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Original Research Article

Sustainable adsorbents from *Moringa oleifera* residues: chemical modifications and nickel removal efficiency

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ABSTRACT

In this work, the potential reuse of solid effluents from a *Moringa oleifera* infusion production company as adsorbents for Nickel removal from aqueous media was evaluated. To enhance adsorption performance, the material was chemically modified through an alkaline pretreatment, followed by carboxymethylation or acetylation. The obtained products were characterized by Fourier Transform Infrared spectroscopy to confirm the incorporation of functional groups. The adsorption capacity of the materials was determined, revealing that the moderate improvement observed for the carboxymethylated sample does not justify its low synthesis yield and the increased process complexity. Therefore, only the raw material, the alkaline-pretreated sample, and the acetylated adsorbent were selected for detailed evaluation. Kinetic and equilibrium experiments, as well as packed-column tests, demonstrated that moringa residues exhibit promising performance for nickel ions removal. Overall, these results highlight moringa-based materials as low-cost and sustainable adsorbents for wastewater treatment applications.

KEYWORDS

Moringa oleífera, Surface-modification, Acetylation, Adsorption, Nickel, Fixed bed reactor

INTRODUCTION

In a production process, not only economic costs but also environmental costs must be considered, defining cost as the sacrifice of resources or what is given up to achieve a specific objective, whether to acquire or produce a good or a service. The current challenge is in this double reduction to improve the feasibility of applying treatments[1]. Regarding environmental costs, although every production process generates unwanted by-products, efforts should be made to minimize their release to the environment and ensure they are not harmful to its inhabitants. One of the *precepts of the Circular Economy is the proposal of the reuse of waste and its inclusion in a new production line, in order to reduce both the entry of new raw materials to the production system and the generation of waste[2].

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In this sense, the Amsterdam Pact focuses on waste management (turning waste into a resource), the shared economy and resource efficiency [3]. In this way, "El Moringuero", a moringa production comp†any that develops its crops in a sustainable way, is looking to solve the problem of the volume of waste generated. On the other hand, the concentration of metals in water bodies has increased due to rapid industrialization, increased industrial waste, increased consumption of agricultural inputs and mining [4]. This work is proposed in order to reduce this pollution and contribute to the fulfilment of the Sustainable Development Goals [5]. *Moringa oleifera* is a fast-growing deciduous tree widely cultivated in tropical and subtropical regions of Africa, Asia and Latin America. It is considered a multipurpose crop due to the wide range of valuable products that can be obtained from its different anatomical parts. The leaves are one of the most exploited fractions because of their high nutritional value and are commonly used for the production of infusions, powders and dietary supplements. The seeds are mainly used for oil extraction, yielding a high-quality edible oil, and the resulting press cake is often applied in water treatment due to its natural coagulant properties. Other parts of the plant, including pods, bark and roots, also have applications in food, traditional medicine [6]. However, despite this versatility, a significant fraction of the biomass generated during cultivation and processing is not utilized. It is estimated that only 20–30% of the total plant biomass is effectively valorised, while the remaining 70–80% corresponds to lignocellulosic residues such as branches, peduncles, stems and seed husks [7]. In the particular case of infusion production, such as that carried out by the company "El Moringuero", only the leaves are harvested and processed, generating solid residues mainly composed of branches, peduncles and trunk fractions. These residues represent a substantial proportion of the harvested biomass (typically above 60% on a dry weight basis) and are usually discarded without further valorization [8]. This underutilization highlights the potential of these materials as low-cost feedstocks for the development of value-added products, such as adsorbents for water treatment.

Metal removal from effluents represents a complex challenge and the methodology to be used must be easy to implement, effective and low cost in order to be widely adopted. Conventional methods are chemical precipitation, electrochemical techniques, reverse osmosis, evaporation and ion exchange, among others. However, these methods can be highly expensive or inefficient as they can create sludge with metals that are difficult to dispose of [9].

An interesting technique is adsorption, a surface phenomenon in which a wide variety of contaminants are removed through physicochemical interactions with rapid and possibly selective kinetics. The performance of this technique is closely linked to the morphological and physicochemical characteristics of the adsorbent. The adsorbents widely used are activated carbons, clays, biopolymers, among others. However, these materials have the disadvantage of being expensive. In recent years, due to the imperative need to address the removal of metals from contaminated waters in an efficient and economically viable manner, research has been encouraged towards the development of affordable alternative adsorbents. In this regard, the exploration of various resources from agroindustry is being promoted in order to produce low-cost, commercially available adsorbents. Therefore, it is essential to study their potential sources and evaluate their effectiveness in metal removal [10].

In Argentina, a country with rich natural and cultural diversity, agriculture plays a crucial role in the economy, and the growing need for agricultural products is an excellent opportunity to foster equitable development in all regions of the country. This is achieved by strengthening primary production and, especially, by promoting added value in agroindustry [11]. One of the ways to add value to a crop is to introduce the material considered as waste into a new

production line to take advantage of it. This reduces waste, diminishes the effect on the environment and reduces the economic cost for the companies responsible for its disposal.

Moringa oleífera is a deciduous tree of which only its leaves and seeds are used in most cases, and the rest of the plant is rejected [12]. It has become one of the crops that are booming today, from the discovery of the high nutritional value and the great variety of properties that it possesses, which has allowed its use in cosmetic, pharmaceutical and food industries [13]. These residues were used in this work as lignocellulosic material for nickel bioadsorption.

