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Competitive adsorption of Cr(III) and Cr(VI) by sugarcane bagasse magnetic nanocomposite in water matrix: A pH study.

Adsorção competitiva de Cr(III) e Cr(VI) por bagaço de cana-de-açúcar nanomodificado magneticamente em meio aquoso: Um estudo de pH.

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RESUMO: A biossorção é um processo ecologicamente correto, com baixo custo, alta rapidez e eficiência na remoção de contaminantes, entre os quais, o crômio se destaca em razão de suas diversas aplicações industriais. Por seus efeitos adversos e pela grande quantidade de efluentes industriais contendo crômio, novas técnicas são propostas para a descontaminação de ambientes aquáticos. Dessa forma, esse trabalho propõe utilizar um biossorvente de bagaço de cana-de-açúcar, resíduo da indústria sucroalcooleira, na forma *in natura* (SB) e nanomodificado (SB-NP), para avaliar a capacidade de remoção de crômio, nas formas mais abundantes, Cr(III) e Cr(VI), em matrizes aquosas. O potencial de descontaminação de águas por estes materiais é avaliado em processo por batelada, investigando-se a sorção dos íons Cr(III) e Cr(VI) de forma monoelementar e competitiva, em função do pH, por Espectroscopia de Absorção Atômica com Chama (FAAS) e Espectrofotometria de Absorção Molecular na Região Ultravioleta-Visível (UV/Vis). Na sorção individual, o melhor resultado de Cr(VI) para SB e SB-NP ocorreu em pH 1, com remoção de 85 e 84% deste contaminante, respectivamente. Para a sorção de Cr(III), o valor de melhor pH de sorção foi 6, atingindo-se remoção de 60% e 40% para SB e SB-NP, respectivamente. Na sorção simultânea, a maior eficiência de remoção foi obtida em pH 4, com remoção de 27% para SB e 52% para SB-NP. Em função do pH, o Cr(III) e Cr(VI) podem ser oxidadas ou reduzidas, o crômio hexavalente pode ser reduzido a Cr(III) devido à oxidação da matéria orgânica na superfície do biossorvente.

Palavras-chave: Biossorvente. Nanomodificação. Descontaminação de água. Sorção.

ABSTRACT: Biosorption is an eco-friendly process with low cost, high speed, and efficiency in removing contaminants, among which chromium stands out due to its various industrial applications. Due to its adverse effects and the large number of industrial effluents containing chromium, new techniques have been proposed to decontaminate aquatic environments. The present work proposes to use sugarcane bagasse as a biosorbent, a residue from the sugar and alcohol industry, *in natura* (SB) and nanomodified (SB-NP), in order to evaluate the chromium removal capacity, in their most abundant forms, Cr(III) and Cr(VI), in aqueous matrices. The potential of these materials in water decontamination is evaluated in a batch process to investigate the effect of pH individual or competitive sorption of Cr(III) and Cr(VI) ions. Chromium was determined by Flame Atomic Absorption Spectroscopy (FAAS) or Ultraviolet-Visible Molecular Absorption Spectrophotometry (UV/VIS). In the individual sorption of these metal ions, the best Cr(VI) result for SB and SB-NP occurred at pH 1, with the removal of 85 and 84% of this contaminant, respectively. For Cr(III) sorption, the best sorption pH value was 6, reaching up to 60% and 40% removal for SB and SB-NP, respectively. In simultaneous studies, the highest removal efficiency was obtained at pH 4, with the removal of 27% and 52% for SB and SB-NP, respectively. Depending on the pH, Cr(III) and Cr(VI) may change their oxidation state, and hexavalent chromium can be reduced to Cr(III) due to the oxidation of organic matter on the surface of the biosorbent.

Keywords: Biosorbent. Nanomodification. Water decontamination. Sorption.

