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Design, Scaling, and Development of Biofilters with E *crassipes* for Treatment of Water Contaminated with Cr (VI)

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Abstract: The heavy metal water treatment process is the subject of worldwide research. Chromium (VI) is a heavy metal that is very dangerous to humans due to it being able to alter genetic material and cause cancer. Cellulose is an interesting material for removing heavy metals, and excellent removals have been achieved in many experiments at the laboratory scale. However, scaling these processes to polluting industries is not easy. The objective of this research is to design, scale, and test a biofilter with biomass of E *crassipes* transformed with iron for treatment of water contaminated with Cr (VI). The biomasses of E *crassipes* (EC) and E *crassipes* with iron (EC + Fe) were evaluated at the batch laboratory scale to determine the adsorption capacities through Langmuir isotherms. With these capacities, a mass balance was formulated, obtaining the design equation to build a biofilter at the pilot scale and providing the required amount of biomass from (EC) and (EC + Fe) for the adequate treatment of the Cr (VI) present in the water. The mass, as suggested by the relevant equations, for the greatest concentration of Cr (VI) of 500 mg/L was 42 g together with a flow rate of 10 mL/min for the biomass of (EC + Fe); for the biomass of (EC), the suggested model for the treatment of the greatest Cr (VI) concentration of 500 mg/L was 64 g of biomass together with a flow rate of 10 mL/min. We conclude that the two pilot-scale treatment systems were consistent with the Cr (VI) removal process and that the equation for the design was adequate.

Keywords: models; E *crassipes*; biofilter; chromium (VI)

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1. Introduction

Water care has become a necessity in environmental policies throughout the world due to the evident deterioration of rivers, lagoons, wetlands, etc. One of the many ways in which our planet's water is being negatively affected is through the outrageous pollution from industries.

Water decontamination is a fundamental goal in sustainable development policies, and due to this, new effective and cheap treatment systems must be researched, designed and developed for the proper care of this valuable resource [1].

Heavy metals are a clear contaminant of water resources. Metals such as mercury, chromium, and nickel, among others, cause serious damage to aquatic ecosystems as well as to human health [2]. Hexavalent chromium (Cr) (VI) is highly carcinogenic as it has the capacity to alter genetic material; due to this, care must be taken in the treatment of water contaminated with this heavy metal [3–6].

However, the conventional treatment of water is very expensive and difficult to carry out—for example, activated sludge, oxidation ditches, and membrane filtration, among others [7].

Biological filters with a static adsorbent material are an alternative for the adequate effective treatment of water contaminated with heavy metals, due to the efficiency, easy assembly, and cheapness of this treatment [8].

The adsorbent material that makes up biofilters can be the cellulose prevenient of waste material, as this biomass has multiple available sites (OH, CH_2, COOH) where heavy metals can house in chemical diffusion processes [9–11].

A native, invasive, and high-impact plant is *Eichhornia crassipes*; due to its high reproduction rate, it can overpopulate a wetland in just a few months from its arrival in the microsystem [12,13].

Currently, this biomass of E *crassipes* (EC) is wasted due to the lack of sustainability policies for its management. Additionally, owing to the high amount of cellulose, it could be used for the design and assembly of cheap and effective treatment systems to treat water contaminated with Cr (VI) from the tannery industry, as in, for example, [13–17].

To optimize the treatment process with the biomass of the (EC) plant, it can be treated with iron chloride; this chemical compound oxidizes the organic matter, creating more active sites and increasing the chemical affinity with heavy metals [18–21].

Tests to determine the removal efficiencies of heavy metals through biomass have been carried out at the batch laboratory scale where adsorption capacities and kinetics of adsorption were obtained [22–26].

Subsequently, these tests could be validated through pilot-scale tests, such as fixed column adsorption assessments, in an arrangement akin to that which is used to treat the effluents of industries [27].

In [28–30], the authors experimented on batch, determining the design parameters and scaling the results by developing fixed columns for the treatment of heavy metals with biochar. The mass balances were used to determine the amount of pollutant load that must be treated, thus achieving the discharge aims [31].

The objective of this research is to design, scale, and evaluate a biofilter with a biomass of (EC) transformed with iron for treatment of water contaminated with Cr (VI). Through the development of a biotreatment in batch, the adsorption capacities of Cr (VI) by (EC) and (EC + Fe) can be determined. With these capacities, a balance of mass was formulated, resulting in the equation of design of a build biofilter at the pilot scale; the removal efficiency of Cr (VI) was subsequently evaluated by part of the biofilter.

