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Agro-Industrial Aloe Vera Waste-Derived Bioadsorbent for Efficient Aluminum Removal from Water

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ABSTRACT

Removing metal pollutants from water sources has become a critical environmental concern, demanding ongoing research and innovation in remediation technologies. Among the treatment methods, adsorption, using organic materials derived from agro-industrial wastes, stands out. The use of bioadsorbents derived from agro-industrial wastes, such as aloe vera, can be an effective and environmentally friendly solution for removing aluminum or other pollutants in irrigation water. This study aims to evaluate the aluminum removal capacity of a bioadsorbent developed from aloe vera processing wastes, using an adsorption column at a laboratory scale. For this purpose, tests were carried out with different doses of bioadsorbent and at different influent pH, using a two-factor, three-level factorial design. FT-IR, RAMAN and SEM analyses were performed to characterize the adsorption mechanism between aluminum and the bioadsorbent, which is quite complex. The present study demonstrated that the developed bioadsorbent has an adsorption capacity between 1.14-0.64 mg/g and achieved aluminum removal rates above 73% in all experiments, with the maximum removal (about 98%) observed at pH 4.5. This high efficiency in aluminum removal, combined with the use of agro-industrial waste as a bioadsorbent source, makes it a cost-effective and environmentally friendly solution. This suggests a promising potential for real-world water treatment applications, particularly in irrigation systems. Such an approach offers a sustainable and efficient solution for contaminant removal, in line with the increasing focus on environmental responsibility in water management.

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INTRODUCTION

Contamination of water sources by metals such as lead, cadmium, aluminum, manganese, and mercury has been identified as one of the major environmental problems in recent years. Water toxicity caused by metals is considered a serious problem for the inhabitants directly supplied by river water, especially when the increase in the concentration of these metals is caused by anthropogenic activities (Pabón et al., 2020).

The main activity generating metal pollution is mining, where the water pollution rate can be around 200 million cubic meters per day (Pabón et al., 2020; Senze et al., 2021). In recent years, the most attention has been focused on the potential adverse effects of aluminum, due to its widespread use in industrial applications, water treatment, food, and packaging (Panhwar et al., 2016; Boeris et al., 2016; Nidheesh et al., 2018).

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Aluminum is the third most abundant element in the earth's crust, occurring as the Al ion^{3+} in rocks, soils, water, plants, and the human body (Das et al., 2023). Although it is recognized that aluminum has no specific biological function in the human body, recent studies have shown that long-term and prolonged exposure to aluminum is associated with several health conditions such as Alzheimer's disease, encephalopathy, bone disorders, cancer, and endocrine diseases. In the human body, aluminum-related toxicity is associated with its ability to form complexes and its absorption, deposition, excretion, and retention in human tissues (Poudyal et al., 2022).

Aluminum enters the trophic chain through plants cultivated with contaminated irrigation water, which eventually deposits it in the soil. Because of this problem, it is of utmost importance to find a method to retain and extract this component from water sources, to guarantee the preservation of ecosystems and human life. One method that is currently attracting interest is bioadsorption. It is easy to use, has low operating costs, and is highly efficient (da Costa et al., 2020; Tan et al., 2022; Amaral et al., 2023; Goswami et al., 2023; Nouioua et al., 2023).

This has led to the search for new materials that are effective as bioadsorbents. Research has been carried out mainly with crop residues or agro-industrial wastes, which are affordable and can be used as a sustainable alternative. Bioadsorbents derived from industrial and agricultural waste have attributes such as broad availability, convenient accessibility, and robust adsorption capacity, contributing to their widespread utilization across various sectors. Examples of such waste include straw, pomelo peel, peanut shells, and biomass paper mill sludge (J. Li et al., 2024). An agro-industrial waste that has been little studied for its use in Colombia is that generated during the processing of aloe leaf for the extraction of aloe vera gel (Torres Gómez et al., 2020).

Aloe vera leaf is used in the pharmaceutical, food, and beauty industries. Considerable amounts of solid waste are produced during processing (Giannakoudakis et al., 2018). These wastes contain biologically active compounds such as vitamins, organic acids, and polysaccharides, which when subjected to different treatments produce materials with electron-donating functional groups (Singh et al., 2021; Wang et al., 2022; Yılmaz et al., 2023; Kalderis et al., 2024). Several studies have shown that these materials have a significant pollutant removal efficiency, associated with an interesting adsorption capacity compared to other adsorbents derived from natural products (Katubi et al., 2021; Nilamsari et al., 2022; Pal et al., 2022; Maia et al., 2023). In this context, the objective of this work is to evaluate the efficiency of aluminum removal in synthetic waters using a bioadsorbent based on aloe vera wastes.

