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Inovações e Soluções Sustentáveis em Engenharia Ambiental

Optimizing an alternative method to remove Pb^{2+} and Ba^{2+} in aqueous solution by using coconut fiber as biosorbent material

Otimização de um método alternativo para remoção de Pb^{2+} e Ba^{2+} de soluções aquosas, empregando fibra de coco como material biossorvente

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ABSTRACT

This work presents the optimization of an alternative method for the removal of lead (Pb^{2+}) and barium (Ba^{2+}) from public supply water using coconut fiber as a biosorbent material. A system of four fixed-bed reactors connected in series, each with a volume of 0.250 L, was employed, operated in an upward continuous flow. A central composite rotational design (CCRD) was used, where the critical values that maximized the removal of Pb^{2+} ions were: pH = 4.8; time = 57.11 min; and biomass concentration = 25.45 g/L. For Ba^{2+} ions, the critical values were: pH = 4.99; time = 53.41 min; and biomass concentration = 19.83 g/L. The removal percentage was determined at the critical values, and an increase in lead removal (from 82% to 88%) and a reduction in barium removal (from 94% to 89.2%) were observed when using four reactors in series. In the multi-elemental analysis containing both lead and barium simultaneously and using the average values found for the ions analyzed separately (pH = 5.0; time = 55.26 min; biomass concentration = 22.64 g/L), there was a slight reduction in lead removal (from 81.2% to 78%), but within the standard deviation, and barium removal remained stable (92.6%). Thus, this alternative method proved to be promising for the removal of potentially toxic metals in public supply water.

Keywords: Environmental pollution; Biosorbent materials; Potentially toxic metals

RESUMO

Este trabalho apresenta a otimização de um método alternativo para a remoção de chumbo (Pb^{2+}) e bário (Ba^{2+}) de águas de abastecimento público, utilizando fibra de coco como material biossorvente. Foi empregado um sistema de quatro reatores de leito fixo ligados em série, cada um com volume de 0,250 L, operados em fluxo contínuo ascendente. Utilizou-se o delineamento composto central rotacionado (DCCR), onde os valores críticos que maximizaram a remoção dos íons Pb^{2+} foram: pH = 4,8; tempo = 57,11 min; e concentração de biomassa = 25,45 g/L. Para os íons Ba^{2+}, os valores críticos foram: pH = 4,99; tempo = 53,41 min; e concentração de biomassa = 19,83 g/L. O percentual de remoção foi determinado nos valores críticos e observou-se um aumento na remoção de chumbo (de 82% para 88%) e uma redução na remoção de bário (de 94% para 89,2%) ao utilizar quatro reatores em série. Na análise multielementar contendo chumbo e bário simultaneamente e utilizando os valores médios encontrados para os íons analisados isoladamente (pH = 5,0; tempo = 55,26 min; concentração de biomassa = 22,64 g/L), houve uma leve redução na remoção de chumbo (de 81,2% para 78%), mas dentro do desvio padrão, e uma manutenção no percentual de remoção de bário (92,6%)0. Assim, este método alternativo mostrou-se promissor para a remoção de metais potencialmente tóxicos em águas de abastecimento público.

Palavras-chave: Poluição ambiental; Materiais bissorventes; Metais potencialmente tóxicos

1 INTRODUCTION

The growing demand for natural resources, driven by the modern lifestyle, has resulted in an alarming global water crisis. This issue stems from rapid population growth and the intensive use of water by sectors such as industry, agriculture, and domestic consumption. Every day, large amounts of pollutants are discharged into water bodies, causing serious environmental impacts. Among the main pollutants are ions of potentially toxic metals, originating from various industrial activities, which pose risks to both human health and the environment (Azimi et al., 2017; De Sá Costa et al., 2021; Lakshmi et al., 2021; Canellas et al., 2023; Xie, 2024).

