

SEM - EDS ON THE STUDY OF THE EFFECTS OF pH FOR THE REMOVAL OF Al (III) USING MORINGA *OLEÍFERA* PODS AS A BIOADSORBENT FROM AQUEOUS SYSTEMS.

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ABSTRACT

Nowadays, water contamination by heavy metals has become a world-wide environmental problem. Recently, bioadsorption has been proposed as an alternative treatment procedure. Moreover, enhancement of agro-industrial waste is the great interest. In this sense, the aim of this study is to perform a chemical characterization of moringa pods and subsequent study as bioadsorbent for removal of aluminum (III) from aqueous systems. The pods used were collected from Guárico, Venezuela. After dried, ground and sieved to a particle size of 595 μm , were characterized by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDS), Fourier transform infrared spectroscopy (FT-IR) and point of zero charge (pH_{PZC}). The potential of pods was tested under different pH conditions (2 to 9), biomass (0.25 g), metal concentration (50.0 mg/L), contact time (30 min) and volume of solution (30.0 ml) by batch system. The residual metal concentration was measured by atomic absorption spectrophotometry (AAS). The results showed that this biomaterial containing functional groups and adequate morphological profile for the retention of metal ions. Additionally, present a point of zero charge of 6. The optimal removal of Al (III) ions was achieved at pH 7 (99%). According to achieved results, moringa pods have promising characteristics for applications aluminum (III) remediation from contaminated waters at low cost, easy acquisition, eco-friendly and relatively neutral pH.

Keywords: Moringa-pods, Bioadsorbent, Aluminum, Aqueous-system

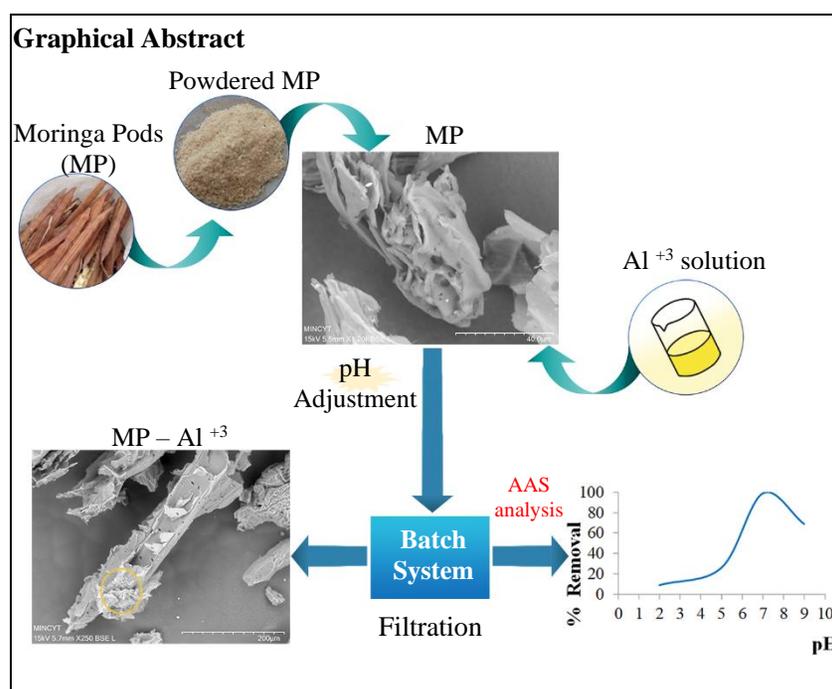
MEB-EDX en el Estudio de los Efectos del pH para la Remoción de Al (III) usando Cápsulas de Moringa *Oleífera* como Bioadsorbente en Sistemas Acuáticos.