In order to enhance the efficiency of ion adsorption, chemical modifications can be made to the surface. These modifications could be considered as pretreatment of the lignocellulosic material and should be carried out in such a way as to keep environmental and economic costs low.

Pretreatment technologies have different objectives, including increasing the accessibility of specific functional groups that could favour adsorption. Acidic or alkaline solutions are frequently used to modify or eliminate lignin and hemicellulose from biomass. This pretreatment can be carried out using concentrated or diluted solutions. For example, in one case of alkaline pretreatment, an increase in surface roughness, surface exposure of the fiber and partial elimination of substances that act as fiber cement (lignin, hemicelluloses and pectin) were observed [14]. On the other hand, the introduction of new functional groups, such as carboxyl groups through carboxymethylation [15], increases the surface polarity and generates negatively charged sites at pH values above 6, thereby favoring the adsorption of metal cations through electrostatic (ionic) interactions [16]. That is why it was thought to work with simple modifications tested by other authors in materials such as coconut shell [17], agave bagasse [18], sugar cane bagasse [19], among others. Some authors also made modifications in the leaves of moringa, with satisfactory results. However, given the high nutritional value they currently have, the leaves are no longer considered crop residues [20].

This approach points to a possible application of these low-cost treatments in industries with metal content effluents. Metal ions are non-biodegradable substances that can bioaccumulate and even produce biomagnification in the environment, so that an inadequate disposal of industrial effluents could put at risk the health of living beings, the ecosystem and therefore the environment.

Nickel is a metal widely used in industry due to its physical and chemical properties, including its resistance to corrosion, electrical conductivity and ability to form alloys. It is used in the manufacture of stainless steel, rechargeable batteries, coins and catalysts, among other products. This metal and certain compounds that contain it are considered harmful to human health and are also bioaccumulative, making it dangerous for the environment. Some of the health effects include dermatitis, gastrointestinal problems, systemic toxicity, and they have also been classified as carcinogenic to humans by several health and regulatory agencies, such as the International Agency for Research on Cancer (IARC) [21] and the United States Environmental Protection Agency (EPA) [22]. Currently, in Argentina, Law No. 24,051 [23] sets the limit of the metal content for the effluent to be discharged or used in various ways, with a concentration range in the receiving water body between 0.1 and 1.0 ppm. Buenos Aires Provincial Law No. 12,257, establishes strict limits for the discharge of liquid effluents, including a maximum allowed for nickel, generally set at 2 mg/L for receiving bodies, seeking to protect water resources [24].

In this sense, the proposal of a solution that combines the removal of nickel from industrial effluents with the reuse of moringa waste would integrate the process within the framework of the Circular Economy. This perspective promotes the supply of raw materials from secondary sources, which contributes to closing the life cycle of products and reducing dependence on primary natural resources. The adoption and application of a treatment system for metal effluents would result in an immediate reduction of the environmental impacts generated by industrial activities, as well as in the preservation of public health and water resources, one of the greatest concerns due to the continuous increase in population. To achieve this objective,

an effective system is required, as mentioned above, easy to implement, requiring little maintenance and economically accessible, such as biosorption [25].

Nowadays, it is considered that the retention of contaminants, in particular metals, on the surface of low-cost adsorbents, which are usually non-porous, does not occur only due to adsorption, but also as a result of other processes, such as microprecipitation, ion exchange and sometimes redox chemical reactions and complex formation with functional groups present on the surface [26]. In relation to this last procedure, it is worth highlighting the common characteristic shared by these wastes: the presence of natural biopolymers in their structures such as cellulose, hemicellulose or lignin [27] that are characterized by the presence of different functional chemical groups, mainly hydroxyl and carboxyl; which play a key role in the adsorption of contaminants [28].

In this work, the possibility of reusing solid effluents from a moringa infusion production company to remove nickel from other industrial effluents was investigated. In order to maximize the adsorption capacity, the surface of these adsorbents was chemically modified. First, an alkaline pretreatment was applied to open the lignocellulosic structure and increase fiber accessibility, allowing the reagents used in the subsequent surface modifications to interact more efficiently with the material. Then, two different chemical modifications were carried out. These two routes were selected because they involve simple, well-established chemical modifications as functional derived from esterified cellulose [29], carboxymethyl cellulose derivatives [30] or functional nanomaterials derived from diverse esterified cellulose compounds [29] that are widely applied in differents industries as food, wastewater treatment, medicine, papermaking, etc.[31], , making them suitable for potential large-scale implementation. On one hand, a carboxymethylation process was performed to introduce negatively charged groups capable of binding positively charged ions as nickel (Ni^{2+}). The working hypothesis was that this modification would enhance the removal of cations through an ion-exchange mechanism. On the other hand, an acetylation process was conducted on a separate fraction of the pretreated material. In this case, the hypothesis was that acetyl groups would disrupt the native hydrogen-bonding network within the lignocellulosic matrix, exposing oxygen lone pairs that could coordinate metal species, thereby improving the removal of cations through a chelation mechanism.

The significance of this work lies in carrying out a systematic evaluation of relevant modification routes of a novel material for industry (alkaline pretreatment, carboxymethylation and acetylation) within a common experimental framework; and the selection of the most suitable treatment considering not only the adsorption yield but also practical criteria such as synthesis yield, process simplicity and scalability.

