

Biosorption of Cr(VI) from Aqueous Solutions Using Chemically Modified Okra Powder

Joelington do C. Conceição¹, Victor Hugo S. Ramos¹, Edilson de Jesus^{1,*}, Antônio Santos Silva² and Antônio Wilson M. de C. Costa³

¹Department of Chemical Engineering, Federal University of Sergipe, São Cristóvão-SE, Brazil

²Department of Mathematics, Federal University of Sergipe, São Cristóvão-SE, Brazil

³Federal Institute of Education of Sergipe, Aracaju-SE, Brazil

Abstract: The main objective of this work was to use the okra powder modified with citric acid as biosorbent for the removal of Cr(VI) from aqueous solutions. Samples of Cr(VI) solution were prepared by diluting the 100 ppm solution diluted from the 1000 ppm stock solution prepared with $K_2Cr_2O_7$. The determination of Cr(VI) was performed spectrophotometrically by the method of complexation with 1,5-diphenylcarbazide, measuring the absorbance at a 540 nm wavelength. The experiments were conducted at 25 ± 1 °C; 2, 5, 7 and 8 initial pH; 10-240 minutes contact time and 0.05 g of adsorbent. The Freundlich and Langmuir models were used to fit the experimental data. The Freundlich model provided $R^2=0.9599$, indicating adsorption on heterogeneous conditions. Analysis of fluorescence of X-rays, SEM and FTIR were used to prove the okra powder efficiency in the removal of Cr(VI) from aqueous solutions.

Keywords: Okra powder, biosorbent, removal of Cr(VI), equilibrium study.

1. INTRODUCTION

The problematic of obtaining water for consumption and adequacy of effluents within specifications for human consumption have instigated new technologies to enable not only meet legal standards, but also the improvement in processes efficiency.

The pollution caused by heavy metals from wastewater generated by various industrial sectors is one of the most serious problems in the environmental area [1]. Hexavalent chromium, which has toxic characteristics, is considered a quite aggressive pollutant to the environment [2, 3]. Cr(VI) is rarely found in natural waters, thus the water contamination by chromium is mainly due to the waste generated by industries such as the steel and cellulose industry and erosion from natural deposits [2, 4]. The treatment of these effluents is a wide field of research, especially with regard to the development of physicochemical treatment techniques [1].

Existing technologies to control heavy metals in environment include precipitation, ion exchange, solvent extraction, reverse osmosis and adsorption. The adsorption process has been adopted for being effective and economical in the removal of metals in waste water [3].

Many studies show removal and recovery of heavy metals with different biosorbents. Some industrial wastes and biological materials have shown potential in removing pollutants. Among those of biological origin with ability to adsorb dissolved metal ions [5], there are the cactus [6], bagasse [7], *cyanobacterium* [8], green algae [9], *Oedogonium* sp. and *Nostoc* sp. [10], walnut shells [11], Osage orange [12], Brown Seaweeds [13] and *Moringa Oleifera* [14].

The okra (*Abelmoschus esculentus*) is widely cultivated in the tropics and subtropics, with immature fruit eaten as a salad, steamed and baked. The seeds are rich in oil and protein. According Abreu-Lima [15] and Amaya-Farfan [16] main chemical constituents are vitamin A, vitamin B2 and B6, calcium, alanine, alpha-tocopherol, arginine, ascorbic acid (vitamin C), aspartic acid, glycosides, acid glutamic, gossypol, histidine, isoleucine, leucine, linolenic acid, myristic acid, oleic acid, palmitic acid, pantothenic acid, pectin, quercetin, riboflavin, starch and stearic acid.

The main advantage of using okra powder is its natural composition. In addition, the powder may be produced from mature fruits often rejected by consumers. Among the main disadvantages is the increased organic matter in the water, which can increase the amount of oxidants [17].

Thus, this work uses okra powder modified with citric acid (CA) for Cr(VI) removal from aqueous solutions. A kinetic study on the sorption process and operational parameters such as the influence of pH,

*Address correspondence to this author at the Marechal Rondon Avenue, Cidade Universitária Prof. José Aloísio Campos, Jardim Rosa Elze, CEP 49100-000, Brazil; Tel: +55-79-21056676; Fax: +55-79-21056890; E-mail: edilsonjs@ufs.br, ejs201107@gmail.com

amount of adsorbent, adsorption time and final concentration in chromium (VI) removal have been investigated.