MATERIALS & METHODS

Analytical grade chemicals were used throughout this research, including sodium acetate 3-hydrate (Panreac), acetic acid (Fisher Chemical), L-ascorbic acid (Fisher Chemical), sulfuric acid (Panreac), and Erichrome cyanine R. (Sigma-Aldrich). The stock solution, containing approximately 2000 ppm of aluminum (Al), was prepared by dissolving 2 g of metallic aluminum in 10 ml of hydrochloric acid and diluting it to 1 L with deionized water.

For each experiment, 5 L of synthetic water was prepared by taking 25 mL of the stock solution and adjusting the volume in a volumetric flask. The concentrations obtained during the assays were found to be in the range of 7.9 to 9.4 ppm. The final concentration varied in an interval of ±0.8 ppm due to the purity of the metallic aluminum used.

Industrial Waste from Aloe Barbadensis Miller (Aloe Vera) processing, such as thorns, tips, and aloe peel, was supplied by the company Veroa S.A.S located in Boyacá, Colombia. The physicochemical analysis was carried out in duplicate according to the methodology established in the Colombian Technical Standards (Normas Técnicas Colombianas or NTC) as presented in Table 1.

Initially, the waste was washed thoroughly with water to remove particulate contaminants.

Subsequently, it underwent desiccation in an oven set at 105°C for 10 hours. This was followed by a 2-hour thermal treatment at 300°C. The resulting biomass was subjected to sieving to select the fraction conforming to -18 +30 mesh ASTM standards, after which it was stored in a glass container (Khaniabadi Omidi et al., 2015).

The bioadsorbent was analyzed by Raman Spectrometry in a Thermo Scientific spectrophotometer, model DXR-SmartRaman with a standard 4 mV laser and a wavelength of 532 nm. The spectra obtained were taken in a range between 500 and 3500 cm^{-1} . The functional groups on the bioadsorbent were identified by Infrared Spectrometry on a JASCO FT/IR 4100 spectrophotometer, using the KBr pellet technique, in a range of 4000-400 cm^{-1}. The morphological characterization of the bioadsorbent was obtained by Scanning Electron Microscopy (SEM) using the JEOL JCM-5000 NeoScope Table Top SEM equipment.

Bioadsorption tests were performed at room temperature in a portable laboratory-scale column. Synthetic water with characteristics like industrial discharges (aluminum content between 7.9 and 9.4 ppm) was deposited into the column from the top using a dosing pump, as illustrated in Figure 1. The height of the bed varied according to the amount of bioadsorbent placed inside the column.

Different bioadsorbent mass and inlet pH were evaluated while maintaining a constant inlet flow rate of 22 ± 3 ml/min. The pH levels and bioadsorbent mass were established through preliminary tests that defined the specified operating conditions in Table 2. A 3^2-factorial design with replication was adopted for the combination of variables in the different trials.

Table 1. Tests for the characterization of aloe vera residues.

Parameter	Methodology
Humidity	NTC 4888:2000
Ashes	NTC 4648:2022
Fats	NTC 6240:2017
pH	NTC 440:2015