RESUMEN

Actualmente, la contaminación de las aguas por metales pesados se ha convertido en un problema ambiental mundial. Recientemente, la bioadsorción ha sido propuesta como un procedimiento de tratamiento alternativo. Por otra parte, la valorización de los residuos agroindustriales es de gran interés. En este sentido, el objetivo del presente estudio es realizar una caracterización química de las cápsulas de moringa y su posterior estudio como bioadsorbente para remoción de aluminio (III) en sistemas acuáticos. Las cápsulas usadas fueron recolectadas en Guárico, Venezuela. Después de secadas, pulverizadas y tamizadas a un tamaño de partícula de 595 μm fueron caracterizadas mediante microscopía electrónica de

barrido /espectroscopia de rayos X de energía dispersiva (MEB/EDX), espectroscopia infrarroja con Transformada de Fourier (FT-IR) y punto de carga cero (pH_{PZC}). El potencial de las cápsulas fue probado bajo diferentes condiciones de pH (2 a 9), biomasa (0,25 g), concentración de aluminio (50,0 mg/L), tiempo de contacto (30 min) y volumen de solución (30 mL) mediante sistema batch. La concentración residual del metal fue medida por espectrofotometría de absorción atómica. (AAS). Los resultados mostraron que este biomaterial posee grupos funcionales y un perfil morfológico adecuado para la retención de iones metálicos. Adicionalmente, presenta un punto de carga cero de 6. La remoción óptima de iones Al (III) fue alcanzada a pH 7(99%). Finalmente, las cápsulas de moringa poseen características prometedoras para aplicaciones en remediación de aluminio (III) desde aguas contaminadas a bajo costo, fácil adquisición, eficiente, amigable al ambiente y a pH relativamente neutros.

Palabras claves: Cápsulas de moringa, bioadsorbente, aluminio, sistema acuoso.



Abbreviations

EDS	Energy-dispersive X-ray spectroscopy
FT-IR	Fourier transform infrared spectroscopy
pH_{PZC}	Point of Zero Charge
MP	Moringa pods
MP-Al	Moringa pods- aluminum
SEM	Scanning electron microscopy

INTRODUCTION

Al-based coagulant such as $\text{Al}_2(\text{SO}_4)_3$ (aluminum sulphate), better known as alum or poly-aluminum chloride (PACl) are commonly used in drinking water

treatment to enhance the removal of particulate, colloidal and dissolved substances via coagulation processes [1]. However, concerns have been raised that the use such coagulants may increase aluminum concentrations in treated water [2]. Based on their toxicity, the Environmental Protection Agency (EPA, 2008) sets the secondary permissible standard for aluminum in drinking water as 0.05–0.20 mg/l [3]. World health organization (WHO) demanded the residual Al concentration in drinking water must be lower than 0.2 mg/l [4]. Therefore, it is important to remove the Al (III) from water before being used as drinking water, given that elevated concentrations of aluminum may pose a potential risk to human health, resulting in brain changes characteristic of Alzheimer's disease [4]. Further, it is implicated in bone softening, renal insufficiency, pulmonary fibrosis, microcytic anaemia [5] and neurological disorder known as autism spectrum disorders (ASD) or simply autism [6].

Several methods have been used for the removal of heavy metals from aqueous system, including chemical precipitation, reverse osmosis, ion exchange, electro-coagulation, flotation, electrochemical treatment and membrane processes, among others [7-12]. However, these methods have many disadvantages, such as generation of toxic chemical sludge, incomplete metal removal, low efficiency, complicated treatment process, high cost and high energy consumption requirement [13]. Hence, new cost effective, safe and economic tools are recommended to reduce the limitations of the conventional methods. In recent years, bioadsorption technique has become an emerging alternative treatment procedure, due to its low cost and high efficiency for the removal of heavy metals at very low concentrations [14]. Moreover, enhancement of agro-industrial waste as raw material for the production of new material is the great interest [14]. Recently, some types of agricultural waste such as sugarcane bagasse [5], rice husk [15], guava leaves [16], rice straw [17], date pit [3] and tea leaves

[18] have been used as bioadsorbent for the removal of Al (III) ions from aqueous solution.

Nowadays, large amounts of agricultural waste, such as moringa pods are produced in Venezuela. Moringa (*Moringa oleifera* Lam.) belong to the Moringaceae family and it originated from the India's northeast. The specie is known for its high nutritional value, and almost all parts of the plant are used as food as well as having medicinal and industrial importance [19]. It is well known that moringa seeds have been used as an adsorbent for the removal of heavy metals such as lead (Pb), cadmium (Cd), and copper (Cu) [20]. Lead (Pb), cadmium (Cd), cobalt (Co) and nickel (Ni) [21]. Manganese (Mn) [22]. Also, copper (Cu), chromium (Cr), lead (Pb) and zinc (Zn) [23] from aqueous solutions. There are few works on metal ions adsorption using moringa pods [14, 24-25]. At present, the use of moringa pods as a bioadsorbent of aluminum in aqueous system has not been reported.