Until now, in the literature, biofilters with these characteristics have not been seen. Although the (EC) plant was using for the adsorption of Cr (VI) and Pb (II) in continuous processes [18,27], these design parameters of the treatment systems were not established. Therefore, the novelty of this study is the implementation of the balance of mass, which provides an equation to determine design variables in the construction of a continuous filter with biomass of (EC) and (EC + Fe).

2. Materials and Methods

2.1. Use of (EC)

The E *crassipes* plants were collected in a wetland in the Mosquera municipality on the outskirts of Bogotá D.C, and were then washed (EC) with distilled water, separating the root from the stem and the leaves. They were dried at 70 °C for 48 h to remove moisture and grind to a diameter of 0.212 mm. The pulverized biomass was sieved through a knife mill to get different sizes of particles.

2.2. Obtaining Cellulose Modified with Iron

Biomass with iron chloride was mixed with 1 g of cellulose (EC) and 200 mL of deionized water; then, 1 g of iron chloride with a pH of 3.0 was added. Then, the suspension was placed in a shaker for 3 h at 175 rpm and 50 °C to impregnate Fe (III) into the surface of the cellulose [23].

Then, the pH of the suspension was adjusted to 10.0 to form FeOOH on the cellulose surface using 40 mg/L NaOH. Finally, the cellulose loaded with iron was obtained by drying in an oven for 3 h at 60 °C. With this process (EC + Fe), was created.

2.3. Chromium Measurement

Cr (VI) laboratory measurement: 50, 100, 200 and 300 mg/L of Cr (VI). Samples were taken in the flask at each time interval, and the residual chromium concentration was analyzed. Then, 20 µm samples were taken and then placed in the centrifuge (KASAI MIKRO 200, Hettich, Föhrenstr Germany). In sampling, aliquots of the reaction mixture were analyzed for residual chromium concentration using a (UV84 Hettich, Föhrenstr Germany).

2.4. Batch Adsorption Experiment

Batch adsorption experiments were carried out in a 100 mL glass vessel with constant stirring (IKA Ks 4000 shaker, Hettich, Föhrenstr Germany) at 20 °C, 150 rpm. Data were recorded every 20 min for 180 min. The sample taken was 20 µm. All experiments were carried out in triplicate, with an average of the final values.

Spectrophotometer (Evolution 300 spectrophotometer, Hettich, Föhrenstr Germany) by monitoring changes in absorbance. All procedures for the determination of chromium, for water and substrates, were carried out using the APHA (Procedure of the American Public Health Association) for standard tests (standard methods for the examination of water and wastewater).

The 1000 mg/L Cr (VI) stock solution was prepared with distilled water using potassium dichromate. This stock solution was used to prepare the test solutions of 50, 100, 200 and 300 mg/L Cr (VI). The batch adsorption study was carried out at room temperature. The adsorption capacity was determined by suspending 0.3 g of biomass in 40 mL of Cr (VI) test solution for 140 min at 200 rpm, taking samples every 20 min and centrifuging at 15,000 rpm before determining the residue and discarding the sediment.

2.5. Continuous Experimentation

Depending on the capabilities presented in the batch experiment, the experimental design was developed to quantify the transformed biomass, volume, fluxes, and other design parameters of a biofilter.

3. Results

3.1. Mass Balance Process

In Figure 1, the representative scheme of the biological biofilter composed of material of (EC) and (EC + Fe) is shown. For the purposes of balance, the accumulation of polluting material in the biomass will be taken into account, as represented by Equation (1).