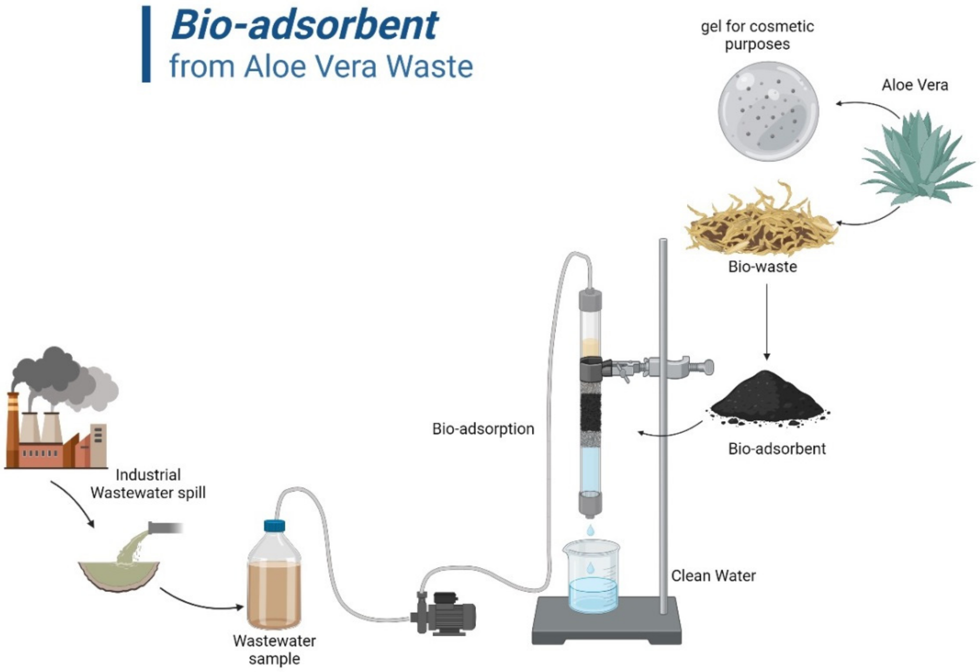


Fig. 1. Set-up used during experimentation

The percentage of Aluminum removed, $\mathrm{Al}(\%)$, and adsorption capacity (mg/g), q_T, were calculated, respectively, with Equations (1) and (2) given below:

$$\mathrm{Al}(\%) = \frac{C_0 - C_T}{C_0} * 100 \tag{1}$$

$$q_T = \frac{C_0 - C_T}{M} * V_T \tag{2}$$

where C_0 is the inlet concentration, C_T is the outlet concentration for a specified operating time (Equation 3), V_T is the total volume treated inside the column, C_i is the effluent concentration, V_i is the treated volume in a time interval and M is the bioadsorbent mass.

$$C_T = \frac{\sum_{i=0}^{t} C_i\ V_i}{V_T} \tag{3}$$

The aluminum concentration for each of the times was determined using a JASCO V-730 spectrophotometer following the methodology established in the NTC 4785:2000 standard.

RESULTS AND DISCUSSION

As can be seen from **Table 3**, the aloe vera waste has a moisture percentage of 90.54%, which indicates a higher moisture content compared to typical agro-industrial wastes, which range between 15% and 80%.

This high moisture content implies a higher energy requirement for waste treatment. However, the associated costs can be offset by the high adsorption capacity of the phenolic compounds, organic acids, and polysaccharides present in 9.46% of the residue (Wang et al., 2022). Furthermore, the study indicates an aloe vera waste pH value of 5.2. Thus, waste can attract cations at lower pH levels and capture anions at higher pH levels.

In addition, as shown in Table 3, industrial waste contains 1.60% of ashes corresponding to inorganic residues with the presence of minerals such as iron, copper, sodium, and potassium. Similarly, a fat percentage of 1.3% was found, related to the content of lipids such as fatty acids

Table 2. Column operating conditions

Bioadsorbent mass ± 0.01 g	**Feed pH ± 0.3**
2.69	3.0
4.04	4.5
5.38	6.0

Table 3. Physicochemical characteristics of aloe vera residues.

Parameter	**Value**			
Humidity	90.54	±	0.01	% w/w
Ashes	1.60	±	0.05	% w/w
Fats	1.3	±	0.2	% w/w
pH	5.2	±	0.2	

and steroids, which due to the presence of functional groups such as OH and COOH can retain metals (Hamman, 2008).

Desirable characteristics in a bioadsorbent include elevated selectivity and absorption capacity, determined through structural, chemical, and morphological characterizations of the material, such as porosity, surface area, carboxylic functional groups, and those containing oxygen (Lu & Zong, 2018). Figure 2 illustrates the Raman spectrum for a bioadsorbent sample after thermal treatment. The band observed around 1350 cm^{-1} is known as band D, while the band around 1580 cm^{-1} is called band G. Both bands are characteristic of carbonaceous structures (Chee et al., 2012). The G-band is related to the vibrations of the carbon atoms sp^2 associated with double bonds, while the D-band is associated with the disordered structure of graphite (Kawakami et al., 2006).

