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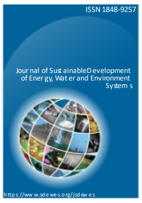

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Original Research Article

Sustainable Adsorbents from *Moringa Oleifera* Residues: Chemical Modifications and Nickel Removal Efficiency

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ABSTRACT

This work evaluated the potential reuse of solid effluents from a *Moringa oleifera* infusion production company as adsorbents for the removal of nickel from aqueous media. To enhance adsorption performance, the material was chemically modified through an alkaline pretreatment, followed by carboxymethylation or acetylation. The obtained products were characterised 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 the removal of nickel ions. Overall, the results highlight moringa-based materials as low-cost, sustainable adsorbents for wastewater treatment.

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, as costs are the sacrifices 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 lies in this double reduction to improve the feasibility of applying treatments [1]. Regarding environmental costs, although every production process generates unwanted byproducts, efforts should be made to minimise their release to the environment and ensure they are not harmful to its inhabitants. One of the precepts of the Circular Economy is the reuse of waste and its incorporation into a new production line, to reduce both the introduction of new raw

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materials in the production system and the generation of waste [2]. Accordingly, the Amsterdam Pact focuses on waste management (turning waste into a resource), the shared economy, and resource efficiency [3].

In this vein, "El Moringuero", a moringa production company that grows its crops sustainably, is looking to address the volume of waste generated. On the other hand, the concentration of metals in water bodies has increased due to rapid industrialisation, increased industrial waste, increased consumption of agricultural inputs and mining [4]. This work aims to reduce this pollution and contribute to achieving 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 because a wide range of valuable products can be obtained from its various anatomical parts. The leaves are among the most exploited fractions due to 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 and traditional medicine [6]. However, despite this versatility, a significant fraction of the biomass generated during cultivation and processing is not utilised. 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 valorisation [8]. This underutilisation 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 is a complex challenge, and the methodology to be used must be easy to implement, effective, and low-cost 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 containing 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. Commonly used adsorbents include activated carbons, clays, and biopolymers, among others. However, these materials are expensive. In recent years, driven by the imperative need to remove metals from contaminated waters in an efficient and economically viable manner, research has been encouraged to develop affordable alternative adsorbents. In this regard, the exploration of various agroindustry resources is being promoted 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 demand for agricultural products offers an excellent opportunity to foster equitable development across the country. It is achieved by strengthening primary production and, especially, by promoting added value in agroindustry [11]. One way to add value to a crop is to introduce waste material into a new production line to make use of it. This approach reduces waste, diminishes environmental impact and lowers the economic cost for the companies responsible for its disposal.

Moringa oleífera has become one of the crops that are booming today, due to the discovery of its high nutritional value and the wide range of properties it possesses, which has enabled its use in the cosmetic, pharmaceutical and food industries [12], [13]. Residues from moringa processing were used in this work as lignocellulosic material for nickel bioadsorption.

To enhance ion adsorption efficiency, chemical modifications can be made to the material surface. These modifications could be considered a pretreatment of the lignocellulosic material and should be carried out 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 fibre, and partial elimination of substances that act as fibre cement (lignin, hemicelluloses and pectin) were observed [14]. On the other hand, the introduction of new functional groups, such as carboxyl groups via carboxymethylation [15], increases surface polarity and generates negatively charged sites at pH values above 6, thereby favouring 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 their high nutritional value, the leaves are no longer considered crop residues [20].

This approach points to a possible application of these low-cost treatments in industries that produce metal-containing effluents. Metal ions are non-biodegradable substances that can bioaccumulate and even undergo biomagnification in the environment, so that inadequate disposal of industrial effluents could put at risk the health of living beings, the ecosystem, and by extension, 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 containing it are considered harmful to human health and are also bioaccumulative, posing a danger to 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 for the metal content of effluent to be discharged or used, with a concentration range in the receiving water body of 0.1−1.0 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 to combine the removal of nickel from industrial effluents with the reuse of moringa waste would integrate the process into the Circular Economy. This perspective promotes sourcing raw materials from secondary sources, which helps close the products' life cycle and reduce dependence on primary natural resources. The adoption and implementation 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, a major concern given the continuous population growth. 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 characterised 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 authors investigated the possibility of reusing solid effluents from a moringa infusion production company to remove nickel from other industrial effluents. To maximise the adsorption capacity, the surfaces of these adsorbents were chemically modified. First, an alkaline pretreatment was applied to open the lignocellulosic structure and increase fibre 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, their two routes selected because they involve simple, well-established chemical modifications as functional groups derived from esterified cellulose [29], carboxymethyl cellulose derivatives [30] or functional nanomaterials derived from diverse esterified cellulose compounds [29] that are widely applied in different industries, such as food, wastewater treatment, medicine, papermaking, etc. [31], making them suitable for potential large-scale implementation. On the one hand, a carboxymethylation process was performed to introduce negatively charged groups capable of binding positively charged ions, such as nickel (Ni^{2+}). The working hypothesis was that this modification would enhance cation removal 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 via chelation.