2. MATERIALS AND METHODS

2.1. Synthesis of Biosorbent

The okra powder used in this study was obtained by drying the okra pods in a Sterilifer model SX 1.0 greenhouse at 150 °C for 4 hours. After drying, the okra pods were crushed in common food processor. The chemical modification of okra powder was performed with 5 g of citric acid (CA) in 250 mL of distilled water. 6 g of okra powder 12 mesh were mixed to the CA solution. The mixture was left in contact with the solution of citric acid for 24 h in oven at 60 °C until evaporation of all the liquid present. The formed product was separated by filtration and then washed several times with distilled water and dried in oven at 60 °C for 2 h.

2.2. Determination of Acidity

1.0 g biosorbent with 12 mesh particle size and 200 mL of distilled water were stirred for 24 h using a magnetic stirrer. After stirring, 10 mL-aliquot was withdrawn to a 100 mL-flask and the volume was completed with distilled water. This solution was titrated with 0.01 M NaOH to the phenolphthalein end point [18].

2.3. Elemental Analysis

The elemental composition was performed by using CNH analyzer Flash EA 1112 Series from Thermo Finnigan. The composition of carbon, nitrogen and hydrogen was carried out at 900 °C under atmosphere of O₂ (oxygen gas) and N₂ (nitrogen gas).

2.4. FTIR Characterization of Biosorbent

The FTIR spectrums of okra powder was studied before and after Cr(VI) biosorption. A Nicolet model iS-10 FTIR Spectrometer was used between 400 and 4000 cm⁻¹ with 4 cm⁻¹ resolution. Samples were analyzed in the form of tablets with KBr.

2.5. Adsorption Experiments: Estimation of Chromium by Diphenylcarbazide Complex Method

Cr(VI) solutions were obtained at concentrations of 0.2, 0.4, 0.6, 0.8 and 1.0 ppm for the drawing the calibration curve. A purple-violet-colored complex was developed in the reaction between hexavalent

chromium and 1,5-diphenylcarbazide in acidic condition [18]. Absorbance was measured at 540 nm wavelength. A standard graph of absorbance vs. concentration was obtained to determine chromium concentrations. The unknown concentration was determined by the absorbance value of sample using UV-Vis spectrophotometer model Cary 60 from Varian. The percentage of hexavalent chromium removal was determined by Equation 1.

$$\% \text{ Chromium removal} = \left[\frac{(C_i - C_e)}{C_i} \right] \times 100 \quad (1)$$

where C_i and C_e are the initial and equilibrium concentration of hexavalent chromium solution (in milligrams per liter), respectively. Removal experiments performed under different conditions: conditions of initial Cr(VI) concentration 2, 3, 4, 5 and 6 ppm; 0.05, 0.1, 0.2, 0.3 and 0.4 g adsorbent amount, 2, 5, 7, 8 pH, 10-240 minutes contact time and 25 ± 1 °C temperature.

2.6. X-Ray Fluorescence Spectroscopy

The biosorbent's chemical composition before and after biosorption was determined by X-ray fluorescence. The excitation energy used was 20 keV. Samples from okra powder before and after biosorption were placed into a bucket and covered with a polypropylene film of 5 µm in thickness using a Bruker Spectrometer Model S8.

2.7. Scanning Electron Microscopy

SEM micrographs of the okra powder before and after adsorption of Cr (VI) were obtained. The measurements were carried out in a Scanning Electron Microscope JEOL SEM Model 5700 JCM-Carry Scope, vacuum, accelerated beam of 5 kV. All samples were previously metallized with gold (12 nm average thickness).

3. RESULTS AND DISCUSSION

3.1. Chemical Modification and Determination of the Acidity of the Modified Okra Powder

The average acidity in modified okra powder was 0.9094 mmol/g. The okra powder is natural organic matter, consisting primarily of hydrophilic colloids, low-molecular-weight acids, proteins and polysaccharides, humic acids and fulvic acids. These polysaccharides (natural polymers) are sometimes effective to the

synthetic flocculants [19, 20] The fruit of okra is a capsule filled with viscous white and round seeds, which has a high nutritional value and nutrient utilization as a complete source of fiber, calcium, minerals and vitamins [21]. The seeds represent 17 % of the fruit and consist of oils (which can reach up to 40 %) and protein (> 15 %), with a duration of 5 years germline [22, 23] tested several cultivars of okra, the starch content ranged from 0.98 to 1.23% and the total sugars from 1.91 to 2.16 % in fresh matter, and the starch content was higher than the overall mean (0.52%) described in the literature [24]. The okra pods used in this paper after drying show up quite fibrous, as can be verified by the SEM analysis, which is in agreement, the average crude fiber content in different cultivars was 0.9 to 1.1 % [23]. Although okra is not a rich source of carbohydrates, fresh fruit offers fiber, protein and vitamin C to human nutrition, and the seeds that are mainly sources of protein and oils [23, 25]. Modification of Okra powder using citric acid led to the formation of carbonyl groups in the material due to the esterification of citric acid [11].