The modified materials were characterized by their surface area and the presence of surface functional groups. Then, nickel adsorption studies were conducted through batch tests, and the kinetic and equilibrium results, as well as the performance in fixed bed columns, were compared.

MATERIALS AND METHODS

The *Moringa oleífera* wastes were gently provided by "El Moringuero SA" company, located in Misiones, Argentine. The supplier previously processed the residues by coarse grinding and packed in 3 kg bags. A representative sampling was carried out from this material using statistical data for bulk material [32], and a finely ground material was collected from the sieve (ASTM N°16-35), with a size between 500 and 850 µm, which was used as adsorbent material in all the tests carried out. A previous washing stage in deionized water at 50°C and drying in an oven at 60°C for 24 h was conducted. This material was designated as raw moringa (RM).

Sodium hydroxide (NaOH), hydrochloric acid (HCl), sulphuric acid (H_2SO_4) and potassium nitrate (KNO_3) were analytical grade and purchased from Merck®. Monochloroacetic acid (MCA) and acetic anhydride (Ac_2O) were both reagent grade (Merck®).

Alkaline pretreatment of raw moringa

The alkaline pretreatment was carried out as previously reported for other biomaterials [33]. Briefly, RM (1.00 g) was suspended in 20.0 mL of NaOH (0.10 M) and stirred for one hour at two different temperatures (25 and 100°C). After this time, the material was filtered, washed with distilled water until neutral pH was reached, and dried at 50°C for 16 h. The resulting solids yielded 0.91 g for the sample treated at 25 °C and 0.75 g for the sample treated under reflux, reflecting the partial extraction of base-soluble components. The materials obtained at both temperatures were characterized in terms of their chemical structure and nickel adsorption capacity. Based on these results, the room-temperature pretreatment was selected for all subsequent experiments, and the resulting material was designated as pretreated moringa (PM).

Carboxymethylation

Carboxymethylation was carried out as previously reported [34]. Briefly, PM (1.00 g) was suspended in 27 mL of ethanol (96 %) and vigorously stirred at 55°C. Then, 2.7 mL of an aqueous NaOH solution (40% w/v) were added dropwise over 30 minutes. The resulting suspension was stirred at 55°C for 2.5 h. Finally, sodium chloroacetate, reagent grade Merck ®), (1.50 g) was added and the mixture was stirred under the same temperature conditions for one hour. After this time, glacial acetic acid, reagent grade (Merck ®), was added until the pH reached 8–9 [35], the resulting material was filtered, washed with ethanol (90%), and dried under vacuum at 50°C for 16 h, yielding 1.16 g of the carboxymethylated product [36]. Subsequently, a fractionation based on water solubility was performed by suspending the material in 250.0 mL of water and stirring for 1 h. The mixture was filtered, and the residue was dried under vacuum at 50°C to recover the water-insoluble fraction, corresponding to a 60% yield, called carboxymethylated moringa (CM).

Acetylation

The acetylation was carried out as previously reported[37]. Briefly, 5.0 mL of Ac_2O were placed in a beaker, and then 10 µL of H_2SO_4 were added. Subsequently, 1.00 g of PM was added, purged with a stream of argon, and sealed to minimize anhydride hydrolysis by moisture. The acetylation was carried out at room temperature, without stirring, for 24 h. Then, the sample was diluted in 100.0 mL of distilled water, filtered, and washed until neutral. Finally, the modified sample was dried under vacuum at 50°C for 16 h, yielding 0.98 g of the acetylated product (AM1). A second modification was carried out under the same conditions but using 10.0 mL of Ac_2O and 20 µL of H_2SO_4, yielding 1.13 g of product (AM2).

Evaluation of impurities release from adsorbents

To assess whether a column packing material does not release its own contaminants into the water it is treating, rigorous standards and laboratory tests are followed [38]. In this work, the inorganic components released into deionized water were quantified by Total Reflection X-Ray Fluorescence analysis (TXRF) with an BRUKER® S2 PICOFOX spectrometer (Germany)[39].

Chemical characterization: Fourier Transform Infrared Spectroscopy (FT-IR)

All the materials were analysed in a Thermo Scientific® Nicolet 6700 spectrometer (USA). Each sample was mixed with potassium chloride (KCl) (Thermo® Spectra-Tech Grade, FT-IR 99+%, USA) and subsequently pressed into a 3 mm disc using a Hand Press accessory from PIKE Technologies®. Spectral data were collected with a resolution of 4 cm^{-1} over the range of 400-4000 cm^{-1} and 32 scans per sample.

Determination of degree of substitution

The degree of substitution of both acetylated materials (AM1 and AM2) was determined by standard saponification methodology [40]. Briefly, 0.100 g of the acetylated sample was added to 100 mL Erlenmeyer flasks containing 10 mL of 75% (v/v) ethanol and a few drops of phenolphthalein. The mixture was then heated at 55°C for 30 min. After this time, 0.1 M NaOH was added dropwise until the phenolphthalein endpoint was reached. Subsequently, 10 mL of 0.1 M NaOH were added, and the solution was heated at 55°C for an additional 15 min. Finally, the samples were left undisturbed for 48 h. The excess NaOH was then back-titrated with 0.1 M hydrochloric acid (HCl), using the PM as a blank.