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 characterised 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* waste was kindly provided by the “El Moringuero SA” company, located in Misiones, Argentina. The supplier previously processed the residues by coarse grinding and packed them in 3 kg bags. A representative sample was obtained from this material using statistical data for bulk material [32], and a finely ground material was collected from the sieve (ASTM No. 16-35) with a size range of 500–850 μm, to be used as the adsorbent material in all the tests carried out. A previous washing stage in deionised water at 50 °C, followed by 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 the pH reached neutrality, 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 characterised 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) was 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 1 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 minimise 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 Released and Chemical Characterisation of Materials

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 deionised water were quantified by Total Reflection X-Ray Fluorescence (TXRF) analysis using a BRUKER® S2 PICOFOX spectrometer [39].

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 the Degree of Substitution and Quantification of the Adsorbate

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 was 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.

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 to determine 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 $NiCl \cdot 6H_2O$ (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 (MN710-125 mm Macherey-Nagel®) [42].

The test results enabled the determination of metal removal percentage (%R) and the adsorbent’s retention capacity (q). Experimental equilibrium data were fitted by using the Langmuir [43] and the Freundlich [44] isotherm models. These models were compared 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 developed by Ho and McKay [47] 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 at a slow upward flow rate (0.5 mL min^{-1}) to increase contact time between the circulating solution and the adsorbent material. Samples were taken every 5 min. The reactor operates in plug flow, as shown in a previous fluid-mic test [48] using inert material [49].

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

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

To improve fibre accessibility and facilitate subsequent functionalisation 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].

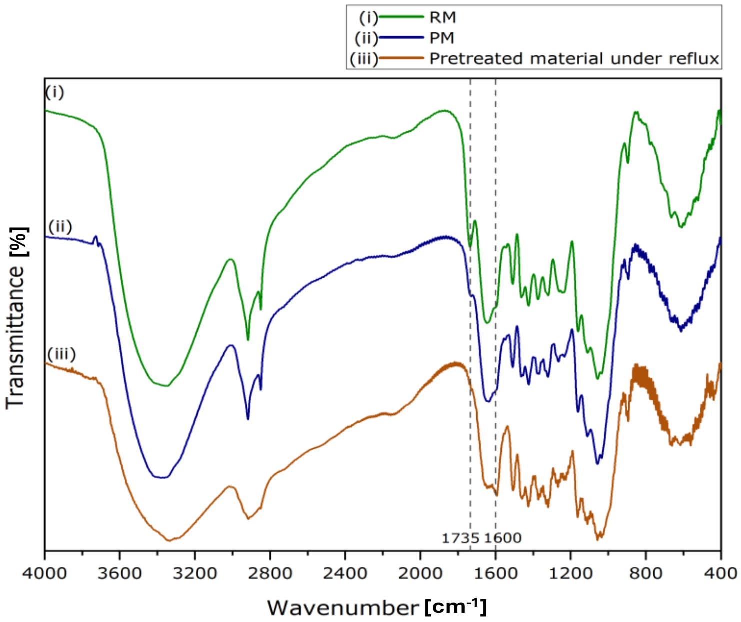

Figure 1. FT-IR spectra of the untreated moringa material and the products of the alkaline pretreatment

Before proceeding with the chemical modification of the fibres, 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 summarised in Table 1.

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

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 analysed ($p>0.05$). Since the pretreatment is expected to achieve fibre separation and 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 material pretreated at room temperature will be referred to as PM.

Characterisation of Modified Materials

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

Chemical characterisation of pretreated and chemically modified materials. **Figure 2** shows the *FT*-IR spectra of the samples 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.