$$Acumulation = Input - output - chemisorption \tag{1}$$

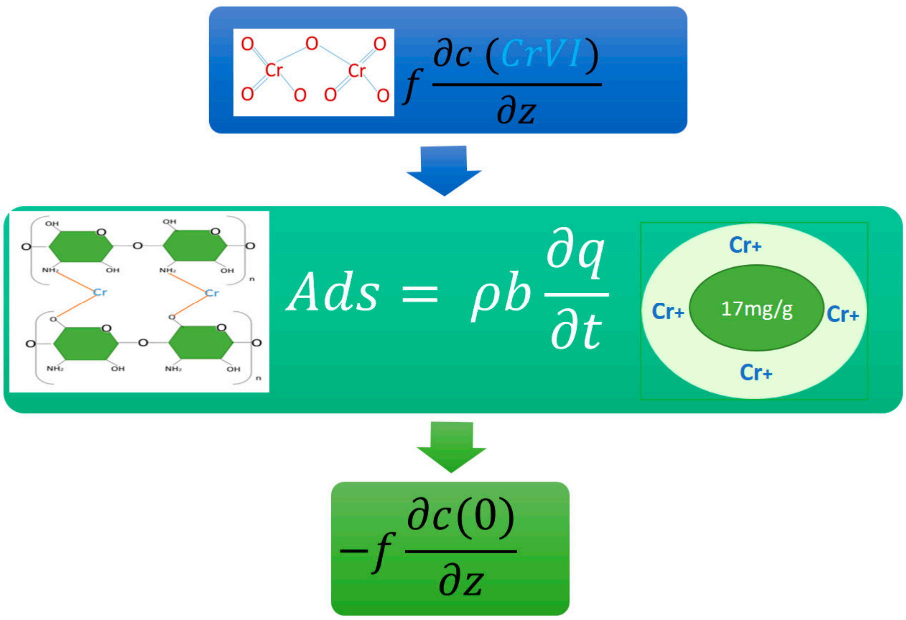


Figure 1. Representation of the filter model in the Cr (VI) treatment process—mathematical model of input, output and internal adsorption.

Each of these will be explained through differential equations, since changes (∂) occur over time in capacity (∂q), concentration (∂c) and porosity ($\partial \varepsilon$).

The input to the biofilter is related to the flow velocity (f), the initial Cr (VI) concentration in the treatment process and section of experimentation of biomass in the horizon longitudinal (z) of the biofilter.

$$Input = fCr(t,\ z) = fV\frac{\partial c(t,z)}{\partial z} \tag{2}$$

The output of the biofilter is related to the flow rate (f) and the final Cr (VI) concentration in the treatment process. Equation (3) assumes that the concentration end of Cr (VI) is (0).

$$Output = fCr(t,\ z) = -fV\frac{\partial c(t,z)}{\partial z} \tag{3}$$

In Equation (4), which is representing in the process of adsorption of the Cr (VI) contaminating material, it can be seen the change in the adsorption capacity (q) is given as a function of time and space within the filter. The particle density (p) and the total volume (V) of the water to be treated also play important roles.

$$chemisorption = \rho Ardz\frac{\partial q}{\partial t} = \rho V\frac{\partial q}{\partial t} \tag{4}$$

The accumulation represents the porosity of the biofilter (ε); this is the relationship between the particle density and the density of the filter medium and is represented by the following equation (Equation (5)).

$$Accumulation = \varepsilon V\frac{\partial c}{\partial t} \tag{5}$$

Equation (1) can give Equation (6), the general mass balance equation in the reactor that filters the water contaminated with heavy metals.

$$\varepsilon\frac{\partial c}{\partial t} = f\frac{\partial c}{\partial z} - \rho\frac{\partial q}{\partial t} \tag{6}$$

In Equation (6), the flow is considered f = $\frac{\partial z}{\partial t}$.

$$\varepsilon\frac{\partial c}{\partial t} = \frac{\partial c}{\partial t} - \rho\frac{\partial q}{\partial t} \tag{7}$$

In the initial treatment process, it could be said that all these design parameters are constant, leaving Equation (8).

$$m\ \frac{\partial q}{\partial t} = v\frac{\partial c}{\partial t} - \varepsilon v\frac{\partial c}{\partial t} \tag{8}$$

where the time (T) will be that of the rupture of the process, leaving Equation (9).

$$mq = QCoT - Co\varepsilon V \tag{9}$$

When clearing (m), Equation (10) remains.

$$m = \frac{QCoT}{q} - \frac{Co\varepsilon V}{q} \tag{10}$$

where Q is the flow rate used (ml/min), Co is the initial Cr (VI) concentration of the process mg/L, (V) is the volume, and (ε) is represented in [31,32]; relating the density of the particle and the density of the bed, together with the stipulated breakthrough curve (T), the parameter measured in batch will be (q), the adsorption capacity for the two biomasses.

Through Equation (10), the biomass necessary to build a biofilter was determined in order to guarantee the removal of Cr (VI).

3.2. Batch Experimentation

Next, the experiments carried out in batch are presented in order to find the parameter of the capacity of biomass to adsorb Cr (VI). Figures 2 and 3 show two adsorption processes, where four types of the initial concentration of Cr (VI) are used, evaluating only the biomass of (EC) in the case of Figure 2; Figure 3 shows the removal percentages by part of (EC + Fe).

