Advances in Chemical Engineering and Science, 2025, 15(1), 31-46
https://www.scirp.org/journal/aces
ISSN Online: 2160-0406
ISSN Print: 2160-0392

Determination of Heavy Metal Adsorption Capacity of Water Contaminated by Metallurgical Tailings Leaching Using Carbohydrates Derived from *Solanum tuberosum*

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How to cite this paper: Aparicio-Aragón, W.B., Ttito-León, S. and Valencia-Pacho, M.Y. (2025) Determination of Heavy Metal Adsorption Capacity of Water Contaminated by Metallurgical Tailings Leaching Using Carbohydrates Derived from *Solanum tuberosum*. *Advances in Chemical Engineering and Science*, **15**, 31-46.
https://doi.org/10.4236/aces.2025.151003

Received: December 19, 2024
Accepted: January 21, 2025
Published: January 24, 2025

Abstract

Water contamination in areas of formal and informal polymetallic mining is a growing concern in the Peruvian highlands. At the same time, there are agricultural activities that use contaminated water from these rivers for irrigation. This could contaminate the agricultural products grown on these lands, such as potatoes. It is, therefore, necessary and a priority to determine the adsorption characteristics of these derivatives and to be able to use these natural adsorbents to decontaminate water contaminated with heavy metals.

Keywords

Heavy Metals, Adsorption, Potato Derivatives, Isotherm, Polluted Water

1. Introduction

The increase of heavy metal pollution in air, soil and water is a potential threat to ecosystem and public health worldwide. Contamination by metals such as Cu^{2+}, Zn^{2+}, Pb^{2+} and Cd^{2+}, due to their high toxicity, non-degradability [1] and bioaccumulation in aquatic species [2] and other living organisms [3], directly affect the food chain [4], according to the WHO, the presence and permanent contact with these metals causes neurological and renal damage [5], they also damage cells, generating free radicals [6] and reactive oxygen species which cause oxidative stress associated with cancer [7], as a teratogenic agent they cause malformations in living organisms [8]. These toxic pollutants come from anthropogenic and industrial activities such as mining and smelting [1] as well as agriculture, where excessive use of fertilisers and pesticides released from effluents pollute the environment [9].

These environmental problems are mainly manifested in the contamination of Lake Titicaca, a natural wonder of the altiplano [10], which has a rich biodiversity of endemic species in danger of extinction in the region [11], with a decrease in phytoplankton due to the accumulation of heavy metals in the sediments [12], which mainly come from mining activities [13]. The lake's water is an essential resource for the subsistence of the population due to polymetallic mining and metallurgical activities. This affects water quality due to the presence of lead in concentrations exceeding permissible limits [14]. This impact, associated with contaminated wastewater discharges [15], facilitates the accumulation of metals in sediments rich in organic matter, as natural reservoirs [11]. While Maldonado [12] reports that the analysis of lake water samples for metals such as lead, copper and cadmium revealed concentrations that exceed the limits allowed by legal regulations, making the water a risk because they are not suitable for uses such as irrigation and animal consumption, as reported in related studies of heavy metal contamination in water bodies [16].

Different heavy metal removal techniques such as chemical precipitation, membrane filtration, ion exchange, photocatalysis, electrochemical methods, and adsorption are used [7] [8]. Saravanan *et al.* report that non-conventional techniques such as bioremediation and microbial reduction are used to remove metals from water bodies. The use of the appropriate technique is determined by the specific characteristics of the contaminated site and its efficiency, which are currently a challenge for the removal of these pollutants due to the high costs involved and low efficiency at low concentrations [17]. Therefore, a physicochemical treatment such as adsorption has become the most attractive water decontamination method, due to its advantages such as ease of operation, low cost, short treatment time, high efficiency and environmental friendliness [17] [18].

Various adsorbent materials of organic origin were studied for the removal of heavy metals from polluted waters, such as wood ash modified biocarbon [19], tamarind peel, banana peel (*Musa paradisiaca*), lemon peel (*Citrus limonium*), orange peel (*Citrus sinensis*) [20], corn husk [21], peanut shell [24], chitosan and sodium alginate for Cd removal [22], potato and sweet potato starches modified in their structure by enzymatic combination [23], potato starch phosphate has been synthesised to remove metals such as Zn(II), Pb(II), Cd(II) [24], orange peel modified with sodium hydroxide and calcium chloride in the adsorption of Cu(II), Pb(II) and Zn(II) [17], sweet potato peels modified with sodium hydroxide to remove Cd (II) [25], Choque *et al.* [26], reports a potato starch copolymer with cactus mucilage with a Pb(II) removal capacity of 102. 20 mg/g. The combined biomass of banana peel and potato peel was used as a biosorbent for Pb(II), reporting an efficiency of 90.742 mg/g [27]. Seven toxic ions were adsorbed with banana, orange and potato peels fixed on sodium alginate beads with the highest removal for Cd(II), followed by Cu [28], Lamiaa, [29] used potato peels in the removal of Cu(II) from mixed metals in solution, reporting a sorption capacity of 39 mg/g, also using raw and burnt potato peels was reported by El-Azazy [30]. The adsorption of Cd, Cu, Pb with an adsorption capacity of 239.64 mg/g with burnt potato peels for Cd. Potato peels were used for Cu removal [31], as well as Cd adsorption with waste potato peels without reporting 7.61 mg of Cd per gram of adsorbent [32].