To mitigate this problem, various pollutant removal techniques have been developed and applied, including biodegradation (enzymatic and microbial), electrochemical methods, advanced oxidation processes, reverse osmosis, chemical precipitation, ultrafiltration, and membrane separation (Jiang, 2018). However, these methods have significant limitations, such as high operational costs and limited efficiency in fully removing metals, in addition to generating considerable waste (Azimi et al., 2017; Jiang, 2018; Jaafer et al., 2021).

In this context, biosorption emerges as a promising, effective, and economical solution for the removal of toxic metals from water. Biosorption is based on the reversible interaction between a biosorbent material and the adsorbate, allowing the reuse of the material after the desorption process in a sustainable manner (Azimi et al., 2017; Santosh et al., 2017). This method exploits the ability of certain biological materials to adsorb metal ions through mechanisms such as physical and chemical adsorption, ion exchange, precipitation, and complexation, offering significant advantages such as low cost and wide availability (Azimi et al., 2017; Babel; Sheikh et al., 2021; Singh et al., 2024; Canellas et al., 2023; Xie, 2024).

Among the materials used as biosorbents, coconut husk waste, from the tree Cocos nucifera Linn, commonly known as the coconut tree or green coconut, stands out. This species, one of the most cultivated in the world, has lignocellulosic fibers with remarkable adsorption capacity (Canellas et al., 2023). The combination of low cost, high availability, and stability makes the coconut tree a promising material for biosorption applications.

Additionally, this study demonstrates the feasibility of an optimized method that complements conventional water treatments, which often have limitations in the removal of toxic metals. The proposed solution involves a system of fixed-bed reactors with continuous flow in series, whose performance has been thoroughly studied and proven feasible for practical application, with the necessary adjustments (Canellas et al., 2023).

2 MATERIALS AND METHODS

Green coconut fiber dried at 60ºC for 24 hours and treated with Sodium Hydroxide solution, NaOH (Sigma-Aldrich, 98%) at 0.1 mol L^{-1} for 3 hours, at room temperature, was the adsorbent material used in the biosorption experiments. Details about this fiber's preparation procedure are better described in a previous publication (Bruno, 2023).

Solutions added with metal ions Pb^{2}+ and Ba^{2}+ were prepared based on using lead II acetate salts, $Pb(H_3CCOO)_2$ (MERCK, 99%), and barium nitrate, $Ba(NO_3)_2$ (MERCK, 99%). All solutions were prepared by using distilled water presenting conductivity lower than 3.0 µS cm^{-1}. An experimental system with fixed acrylic bed reactors in series, with continuous flow and circular cross-section, was used to optimize potentially toxic metals' removal (see reference, Bruno 2023).

An experimental matrix was developed by defining a Rotated Central Composite Design (RCCD), under triple repetition at the central point for each of the herein analyzed variables, namely: pH, residence time and biomass concentration. RCCD was performed at two levels, the lower (-1) and the upper (+1) ones. After defining the parameters and limits, the rotational planning foresaw the addition of two extra points to each assessed parameter, the so-called axial points, which allowed the planning rotation. Thus, six additional points paired in –α and +α were defined based on triplicates located at the central points, which totaled 17 experiments, as shown in Table 1.

Table 1 – Values recorded for parameters used in RCCD applied to Pb^{2+} and Ba^{2+} ions

	- α	**-1**	**0**	**+1**	**+α**
pH	4.42	4.5	5.0	5.5	5.58
Residence time	30	40	50	60	70
Biomass Concentration	2.5	5.0	20.0	35.0	38.0

Source: Authors (2024)

Table 2 presents the experiments performed through RCCD applied to Pb^{2}+ and Ba^{2}+ ions.Initial concentration of metal ions (Pb^{2}+ and Ba^{2}+) = 2.5 g L^{-1} and flow rate = 10 L h^{-1} were used to conduct the experiments through RCCD and the solution was subjected to continuous flow in 1 reactor, alone. These values were defined based on previous results recorded by Canellas et al. (2023).