INTRODUCTION

The use of biomasses as biosorbents becomes advantageous due to their abundance, high sorption capacity for cationic and anionic species, and low cost (Milani et al., 2018a). A promising adsorbent has many active sites available for interaction with substances under analysis (Milani et al., 2018a; Silva et al., 2014). The sites are places where adsorption occurs and are formed by one or more functional chemical groups (Moreira, 2010). The performance in the sorption of a metal ion by the biosorbent involve parameters such as the presence of competing ions, the adsorbent nature and surface area, temperature, pH, and the ion concentration (Silva et al., 2014).

The compounds most used in the adsorption of metal ions include secondary products of the industry, such as fungi, algae, bacteria, plants, and animals (Labuto and Carrilho, 2016). Sugarcane bagasse is a fibrous residue formed by several components, including cellulose, hemicelluloses, and lignin, ashes, and a small number of extracts that show strong attractive force for the connection with these ions (Milani, 2017). This material is formed by functional groups such as carboxylic acids, alcohols and exhibits great affinity for metal ions (Sarker et al., 2017). In addition to these favorable characteristics, there are large quantities of this waste in Brazil, therefore having a low cost (Silva, 2017). Currently, in order to reduce costs and environmental impacts, SB has been studied as a potential adsorbent in their products *in natura* and modified forms (Carvalho et al., 2020; Abilio et al., 2021; Milani et al., 2018a; Milani et al., 2018b; Do Carmo Ramos et al., 2016).

Nanotechnology has application in the decontamination of the environment due to its high chemical reactivity. However, it is rarely applied in contaminated fluid media (Paschoalino et al., 2010). Thus, a new area has emerged, which combines nanotechnology with biosorption, which can be an alternative to leverage the bioeconomy, producing hybrid materials with their characteristics and more suitable for specific uses (Labuto and Carrilho, 2016).

Chromium is a transition metal with several oxidation states, and Cr(III) and Cr(VI) are the most abundant and stable. In trivalent form, it is not toxic, being essential for human metabolism, as it acts on the metabolism of lipids, glucose, and proteins. In contrast, in the hexavalent form, it is highly oxidizing, toxic, and carcinogenic. However, Cr(VI) is quickly reduced to Cr(III) by reducing agents or organic compounds (Pina, 2011). Due to Cr(VI) toxicity, redox speciation of chromium has become essential for adequate environmental monitoring.

This work aimed to evaluate the potential of SB and SB-NP as efficient biosorbents for the removal of Cr(III) and Cr(VI), a sustainable alternative in the decontamination of aquatic environments, with a low cost for using waste from local alcohol industries.

MATERIAL AND METHODS

Preparation of *in natura* (SB) and magnetic nanomodified sugarcane bagasse (SB-NP)

Firstly, sugarcane bagasse from agroindustrial waste was washed with distilled and deionized water to remove particulate solids and impurities and was dried in an oven at 50° C. The resulting material was ground to 1 mm particle size and used as an *in natura* biosorbent and to prepare the nanocomposite SB-NP.

For the synthesis of magnetic nanoparticles (NP), the coprecipitation method was used. It consists of mixing $FeCl_2$ and $FeCl_3$ solutions, in an acidic medium, in a 1:2 molar ratio, under constant agitation. Then, 0.7 mol/L NH_4OH solution was slowly added to obtain the magnetite (Reaction 1). The synthesis of the SB-NP nanocomposite was performed by adding SB into the NP suspension at 5:1 SB:NP ratio and kept under heating at 80°C for 30 min. The resulting magnetic nanocomposite (SB-NP) (Reaction 2) was washed with distilled deionized water to remove excess reagents (Labuto et al., 2018; Alomá et al., 2013; Carvalho et al. 2020; Abilio et al. 2021). The materials SB and SB-NP were tested for their effectiveness in the process of Cr(III) and Cr(VI) sorption under different pH.

$$Fe_{(aq)}^{2+} + 2Fe_{(aq)}^{3+} + 8OH_{(aq)}^{-} \rightarrow Fe_3O_{4(s)} + 4H_2O_{(l)}$$