The potato (*Solanum tuberosum* L.) which contains approximately 80% water, and 20% dry mass, of which 70% is starch, cereal-like proteins [33], carbohydrates, aspartic and glutamic acids and their amides, phenolic compounds and flavonoids [34] as well as their derivatives, chuño, known as freeze-dried and dried black potato contains polyphenols such as epicatechin and chlorogenic acid [35], white chuño also known as tunta or moraya obtained by a freeze-drying process with four weeks of soaking in water [36] and dried potatoes that are blanched and then dried, is an option for use as a natural adsorbent to mitigate water contaminated by toxic metals, because these derivatives have chemical characteristics that have an affinity for the adsorption of metals such as Cd, Pb, Zn and Cu, due to the presence of functional groups such as hydroxyl and carboxyl.

Escobar [37] reported in a study around the bay of Lake Titicaca, found arsenic content of 41 $mg \cdot kg^{-1}$, which far exceeds the maximum permissible limits, Soloisolo, D. [38] in a study of heavy metal determination in the Llallimayo river, (Umachiri-Melgar-Puno). The concentrations of lead and mercury in the river water of the Llallimayo basin are 1.486, 0.2640, 0.2600, $mg \cdot dm^{-3}$ respectively. These values exceed the WHO and MINSA standards.

The objective of this work is to report the adsorption capacity of these potato-derived bioadsorbents to remove dissolved contaminants in water, such as Cu, Pb, Zn and Pb metals, which contaminate rivers and lakes in the Peruvian Altiplano due to formal and informal mining activities.

2. Experimental Part

2.1. Chemicals

Ethanol, CH_3CH_2OH (HPLC grade, Fisher), Water, distilled and deionised, Acetone, CH_3COCH_3 (analytical grade, Aldrich), Metal salts of lead, copper, zinc and cadmium as nitrate (analytical grade, Aldrich), Atomic absorption standards of metals lead, cadmium, copper and zinc nitrates (analytical grade, Aldrich), Calcium chloride, $CaCl_2$ (98%, Fisher), Eryo chromium black T (indicator), Ammonium solution (analytical grade, Aldrich), Buffer solution pH 10, Universal indicator paper.

2.2. Adsorbent Material

The experimental tests of adsorption of metallic cations, Pb^{2+}, Cd^{2+}, Zn^{2+} and Cu^{2+}, have been carried out using the tuber called potato, which is an agricultural product that is cultivated in the highlands of Perú. The variety used is the potato compis (*Solanum tuberosum*), from which derivatives such as:

The chuño is processed by exposing the potato to alternating cycles of freezing and sunshine. In each cycle, the tuber loses water due to the effect of freezing and the heat of the sun, and the harvested potato is placed on flat ground covered with grass or Andean straw. It is left to freeze due to the frost for about 3 nights. It is dried in the sun for several days. The whole process takes an average of 20 days; at the end, the product extracted from the potato (chuño) is a dried tuber. Certain substances present in the potato during processing in contact with the air oxidise, giving it a brown to black colour.

The moraya is obtained by processing the harvested potato, subjecting it to freezing following the same technique of obtaining chuño, but avoiding that the frozen potato is in contact with the sun during the 3 days of freezing. The frozen potato is dehydrated by pressing the feet against the floor to squeeze, then put in a stream of water for 15 days to remove substances that can oxidize with air, thus obtaining a white product after several days of drying.

The dried potatoes are obtained from a tuberous plant (potatoes), which are homogeneously sized and boiled for 20 minutes at the temperature of boiling water. The skin is removed manually from the tuberous plant, cut up, and subjected to drying in the shade for 8 days. At the end of this, a slightly yellowish product is produced, which is completely dried. These derivatives are reported and discussed in the bromatological analysis.

2.3. Treatment of Potato Derivatives (Chuño, Moraya and Dried Potatoes) for the Purpose of Adsorption

The treatment applied to potato derivatives such as chuño, moraya and dried potatoes, ground and sieved at 100 mesh, is 1) determination of the bromatological properties of each of these derivatives, 2) determination by infrared spectroscopy of the bands of the vibrational spectra and 3) adsorption tests of each of the potato derivatives.

2.4. Determination of Bromatological Properties

The bromatological analysis of the potato derivatives (chuño, moraya and dried potato), such as moisture, ash content, fat and protein, were determined using the AOAC Technique [39], whose calculation formulas are respectively:

$$\%\text{Humidity} = \frac{\text{Total weight} - \text{Final weight}}{\text{Sample weight}} * 100 \tag{1}$$

$$\%\text{Ash} = \frac{\text{Ash weight}}{\text{Sample weight}} * 100 \tag{2}$$

$$\%\text{Fibre} = \frac{\text{Weight of sample flask} - \text{Weight emply flask}}{\text{Sample weight}} * 100 \tag{3}$$

$$\%\text{Protein} = \%\text{Nitrogen} * 6.25 \tag{4}$$

$$\%\text{Nitrogen} = \frac{V(\text{ml}) \text{ of HCl} * \text{Normality} * \text{Meq of N}_2}{\text{Sample weight}} * 100 \tag{5}$$

2.5. Determination of Infra-Red Spectra

Firstly, using Fourier Transform Infrared-Attenuated Total Reflectance (FTIR-ATR) equipment. PERKIN ELMER, FRONTIER. The spectra of the adsorbents were determined before and after contact with the respective metal cation solution, using approximately 200 mg of the respective dry powder sample.