3.2. Elemental Analysis (CHN)

The elemental analysis indicated contents of C, H and N equal to 14.04 ± 0.2 ; 41.16 ± 0.2 and $0.81 \pm 0.10\%$, respectively. The CHN elemental analysis did not allow describing molecular formulas since okra powder is a natural compound made up of several substances (proteins, polysaccharides and organic acids). Elemental analysis allows us to qualify and quantify the sample, identifying the presence of carbon, nitrogen, hydrogen atoms. The percentages of carbon, hydrogen and nitrogen suggest the presence of organic compounds such as polysaccharides and proteins in the okra powder [23, 25].

3.3. pH Effect of on Biosorption

Chromium has different types of equilibrium depending on the pH in aqueous solutions. The adsorption of chemical species is strongly affected by the pH in the medium. The adsorption of Cr(VI) was more effective in acid medium, showing a removal at pH 2.0.

In this work, the pH influence on the Cr(VI) removal was studied in the initial concentration of 5 ppm and pH 2.0, 5.0, 7.0 and 8.0, as shown in Figure 1. The removal percentage was higher at pH 2.0. As there was pH variation, there is change in the balance of characteristic species of hexavalent chromium. In the

pH range 2.0-6.0 are predominant the HCrO_4^- species and Cr_2O_7^- ions are in equilibrium. It is noted that at pH 2.0 there was greater Cr(VI) removal; this is probably due to the protonation of the adsorbent surface and the pH increase promotes competition between OH^- and Cr(VI) species. At pH 2.0 the removal was approximately 45 % Cr(VI).

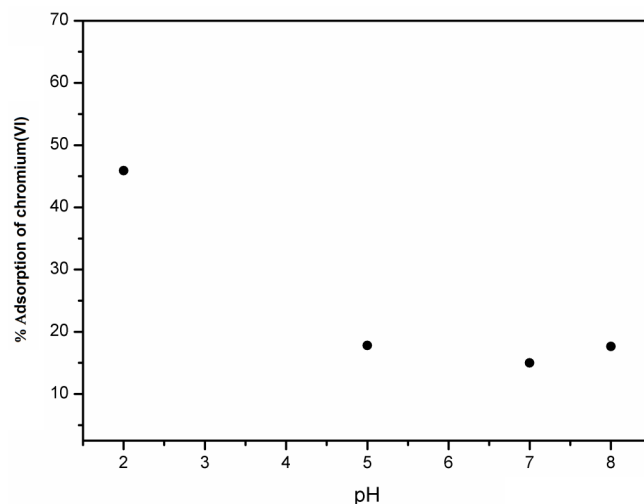


Figure 1: Effect of pH on the sorption of Cr(VI) by okra powder. (Conditions; initial concentration of Cr(VI), 5 ppm; amount of biosorbent, 0.05 g; 12-mesh particle size; volume of biosorption medium, 100 ml; temperature, 25 ± 1 °C; stirring time, 200 min; stirring rate 180 rpm).

3.4. Contact Time Effect

The balance in the Cr(VI) removal can be explained by the use of the entire active site of okra powder. During adsorption, the hexavalent chrome is

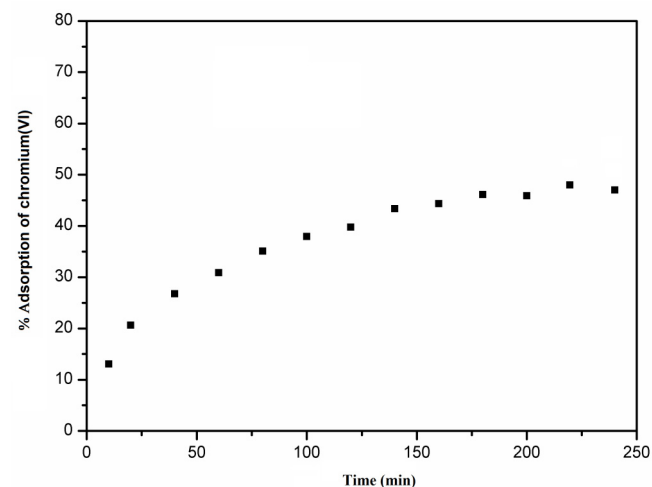


Figure 2: Effect of contact time on the sorption of Cr(VI) by Okra powder. (Conditions; initial concentration of Cr(VI), 5 ppm; amount of biosorbent 0.05 g; 12-mesh particle size; volume of biosorption medium, 100 ml; temperature, 25 ± 1 °C; stirring rate 180 rpm; initial pH 2.