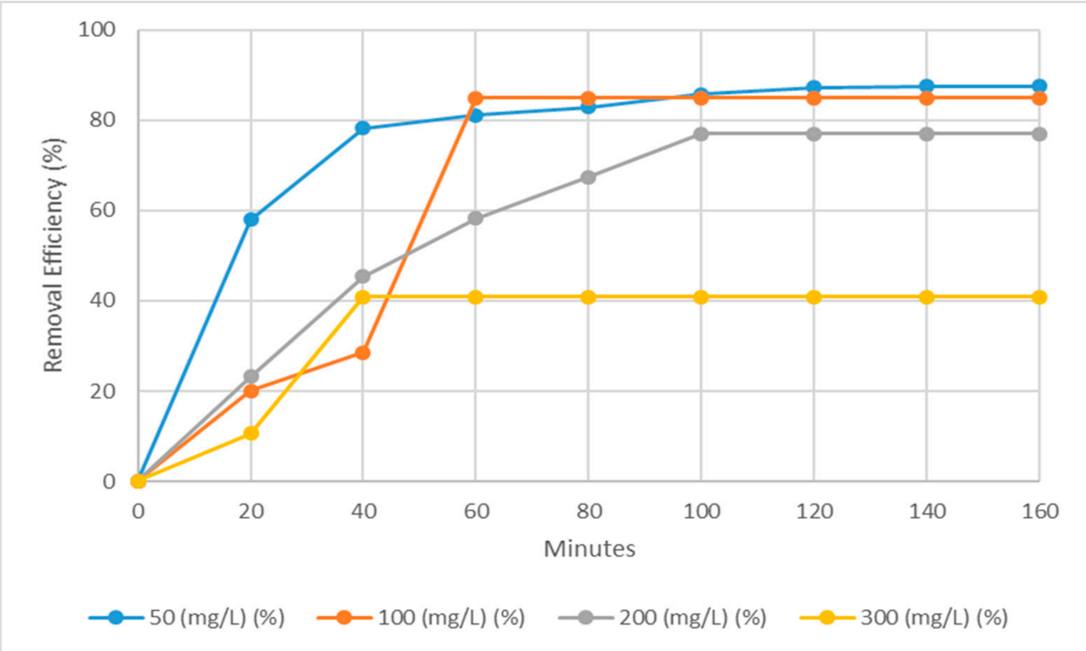


Figure 2. (EC) removal percentages.

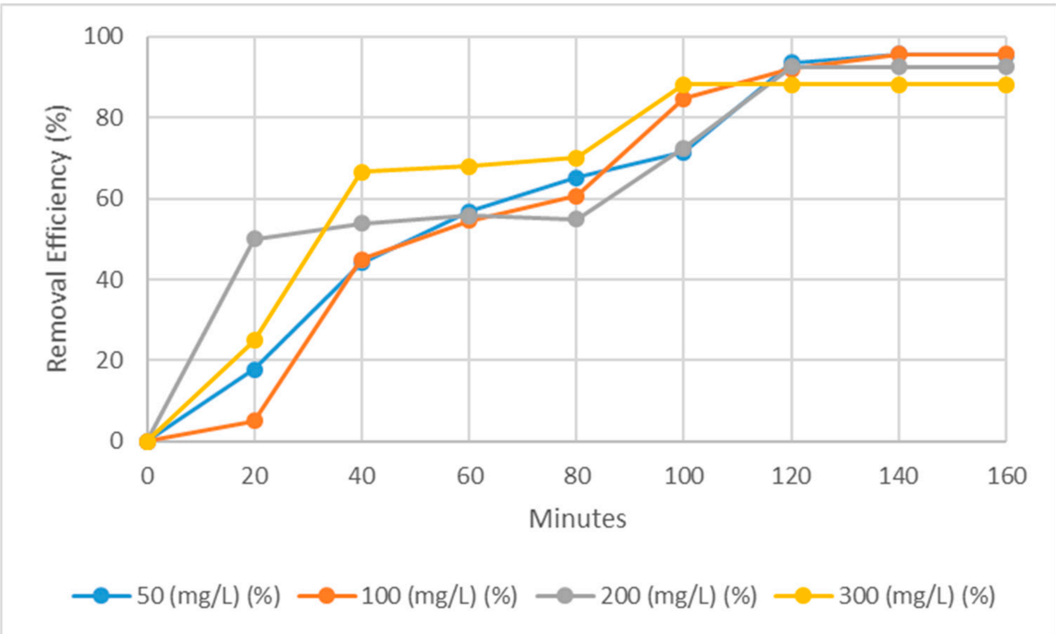


Figure 3. (EC + Fe) removal efficiency.

In the percentage removal of Cr (VI), the saturation of the biomass of (EC) removed only 40% of the initial concentrations of 300 mg/L. However, with the initial Cr (VI) concentrations of 200, 100, and 50 mg/L, significant removals of more than 80% could be observed. Figure 3 shows the removals of (EC + Fe).