Quantification of adsorbate

The solutions used in the calibration curve were prepared by diluting a standard solution of 1000 mg Ni^{2+} L^{-1} (SCP SCIENCE®). Atomic Absorption spectrometry (GBC®-XplorAA) was used for the determination of nickel in solution, following the standard method APHA 3111B[41].

Adsorption discontinuous tests

The discontinuous tests were carried out using 0.100 g of adsorbent and 50 mL of Ni^{2+} solution (20 mg Ni^{2+} L^{-1}), prepared from $NiCl.6H_2O$ (Merck® 99%). Tests were performed under controlled conditions according to previously adjusted procedures (25 ± 2 ºC, pH = 6.0 ± 0.2, 200 rpm for 24 h) [39]. Samples were then filtered (MN710-125mm Macherey-Nagel®) [42].

Metal removal percentage (%R) and the retention capacity of the adsorbent (q) were calculated. Experimental equilibrium data were fitted by using adsorption Langmuir [43] and Freundlich [44] isotherm models. These models were compared with each other using IQAI's ADSOLAB program [45]. For fitting experimental data obtained in the kinetic tests, pseudo-first order and pseudo-second order models were employed. The mathematical expression for the pseudo-first order kinetics model [46] is widely used for adsorption studies of molecular liquids. The pseudo-second order model was developed by Ho and McKay [47], it assumes that the sorbate is bound to two active sites on the sorbent and is generally used in the case of ionic species.

Continuous tests

For the continuous tests, a column filled with 0.30 g of adsorbent and a total volume of 30.2 cm^3 was used. The contaminant solutions (60 mg Ni^{2+} L^{-1}) were circulated with a slow upward flow (0.5 mL min^{-1}) to longer the contact between the circulating solution and the adsorbent material. Samples were taken every 5 min. The reactor works as a plug flow according to a previous fluid dynamic test [48] with inert material [49].

The obtained breakthrough curves were fitted using Thomas [50], Adams-Bohart [51] and Yoon-Nelson [52] models. The Tomas model, based on the 2nd order kinetic model, considers that the adsorption reaction is controlled by the mass transfer at the interface. The Adams-Bohart model assumes that the rate of adsorption is proportional to the adsorption capacity and the remaining concentration of the adsorbate. The Yoon-Nelson model considers that the adsorption rate depends on the adsorbate concentration and the available capacity on the adsorbent.

RESULTS AND DISCUSSION

The release of impurities by 1.00 g of RM in contact with 50.0 mL of pure water was determined by TXRF. The results show that this material can be safely used as an adsorbent, as no hazardous leaching of potentially toxic elements was observed (Calcium 621 µg g^{-1},

Magnesium 179 μg g^{-1}, Potassium 306 μg g^{-1}, Iron 40 μg g^{-1}, Manganese and Zinc 14 μg g^{-1} and Nickel 2 μg g^{-1}).

Alkaline pretreatment of moringa: comparison with untreated material and evaluation of temperature effects

In order to improve fiber accessibility and facilitate subsequent functionalization reactions, an alkaline pretreatment at two different temperatures was carried out. This pretreatment induced clear morphological changes compared with the untreated material, including surface roughening, enhanced agglomeration, and a slight increase in fine, dust-like particulates.

Figure 1 shows the FT-IR spectra of the three samples: moringa before (RM) and after alkaline pretreatment at 25°C (PM) and under reflux. Although the pretreatment was intended mainly to improve physical accessibility of the lignocellulosic matrix, the spectra revealed specific chemical alterations. Notably, the band at 1735 cm^{-1} showed a pronounced decrease for the sample treated at 25°C, whereas it disappeared completely in the material treated under reflux. This behaviour is primarily attributed to the saponification of ester groups present in pectins and/or hemicelluloses, which converts esters into acid salts, resulting in a concomitant increase in the carboxylate band (1600 cm^{-1}). Additionally, the partial solubilization of these components (pectins and hemicelluloses) in the alkaline medium explains the mass loss observed during the process. Despite these changes, the remainder of the *FT*-IR profile showed no substantial modifications in the main functional-group regions, in agreement with previous reports [53].

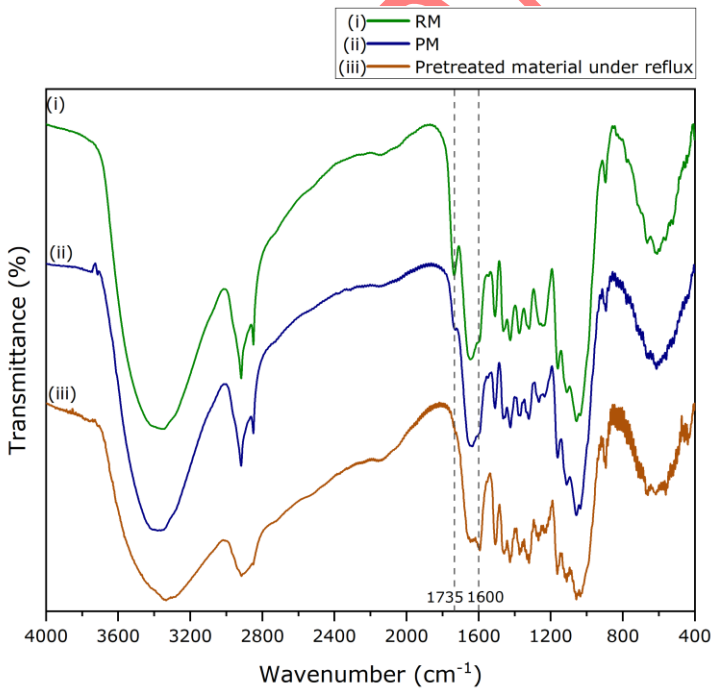

Figure 1. FT-IR spectra of the untreated moringa material and the products of the alkaline pretreatment.

