage of 15.04%, which far less than that of the original state (96.76%). The case has been reverse using HCl, where 86.28% was the value of the chromium removal percentage. Accordingly, metal sorption by the biomass decreased greatly and slightly after each successive cycle using NaOH and HCl, respectively. After five sorption/desorption cycles, 91.8% and 22% decrease in sorption was occurred using NaOH and HCl, respectively. Taking into account the loss of biomass ( $\approx 15\text{--}20\%$ ) at the end of the fifth cycle, it is apparent that the metal sorbing ability of the biomass did not considerably diminish during repeated sorption / desorption cycles using HCl. This means that the regeneration process for chromium-loaded biomass was highly effective using HCl than NaOH. Generally, the superiority of HCl for desorption of metals from the biomass may be attributed to that its efficiency is based on the competition between its protons and the heavy metal ions bound to active sites, which will be released if elutant concentration is high enough. i.e., the metal ions were displaced from the biomass by acid protons showing probable involvement of ion exchange. Similar findings were obtained by **Lodeiro *et al.* (2006)**, who reported the acids as one of the best desorbent agents for metal desorption. Increasing the acidity generally leads to an

effective removal of metals from the biomass. As acids do not deteriorate *Oedogonium sp.* biomass, this finding may have considerable bearing on the use of acids for metal desorption (Gupta *et al.*, 2000; Gupta and Rastogi, 2008). On the other hand HCl is not only efficient in desorbing metal ions but is inexpensive as well.

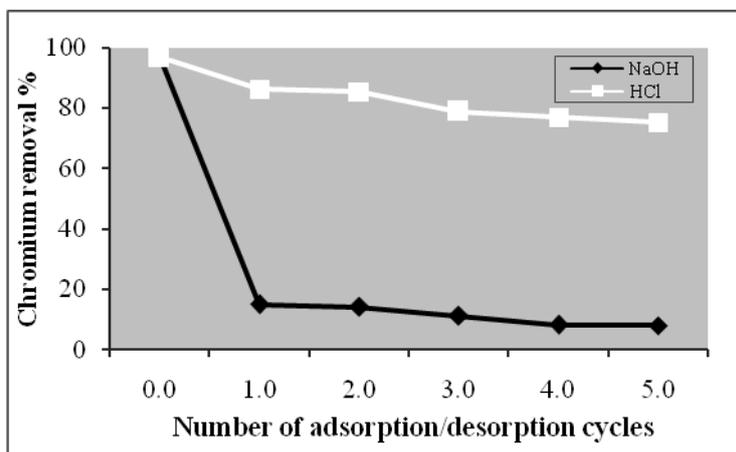


Figure (4): Effect of 0.1 M NaOH and HCl as desorption agents on the removal percentage of chromium by *S. hornschurchii* dry biomass

### Conclusion

The performance of Mediterranean brown macroalga *Sargassum hornschurchii* for the removal of Cr (VI) from aqueous solution was evaluated through batch experiments. The removal percentage of metal was estimated as a function of contact time, algal dosage and pH. It increased with increase in time and algal dosage and reached a plateau at 36 hrs and 8 g L<sup>-1</sup>, respectively. The adsorption process was solution pH dependent and a maximum adsorption of metal was determined at pH 3. The biomass was also employed for five times adsorption / desorption cycles with 0.1 M NaOH and 0.1 M HCl as desorbing agents. Lack of considerably diminishing in metal sorbing ability of the biomass during repeated sorption / desorption cycles using HCl favors it to be employed as a suitable metal desorbing agent for *Sargassum hornschurchii*. Consequently, dried biomass of *Sargassum hornschurchii* could be used as an alternative, inexpensive and effective material to remove high amounts of chromium ions from aqueous phase.

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## ادمصاص الكروم باستخدام الكتلة الجافة من الطحلب البني سارجسام هورنشوشاي

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تهدف هذه الدراسة الي ازالة عنصر الكروم سداسي التكافؤ باستخدام الكتلة الجافة من طحلب سارجسام هورنشوشاي تحت ظروف معملية. تمت دراسة تجريبية لمعرفة مدي تأثير بعض العوامل في عملية الادمصاص مثل وقت الاتصال، جرعة الكتلة الحيوية من الطحلب قيد الدراسة ودرجة الحموضة و ذلك باستخدام تركيز 25مجم/لتر من محلول الكروم. كان ادمصاص الكروم سريعا في المرحلة الأولى من الدراسة وتدرجيا مع مرور الوقت أصبح الادمصاص بطئ. في دراسة ذات مرجعية إحصائية بلغت نسبة الإزالة القصوى من الكروم 99.36% في 36 ساعة ودرجة حموضة 3 و ذلك باستخدام 8 جرام/لتر كوزنة جافة من الطحلب. تم تشغيل خمس دورات من الادمصاص / الاستعادة متتالية باستخدام 0.1مولر من هيدروكسيد الصوديوم و0.1مولر من حمض الهيدروكلوريك. وقد تبين أن ازالة الكروم انخفضت عقب كل دورة. أشارت النتائج الي تفوق حمض الهيدروكلوريك لاستعادة الكروم من الكتلة الحيوية الجافة للطحلب. و قد تبين من الدراسة الحالية أن طحلب سارجسام هورنشوشاي المستوطن في شواطئنا بالبحر المتوسط له قدرة كبيرة على ازالة الكروم من المحلول المائي.