Table 2 – Experiments carried out through RCCD

Experiment	pH	Time (min)	Biomass concentration (g L^{-1})
01	4.5	40	5.0
02	5.5	40	5.0
03	4.5	60	5.0
04	5.5	60	5.0
05	4.5	40	35.0
06	5.5	40	35.0
07	4.5	60	35.0
08	5.5	60	35.0
09	4.42	50	20.0
10	5.58	50	20.0
11	5.0	30	20.0
12	5.0	70	20.0
13	5.0	50	2.5
14	5.0	50	38.0
15	5.0	50	20.0
16	5.0	50	20.0
17	5.0	50	20.0

Source: Authors (2024)

Furthermore, biosorption experiments were carried out by using 4 reactors in series, all of them filled with coconut fibers, at equal ratios.

3 RESULTS AND DISCUSSION

Table 3 presents the experiments and results recorded for Pb^{2+} and Ba^{2+} ions under RCCD. These experiments were performed by using 1 reactor, alone.

The data presented in Table 3 illustrate the efficiency of the percentage removal of Pb^{2+} and Ba^{2+} ions by coconut fibers. In most experiments, the removal rate exceeded 80%. The best performance was observed for the Pb^{2+} ion, with a removal rate of 96% at pH 5.0, 50 minutes of residence time, and a biomass concentration of 2.5 g L^{-1}. For the Ba^{2+} ion, the optimal removal of 92% occurred within a pH range of 4.5 to 5.5, with contact times varying between 40 and 70 minutes, and biomass concentrations ranging from 5.0 to 38.0 g L^{-1}.

Table 3 – Results recorded for Pb^{2+} and Ba^{2+} ions under RCCD

Experiment	pH	Time (min)	Biomass concentration (g L^{-1})	Final concentration (mg L^{-1})		Removal %	
				Pb^{2+}	Ba^{2+}	Pb^{2+}	Ba^{2+}
01	4.5	40	5.0	0.40	0.35	84	86
02	5.5	40	5.0	0.30	0.20	88	92
03	4.5	60	5.0	0.25	0.30	90	88
04	5.5	60	5.0	0.20	0.30	92	88
05	4.5	40	35.0	0.75	0.35	70	86
06	5.5	40	35.0	0.55	0.25	78	90
07	4.5	60	35.0	0.35	0.20	86	92
08	5.5	60	35.0	0.15	0.20	94	92
09	4.42	50	20.0	0.60	0.25	76	90
10	5.58	50	20.0	0.55	0.25	78	90
11	5.0	30	20.0	0.25	0.45	90	82
12	5.0	70	20.0	0.25	0.20	90	92
13	5.0	50	2.5	0.10	0.25	96	90
14	5.0	50	38.0	0.40	0.20	84	92
15	5.0	50	20.0	0.15	0.30	94	88
16	5.0	50	20.0	0.25	0.30	90	88
17	5.0	50	20.0	0.20	0.35	92	86

Source: author (2024)

Previous studies show that the adsorption capacity is influenced by various parameters, including contact time, solution concentration, adsorbent characteristics, and particle size. Lower concentrations of ions tend to require less contact time to saturate the active sites (ARPA et al., 2000; Pierangeli et al., 2007). Additionally, the time required to reach adsorption equilibrium may vary among different potentially toxic metals (Jeronimo, 2019). Thus, certain ions require more time in contact with the specific adsorbent (Westrup et al., 2005). Moreover, it has been observed that lower initial concentrations of adsorbate in aqueous solution can increase the percentage of removal (Antunes et al., 2018). While increasing the concentration of the adsorbent may raise the removal percentage, it can also lead to the occupation of some active sites on the surface of the adsorbent material, which are energetically unfavorable (Reddy; Verma; Subrahmanyam, 2016). Other studies indicate that, at higher concentrations, some ions such as Pb, Cr, and Cu maintain a high affinity for the adsorbent surface, while ions such as Ni, Zn, and Cd may be displaced (Fontes and Gomes, 2003). Based on the results from Table 3, Figures 1 and 2 present the response surfaces obtained for Pb^{2+} and Ba^{2+} ions, respectively, as a function of the working parameters, along with the corresponding Pareto diagrams.