(Reaction 1)

$$Fe_3O_{4(s)} + SB_{(s)} \rightarrow SB{-}Fe_3O_{4(s)}$$

(Reaction 2)

Determination of pH at the point of zero charge

The pH at the point of zero charge (pH_{PZC}) was determined using 100 mg of SB or SB-NP and 10 mL of 0.1 mol/L NaCl solution at initial pH values ranging from 2 to 12, adjusted with 0.1 mol/L HCl or NaOH. This resulting solution was kept under stirring for 24h in an incubator shaker at 185 rpm. The pH value corresponding to the pH_{PZC} was determined by the graphical representation of the initial pH variation as a function of the final pH. After determining the pH_{PZC}, the best pH range for sorption was assessed. After determining the pH_{PZC}, the best pH range for sorption was assessed. (Abilio et al., 2021; Pina, 2011).

Sorption pH assessment and chromium species determination

The sorption pH was assessed by mixing 100 mg of *in natura* (SB) or nanomodified (SB-NP) biomass with 10 mL of 10 mg/L Cr solutions [a 5 mg/L Cr(III) + solution Cr(III) and 5 mg/L Cr(VI)], previously adjusted at a pH range of 1 to 6, under constant agitation at 185 rpm for 10 minutes. The supernatants were analyzed by Flame Atomic Absorption Spectroscopy (FAAS) (AAnalyst 400, PerkinElmer, USA) to determine total Cr, employing a calibration curve of 1.0 to 6.0 mg/L Cr. The FAAS operational conditions were: acetylene flow rate (3.3 L/min), nebulizer flow (5.0 L/min), air (10.0 L/min), and nebulizer flow rate (2.0 L/min). Chromium was determined employing a Cr cathode lamp at 357.87 nm. The assays were performed in triplicate.

Chromium(VI) was determined as a Cr(VI)-diphenylcarbazide (DPC) red-violet complex analyzed by Ultraviolet-Visible spectroscopy (UV/Vis) (Genesys, 10S, Thermo Fisher Scientific, USA) at 540 nm, employing a calibration curve of 1.0 to 6.0 mg/L Cr(VI). Cr(III) was determined in the competitive tests by subtracting Cr(VI) contents from the total Cr contents quantified by FAAS since it does not react with DPC.

Results and Discussion

Preparation of magnetite nanoparticles (NP) and sugarcane bagasse nanocomposites (SB-NP)

The aim of impregnating SB with nanoparticles is to allow the biomass to acquire paramagnetic properties, allowing the material to be removed by a magnetic field such as a magnet after being applied for remediation in an aqueous medium and thus use on an industrial scale. Magnetite has unique intrinsic properties. Its structural shape is cubic, and magnetite crystals have a high inverted polarity, allowing this to be considered one of the nanoparticles with the highest magnetic values (Abilio et al., 2021; Carvalho et al., 2020).

Determination of pH at the point of zero charge and sorption pH assessment

The pH_{PZC} indicates the pH at which the surface charge of the biosorbent is electrically zero. When present at a pH below the pH_{PZC}, the material is positively charged; however, if the solution is at a pH higher than the pH_{PZC}, the charges on the material surface will be negative. Therefore, this assessment makes it possible to predict the surface charges and obtain more satisfactory conditions for the adsorption process (Bakatula et al., 2018).

The pH_{PZC} was calculated from the arithmetic mean of pH values that remained constant. The pH_{PZC} for SB and SB-NP was 6.1 and 5.8, respectively (Abilio et al., 2021). Below these pH values, the biosorbents surface exhibits positive charges, which would favor the sorption of Cr(VI). With pH above pH_{PZC}, the material's surface is negatively charged and is attracted to positive chromium species. Figure 1 shows the graphs of initial versus final pH values used to determine the pH_{PZC} of SB and SB-NP.