2.6. Adsorption Tests for Potato Derivatives (Chuño, Moraya and Dried Potato)

Prepared solutions are required for exchange tests, such as:

2.7. EDTA Solution

0.1 mol·dm^{-3} standard EDTA solution was prepared from 37.22 grams of ethylene diamino disodium salt in one litre of solution. Standardised with dried calcium chloride, using Eriochrome Black T as an indicator, this EDTA standard solution was used to standardise the concentration of the heavy metal standard solutions.

2.8. Buffer Solution pH 10

This solution was prepared following the technique proposed by H. A. Flaschka [40], "*EDTA Titration*". 70 grams of ammonium chloride (NH_4Cl) is dissolved in 570 mL of ammonium solution (NH_4OH), then diluted with distilled water to a final volume of 1.0 litre, which is stored in a plastic bottle.

2.9. Standard Solution of Heavy Metals

In order to determine the adsorption isotherms of heavy metal cations as: lead, cadmium, copper and zinc, all as nitrate, standard solutions of these metals were prepared in deionised water with a concentration ~2×10^{-3} mol·dm^{-3} on average, which in each case to determine the exact concentration were standardised with EDTA 0.0148 mol·dm^{-3}, these solutions were used in the following section, for the adsorption tests and determination of adsorption equilibrium curves.

2.10. Procedure

The adsorption of metal cations, Pb^{2+}, Cd^{2+}, Cu^{2+}, Zn^{2+}, nitrate as anion, was carried out by preparing solutions of concentration ~2×10^{-3} mol·dm^{-3} approximately, which were standardised with EDTA using Eriochrome black-T as an indicator to determine the exact concentration of each metal ion, using 10 test tubes of 30 mL capacity for each adsorbent, approximately 01 gram of adsorbent (chuño, moraya and dry potato) was deposited respectively in each test tube, 10 mL of the respective metal cation solution was added into the test tube, with a concentration range, (~2×10^{-4} to ~2×10^{-3} mol·dm$^{-3)}$, shaken vigorously and left overnight in a thermostatic bath at 25˚C, aliquots of the liquid part were removed from each experimental tube and analysed by Atomic Absorption Spectroscopy, using the AnalytikJenna noVAA 800, in order to determine the final concentration of the respective metal cation remaining in solution.

Before determining the concentration of metal cation remaining in solution, the atomic absorption instrument was calibrated with the appropriate standard and at the wavelength recommended by the instrument characteristics.

3. Results and Discussion

3.1. Determination of Bromatological Properties

Using the technique described in the experimental part, the bromatological characteristics of derivatives of the "compis" potato (*Solanum tuberosum*), such as chuño, moraya and dried potato, were determined and are shown in Table 1. The analysis shows that these three potato derivatives have, on average, similar characteristics in terms of moisture, ash and fibre content, with a slight variation in terms of protein content, where the dry potato derivative retains the protein content of the original potato product (13.00%), while the moraya has the same protein content (13.00%). All these results indicate that potato derivatives generally increase the starch concentration, since this component is a water-insoluble carbohydrate, which favours the adsorption of heavy metals.

Table 1. Humidity, ash, protein and fibre content in the 3 potato derivatives of the variety "compis" (*Solanum tuberosum*).

SAMPLE	% HUMIDITY	% ASH	% PROTEIN	% FIBRE
Moraya	9.370	5.940	3.890	27.790
Chuno	8.480	2.330	7.280	28.790
Dried potato	7.100	4.360	11.640	28.300

3.2. Infrared Spectroscopic Studies of Adsorbents

Potato derivatives, used as bioadsorbents, have starch molecules in their chemical structure; as other impurities have been removed during processing, the infrared spectra only show the vibration of atoms that are part of the starch molecule, so it is concluded that the functional groups that immobilise the metal cations studied in this article are the ethereal oxygens and hydroxyl groups that are part of the structure of the starch molecule.

Figures 1(a)-(c) show the analysis by infrared spectroscopy studies of the potato-derived adsorbents compis (dried potato, chuño and moraya) before contacting the respective metal cation, the figures show the % Transmittance versus vibrational frequency in cm^{-1} of each derivative.

Figures 1(a)-(c) show that the 3 potato derivatives present the same signal with the O-H stretching at 3000 cm^{-1} and C=O at 2900 cm^{-1} of vibration frequencies. These derivatives give the same signals at different frequencies of vibration, concluding that they correspond to a starch molecule. These adsorbents were analysed by the same technique after contact with different solutions of metal cations such as lead, cadmium, zinc and copper, whose representative results are shown for lead and copper cation in the following figures.