Based on these peaks, several parameters have been developed to characterize the carbon structure. However, a universal characterization method for carbon materials has not yet been established (Kawakami et al., 2006).

Since aloe vera is chemically characterized by the presence of phenolic constituents, which are mainly constituted by C, H, and O, there are a limited number of binding vibrations that can be assigned to the FTIR peaks in Figure 3. The band around 3400 cm^{-1} is associated with the

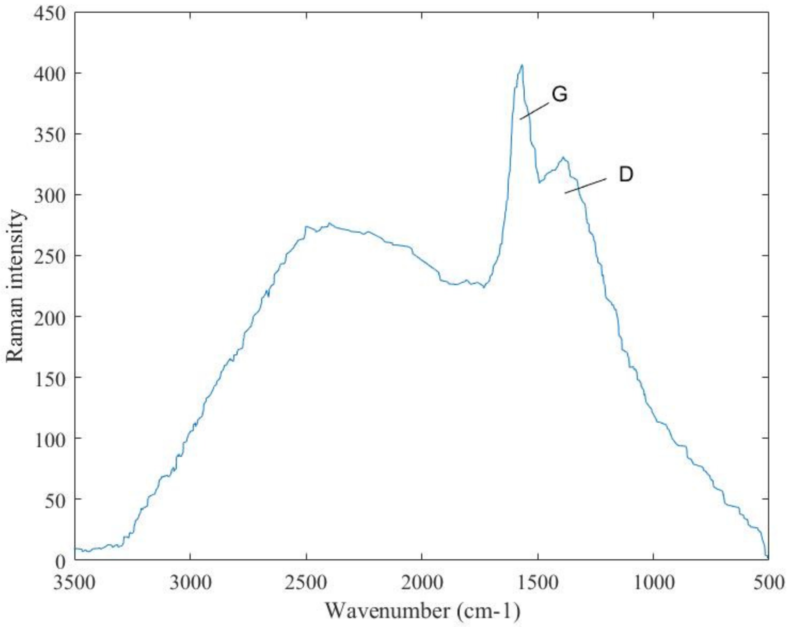


Fig. 2. Raman spectrum of the bioadsorbent.