impregnated on the adsorbent surface until it is entirely covered with metal ions, at this point the Cr(VI) concentration in the solution ceases to decrease.

Figure 2 shows the equilibrium curve constructed from the removal percentage depending on the contact time. The balance time determined was 200 min.

3.5. Effect of the Adsorbent Mass

In the study on the behavior of Cr(VI) reduction with increasing biosorbent mass was used 100 mL of chromium with an initial concentration of Cr(VI) 5 mg L⁻¹, pH 2, 25 ± 1 °C and 200 minutes contact time.

Figure 3 shows that Cr(VI) adsorption increases with increasing adsorbent mass. This behavior is attributed to the increase in the number of active sites. The okra powder is a material consisting mainly of organic substances (hydrophilic colloids, proteins, polysaccharides and carboxylic acids). These compounds have carboxyl groups, carbonyl and hydroxyl that may be involved in the adsorption of metallic pollutants from aqueous solutions.

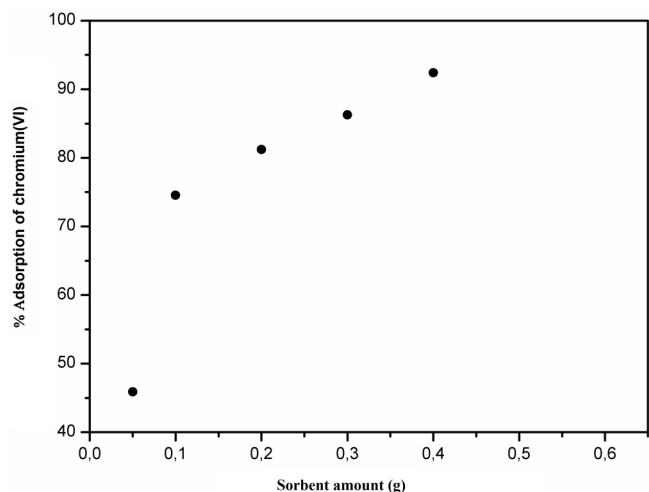


Figure 3: Effect of biosorbent amount on the sorption of Cr(VI) by Okra powder. (Conditions; initial concentration of Cr(VI), 5 ppm; volume of biosorption medium, 100 ml; temperature, 25 ± 1 °C; stirring time, 200 min; stirring rate 180 rpm; initial pH 2).

3.6. Adsorption Isotherms

From the experimental data it was possible to evaluate the adsorption capacity of the ion Cr(VI) in solution. Experiments to determine the adsorption parameters were at operational conditions: conditions of initial Cr(VI) concentration 2, 3, 4, 5, and 6 mg/L, amount of adsorbent 0.05, pH 2, 200 min contact time and 25 ± 1 °C.

In the literature several models are used for the determination of kinetic parameters of adsorption, with the most important being the Freundlich and Langmuir [12, 26, 27].

Equation 2 represents the Freundlich isotherm, which is widely used to accurately describe experimental results of adsorption kinetics. Equation 3 Represents the Freundlich isotherm in the linear form.

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

or

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \quad (3)$$

where Q_e is the amount of adsorbate per unit of adsorbent (mmol of adsorbate per gram of adsorbent); C_e is the concentration of adsorbate in the equilibrium (mmol/L); K_f and n are coefficients to be determined experimentally. K_f corresponds to the Freundlich constant and it is related to the biosorption ability and n relates to biosorption intensity. Values of n in the range $1 < n < 10$ indicate favorable biosorption.

For fixed values of C_e and $1/n$, q_e will be as greater as K_{ad} and C_e , binding of biosorption will be stronger as smaller the value of $1/n$. The lower $1/n$, the biosorption capacity is independent of C_e and the biosorption isotherm (ratio between C_e and Q_e) is close to the horizontal. The higher the $1/n$ value the smaller the binding intensity on the biosorption.