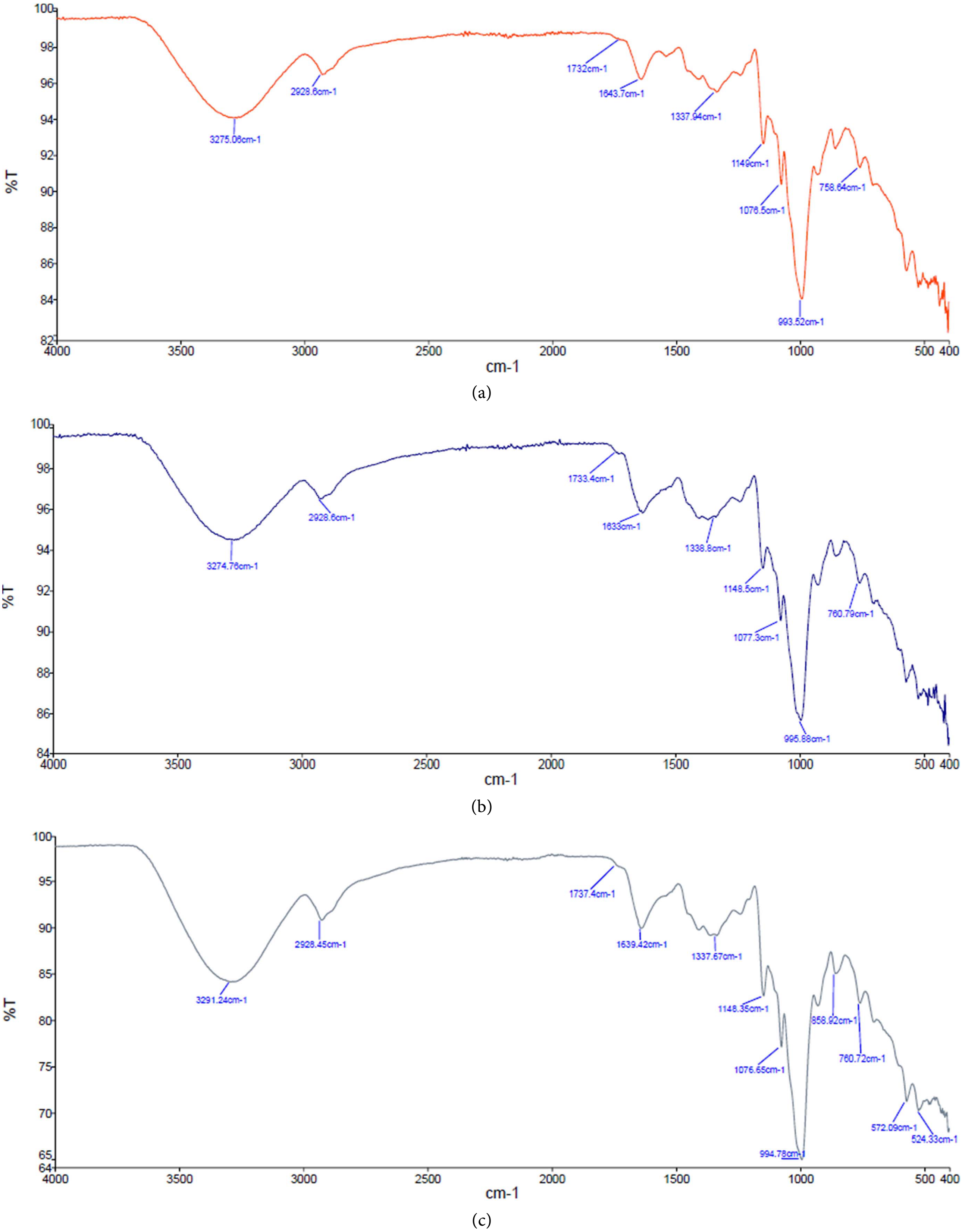
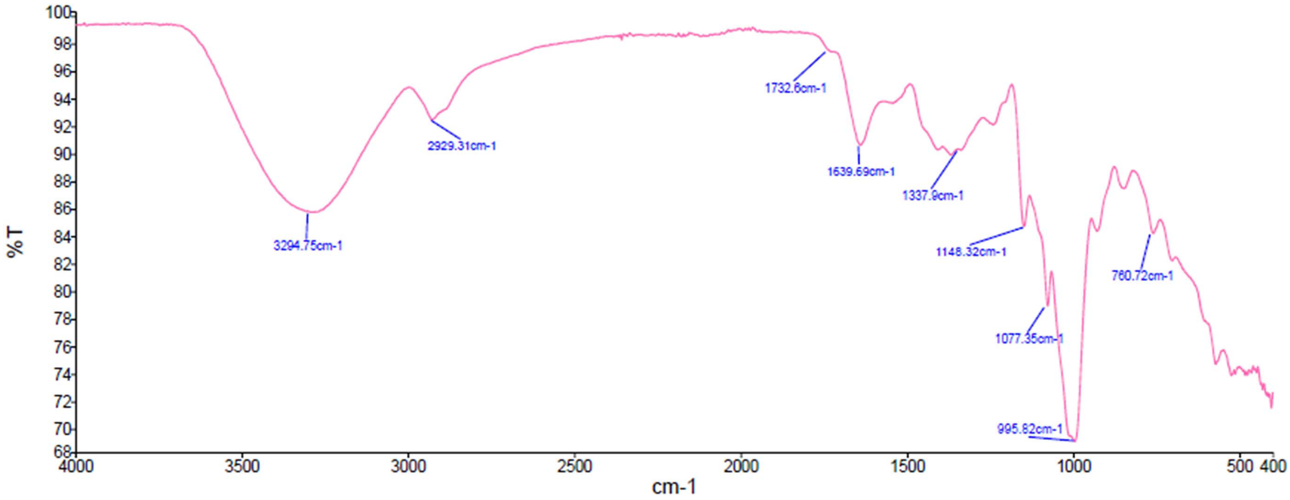
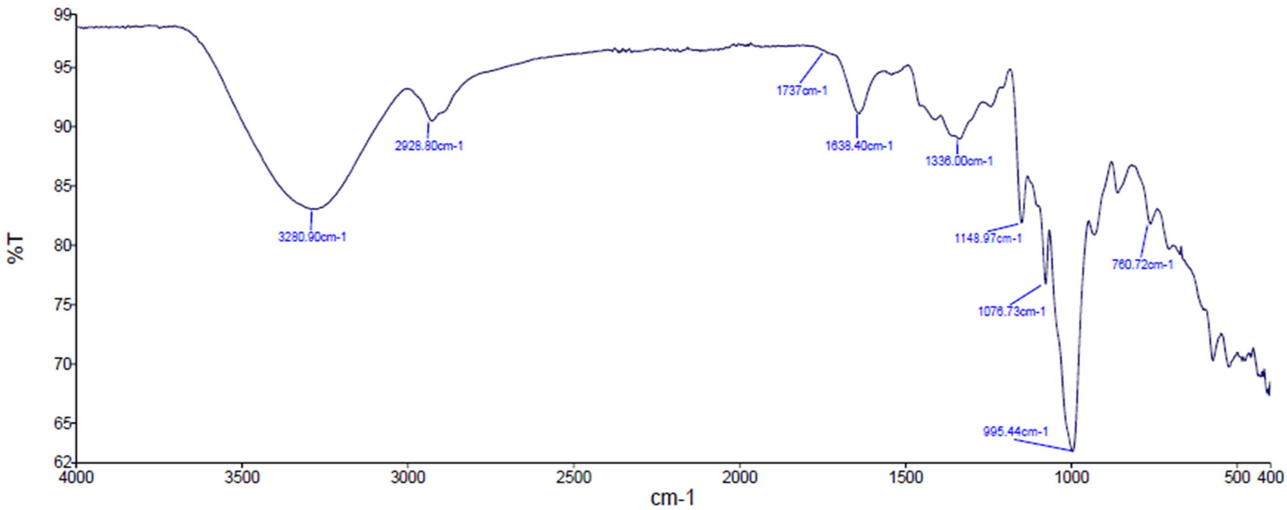