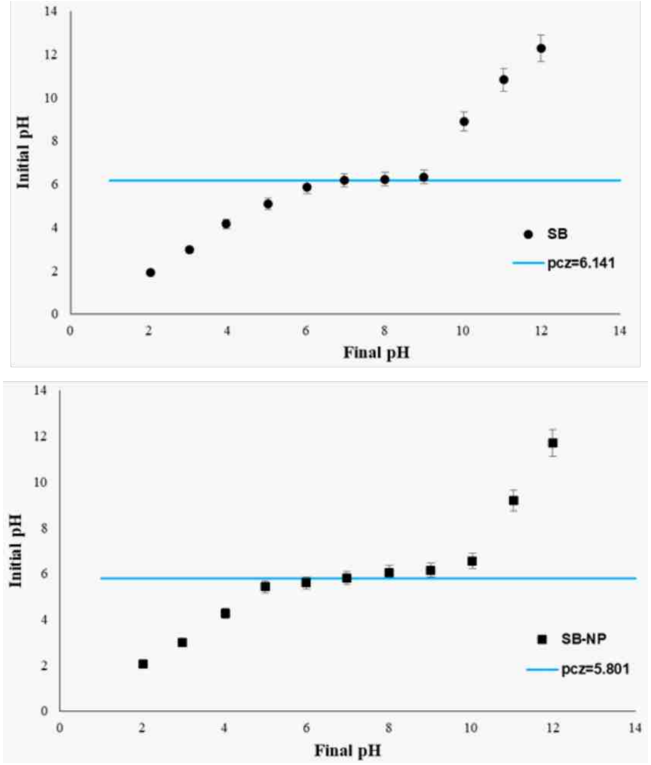

Figure 1: Point of zero charge (pH_{PZC}) of *in natura* (A) SB and the nanocomposite (B) SB-NP, using 10 mg of the biosorbent with 10 mL of 10 mg/L Cr(VI) solution, n = 3.

Chromium determination by UV/Vis

Total Cr in the supernatants from each biosorbents essay was determined by FAAS. Chromium (VI) was quantified by UV/Vis, at 540 nm, by forming a red-violet Cr(VI)-diphenylcarbazid complex. In the UV-Vis analysis, the analytical curve of 1 to 6 mg/L Cr(VI) and calculations performed according to the Lambert-Beer equation to quantify Cr(VI) in the supernatant. Figure 2 depicts the Cr(VI)-diphenylcarbazide red-violet complex formation using a bielement solution of 5mg/L Cr(III) and 5mg/L Cr(VI) at a pH range of 1 to 6.

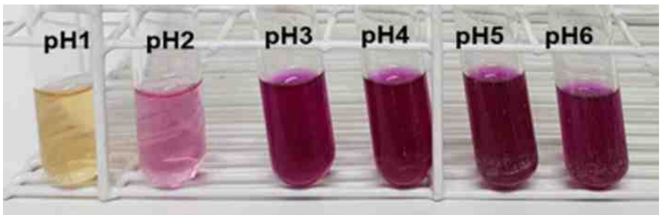

Figure 2: Illustration of the reaction of Cr(VI), remaining in the supernatants, and DPC, after Cr sorption in 5 mg/L of Cr(III) + 5 mg/L of Cr(VI) solution, at pH 1 to 6.

Effect of pH on the sorption of chromium

This difference depends on the pH of the solution, the total Cr concentration, and the presence of oxidizing and reducing compounds, as there is a competition between hydrogen ions and metal ions to bind to the functional groups of the nanocomposite by electrostatic attraction between adsorbent and adsorbed.

At very low pH values, there is a high concentration of H^+ ions, which neutralizes the negative charge surface of the adsorbent. In contrast, in higher pH values, the concentration of OH^- increases. The total charge on the adsorbent surface becomes negative, occurring a repulsion with the negative charge of Cr(VI) species (Alomá et al., 2013). The distribution of Cr(III) and Cr(VI) is strongly dependent on the pH of the solution, the initial concentration of metal ions, and the dosage of the biosorbent.