The aim of this research work was to investigate the ability of Moringa pods as a bioadsorbent for removal of Al (III) ions from aqueous solutions. The effect of solution pH was evaluated.

MATERIALS AND METHODS

Moringa oleifera pods (MP) were collected from Guárico State, Venezuela. Pods were soaked and washed with distilled water; oven dried at 60 °C for a 24h period. After drying, pods were grounded using a commercial blender (FISHER). Subsequently, the resultant powders be sieving to a particle size of 595 µm. Finally, were collected in a plastic bag at room temperature and used as bioadsorbent for adsorption experiments. The MP powders were characterized using several techniques. Fourier transform infrared spectroscopy (FT-IR) was used to identify the different functional groups available on the bioadsorbent sites and their effect on metal ion adsorption. The FT-IR spectra of the adsorbent were taken before and after adsorption using FT-IR

spectrophotometer (Elmer Perkin Spectrum 100) within the wave number range 4,000 – 500 cm^{-1} using the KBr standard method. The surface morphology of moringa pods before and after Al^{3+} adsorption was evaluated by scanning electron microscopy (SEM). Elemental analysis was then recorded by energy-dispersive X-ray spectroscopy (EDS) (Hitachi model microscope coupled at EDS). As further characterization, the point of zero charge (pH_{PZC}) of the moringa pods was determined according to literature [26]. The procedure consisted of adding 50 mg of the bioadsorbent in 50 ml aqueous potassium chloride solution (KCl) at 0.05 and 0.5 molL^{-1} at various initial pH values from 2.0 to 9.0 which were adjusted with solutions of hydrochloric acid (HCl) and sodium hydroxide (NaOH) both at 0.1 mol L^{-1} . After the mixtures were agitated (200 rpm) for 24 h at room temperature. Followed, the phases were separated by centrifugation, and each solution's pH was measured using a pH-meter (FISHER ACCUMET model 230A) resulting in a graph with initial pH depending on the final pH, being that the pH range there was no change to the final pH corresponds to the pH_{PZC} .

Adsorption studies of Al (III) ions were carried out using the batch system under different pH conditions (2 to 9), biomass (0.25 g), metal concentration (50.0 mg/l), contact time (30 min), volume of solution (30ml) at 150 rpm and room temperature (25°C). Finally, the supernatants were filtered through a Whatman filter paper (0.45 μm pore size).

The residual metal concentration in the solution was measured by atomic absorption spectrophotometry (AAS) using Agilent 55 AA equipment and it was operated under the conditions recommended by the manufacturer.

The Al (III) ions removal percentage (R) was calculated by the following equation:

$$R = \frac{C_0 - C_f}{C_f} \times 100$$

where C_0 and C_f are the ions concentrations at the initial and final time (mg/l), respectively.

RESULTS AND DISCUSSION

In order to determine the characteristic functional groups responsible for biosorption of Al (III) ions on moringa pods surface FT-IR spectroscopy was applied. Figure 1 shows FT-IR spectra obtained for moringa pods before and after the adsorption process. In general, it is observed that present similar profiles, with characteristic bands of lignocellulosic materials (cellulose, hemicellulose and lignin), which contain carboxylic groups (-COOH) and are identified by several authors as the functional groups responsible for adsorption process metal ions [27]. Figure 1 (a) illustrate a broad band centered at 3,434 cm^{-1} which may be attributed to the stretching of the OH and N-H groups present in proteins, fatty acids, carbohydrates (cellulose and hemicelluloses), lignin and water absorbed [28]. The peaks present at 2,918 and 2,850 cm^{-1} , respectively, correspond to asymmetric and symmetric stretching of the C-H bond of the CH_2 group. In the region of 1,800-1,500 cm^{-1} a number of overlapping bands are observed and between 1,714 and 1,631 cm^{-1} this can be attributed to C=O stretching. Due to the heterogeneous nature of the pods, the carbonyl group may be bonded to different neighborhoods as part of the fatty acids of the lipid portion or amides of the protein portion. The peak observed at 1,509 cm^{-1} may be attributed to stretching connecting C-N and also the deformation of the N-H bond [29] present in proteins of moringa pods. The peaks at 1,110 – 1034 cm^{-1} may be attributed to the vibrational stretching of C-O bond of amide and carboxylic groups (-COOH), which are characteristic bands of lignocellulosic materials. The band at 1,059 attributed to C-O stretching also suggests the presence of lignin which has compounds such as carboxyl groups that provide sites for adsorption of metal ions [30]. Thus, according to Sharma *et al.*, 2006 the adsorption of metals by vegetable wastes, natural