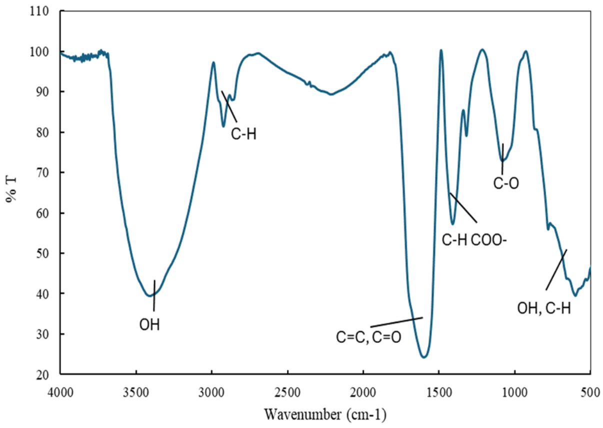


Fig. 3. Characteristic IR spectrum of the bioadsorbent before adsorption

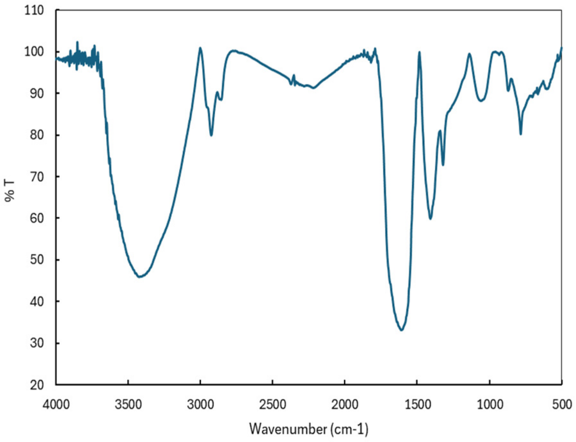


Fig. 4. Characteristic IR spectrum of the bioadsorbent after adsorption

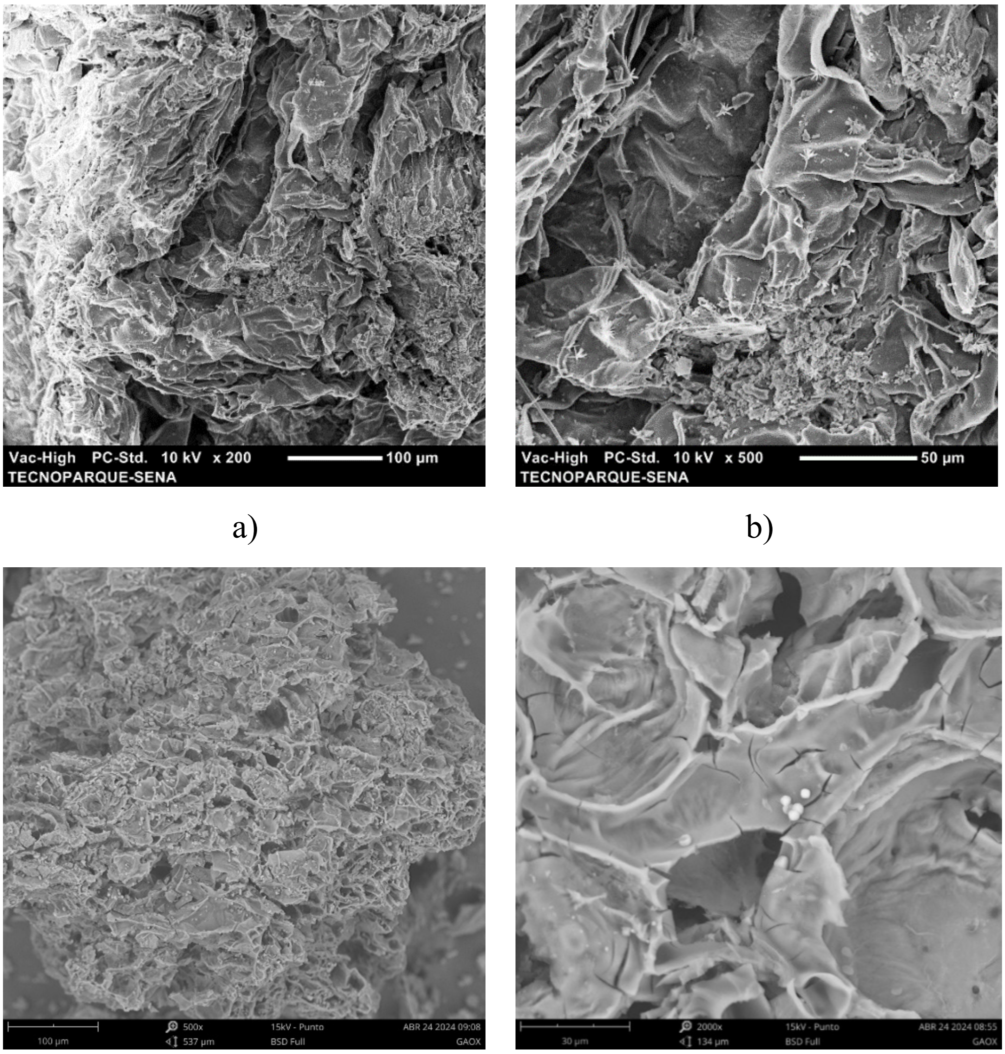
stretching vibration of O-H, as the phenolic compounds present are rich in hydroxyl groups that can persist even after thermal treatment. The peaks observed around 2900 cm^{-1} are attributed to the symmetric and asymmetric stress vibrations of the C-H bond of the methyl and methylene groups. Likewise, the peak observed at 1600 cm^{-1} corresponds to the stretching vibration of the C=C bond, indicating that the organic compounds present in the agro-industrial residues have not been completely carbonized (Chee et al., 2012). It also indicates the presence of the C=O bond (Moosa et al., 2016), typical of carboxylic and phenolic compounds of a polar nature. The band around 1400 cm^{-1} indicates the presence of COO^- functional groups, along with the asymmetric deformation of the methyl group and the scissoring deformation of the methylene group. At 1080 cm^{-1}, the observed band corresponds to the stretching vibration of the C-O bond, a characteristic feature of diverse compounds found in the aloe vera leaf. The wide band depicted between 500 and 900 cm^{-1} comprises a series of overlapped peaks representing the out-of-plane deformation of the O-H and C-H bonds.