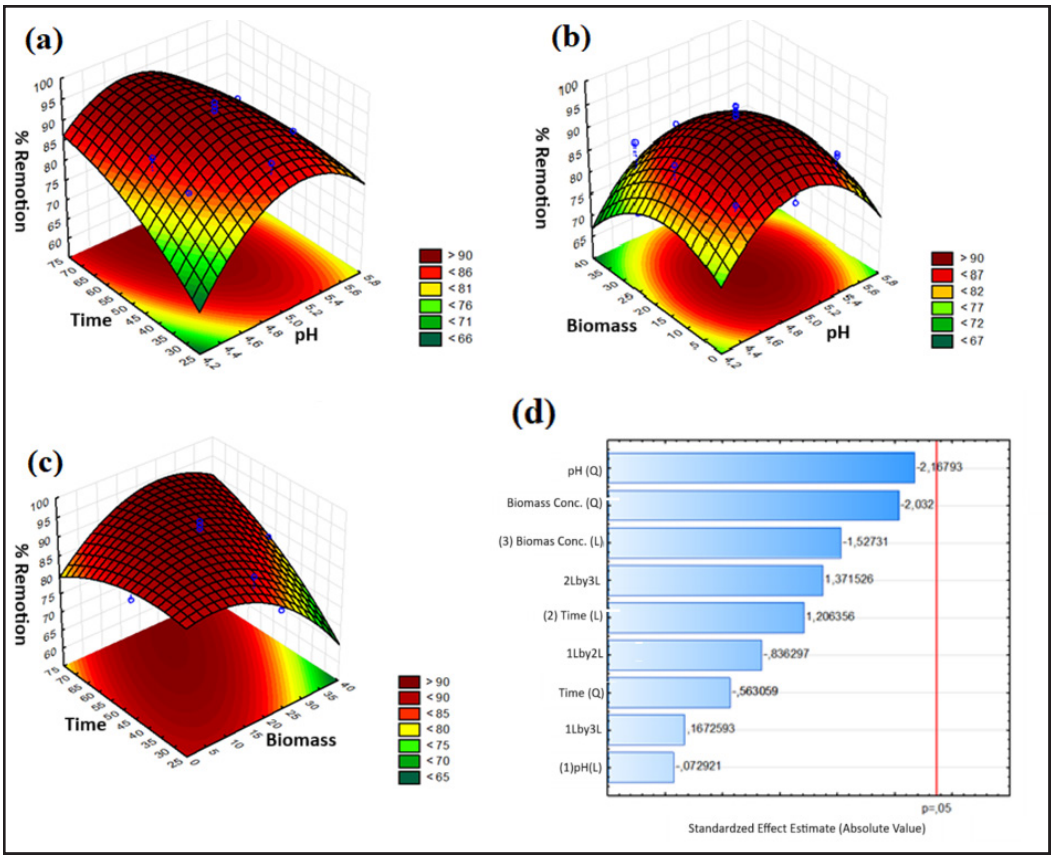
Figure 1(a) reveals that the highest percentage removals of Pb^{2+} were achieved close to pH 5, with biomass contact times exceeding 50 minutes. These results highlight the importance of pH in the removal of metals through biosorption. pH influences the competition between metal ions and H^+ ions present in the solution for the active sites of *Cocos nucifera* L. fiber (Chubar et al., 2004; Selatnia et al., 2004).

In Figure 1(b), the highest percentage removals were observed at a pH close to 5 and at intermediate biomass concentrations within the range used. Therefore, the ratio between the mass of the biosorbent and the volume of metal ions is crucial for the retention of the ion, highlighting the direct relationship between biomass concentration and the efficiency of removing potentially toxic metals.

Based on Figure 1(c), the best removal rate outcomes also happened at intermediate biomass concentrations and contact time longer than 50 min. Contact time required between adsorbate and adsorbent is essential to reach adsorption equilibrium. Oftentimes, adsorptive processes based on short time processes related to ion exchange take place; however, complexation processes require longer times to happen, and it justifies the optimum time analysis and determination of each process (Tarley; Arruda, 2003).