materials, and agro-industrial byproducts can be attributed to the presence of some functional groups such as lignin, proteins, and carbohydrates, which contribute to the adsorption of metal ions [31]. FT-IR spectrum of MP after Al (III) adsorption (Figure 1 (b)) showed similar characteristics as that of moringa pods with slight changes at some absorption bands, which indicated the participation of specified functional groups of MP in Al^{3+} adsorption. The main differences between two spectra are (i) a small decrease and slight shift in the band intensity around at $3,434\text{ cm}^{-1}$ (OH), 1714 and 1631 cm^{-1} (C=O), (ii) an increase and slight shift in the band intensity around at $2,918$ and $2,850\text{ cm}^{-1}$, (iii) the appearance of a small band in around 2100 cm^{-1} and (iv) significant changes at absorption bands between 700 to 500 cm^{-1} , which it could be assigned to the interaction between functional groups present in the bioadsorbent with the metal ion.

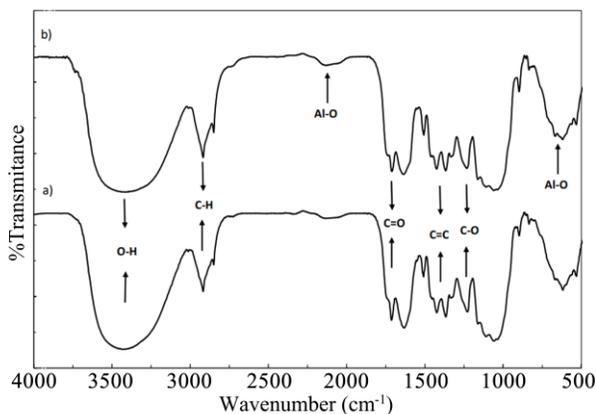
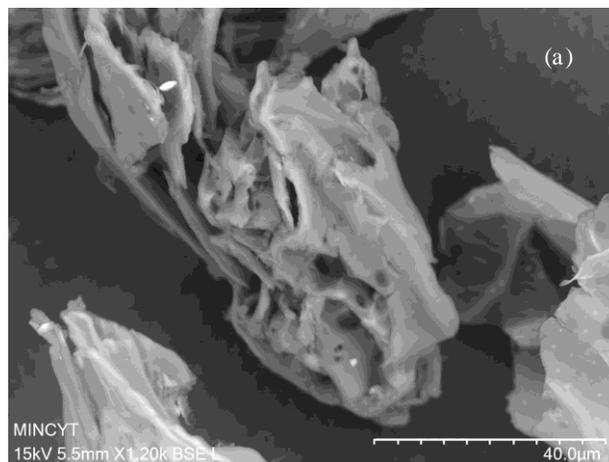


Fig.1. FT-IR spectra for MP (a) and (b) MP-Al at pH = 7

Figure 2 shows SEM micrograph and EDS spectrum of moringa pods before Al (III) ions adsorption process. Fig. 2 (a) indicates that the material surface exhibits a relatively porous and heterogeneous structure. This feature is attributed to the fact that the whole pod comprises a wide variety of material components. The presence of some deformations on the surface of material can be observed, containing available sites, from which it