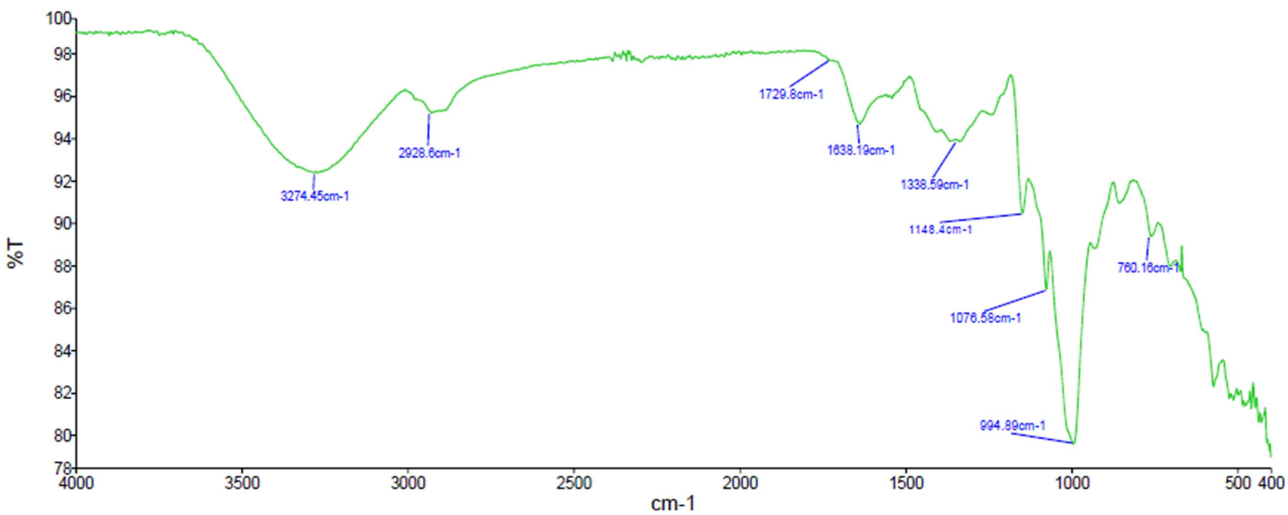
Figure 1. Percentage of transmittance versus vibrational frequency in cm-1 for the absorption bands of dried potato, (a); moraya, (b) and chuño, (c) respectively.



(a)