The functional groups identified in the bioadsorbent are characteristic of compounds such as alcohols, aldehydes, and phenols that play an important role in the bioadsorption process. Metals donate a pair of electrons to these functional groups to form complexes, allowing the bioadsorption process to take place (Duany et al., 2022). The observed changes in the functional group peaks (Figure 4) support this interaction between the bioadsorbent and aluminum ions.

The porosity of the bioadsorbent material is essential for its adsorption capacity and depends on the raw material used in its production (Lu & Zong, 2018). The formation of pores along the surface is attributed to the release of gases during the thermal treatment process (Balaba et al., 2023). Therefore, the pore size may vary according to the process temperature and the lignin and cellulose content of the raw material (Shetty et al., 2021).

As shown in Figures 5a and 5b, the morphology of the bioadsorbent exhibits a rough structure and irregular pores with large folds before the adsorption process. The pores or active sites indicate that the adsorbent is efficient in capturing and retaining metals in aqueous solutions (Abedi et al., 2016). In contrast, a more complex surface morphology was observed after Al biosorption (Figure 5c). This phenomenon can be attributed to the adherence of Al to the surface of the biosorbent (Figure 5d).

The breakthrough curve for the aluminum bioadsorption process is shown in Figure 6 For a time, a less than ten minutes the outlet concentration decreases to almost zero because, at the beginning of the process, the bioadsorbent does not contain adsorbate, which facilitates a higher mass transfer (McCabe et al., 2007). When the bioadsorbent reaches its maximum adsorption capacity and its breakpoint, it is necessary to interrupt the flow. Often, the breakpoint



a) b)

c) d)

Fig. 5. SEM image of bioadsorbent before adsorption a), b) and after Al adsorption c), d)

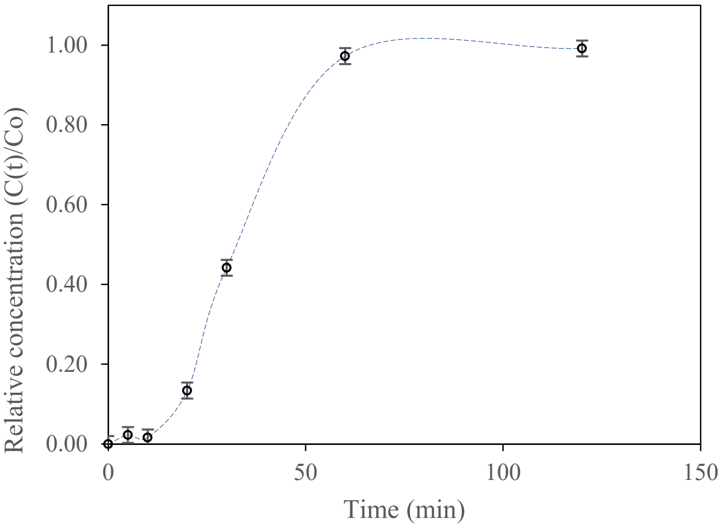


Fig. 6. Breakthrough curve for aluminum adsorption (pH 3, adsorbent mass 4.04 g).

is equivalent to a relative concentration (C(t)/Co) of 0.05 or 0.10 (McCabe et al., 2007). If adsorption continues beyond the breakpoint, the concentration in the effluent would increase very rapidly and then more slowly approach a relative concentration of 1.0.

Based on the results presented in Figure 6, an operating time of 20 minutes has been maintained for all tests.

The pH of the solution significantly affects the adsorption of metal ions by regulating both the surface charge density of the adsorbent and the charges of the metal species present. Depending on the pH, the bioadsorbent undergoes protonation or deprotonation reactions, changing its surface charge and enabling it to function as an anion or cation exchanger. In this study, the effect of pH on aluminum removal was investigated within a range of 3 to 6 with varying amounts of bioadsorbent. To avoid precipitation at higher pH, the pH range was selected based on preliminary batch tests demonstrating stable aluminum determination and higher removal efficiency. As shown in Figure 7, maximum aluminum removal occurs at pH 4.5 due to the greater availability of soluble or exchangeable aluminum (Al^{3+}), which is able to associate with various organic and inorganic ligands present on the bioadsorbent. Conversely, an opposite phenomenon was observed when the pH exceeded 5.0. Under these conditions, the formation of mononuclear and polynuclear species significantly reduces the amount of exchangeable aluminum since these species have lower exchange capacities.