is possible to infer that the bioadsorbent provides favorable conditions for the adsorption of ionic species in the interstices [14]. As shown in Fig. 2(b), the main elements were C and O. The carbon and oxygen elements are originated from ligno-cellulosic nature of bioadsorbent. Figure 3 illustrates SEM image and EDS spectrum obtained for moringa pods after adsorption Al (III) solution at pH = 7, where it can be clearly seen the variation of the morphology of MP, with obvious particles of amorphous precipitate, possibly due to deposition of Al (III) ions on bioadsorbent surface. It could be attributed to the bridging ability of functional groups on MP surface with metal ions. The loading of Al^{3+} onto MP was confirmed by EDS spectra. In contrast, a new Al peak was found in MP-Al sample (0.83 Wt. %). These findings suggested that MP could adsorb Al^{3+} from aqueous solution via a physical process. Van der Waals interactions between Al ion and MP surface functional groups can be suggested for this process. These interactions are of main interactions in physical adsorption [18].



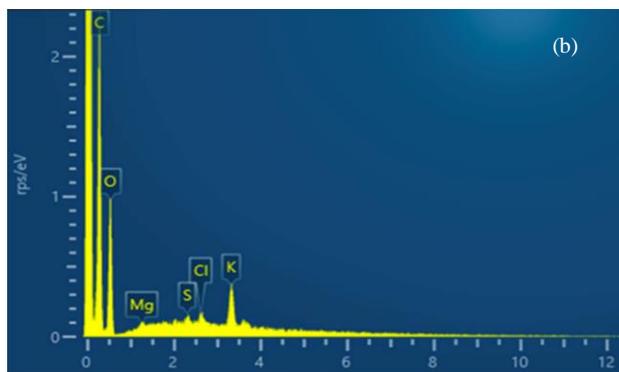


Fig.2. SEM micrograph and EDS spectrum of MP.

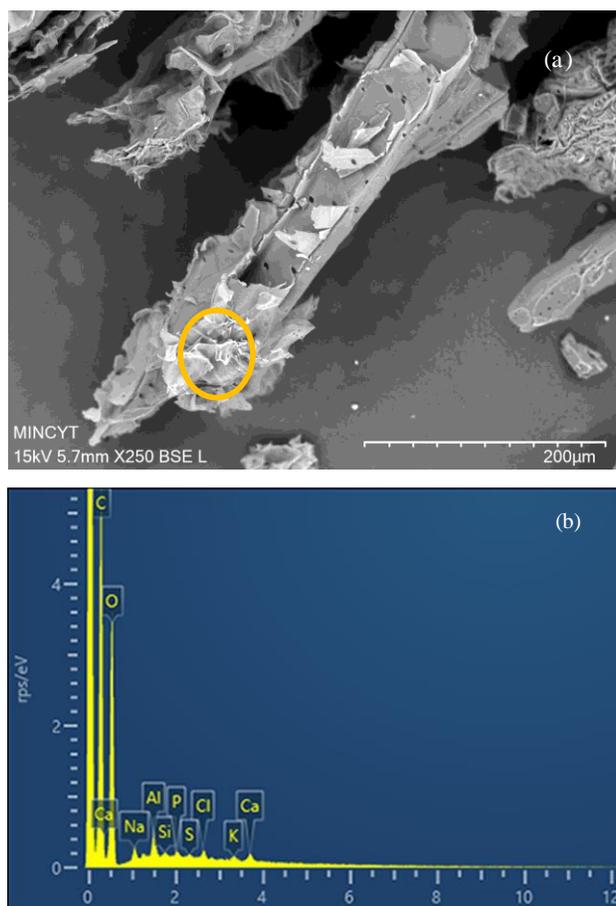


Fig.3. SEM micrograph and EDS spectrum of MP- Al at pH = 7.

The pH is one of the parameters with more importance in the adsorption process, since it interferes in the solid–solution interface, influencing the charges of the active sites of the biomass and also on the metal behavior in the

solution [19]. A decrease in pH means that more H⁺ ions are present in solution, and the biomass is then protonated; thus, the active sites of adsorption are occupied by H⁺ ions before the metal can occupy them. [19]. In the case of the carboxylic groups present in moringa byproduct, when the solution pH has highly acidic character (value less than 3.0), it acts as positively charged species and then attract anions [19]. However, with the increase in pH, there is a deprotonation of these groups due to a higher amount of negative charges. The adsorption process is favored by the attraction of the positive charges (ions) of the heavy metals [19].