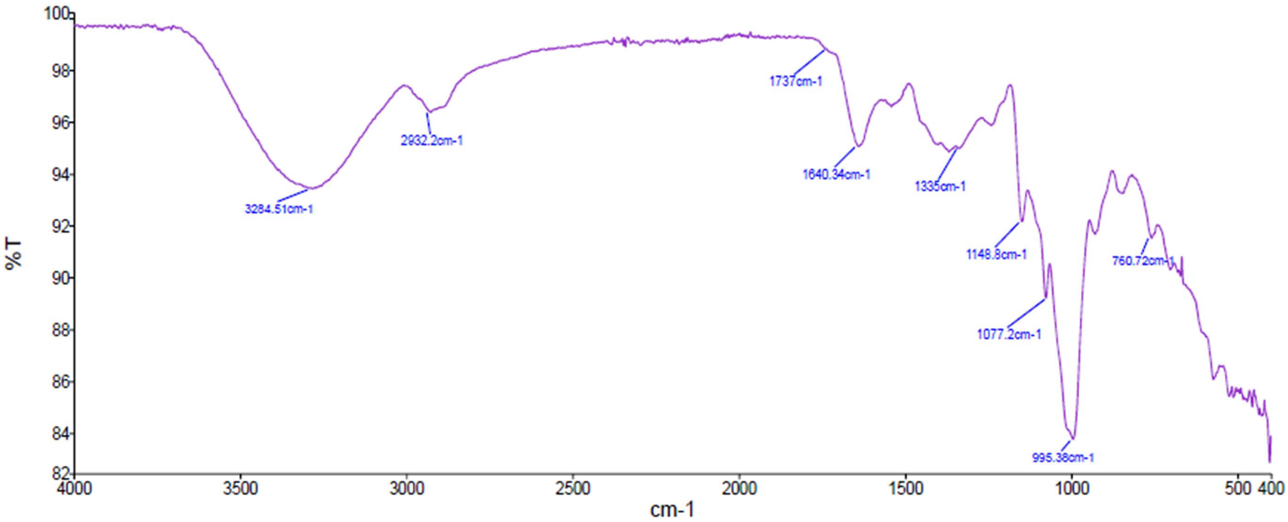


(b)

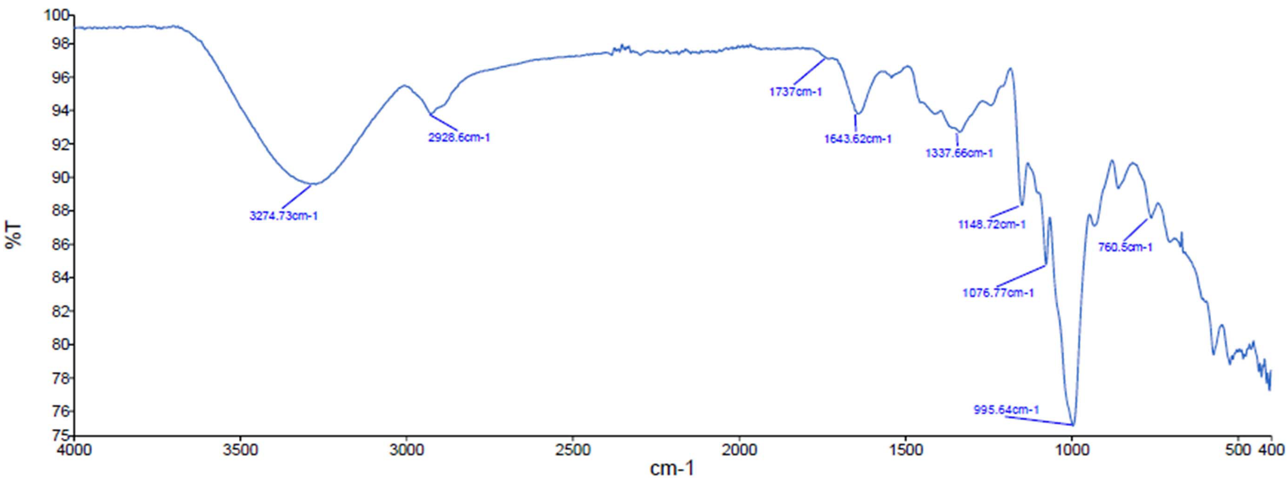


(c)

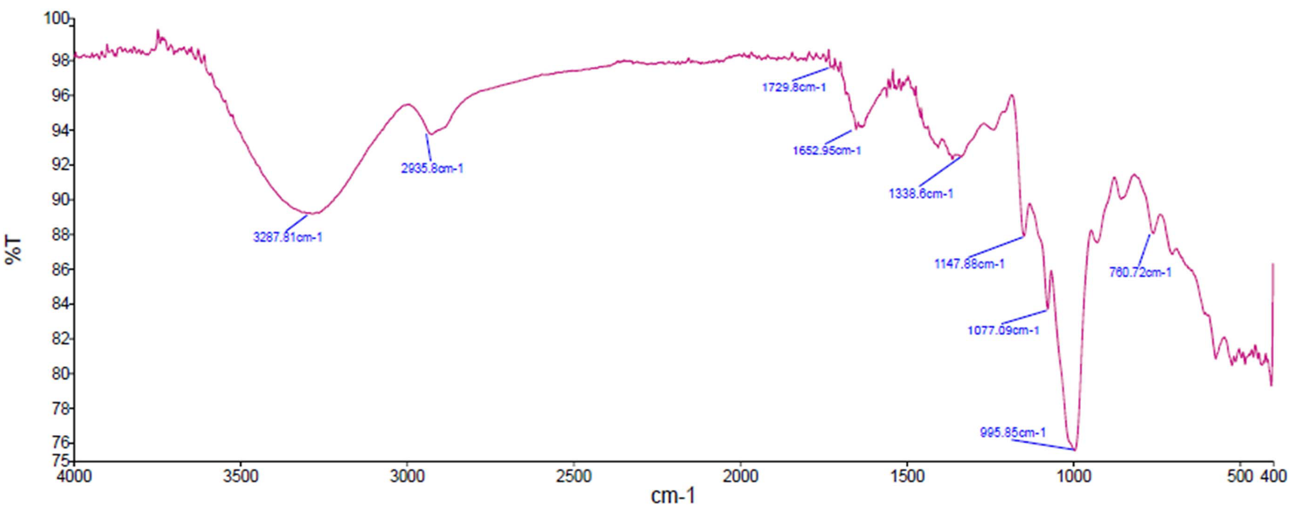
Figure 2. (a)-(c): Percentage of transmittance versus vibrational frequency in cm-1 for the adsorption bands of dried potato, (a); moraya, (b) and chuño, (c) after adsorption of lead respectively.