Removals under initial concentrations of (EC + Fe) were significant; at minute 80, the initial concentrations of 50 and 100 mg/L were already above 60% removed, showing that removal and subsequent saturation is much greater than the vegetable cellulose of (EC), removing around 96% of the initial Cr (VI) in all concentrations.

Adsorption Isotherms

There were four different starting points—initial concentrations of 50, 100, 200, and 300 mg/L of Cr (VI). Each process yielded four final concentrations (C_e), which could be defined as the equilibrium concentration, and four final capacities (q), which could be called equilibrium capacity; with these clear parameters the Langmuir isotherms will be obtained [33,34].

Langmuir adsorption isotherms are shown in Figures 4 and 5, the graph of (C_e/q). C_e for Cr (VI) adsorption, representing the Langmuir equation; with this graph it was possible to obtain the maximum capacity (q) of adsorption of these biomasses.

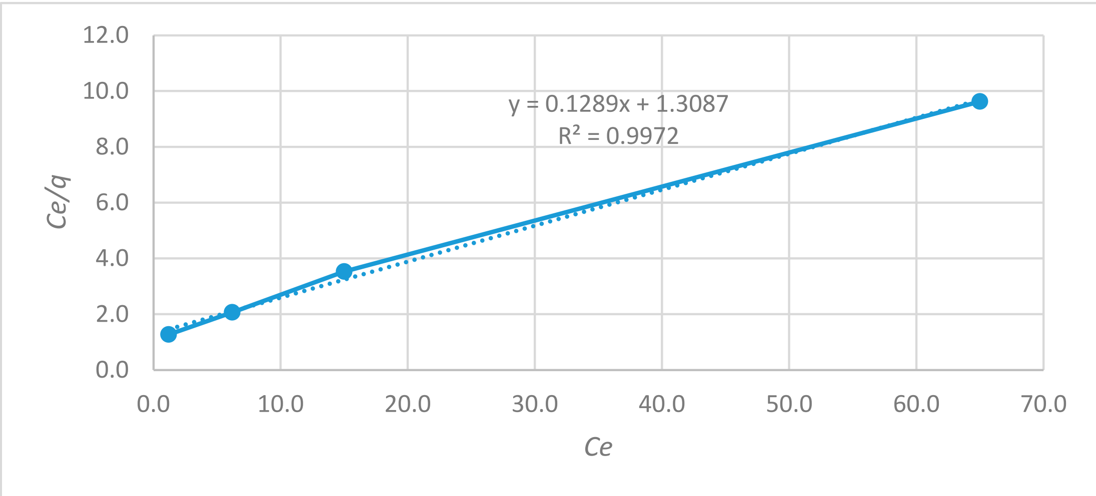


Figure 4. Langmuir isotherm EC.

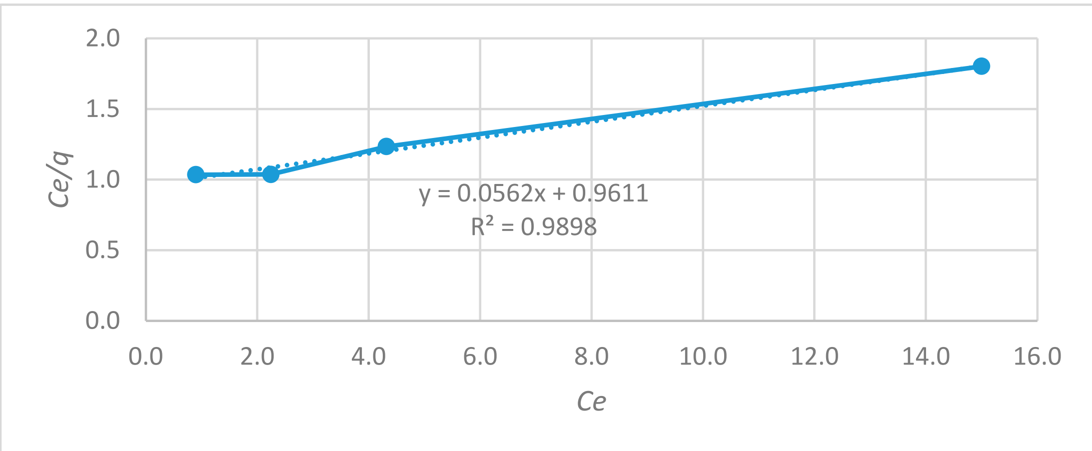


Figure 5. Langmuir isotherms (EC + Fe).