The dose of bioadsorbent is one of the parameters that positively or negatively affects the adsorption process in an aqueous solution. Therefore, the present study investigated the influence of bioadsorbent dosage on the removal efficiency of Al^{3+} ions from solution. Figure 8**.** illustrates the interaction between varying bioadsorbent masses and distinct pH values, providing valuable insights into the optimization of the adsorption process. Within the studied pH range, increasing bioadsorbent dosage consistently yielded higher aluminum removal efficiencies. This trend can be attributed to the increase in available surface area, as a larger bioadsorbent mass translates to more active sites capable of retaining aluminum ions (Malakootian et al., 2015). To understand the mechanism behind the observed increase in removal efficiency with bioadsorbent dosage, the surface chemistry of the bioadsorbent was investigated.

As shown in Table 4, all experimental conditions resulted in aluminum removal percentages greater than 73%. This demonstrates the remarkable efficacy of the bioadsorbent derived from aloe vera processing waste in reducing the aluminum content in water. In particular, it achieved a maximum removal rate of up to 98%, demonstrating its competitiveness with synthetic alternatives (Alalwan et al., 2022; Ali et al., 2022). However, its adsorption capacity is similar

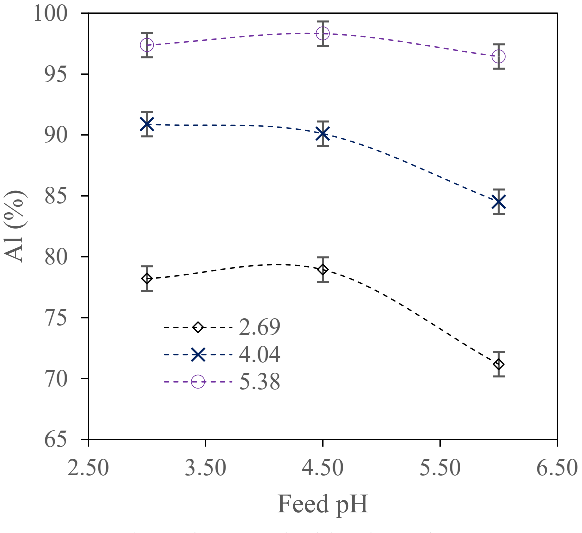


Fig. 7. Effect of pH on the bioadsorption process.

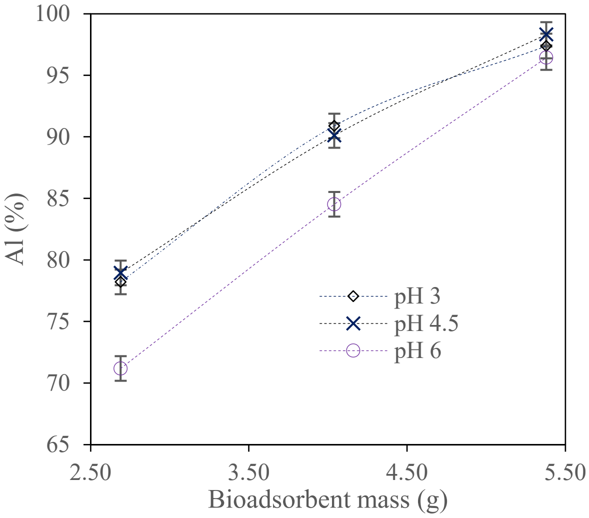


Fig. 8. Effect of adsorbent mass on the bioadsorption process.

Table 4. Adsorption Capacity and Al Removal

Bioadsorbent mass ± 0.01 g	**Feed pH ± 0.3**	**q_T (mg/g) ± 0.01**	**Al Removal (%)**
2.69	3.0	1.14	78
	4.5	1.06	79
	6.0	1.04	73
4.04	3.0	0.93	91
	4.5	0.81	91
	6.0	0.82	86
5.38	3.0	0.75	97
	4.5	0.78	98
	6.0	0.64	97

to that of other industrial waste-derived bioadsorbents due to the nature of the raw material (J. Li et al., 2024; Olawale et al., 2022). Furthermore, the effect of bioadsorbent mass on adsorption capacity suggests that using less adsorbent results in access to more sites, in contrast to using a larger amount of adsorbent (Mohammed et al., 2022).