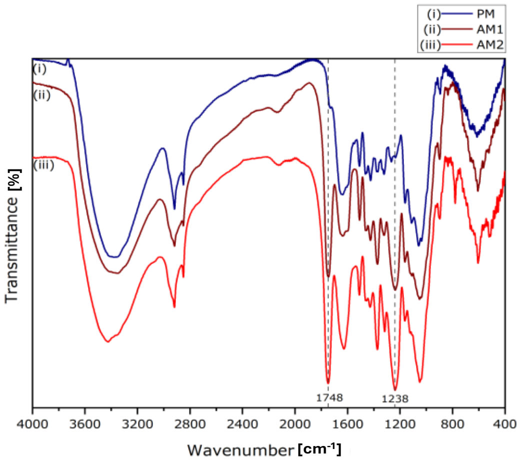

Figure 2. FT-IR spectra of the moringa samples before and after acetylation

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-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).

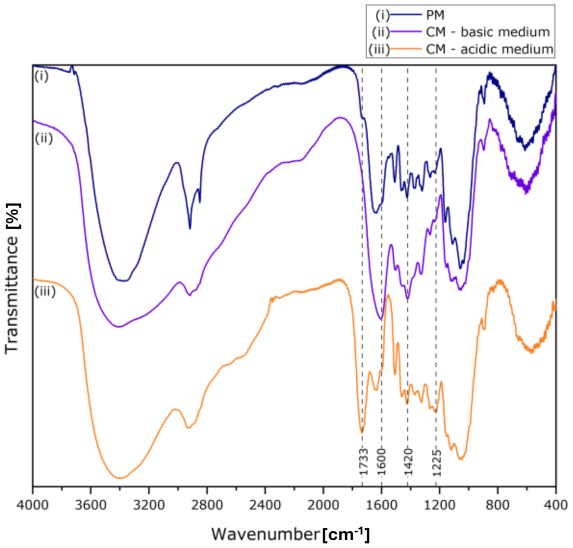

Figure 3. FT-IR spectra of the moringa samples before and after carboxymethylation

Determination of the degree of substitution. For the acetylated samples, the degree of substitution was determined using the standardised methodology for cellulose, with the pretreated 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 volumes 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 performed in triplicate using an initial concentration of 20 mg Ni^{2+} L^{-1}. **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).

Material selection. As shown in **Figure 4**, the CM exhibited an approximately 20% increase in adsorption capacity 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. The determined 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 and PM was selected for investigation.

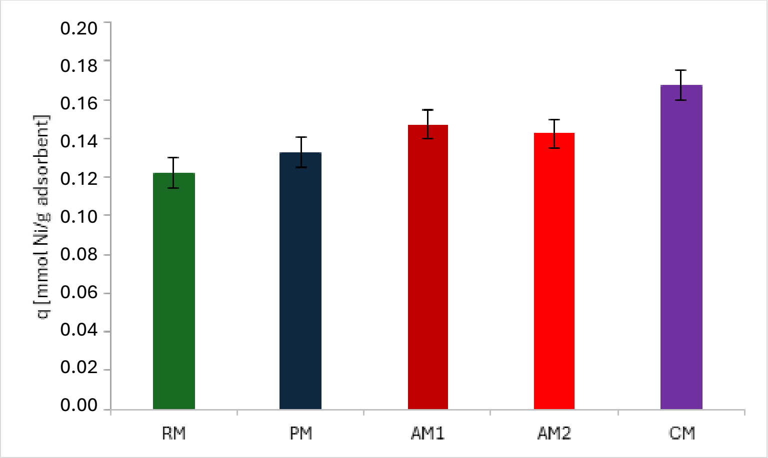

Figure 4. Ni2+ adsorption capacity of each studied adsorbent

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, suggesting that acetylation primarily plays a structural role rather than a direct functional role in adsorption. 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 disrupt hydrogen bonds effectively, 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 maximise 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, thereby limiting interactions with the adsorbate. Since AM1 requires fewer reagents and milder conditions than AM2, it emerged as the more sustainable and scalable option.

Summing up material considerations, based on the adopted criteria (adsorption performance, synthesis yield, and reagent efficiency), 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, their adsorption performance was comprehensively evaluated in batch systems. This study aimed to elucidate the adsorption mechanisms, determine the rate-controlling steps, and quantify the maximum adsorption capacity of the materials. First, equilibrium isotherms were studied 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. For equilibrium and kinetics assays, the procedures explained by Boeykens *et al.* [42] were employed.