The Langmuir isotherm is given by

$$Q_e = \frac{q_{max} C_e b}{1 + b C_e} \quad (4)$$

or

$$\frac{C_e}{Q_e} = \frac{1}{b q_{max}} + \frac{1}{q_{max}} C_e \quad (5)$$

where b (L/mmol) and q_{max} (mmol/g) are coefficients to be determined experimentally.

The q_{max} coefficient corresponds to the surface concentration in a single coating layer; the coefficient b is related to the adsorption energy and increases with increasing binding adsorption strengths. Values of b and q_{max} can be determined experimentally by constructing the curve $1/Q_e$ versus $1/C_e$.

Figure 4 shows the linearized Freundlich isotherm built using the Equation 3, and with experimental data or experimental results. From the slope, the values of Freundlich parameters were calculated and arranged in Table 1. The value of n was 1.062 indicating that adsorption is favorable [26].

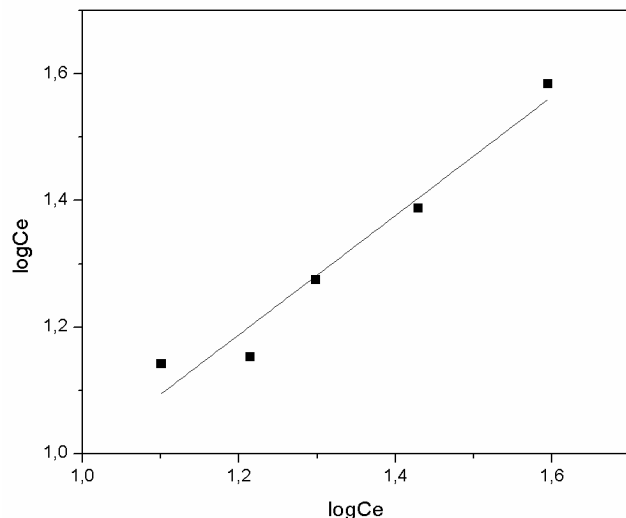


Figure 4: Freundlich isotherm for adsorption of Cr(VI) onto okra powder (Conditions: initial concentration of Cr(VI), 2, 3, 4, 5 e 6 ppm; amount of biosorbent, 0.05 g; 12-mesh particle size, volume of biosorption medium, 100 ml; temperature, 25 °C; stirring time, 200 min; stirring rate 180 rpm; initial pH 2.

Table 1: Freundlich Isotherm Parameters

Parameters	values
R^2	0.9599
$\log K_f$	0.0593
$1/n$	0.9409

Several studies involving physical and chemical modification of biological materials have been developed. Owlad *et al.* [28] offer a review that indicates the advantages and disadvantages of modification methods. In chemical modification using acid, a disadvantage is the closure of pores of the material.

3.7. IR Spectra of Modified Okra Powder

Infrared analysis of the treated okra powder was performed before and after the adsorption (Figure 5) in order any changes in the biomass surface when binding with the metal was observed.

The broad and strong band at 3400 cm^{-1} was attributed to the -OH group of carboxylic acids [11, 12]. The peak at 2922 cm^{-1} indicates the C-H vibration of

CH_2 group [12]. The peak observed at 1735 cm^{-1} was assigned to bending vibration of the ester's C=O. The band around at $1645\text{-}1650\text{ cm}^{-1}$ was attributed to the C=O vibration of carboxylic acids and N-substituted amide. Altun *et al.* [11] associate the variable bands around $1350\text{-}1480\text{ cm}^{-1}$ to bending vibration of the -C-H alkane group. The peak around 1374 is associated with carboxylate group ($-\text{COO}^-$). The peaks 1300 to 1000 cm^{-1} are assigned generally to the C-O stretching vibration in carboxylic acids and alcohols [29].

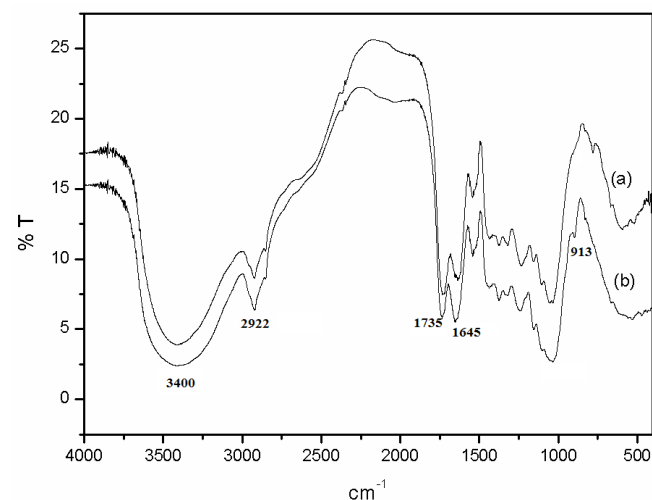


Figure 5: FTIR spectra of biosorbent (a) okra powder - Cr(VI) (b) okra powder.