Before proceeding with the chemical modification of the fibers, it was considered worthwhile to assess whether the alkaline pretreatment could influence the nickel adsorption capacity of the material. To this end, adsorption experiments were performed using the samples pretreated at both temperatures and the results were compared with those of the untreated material. The results of the adsorption tests carried out with the untreated sample (RM) and the pretreated samples (PM) are summarized in Table 1.

Table 1. Comparison of Ni^{2+} removal efficiency. Results are presented as mean ± standard deviation (n = 3). No statistically significant differences were observed among samples (one-way ANOVA followed by Tukey's HSD test, $p > 0.05$).

Sample	Ni^{2+} removal [%]
RM	65 ± 1
PM at 25 °C	68 ± 3
PM under reflux	66 ± 2

As shown in Table 1, no significant differences in Ni^{2+} removal were observed among the three samples of adsorbent analyzed ($p>0.05$). Since the pretreatment is expected to achieve fiber separation and considering that no significant differences were observed between the two pretreatments, the alkaline pretreatment at room temperature was selected for all subsequent studies. Furthermore, as increasing the temperature did not improve nickel adsorption, the room-temperature process is preferable because it reduces energy consumption and offers clear advantages in terms of operating cost and scalability. From now on, the room-temperature pretreated material will be referred to as PM.

Characterization of modified materials

This section shows the characterization of all the materials obtained at each stage of the surface modification of moringa.

Chemical characterization: *FT*-IR spectra of pretreated and chemically modified materials. Figure 2 shows the *FT*-IR spectra of the sample before (PM) and after acetylation (AM1 and AM2). The chemical modification is evidenced by the appearance of two new bands in the spectra of the modified materials: at 1748 cm^{-1} (C=O stretching of esters) and at 1238 cm^{-1} (Csp^2-O stretching), confirming the success of the esterification.

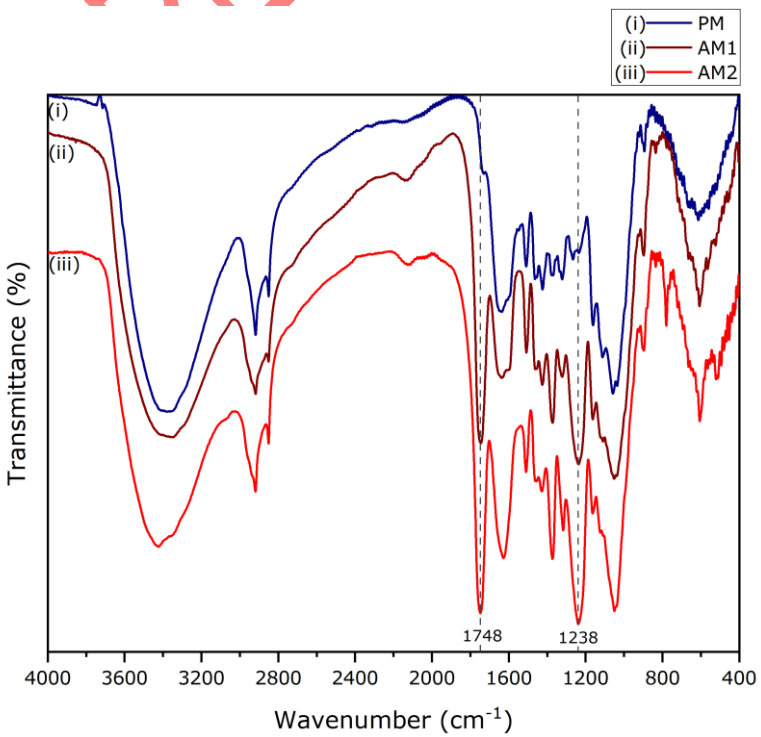

Figure 2. FT-IR spectra of the moringa sample before and after acetylation.

On the other hand, Figure 3 displays the *FT*-IR spectrum of the moringa sample before (PM) and after carboxymethylation (CM). The success of the modification is indicated by the appearance of an intense signal at 1600 cm^{-1} (carbonyl stretching of the carboxylate group) and a signal at 1420 cm^{-1} (asymmetric C-O stretching). Under acidic conditions, a shift in the carbonyl group signal is observed, from 1600 cm^{-1} (carboxylate group, $R\text{-}COO^-$) to 1730 cm^{-1} (carboxyl group, R-COOH). In the acidic spectrum, the bending signal of water appears at approximately 1630 cm^{-1}, while the carbonyl group signal is defined at 1733 cm^{-1}. Additionally, a new band is observed at 1225 cm^{-1} (Csp^2-O stretching).