The influence of pH on adsorption can be describe on the basis point zero changes (pH_{PZC}), which is the point at which the net charge of the adsorbent is zero [32]. Depending on the pH of the solution, their surfaces can be positively or negatively charged. At pH values greater than pH_{PZC}, the biomass surface becomes negatively charged, favoring the adsorption of cationic species, and the process is highly favored though electrostatic force of attraction [32]. However, adsorption of anionic species will be favored at pH < pH_{PZC} [32]. In order to understand the mechanism involved in the adsorption process of the Al³⁺ on the bioadsorbent, it is necessary to determine the point of zero charge (pH_{PZC}) of moringa pods. The results obtained in KCl solutions indicated that the pH_{PZC} for moringa pods is about pH 6.0 (Fig.4). Thus, the adsorption of cations in case of metal ions is favored at pH values above the pH_{PZC}.

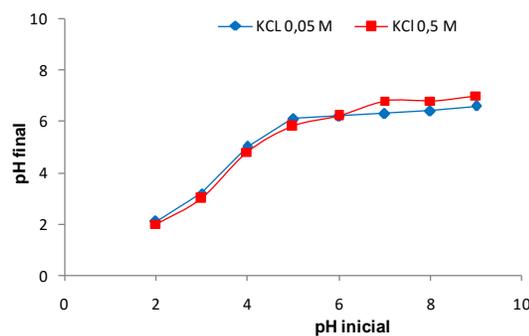


Fig.4. Point of zero charge of MP.

The influence of solution pH on aluminum (III) removal using moringa pods is shown in Fig.5. The effect of pH was examined in the range of 2.0 to 9.0 under conditions of room temperature. The removal of Al increased with the increase in pH of solution from 2 to 5. The highest metal ion removal occurs at pH 7 (~99 %). Similar results were reported for adsorption of aluminum on tea leaves [18] and other natural compound [33]. For this case, at pH 7, surface of moringa pods was negatively charged to its maximum extent, confirming the results obtained in the study of pH_{PZC} . However, it is apparent that in both acidic and alkaline pH, the adsorption of Al^{3+} decreased than the neutral pH. This could be due to the competition between the cation H^+ and Al^{3+} in the acidic medium and the existence of OH^- ions and formation of hydrous Al precipitate ($Al(OH)_3$) in alkaline medium [19]. A similar trend has been observed for other authors [1, 16, 18, 34]

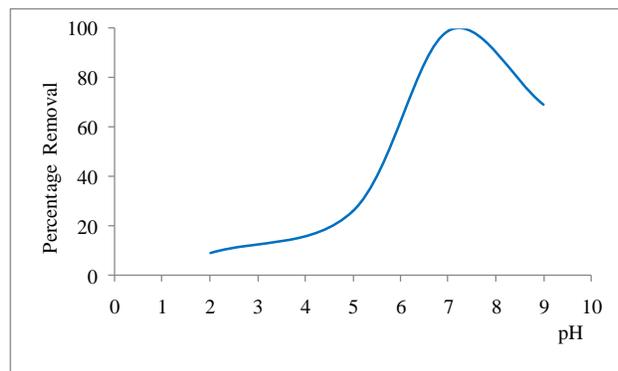


Fig.5. pH influence on percentage removal of Al (III) using MP as bioadsorbent.

CONCLUSIONS

Moringa pods (MP) can be used as an effective alternative low-cost bioadsorbent for the removal of aluminum (III) ions from aqueous solutions. The characterization by Fourier transform infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM) showed that this biomaterial containing functional

groups and adequate morphological profile for the retention of metal ions. In addition, energy dispersive X-ray spectroscopy (EDS) analysis revealed that bioadsorption process is strongly influenced by solution pH. In this case, to very low pH values (pH= 2.0) are not favored adsorption process, observing the optimal percentage at about pH= 7.0 (~99%). This can be attributed mainly to electrostatic attraction between the carboxylate ions negatively charged on MP surface and the positively charged cationic ions (Al^{3+}). Finally, moringa pods have promising characteristics for applications aluminum (III) remediation from contaminated waters at low cost, easy acquisition, eco-friendly and relatively neutral pH.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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