Chromium(VI) can form several species in aquatic environments such as $Cr_2O_7^{2-}$, CrO_4^{2-}, H_2CrO_4 and $HCrO^{4-}$, according to the solution pH, chromium concentration, and the presence of reducing and oxidizing agents

(Pina, 2011). Reactions 3 and 4 demonstrate the predominant species at each pH.

$$H_2CrO_4 \rightarrow H^+ + HCrO_4^- \quad (pH = 1 - 6)$$

(Reaction 3)

$$HCrO_4^- \rightarrow H^+ + CrO_4^{2-} \quad (pH > 7)$$

(Reaction 4)

Since Cr(VI) is toxic while Cr(III) is harmless, the oxidation and chromium reduction relationships become important. Both species must be analyzed. There must be monitoring to obtain more accurate data and better environmental control, thus comparing the quantification of the total content of the Cr element (Matos et al., 2008).

The increase in Cr(VI) adsorption with the decrease of the pH follows two mechanisms to describe the adsorption of Cr(VI) by biosorbents. The first involves the adsorption of Cr(VI), followed by its reduction to Cr(III) due to the oxidation of organic matter, and the second suggests that the oxidation of Cr(III) to Cr(VI) by the biomass is subsequently adsorbed to its surface (Yao et al., 2010). These mechanisms are not exclusive and can occur simultaneously, but both depend on the pH.

It may be implied that the nanoparticle also performs chromium sorption due to the uptake sites available on its surface. Therefore, besides the increase in SB sorption capacity, the use of NP is justified by the paramagnetic property attributed to SB to facilitate removing the contaminant-containing biosorbent from the medium (Pina, 2011).

The results obtained in the competitive adsorption of Cr(III) and Cr(VI) by the *in natura* and nanomodified biosorbent demonstrated that at low pH, the process favors the reduction of Cr(VI) to Cr(III) due to the oxidation of the organic matter in the adsorbent, in a short contact time (Pina, 2011). Figure 3 depicts the effect of pH on the sorption of chromium by SB and SB-NP, using 10 mg of the biosorbent with 10 mL solution of 10 mg/L of Cr(III), Cr(VI), and total Cr.

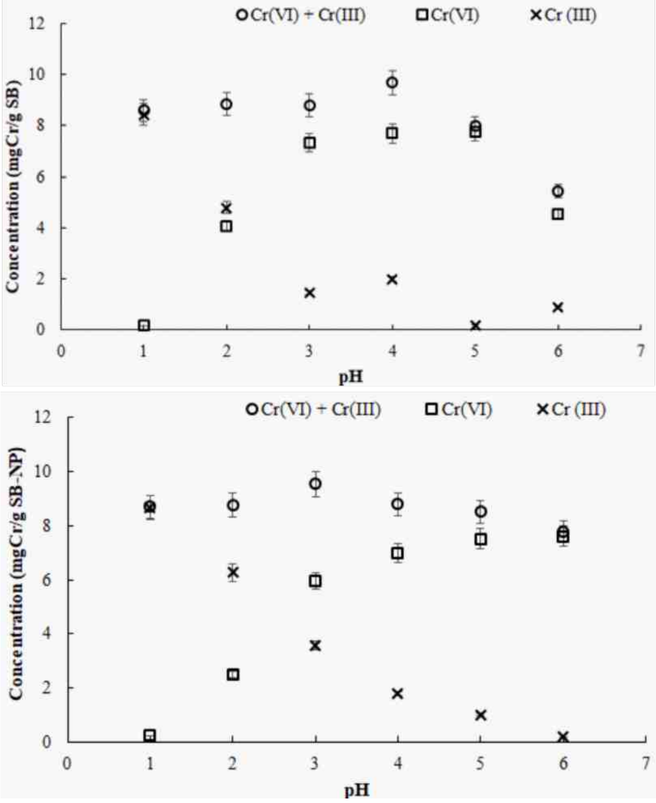

Figure 3: Effect of pH on the sorption of chromium by *in natura* (A) SB and the nanocomposite (B) SB-NP, using 10 mg of the biosorbent with 10 mL solution of 10 mg/L of Cr(III), Cr(VI), and total Cr, n = 3.