A representative fit of 0.992 R^2 can be observed. Taking as reference the Langmuir equation, which is:

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \left(\frac{1}{q_m B} \right) \quad (11)$$

The maximum capacity variable (q_m) was obtained by solving for the expression observed in Figure 4 y = 0.128x + 1.3087. The expression 0.128x is akin to $1/(q_m)C_e$, where the maximum capacity (q_m) of the biomass of (EC) obtained the design parameter of maximum Cr (VI) retention capacity by part of the biomass of (EC) of 7.66 mg/L; similar results were seen in the experiments reported by [8,34–36], where they used biomass of (EC) without modification. Figure 5 shows the Langmuir isotherms of the biomass experiment (EC + Fe).

Observing Figure 5, the maximum capacity was obtained from the equation of the line, clearing 0.0586, and the maximum capacity (q_m) of the cellulose of (EC + Fe) was obtained—17 mg/g; similar results were seen in the reported experiments [20].

Comparing (EC) and (EC + Fe), it can be concluded that the latter has a capacity almost 2.5 times greater (EC) as iron oxidizes the organic matter, creating more active groups favoring chemisorption.

The adsorption capacities obtained of (EC+Fe) and (EC) were using for the followed process of design of the biofilters.

3.3. Continuous Experimentation

For the development of the biofilter, equation (10) was used to find the biomass ideal in the removal of Cr (VI).

$$m = \frac{20\left(\frac{\text{ml}}{\text{min}}\right) * 0.5\left(\frac{\text{mg}}{\text{ml}}\right) * 92(\text{min})}{17\left(\frac{\text{mg}}{\text{g}}\right)} - \frac{0.5\left(\frac{\text{mg}}{\text{ml}}\right) * 0.5 * 250(\text{ml})}{17\left(\frac{\text{mg}}{\text{g}}\right)} \quad (12)$$

m = 42 g
Q = 20 mL/min
V= 250 mL
ε = 0.5 [32]
q = (EC + Fe) 17 (mg/g)
C_o = 0.5 mg/mL
T = 92 min

The biomass used in the biofilter was 42 g of (EC + Fe). The theoretical breakthrough curve for the biomass of (EC + Fe) was 92 min, and the adsorption capacity found in the batch helped to find the required biomass along with the design flow. This value represents the possible capacity that the biomass of (EC + Fe) will have to initially support 500 or 0.5 mg/mL.

For the biomass of (EC), it was calculated similar to (EC + Fe), but with an adsorption capacity of 7.66 mg/g, and the terms were replaced in the equation:

$$m = \frac{20\left(\frac{\text{ml}}{\text{min}}\right) * 0.5\left(\frac{\text{mg}}{\text{ml}}\right) * 55(\text{min})}{7.66\left(\frac{\text{mg}}{\text{g}}\right)} - \frac{0.5\left(\frac{\text{mg}}{\text{ml}}\right) * 0.5 * 250(\text{ml})}{7.66\left(\frac{\text{mg}}{\text{g}}\right)} \quad (13)$$

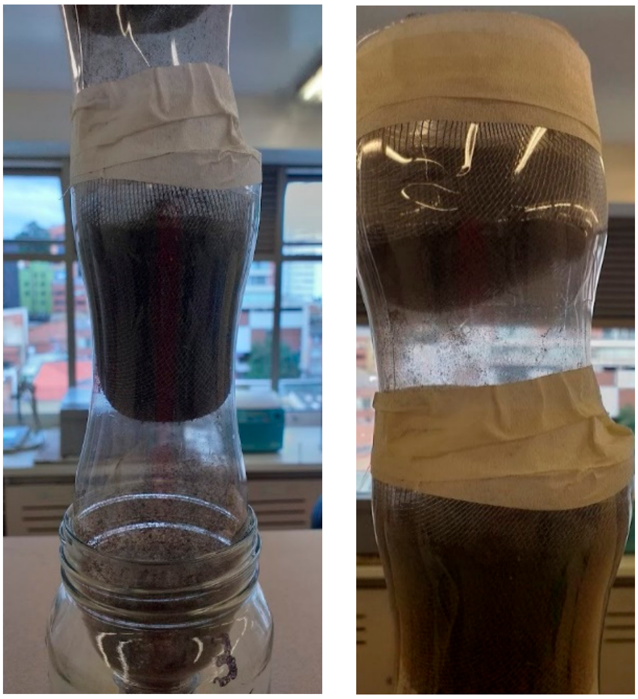
m = 64 g
Q = 20 mL/min
V = 250 mL
εb = 0.5 [32]
q = (EC) 7.66 mg/g
C_o = 0.5 mg/mL
T= 55 min

The biomass used in the biofilter was 64 g—biomass of (EC) to incorporate in the biofilter. The theoretical breakthrough curve for the biomass of (EC) is 55 min; due to it is greater capacity, the biomass of (EC + Fe) can last longer before saturation. For this reason, a higher biomass of (EC) was required. After determining how much biomass was needed and establishing the assembly conditions, the biofilter was developed with the biomasses.