The impact of each factor and their interactions on aluminum removal was assessed using JMP software. Figure 9 presents a Pareto chart, where factors and factor combinations are ranked in descending order of influence. Factors exceeding the orange band represent statistically significant effects. Thus, varying bioadsorbent mass emerged as the sole significant factor influencing the percentage of aluminum removal.

The bioadsorbent exhibited remarkable efficiency in removing aluminum from water through a diverse array of mechanisms. These include complexation, electrostatic attraction, ion exchange, and surface co-precipitation. (Shetty et al., 2021). The ability of the bioadsorbent to remove aluminum involves several mechanisms, each with different characteristics. Complexation, which depends on the ionic properties of the metal, involves the formation of macromolecules through the binding of surface-active groups to metal ions. For instance, J. Li et al. (2020) indicated that a pH of 4 is suitable for aluminum removal from complexation. Conversely, co-precipitation originates from reactions between metal ions and surface compounds, influenced

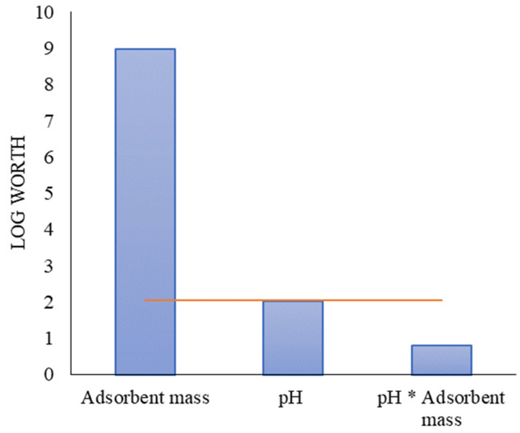


Fig. 9. Pareto diagram for factorial design.

by environmental factors like pH, for removing Al, co-precipitation occurs in pH higher than 6 (W. Li et al., 2024). Ion exchange, despite the variability of its binding nature (physical to chemical), contributes to aluminum reduction in water. Finally, electrostatic attraction, governed by the types and quantity of active sites on the biomass and modulated by the pH of functional groups, plays a crucial role in the adsorption process (Sanchez-Silva et al., 2020; Alalwan et al., 2022).

Based on the findings in Figures 7 and 8, complexation and ion exchange emerge as the dominant mechanisms governing aluminum bioadsorption. While co-precipitation and electrostatic attraction contribute to the overall process, their dependence on pH exhibits minimal impact on the percentage of aluminum removal. The fact that the bioadsorption process is controlled by the formation of surface complexes between the aluminum and the functional groups is supported by the IR spectra in Figure 3. where the presence of oxygen-containing functional groups on the surface of the bioadsorbent is marked.

Figures 7 and 8 reveal a synergistic effect between increasing bioadsorbent mass and decreasing pH, leading to enhanced aluminum removal. This suggests that employing a higher mass at lower pH values is an effective strategy for maximizing aluminum adsorption.

CONCLUSIONS

The studied bioadsorbent distinguishes itself by its robust framework and intricate network of surface pores, accompanied by an abundance of functional groups characteristic of alcohols, aldehydes, and phenols. This combination of structural features positions it as a highly promising candidate for bioadsorption applications.

The influence of influent pH on aluminum removal capacity by the bioadsorbent was investigated within a range of 3 to 6. Notably, no significant effect was observed, suggesting a robust adsorption performance across this pH range. In contrast, bioadsorbent dosage exhibited a positive correlation with aluminum removal, implying that higher removal percentages can be achieved by employing the maximum tested dose. This is further supported by the observation that all experimental conditions yielded aluminum removal exceeding 73% with the maximum removal (about 98%) observed at pH 4.5 and an adsorption capacity between 1.14-0.64 mg/g. These findings highlight the potential of the bioadsorbent for implementation in water treatment systems targeting aluminum concentrations between 7.9 and 9.4 ppm.

To assess the efficiency of the bioadsorbent derived from agro-industrial aloe vera waste, further studies are warranted to explore the influence of additional variables governing the bioadsorption process, such as contaminant concentration and influent flow rate. Moreover, evaluating its removal efficiency towards other pollutants, including heavy metals and dyes, would broaden its potential for diverse environmental treatment applications and showcase its versatility.

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CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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