The Pareto diagram presented in Figure 1(d) points out that, despite influencing less than 95% of values predicted by the mathematical model, the quadratic terms ‘pH’ and ‘biomass concentration’ were the most significant effects.

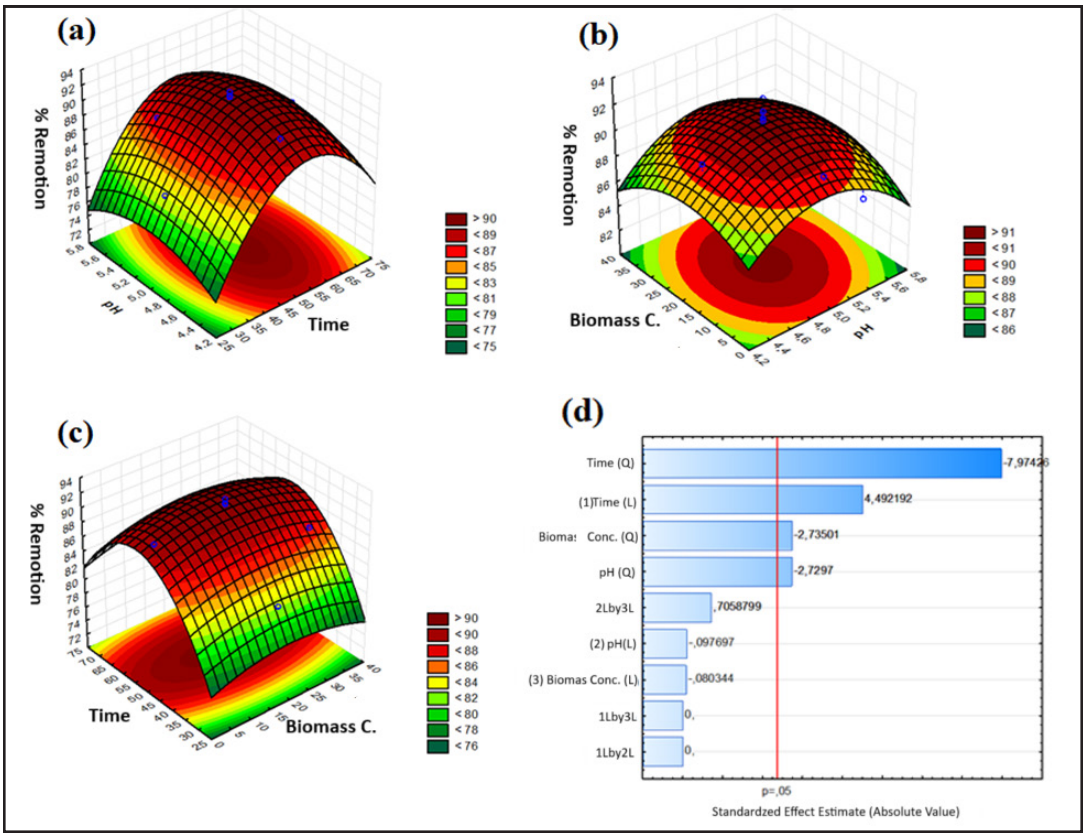
Figure 1 – Surface pH response versus time (a); surface pH response versus biomass concentration (b); biomass concentration surface response versus time (c); Pareto diagram generated for the Pb^{2+} ion (d)



Source: author (2024)

Figure 2(a) evidences that the best results, for Ba^{2+} ions’, were recorded at intermediate pH values and contact time within the applied range. Very high pH values can lead to potentially toxic ions’ complexation and precipitation, whereas very low pH values reduce removal rates through the absorbent material’s protonation. Accordingly, intermediate values, such as the herein recorded ones, favor the retention of potentially toxic metals.