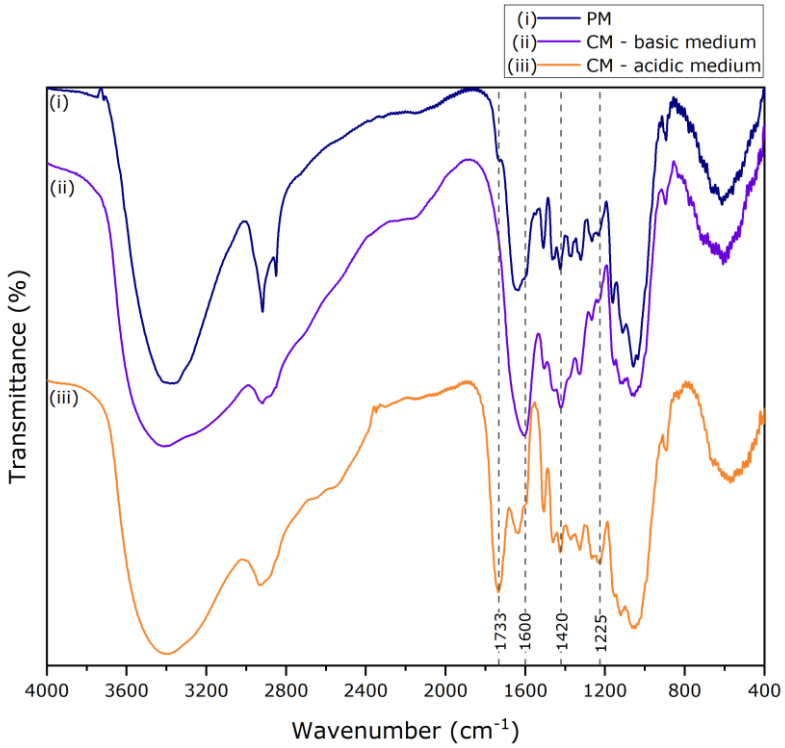

Figure 3. FT-IR spectra of the moringa sample before and after carboxymethylation.

Determination of degree of substitution. In the case of the acetylated samples, the degree of substitution was determined using the standardized methodology for cellulose, with the pre-treated sample serving as the reference. As shown in Table 2, the degree of substitution increases significantly when the volume of acetic anhydride is doubled, highlighting the importance not only of the amount of reagent but also of its role as a solvent, as it improves effective contact with the material.

Table 2. Degree of acetyl substitution obtained for different volume of acetic anhydride used.

	Degree of substitution [mmol acetyl groups g^{-1}]
AM1	1.4 ± 0.1
AM2	4.1± 0.3

Discontinuous tests

To evaluate the impact of the chemical modifications on the removal efficiency, a preliminary adsorption test was conducted using a single initial concentration (20 mg Ni^{2+} L^{-1}) performed in triplicate. Figure 4 compares the adsorption capacities of the untreated material (RM) [54], the alkaline-pretreated sample (PM), the carboxymethylated derivative (CM), and the acetylated samples (AM1 and AM2).

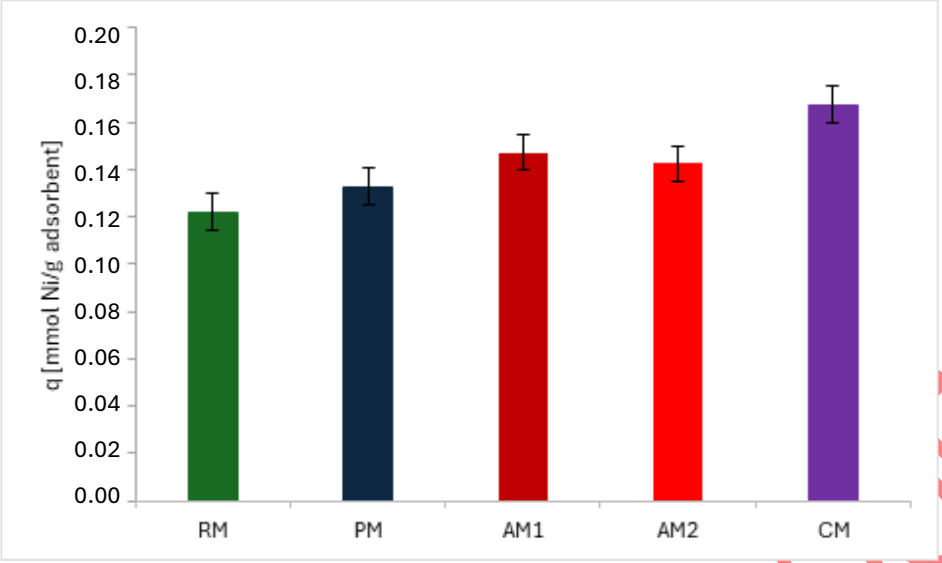

Figure 4. Ni^{2+} adsorption capacity (q) of each studied adsorbent.

As shown in Figure 4, the CM exhibited an increase in adsorption capacity of approximately 20% compared to PM. This enhancement was expected, as the incorporation of carboxylate groups introduces additional negative surface charges that favour electrostatic interactions with Ni^{2+} cations. However, as described in the experimental section, the overall yield of CM was low because a significant fraction of the carboxymethylated material became water-soluble and was separated and discarded with the liquid phase during the aqueous fractionation step. Therefore, the moderate improvement in adsorption performance does not justify the low synthesis yield or the increased process complexity. Consequently, the CM material was excluded from subsequent studies.