3.4. Design and Construction of the Biofilter at the Pilot Scale

The biofilter was built with plastic (PET) bottles, where two compartments of 32 g were designed for the biomass of (EC) and for the biomass of (EC + Fe), 21 g. It had a fine nylon mesh and consisted of two beds in series, with a diameter of 3.5 cm and a height of 7.5 cm; it had orifices at the entrance to control the flow at 20 mL/min, with a pH 6 (aqueous solutions of 0.81).

The density of the bed was constant, and under temperature and pressure conditions of 20 °C and 1 bar, respectively, the system had manual flow control. Samples were taken when 100 mL passed through the system until the saturation of the system was completed. Then, the samples were analyzed in the atomic absorption equipment. It performance the experiment by duplicate, taking the resulting average in the figures. Figure 6 shows three photographs of the biofilter used in the process of remotions of Cr (VI).



(**a**) (**b**)

Figure 6. Biofilters used in the adsorption process: E *crassipes* (EC) (**a**); E *crassipes* with iron (EC + Fe) (**b**).

Experimental Process

Regarding the experimental process of the biofilter, the removals are shown in Figure 7, with a biofilter with biomasses of (EC) and (EC + Fe). At an initial Cr (VI) concentration of 500 mg/L, the (EC) biofilter efficiencies were 75%, with the breakthrough curve being at minute 55 when it had to treat around 300 mL of water. The biofilter with biomass of (EC + Fe) obtained efficiencies above 85% with initial concentrations of 500 mg/L or Cr (VI) and with a breakthrough curve at minute 100, treating around 400 mL of water.

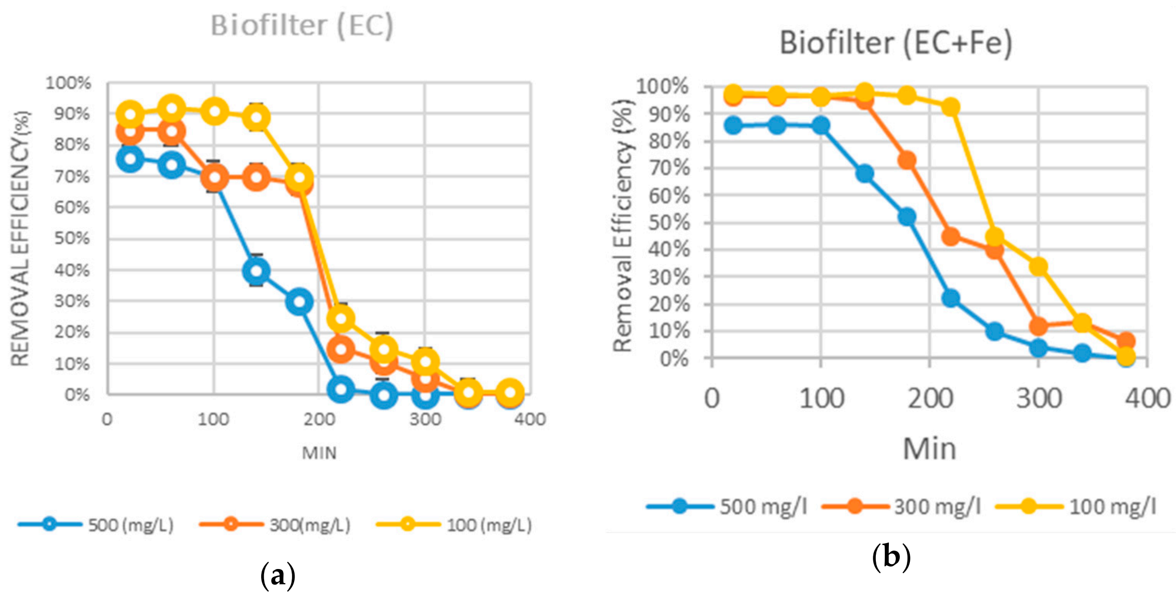


Figure 7. (EC) and (EC + Fe) removal percentages: Biofiliters EC (**a**); Biofilters EC + Fe (**b**).