Figure 2 – Surface time response versus pH (a); surface pH response versus biomass concentration (b); surface biomass concentration response versus time (c); Pareto diagram generated for the Ba^{2+} ion (d)



Source: author (2024)

The pH and biomass concentration shown in Figure 2(b) depict the best results for Ba^{2+} ions' removal rates when values close to pH 5 and biomass concentration = 20 g L^{-1}. It was reported that the amount of Ba^{2+} ions removed by orange peel increased from 58% to 80% with an increase in biomass from 2 to 7 g, which was more significant than the increase from 2 to 3 g at a fixed amount of metal ions, demonstrating the direct relationship between the amount of adsorbent and ion removal (Torab-Mostaedi et al., 2011).

In Figure 2(c), better removal results of potentially toxic ions are observed when average values are used within the parameters of biomass concentration and time. The adsorption capacity of Ba^{2+} ions, using zeolite as the adsorbent material, has been reported, and it was noted that adsorption increases with time until a constant value (saturation) is reached, with this saturation related to the initial concentrations of the metal, thereby demonstrating the relationship between time and the retention of Ba^{2+} ions (Barbosa, 2011).

The Pareto diagram presented in Figure 2(d) evidenced that all parameters were significant in more than 95% of data predicted by the mathematical model, with emphasis on parameter ‘contact time’.

Table 4 presents the critical values to maximize Pb^{2+} and Ba^{2+} ions removal rates, which were analyzed separately, when only 1 reactor was used.

Table 4 – Critical values to maximize Pb^{2+} ions removal rates

Critical values (maximize Pb^{2+} removal rate)					
pH		**Time (min)**		**Biomass concentration (g L^{-1})**	
Pb^{2+}	**Ba^{2+}**	**Pb^{2+}**	**Ba^{2+}**	**Pb^{2+}**	**Ba^{2+}**
4.80	4.99	57.11	53.41	25.45	19.83

Source: author (2024)

Using the critical values from Table 4, the influence of using 4 reactors in series on the removal of Pb^{2+} and Ba^{2+} ions was investigated, initially in unielemental standard solutions, meaning containing only 1 metal ion. For the experiments, an initial concentration of Pb^{2+} ions of 2.5 mg L^{-1} and a flow rate of 10 L h^{-1} were used. The results are presented in Table 5.

Table 5 – Pb^{2+} and Ba^{2+} ions removal rate based on using 4 reactors in series

ion	**Final concentration (mg L^{-1})**	**% Removal**
Pb^{2+}	0.30 ± 0.10	88.0
Ba^{2+}	0.27 ± 0.15	89.2

Source: author (2024)

From Table 5, it can be observed that the percentage removal of Pb^{2+} and Ba^{2+} ions did not increase with the addition of 3 more reactors in series. This fact can be explained, among other factors, by the low concentration of the solution from the second reactor onward. Extremely diluted solutions hinder the contact of ions with the active sites of the biomass, making biosorption more difficult (Wan Ngah; Hanafiah, 2008). Therefore, we can say that the increase in the number of reactors had little effect on the removal process of the ions in question.

Finally, the removal results are presented when Pb^{2+} and Ba^{2+} ions were simultaneously subjected to the biosorption process, which we will refer to as a multielement solution. Table 6 presents the critical values used to enhance the removal of Pb^{2+} and Ba^{2+} ions in the multielement solution, using 1 and 4 reactors (initial concentrations of Pb^{2+} and Ba^{2+} ions of 2.5 mg L^{-1} and a flow rate of 10 L h^{-1}). The values used correspond to the averages obtained from the maximum percentage removal values for Pb^{2+} and Ba^{2+} ions when analyzed individually.

Table 6 – Critical values to maximize the Pb^{2+} and Ba^{2+} ions removal rates in multi-element solution

pH	**Biomass concentration (g L^{-1})**	**Time (min)**
4.90	22.64	55.26

Source: author (2024)

Table 7 presents the results of Pb^{2+} and Ba^{2+} ions removal in a fixed-bed system with continuous flow, using 1 and 4 reactors in a multielement solution.