Regarding the acetylation process, no significant differences in adsorption capacity were observed between AM1 and AM2. Although AM1 and AM2 exhibit markedly different degrees of substitution, their adsorption capacities are comparable, leading to the conclusion that the role of acetylation in these materials is primarily structural, rather than directly functional, in the adsorption process. Specifically, acetylation was introduced to disrupt the intermolecular hydrogen bond network of the native polymer, thereby increasing chain mobility and improving the accessibility of active adsorption sites. In this context, the acetyl groups themselves do not participate significantly in the adsorption mechanism. Therefore, once a sufficient level of acetylation is achieved to effectively disrupt hydrogen bonds, further increases in the degree of substitution do not result in improved adsorption performance. The results suggest that the degree of substitution achieved in AM1 is already sufficient to disrupt the hydrogen bond network and maximize site accessibility; therefore, further substitution in AM2 does not lead to any additional improvement in adsorption performance. Furthermore, excessive acetylation can even be counterproductive, as it can reduce the number of available hydrophilic or active sites and introduce steric hindrance, which could limit interactions with the adsorbate.

Since AM1 requires a lower amount of reagents and milder conditions compared to AM2, it was selected as the more sustainable and scalable option. Based on these criteria—adsorption performance, synthesis yield, and reagent efficiency— the alkaline pretreated moringa (PM), and the acetylated sample (AM1) were selected for comparison of their adsorption efficiency with that of raw moringa.

Adsorption kinetics and equilibrium studies. Following the selection of PM, and AM1, a comprehensive evaluation of their adsorption performance was conducted in batch systems. This study aims to elucidate the adsorption mechanisms, determine the rate-controlling steps, and quantify the maximum adsorption capacity of the materials. First, equilibrium isotherm

studies were performed to evaluate the distribution of Ni^{2+} ions between the solid and liquid phases at different initial concentrations (5 - 40 mg Ni L^{-1}), followed by kinetic tests to analyse the effect of contact time on nickel uptake. The procedures explained by Boeykens et al. [42] were followed for equilibrium and kinetics assays.

Table 3. Langmuir and Freundlich parameters obtained. shows the results of the Langmuir and Freundlich models fits to the experimental data. It is observed that the Langmuir model provides a better fit, which is consistent with previous studies on nickel removal by bioadsorbents [55]. This model assumes that reversible adsorption occurs in a monolayer on a solid surface with homogeneus active sites and no interactions between the adsorbed molecules. Other raw bioadsorbents have an adsorption capacity between 0.038 y 0.27 mmol g^{-1} as sugarcane bagasse [53], *Moringa oleifera* leaves and seeds [55], *Thuja orientalis* [56], *Saccharum bengalese* containing cellulose [57], *coconut copra* meal [58], walnut shell, chestnut shell, pine wood, burnt pine wood [59], and others [60].The adsorption capacity of raw moringa and the chemical modified samples obtained in this work is comparable and also falls within this range, considering it has the advantage of being an industrial waste product, thus creating a circular economy cycle. Adsorption capacities 10 times greater have been reported in cases where energy and money were invested in the process to create activated carbon with this type of material [61]. A recent study with moringa leaves and seeds also showed a better fit to the Langmuir model, with q_{max} of 0.17 and 0.062 mmol/g, respectively [57]. These results suggest a single layer reversible adsorption mechanism and that the biosorbent used in this study has a nickel adsorption capacity comparable and in some cases superior to other materials previously investigated.

Table 3. Langmuir and Freundlich parameters obtained.

Adsorbent	Langmuir			Freundlich		
	K_L	q_{max} [mmol g^{-1}]	R^2	K_F	N	R^2
RM	9 ± 4	0.20 ± 0.03	0.961	0.25 ± 0.04	2.2 ± 0.5	0.936
PM	18 ± 5	0.21 ± 0.02	0.980	0.27 ± 0.06	2.8 ± 0.6	0.944
AM1	13 ± 7	0.20 ± 0.04	0.912	0.23 ± 0.05	3 ± 1	0.84

Table 4 shows the results of the fitting of the experimental kinetic curves using the pseudo-first and pseudo-second order kinetic models. The pseudo-second order model had the best fit, coinciding with previous studies on biosorption of divalent metal ions on moringa and other sorbents [62]. This suggests that chemisorption could be the rate-limiting mechanism with the intervention of two sites of adsorption.

Table 4. Obtained parameters from the different models tested.

Adsorbent	Lagergren			Ho y McKay		
	k1 [min^{-1}]	Qe [mmol/g]	R^2	k2 [g mmol^{-1} min^{-1}]	qe [mmol/g]	R^2
RM	0.18 ± 0.03	0.104 ± 0.003	0.961	2.7±0.5	0.112 ± 0.003	0.986
PM	0.26 ± 0.03	0.121 ± 0.002	0.987	4 ± 1	0.125 ± 0.003	0.988
AM1	0.8 ± 0.3	0.115 ± 0.001	0.995	32 ± 14	0.117 ± 0.001	0.997

Continuous tests

Figure 5 shows the results obtained for the breakthrough curves using the RM, PM and AM1 adsorbents as column fillers. It can be observed that the three curves had similar characteristics. From these curves, it can be seen that the breakthrough points (T10) for 10% of the adsorbent saturation are approximately 60 minutes and the T90 is reached at 140 minutes [49]. These results are an incentive to conduct further studies in continuous systems using these materials as fillers. Some researchers studied other biosorbents with similar breakthrough points [63], although sometimes they required more material in the bed [64]. This suggests that the studied materials offer competitive performance in adsorption efficiency and use.