Regarding the biofilter with (EC) in the treatment of initial concentrations of 300 mg/L, there were efficiencies of 85% of Cr (VI), with a breakthrough curve of 100 min, treating around 300 mL of water; compared to the yields of the biofilters under the same initial concentrations, the biofilter of (EC + Fe) removed 95% of the Cr (VI) in the water. The breakthrough curve was at minute 100, treating around 500 mL of water. The optimization of the biofilter was also evident with the initial concentration of 100 mg/L, where the biomass with (EC + Fe) was above 96% for the removal of Cr (VI) and the biofilter with this initial concentration was around 92% for the efficiency of this heavy metal. The treatment with iron (Fe), optimized the efficiencies of Cr (VI), showing more treatment time and treating about 25% more water. The experiment of [37] had an efficiency of 73% of Cr (VI) with the (EC); in [25], this biomass also had an efficiency of 85% for Cr (VI), and in [30] the efficiency of Cd (II) was 85% (see Table 1).

Table 1. Research with system in continuo with the different biomass.

Reference	Biomass	Initial Concentration mg/L	Removal Efficiency (%)	Heavy Metal Removed
Present article	EC	100	85	Cr (VI)
Present article	EC + Fe	100	98	Cr (VI)
[37]	EC	100	73	Cr (VI)
[25]	EC	100	85	Cr (VI)
[30]	EC	100	85	Cr (VI)
[18]	EC + Biochar	200	95	Cd (II)
[38]	Biochar	200	90	Cr (VI)
[39]	Algae	200	90	Cu (II)
[40]	Algae	300	85	Cu (II)
[41]	Almond shell	100	95	Cr (VI)
[42]	Biomass + Fe	100	96	Cr (VI)
[28]	Bacillus + Biochar	100	95	Cr (VI)
[42]	Biochar + Fe	100	98	Cr (VI)

Biochar removals are very interesting [18,28,38], but the energy expenditure is very expensive. Algae have cellulose compositions similar to (EC) [39,40], and in the development of a biofilter with this biomass, they presented interesting results when removing Cu (II), but (EC) is more abundant and easier to acquire than algae. The authors of [41] used almond shell, presenting removals above 95% to remove Cr (VI); a special combination designed was used in [42], where the realized composite with biochar and Fe removed around 99% of Cr (VI). The present research is based on some of the parameters used in [27]; the process in this study removed around 90% of Cr (VI) and 85% of Pb (II), with the biomass of (EC).

4. Discussion

The biofilter with (EC) is an economic alternative for industries with effluents contaminated with heavy metals, it is also easy to develop. The pilot-scale biofilter for treatment of almost 10 L of water with Cr (VI) would cost around USD $10 [1,27]. The iron-modified biofilter can be used in industry where the contamination of its effluents is very high. Although this biofilter is more difficult to develop, it can treat more complex pollutants. The pilot-scale biofilter, for treatment of almost 10 L, costs about USD $16 [1,23].

Equation (10) could be used for different treatment processes due to the adjustments to caudal, volume, mass, breakthrough curve, and capacities of remotions and relating the density of the particle to the density of the bed. With this equation, a system of treatment can be designed with the aim of removing different contaminates, as Equation (10) is not limited to removing the Cr (VI) alone and can also adjust to the limit of dumping.

Cr (VI) saturated biomass was finally disposed of as a hazardous waste. It is recommended to continue with sustainable projects for the production of biofuels with this material, taking advantage of its cellulose.

5. Conclusions

In the batch experiment carried out at the laboratory scale to determine the capacity parameter of the biomasses of (EC) and (EC + Fe), it was concluded that these biomasses are 7.66 mg/g and 17 mg/g, respectively. A mass balance was carried out with the goal of finding the equations ideal for design of systems at the pilot scale, determining the mass adequacy of (EC) and (EC + Fe) for the treatment of Cr (VI). The masses suggested that for a maximum Cr (VI) concentration of 500 mg/L, 42 g together with a flow rate of 10 mL/min for the biomass of (EC + Fe) was required, eliminating approximately 90% of the Cr (VI). For the biomass of (EC), the model suggested that, for the treatment of maximum Cr (VI) with a concentration of 500 mg/L, the biomass should be 64 g together with a flow rate of 10 mL/min, removing around 80% of the Cr (VI) in a liter of water. Both biofilters are able to adapt to the conditions of the effluents of industries—for example, the biofilter with biomass of (EC + Fe) can treat water with high concentrations of Cr (VI) for above 500 mg/L and the biofilter with (EC) could treat waters with concentrations of under 500 mg/L of Cr (VI).

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