(a)



(b)



(c)

Figure 3. (a)-(c): Percentage of transmittance versus vibrational frequency in cm-1 for the adsorption bands of dried potato, (3a), moraya, (3b). chuño, (3c) after adsorption of copper respectively.

From Figures 2(a)-(c) and Figures 3(a)-(c), we observe shifts of the vibrational frequency to high values and a decrease in the % transmittance of the different vibrational and stretching signals after lead adsorption. The transmittance decreases which indicates that within the starch molecule, the metal cation has been adsorbed, which modifies the parameters of the original spectrum shown in Figures 1(a)-(c). The same behaviour is observed in the adsorption of copper by these derivatives, with a slight decrease in intensity. Similar behaviour is observed for the other metals such as cadmium and zinc, so it is concluded that these derivatives adsorb these metal cations which in the next section we will discuss the quantification of the same.

3.3. Adsorption of Metals (Pb^{2+}, Cd^{2+}, Zn^{2+} and Cu^{2+}) through the Biopolymer Derived from Potato (Dried Potato, Moraya and Chuño)

From the results of the adsorption analysis, taking into account the values of the initial and final concentration of the metal cation solution (Pb^{2+}, Cd^{2+}, Zn^{2+} and Cu^{2+}) retained per gram of adsorbent biopolymer respectively, the results show that the adsorption increased when the initial concentration increases, using these data the adsorption isotherms were determined for the extraction of these heavy metal cations, as shown in Figure 4, these adsorption isotherms coincide with the Freunlich isotherm, as shown in the following equation:

$$\frac{X}{M} = k\left(\frac{X}{L}\right)^{\frac{1}{n}} \qquad (6)$$

In Equation (6), X, M, L, k and n represent the moles of metal cation, grams of potato-derived biopolymer, litres of solution and the characteristic constants for each type of cation and adsorbent respectively.

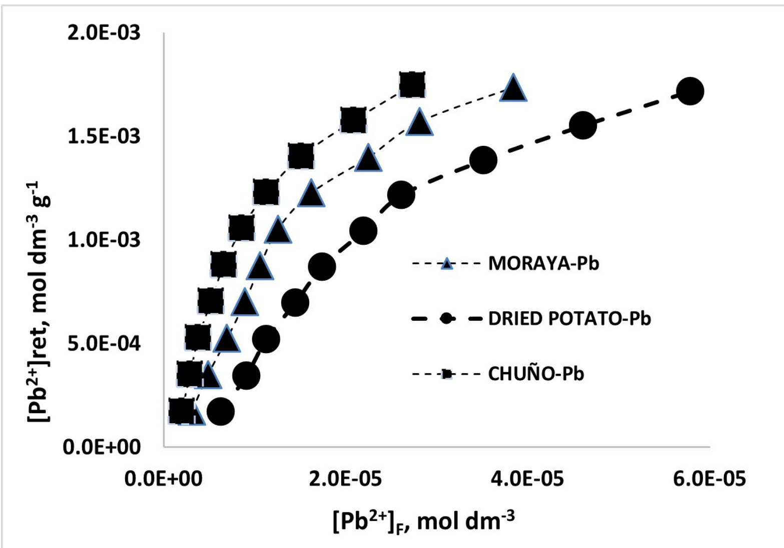


Figure 4. Adsorption isotherms of lead (nitrate as anion) using potato-derived biopolymer (dried potato, moraya and chuño) respectively at 25˚C.

The adsorption isotherms show similar behaviour to that reported by Aparicio, *et al.* [41] who used cocoa shells as adsorbent to remove heavy metals. These results are in line with those reported by Bashir [24] who used potato starch to remove heavy metals, and El-Asazy [29], who used natural potato peel to remove metals, whose results agree that potato, due to its high starch content, adsorbs metals as reported in this work.

Below are shown two representative examples of the adsorption isotherm of lead and zinc using potato-derived biopolymers (chuño, moraya and dried potato), shown in **Figure 4** and **Figure 5**. In these figures, it can be seen that the chuño derivative has a greater affinity for adsorbing these metals, followed by moraya and finally dried potato. The same behaviour is observed for the other cations when the adsorption results are analysed.

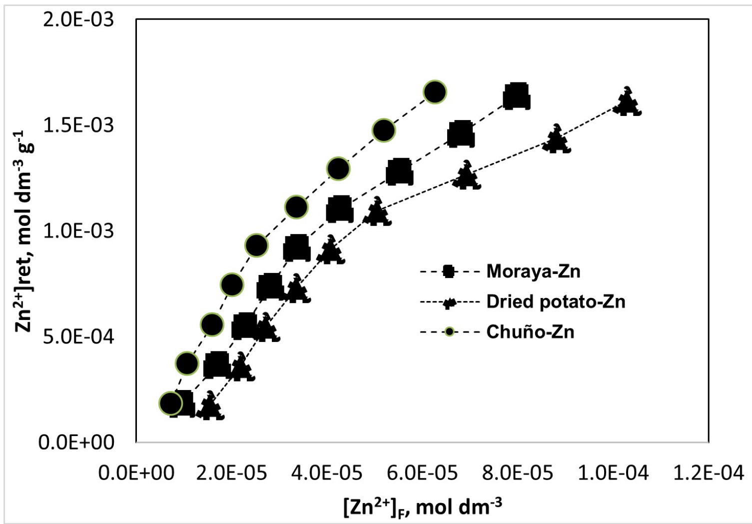


Figure 5. Adsorption isotherms of Zinc (nitrate as anion) using potato-derived biopolymer (dried potato, moraya and chuño) respectively at 25˚C.