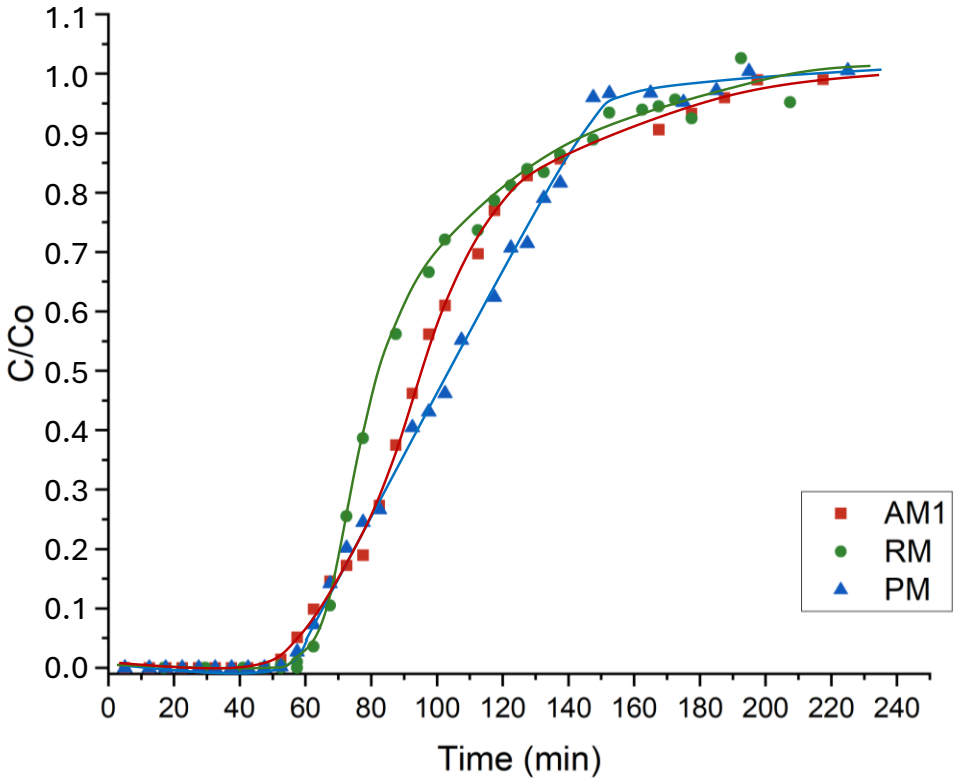

Figure 5. Experimental breakthrough curves obtained with RM, PM and AM.

Table 5 shows the results of the fit using the Thomas, Yoon-Nelson and Adams-Bohart models. The three models provide essential parameters for the scale up of the process. The Thomas and Yoon-Nelson models fit better to the experimental data in all cases, which was predictable given the similarity in their premises. The Adams-Bohart model, which is used to describe the first part of the breakthrough curve, showed a worse fit. These results are consistent with studies on other lignocellulosic materials, such as *banana* peel with dolomite for chromate and phosphate removal [65]. With the obtained parameters, it is possible to evaluate the efficiency of the column, to begin the optimization and scaling of column design at an industrial level, allowing estimation of operating time and the volume of effluent treated.

Table 5. Thomas, Adams Bohart and Yoon Nelson models parameters obtained.

Ads.	Thomas			Adams Bohart			Yoon Nelson		
	K_{TH} [mL min^{-1}g^{-1}]	q_0 [mmol g^{-1}]	R^2	K_{AB} [cm^3 mmol^{-1} min^{-1}]	N_0 [mmol cm^{-3}]	R^2	K_{YN} [min^{-1}]	τ [min]	R^2

RM	67 ± 6	0.147 ± 0.003	0.97	53±5	0.002 ± 2.4E-5	0.908	0.064 ± 0.006	92±2	0.97
PM	54 ± 2	0.166 ± 0.001	0.993	31± 2	0.002 ± 2. E-5	0.932	0.053 ± 0.003	104±1	0.992
AM1	64 ± 3	0.154 ± 0.001	0.992	39± 3	0.002 ± 2. E-5	0.937	0.061 ± 0.003	97±1	0.991

CONCLUSIONS

The results obtained in this study are promising for the treatment of nickel-contaminated water using *Moringa oleifera* residues as adsorbents. Laboratory experiments showed modest but consistent improvements in Ni^{2+} removal after chemical surface modification. However, these differences were less pronounced under continuous-flow conditions, suggesting that the use of raw moringa remains economical and environmentally advantageous. Even so, continued optimization of the modification procedures may further enhance adsorption efficiency and selectivity.

Overall, *Moringa oleifera* represents a highly promising low-cost biomass, both as a precursor for the development of tailored adsorbent materials and particullary as an effective Nickel sorbent in its unmodified form, without requiring additional reagents or processing steps.

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NOMENCLATURE

AM1: acetylated moringa using 5.00 mL of acetic acid

AM2: acetylated moringa using 10.00 mL of acetic acid

CM: carboxymethylated moringa

PM: alkaline pretreated moringa

RM: raw moringa

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