The results of Cr sorption in the monoelement solutions were assessed. Best Cr(VI) sorption was at pH 1, with a removal efficiency of 85% and 84% for SB and SB-NP, respectively. For Cr(III), sorption was more efficient at pH 6 for both SB (60%) and SB-NP (30%). For the simultaneous sorption assessment, the best results were found at pH 4, with 27% for SB and 52% for SB-NP. These findings are presented in Figure 4.

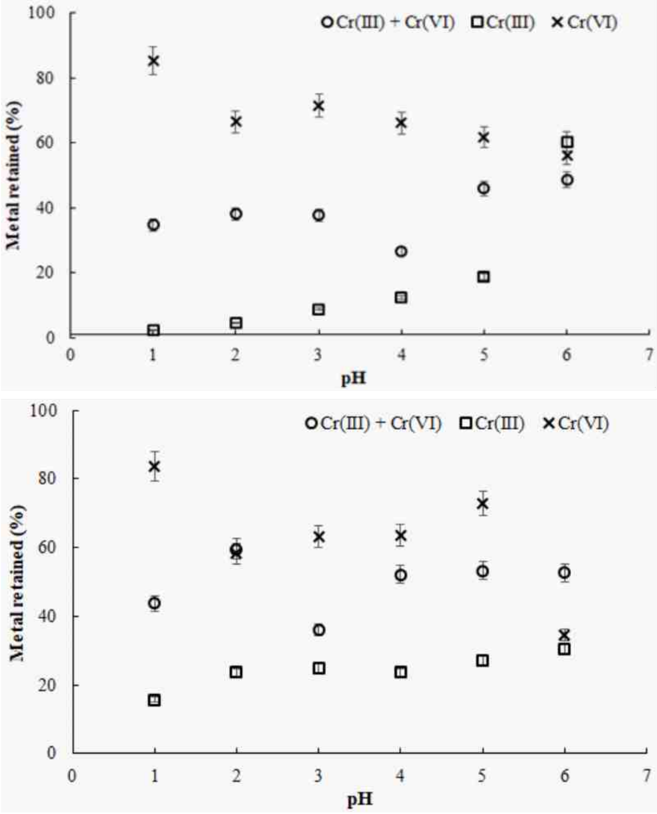

Figure 4: Effect of pH on the sorption of chromium by *in natura* (A) SB and the nanocomposite (B) SB-NP, using 10 mg of the biosorbent with 10 mL solution of 10 mg/L of Cr(III) or Cr(VI), and total Cr, n = 3.

Conclusion

According to our findings, the nanomodified sugarcane bagasse showed great potential for removing Cr(III) and Cr(VI) in an aqueous medium. The coprecipitation method promoted an efficient synthesis with low reagent consumption and operational time, and high yield, facilitating its removal in aqueous matrices. It was observed that the best sorption of Cr(VI) by SB and SB-NP, respectively, 85 and 84%, found at pH 1, improved with the pH decrease. As for Cr(III), sorption was improved at pH 6, reaching up to 60% and 40% removal for SB and SB-NP, respectively. In the simultaneous sorption studies of Cr(III) and Cr(VI), the maximum removal of 27% and 52% for SB and SB-NP, respectively, was achieved at pH 4. It was also possible to analyze the oxidation and reduction of Cr(VI) by the action of the biosorbent according to pH, with adsorption of Cr(VI), followed by a reduction to Cr(III) due to the oxidation of organic matter.

The proposed low-cost biosorbent SB from agro-industrial waste and its magnetic nanomodified form SB-NP are promising materials for removing Cr(III) and Cr(VI) in an aqueous medium.

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