3.4. Adsorption Selectivity

From the calculations made for the determination of the Freunlich adsorption isotherms summarised in **Table 2** at 25˚C, it is observed that the Freunlich constants, "*K*" stated in Equation (1), increases as the size of the metal cation increases, so that for the smallest cation "copper" presents a value of 11.49, for zinc 15.699, cadmium 23.174, while for lead the largest cation shows a value of 25.834, when the bioadsorbent used is the potato derivative, (CHUÑO). similar behaviour is observed with the other two bioadsorbents (moraya and dried potato) where it is corroborated that the largest cation always presents the highest value of "*K*", while the smallest cation shows the lowest value of this constant, while the constant "*n*" of the same equation shows an opposite behaviour in all cases. This is easily explained from the thermodynamic point of view; the adsorption experiments of these metals with the natural adsorbent are performed using water as solvent, which has a nitration free energy, which explains the affinity with water of these cations.

Table 2. Summary of Freunlich constants (*k* and *n*), obtained from adsorption isotherms, using the potato derived bioadsorbent at 25˚C.

Cation	Bioadsorbent					
	Chuño		Moraya		Dried Potato	
	k	n	k	N	K	n
Cu^{2+}	11.490	1.231	20.784	1.116	31.179	1.023
Zn^{2+}	15.699	1.124	23.249	1.050	34.121	0.997
Cd^{2+}	23.174	1.030	31.546	0.971	40.577	0.925
Pb^{2+}	25.834	0.923	34.015	0.885	46.552	0.845

Considering the free energies of hydration of these metal cations with water ($\Delta G \cdot h \cdot Kcal \cdot Mol^{-1}$) of −357.80, −430.50, 484.60 and 497.70, respectively, for lead, cadmium, zinc and copper cations [42]. Correlation of these values with Freulinch constant "k" can be observed in **Figure 6**, where it can be seen that the less hydrated cation such as lead presents greater adsorption by these potato-derived biopolymers, while the more hydrated cation such as copper presents a tendency to be less removed, observing the adsorption of the other metallic cations between these two limits.

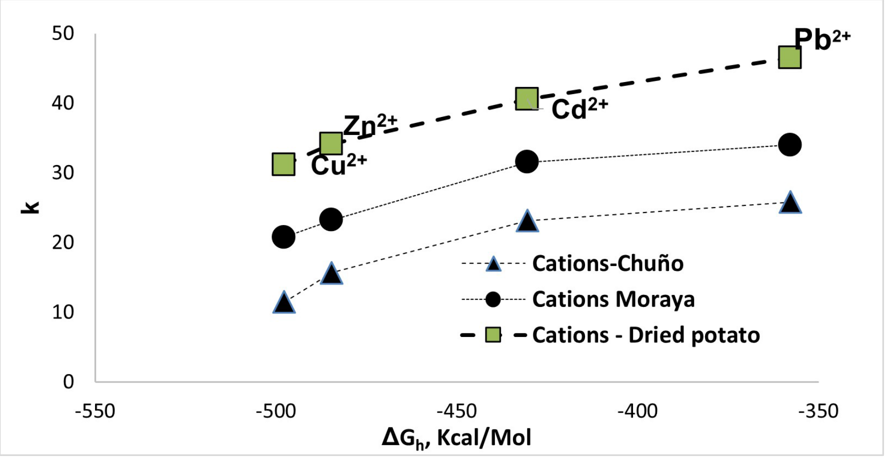


Figure 6. Plot of Freunlich constant, (k) versus Free Energy of Hydration of heavy metals, (Kcal/Mol) at 25˚C.

The difference in adsorption behaviour of potato-derived biopolymers towards these cations is small because these biopolymers contain mostly starch in their chemical composition, and due to the processes of obtaining these derivatives, there are differences in moisture, fibre, ash and protein content, which justifies these small differences in adsorption.

4. Conclusions

From the above discussion, it is concluded that:

1) Potato derivatives (*Solanum tuberosum*) adsorb heavy metals from water

contaminated with these metals.

2) The 3 derivatives, the one with the highest adsorption is the bioadsorbent chuño, followed by moraya and finally dried potato.

3) All these derivatives, due to the presence of the carbohydrate starch in their chemical composition, show a selectivity of adsorption $Pb^{2+} > Cd^{2+} > Zn^{2+} > Cu^{2+}$ in all cases.

Acknowledgements

The authors would like to thank the Universidad Nacional del Altiplano-Puno-Peru. This research was funded by the Vicerrectorado de Investigation.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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List of Abbreviations

WHO:	World Health Organization
AOAC:	Association of Official Analytical Collaboration
FTIR-ATR:	Fourier Transform Infrared-Attenuated Total Reflectance
Pb:	Lead
Cd:	Cadmium
Zn:	Zinc
Cu:	Copper