Table 7 – Pb^{2+} ions removal rates in multi-element solution based on using 1 and 4 reactors

# of reactors	**Final concentration (mg L^{-1})**		**Removal %**	
	Pb^{2+}	Ba^{2+}	Pb^{2+}	Ba^{2+}
1 Reactor	0.47 ± 0.13	0.18 ± 0.03	81.2	92.6
4 Reactors	0.55 ± 0.23	0.18 ± 0.03	78.0	92.6

Source: author (2021)

The data from Table 7 show that the percentage removal of Pb^{2+} ions in the multielement solution, when compared to the retention in the unielement solution, is lower; however, it is within the standard deviation of the analyses performed. For Ba^{2+} ions, the percentage removals in the multielement solution, when compared to the retention in the unielement solution, also did not show a significant difference.

When comparing the percentage removals of Pb^{2+} ions with Ba^{2+} ions, it can be observed that there is a greater retention of barium metal. This fact can be explained by the higher affinity of the active sites present in coconut fiber for Ba^{2+} ions and by the specific characteristics of each ion. It should be emphasized that the competition of potentially toxic metals for the active sites present in various biomasses is due to the specific properties of each ion, such as electric charge and size (Castillo, 2017).

Several factors can influence the adsorption process at the active sites present in biomasses. Among these factors, the selectivity or preference of one ion over another stands out, which is associated with the hydrated ionic radius and the hydration energy of cations with the same valence. For example, ions with a large ionic radius tend to be retained with less intensity, while those with a smaller radius approach the surface of the adsorbent more closely, resulting in greater attraction. Higher valence ions possess a more significant displacement force. Additionally, the retention strength of the ions is determined by the type of interaction between the potentially toxic ions and the surface of the adsorbent material (JI; LI et al., 1997).

Due to various factors, including the type of adsorbent and adsorbate, pH, metal concentration, and others, different affinity sequences can emerge among potentially toxic ions, making it nearly impossible to establish a universal order. If this affinity were based solely on electrostatic interactions, the strongest bonds would be formed by the metal with the highest charge-to-ionic radius ratio. For metals with the same charge, this affinity would be inversely proportional to the hydrated ionic radius, meaning that the affinity is proportional to the ionic potential (Z^2/r), where Z represents the charge and r denotes the ionic radius of the metal (Mcbride, 1989; Basta; Tabatabai, 1992). Research has demonstrated that increasing the concentrations of certain metals, such as Cu, Cr, and Pb, sustained their high affinity, whereas other metals, like Ni, Zn, and Cd, were displaced from the adsorbent surfaces (Fontes; Gomes, 2003).

4 FINAL CONSIDERATIONS

This study demonstrated the use of coconut fiber from *Cocos nucifera* L. in the development of an alternative and optimized method for the removal of potentially toxic ions from water, for human consumption. Using a central composite rotational design (CCRD) for Pb^{2+} and Ba^{2+} ions, the critical values that maximize the removal of Pb^{2+} ions (pH = 4.8; time = 57.11 min; biomass concentration = 25.45 g/L) and Ba^{2+} ions (pH = 4.99; time = 53.41 min; biomass concentration = 19.83 g/L) were determined. An increase in lead removal and a reduction in barium removal were observed when using four reactors in series; however, this difference was not significant. In the multielement analysis of lead and barium, using average values of the ions analyzed individually, a slight decrease in lead removal was observed, but within the standard deviation, and the percentage of barium removal remained constant. The percentages of lead and barium removal in the multielement solution were lower compared to the unielement solution, but still within the standard deviation. In addition to its efficiency, the alternative method developed with the fixed-bed, continuous-flow reactor system, using coconut fibers as the adsorbent material, proved to be promising for the removal of potentially toxic metals in public water supply. This is due to the ease of obtaining the adsorbent material, the low operational cost, the absence of toxic by-products, and the possibility of proper disposal or reuse of the metals after the process.

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