After adsorption, the interaction of Cr(VI) with organic groups present on the powder surface was evident, with reduction in the transmittance percentage of bands that indicate hydroxyl (-OH) and carbonyl (C = O) groups of carboxylic acids, indicating the involvement of these groups in the Cr(VI) adsorption. The peak indicating adsorption of hexavalent chromium was identified by Santhana *et al.* [30] at 913 cm^{-1} , attributed to the possible bond Cr = O.

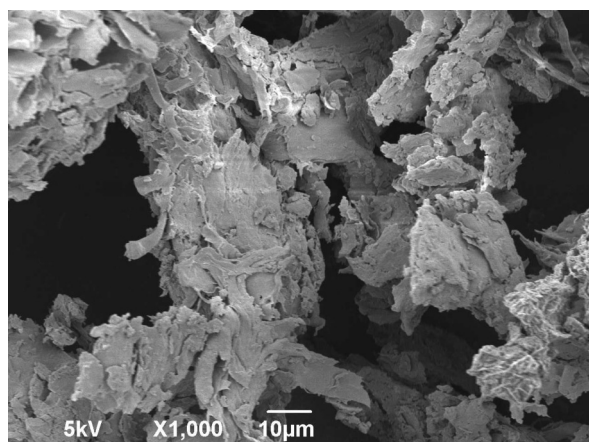
3.8. X-Ray Fluorescence

The main elements expressed in oxides found before and after biosorption. It is noted the appearance of chromium in the biosorbent after adsorption. The analysis of fluorescence of X-ray by dispersive energy (XRF) showed the presence of chromium after biosorption. The main elements expressed as oxides found before biosorption were 29.20% CaO, 5.16% SO_3 , 43.2% K_2O , 10.01% P_2O_5 , 2.49% MgO, 1.76% SiO_2 , 1.25% Fe_2O_3 . After biosorption was found 6.43% CaO, 18.01% SO_3 , 7.45% P_2O_5 , 4.65% MgO, 9.39% SiO_2 , 26.17% Fe_2O_3 and 17.76% Cr_2O_3 . There was hexavalent chromium adsorption by biosorbent. The

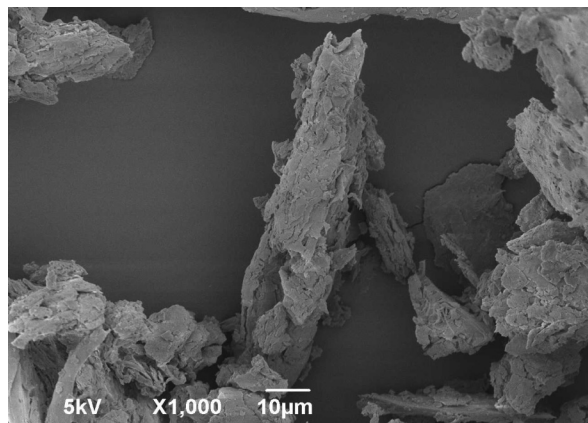
infrared analysis showed that the okra powder surface and organic groups, such as hydroxyl and carbonyl, were involved in the adsorption because there was a change in the transmittance intensity.

3.9. Scanning Electron Microscopy

The surface of the modified okra powder in certain regions has porosity (Figure 6a) showing a structure of networks. Clustering (Figure 6b) on the okra powder surface was observed after with obstruction of porous network indicating the interaction of the chromium with the powder surface after adsorption.



(a)



(b)

Figure 6: SEM micrographs of okra powder (a) before biosorption (b) after biosorption.

4. CONCLUSIONS

Considering the presented results it can be concluded that there was Cr(VI) removal by the okra powder, and the Freundlich model fitted well to describe the adsorption behavior, indicating a multilayer adsorption. The okra powder presented structure containing organic functional groups. The n value was 1.062, following the Freundlich model that

indicates the adsorption is favorable. These results indicated that okra powder could be employed as a low-cost adsorbent in the Cr(VI) removal.

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