Table 3 shows the results of the Langmuir and the Freundlich models fits to the experimental data. The Langmuir model provides a better fit, consistent with previous studies on nickel removal by bioadsorbents [55]. This model assumes reversible adsorption in a monolayer on a solid surface, with homogeneous active sites and no interactions between adsorbed molecules. Other raw bioadsorbents have an adsorption capacity between 0.038 and 0.27 mmol g^{-1}, such 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 chemically modified samples obtained in this work is comparable and falls within this range, with the added advantage of being an industrial waste product, thus creating a circular economy. 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} values of 0.17 and 0.062 mmol g^{-1}, 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 to, and in some cases superior to, that of 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 provided the best fit, consistent with previous studies on the biosorption of divalent metal ions by moringa and other sorbents [62]. This observation suggests that chemisorption could be the rate-limiting step, with the involvement of two adsorption sites.

Table 4. Obtained parameters from the tested models

Adsorbent	Lagergren			Ho and McKay		
	k_1 [min^{-1}]	Q_e [mmol g^{-1}]	R^2	k_2 [g mmol^{-1} min^{-1}]	q_e [mmol g^{-1}]	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 breakthrough curves obtained using the RM, PM, and AM1 adsorbents as column fillers. The three curves show similar characteristics: the breakthrough point (T10) for 10% adsorbent saturation is 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], though they sometimes required more material in the bed [64]. This observation suggests that the studied materials offer competitive performance in adsorption efficiency and use.

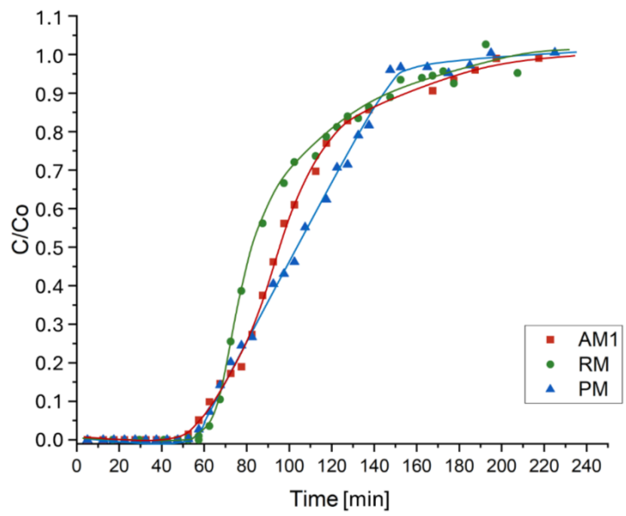

Figure 5. Experimental breakthrough curves obtained with RM, PM, and AM

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 process's scale-up. The Thomas and Yoon-Nelson models fit the experimental data better in all cases ($R^2 > 0.97$), as expected given their shared premises. The Adams-Bohart model, used to describe the initial portion of the breakthrough curve, provided a poorer fit ($R^2 = 0.937$). 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 column's efficiency and to begin optimising and scaling the column design at an industrial level, allowing the estimation of operating time and the volume of effluent treated.

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

Adsor-bent	Thomas		Adams-Bohart		Yoon-Nelson	
	K_{TH} [mL min^{-1} g^{-1}]	q_0 [mmol g^{-1}]	K_{AB} [cm^3 mmol^{-1} min^{-1}]	N_0 [mmol cm^{-3}]	K_{YN} [min^{-1}]	τ [min]
RM	67 ± 6	0.147 ± 0.003	53±5	0.002 ± 2.4 E−5	0.064 ± 0.006	92±2
PM	54 ± 2	0.166 ± 0.001	31± 2	0.002 ± 2.0 E−5	0.053± 0.003	104±1
AM1	64 ± 3	0.154 ± 0.001	39± 3	0.002 ± 2.0 E−5	0.061 ± 0.003	97±1

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 Ni2+ removal after chemical surface modification. However, these differences were less pronounced under continuous-flow conditions, suggesting that using raw moringa remains economical and environmentally advantageous. Even so, continued optimisation 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 particularly as an effective nickel sorbent in its unmodified form, without requiring additional reagents or processing steps.

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NOMENCLATURE

Abbreviations

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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