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Chapter

Use of Porous no Metallic Minerals to Remove Heavy Metals, Precious Metals and Rare Earths, by Cationic Exchange

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Abstract

This chapter is related with the preliminary study of some non-metallic minerals to evaluate their cationic exchange capacity, to remove heavy and precious metals, as well as rare earths elements. The minerals and materials used to execute the ion metals removal were bentonite, phosphorite, and diatomite. The chapter shows the physicochemical behavior of all these minerals, which were used to remove the mentioned elements from solutions coming from ore leaching. It was found that in all cases, the removal of heavy and precious metals, as well as rare earths elements reached over 90%. Although, there were minimal differences in efficiency for all minerals used (bentonite, phosphorite, and diatomite), it could be pointed that the phosphorite has the best results going from 99.43% of removal of Gd, to 99.95–100% for the case of Ce, Nd, La, Yb, Eu, Er, Sm, Tb, Ge, Pd, Pt, and Au.

Keywords: metals removal, cationic exchange, non-metallic minerals, bentonite, phosphorite, diatomite

1. Introduction

As is known, the cation exchange capacity (CEC) is the property of a solid to adsorb cations from the liquid phase, exchanging them for an equivalent amount of other cations. In the solid-liquid system, a dynamic equilibrium is established between the cations of the solution and those adsorbed on the active points of the surface of solid [1]. Similarly, a cation exchange capacity (CEC) can be defined as the measurement of the adsorption equilibria of anions. Cations retained in clays can be replaced by other cations; therefore, they are interchangeable.

There are many kinds of materials used for ion exchange of metals; firstly, the natural and synthetic inorganic minerals such as zeolites, clay minerals, and permutes can be considered; secondly, a great variety of organic materials like synthetic

resins, polysaccharides, and proteins can be found; and finally, carbonic materials can be found. Among all the above mentioned materials used for ion exchange, natural clays are of great interest because they are cheaper than the synthetic and organic materials; therefore, the evaluation of the ion exchange capability of some clays and natural materials could be of great interest to recover heavy and precious metals and also rare earths elements easily.

Particularly, the word “rare earth” comes from some oxides that are classified as “rare,” not by the difficulty of finding them, but because it was difficult to separate the elements from their minerals. Although rare earths are found in hundreds of minerals, only about 20 have favorable conditions to process them, such as bastnasite, monazite, aluminous clays, xenotime, loparite, and parisite [2]. A group of rare earths belong to a series of elements that are very difficult to separate by traditional techniques, since they have very similar chemical properties. Currently, rare earths are in great demand, because in the study of their properties, and have been discovered multiple industrial and technological applications, which currently have placed them as strategic chemical elements for the development of high-tech products and in high demand [2, 3].

The increasing demand for these elements in modern industry, especially for various advanced technologies, has required obtaining them in a very pure state, impossible to achieve with conventional techniques, which is why a novel researches and efforts have been carried out, into important development in the different fields of obtaining and recovering rare earths, from the different mineral resources that contain them [3–7]. For the recovery of these elements, there are different processes of separation of rare earths, such as photochemical separation, supercritical extraction, and extraction with solvents and ion exchange [8].

According to heavy metals, almost all of them are too toxic due to their industrial sources and the permitted levels in environment for their discharge. Toxicity of heavy metals depends on their bioaccumulation in environment, that also, depends about the chemical speciation, persistence and tendency of accumulation or bioaccumulation [9]. The classification of the techniques for the treatment of heavy metals in wastewaters depends on different factors, and these technique are conventional and nonconventional, principally. Among the conventional techniques, filtration by membrane, electrodialysis, inverse osmosis, nanofiltration, ultrafiltration, ionic exchange, chemical precipitation, and others can be found [10]. So, ion exchange using natural minerals could be of importance, due to the low cost of these minerals and the possibility of finding a better way to remove heavy metals from contaminated effluents.

According to the precious metals ions, their recovery traditionally was carried out by chemical precipitation; however, the process showed some disadvantages like the formation of toxic subproducts which avoid a complete recovery of the precious metals such as Ag and Au [11]. For the abovementioned, some processes of ion exchange have been developed, using resins and activated carbon, interchanging Ag and Au ions contained in residues or dissolved solutions from cyaniding [12].

On the other hand, various works on the ion exchange of metals with natural adsorbents (aluminosilicates such as zeolites, mineral clays, feldspars, and zeolites) show that these can be good candidates for the recovery of rare earths, as well as heavy and precious metals, taking into consideration some factors that affect the ion exchange such as pH, temperature, contact time, particle size, nature of the cations (size, ionic charge, shape, and concentration), anions associated with the cations in solution, solvent (water, organic solvents) and material selectivity, as well as the ion exchange rate [13–16].

It is for this reason and due to all the efforts made for the recovery of these metals, extraordinary growth has been generated in the last two decades due to their multiple maps in vital sectors for economic development, such as those of computer

science and renewable energies, but they are also of great interest in the defense sector. However, global production of rare earths is dominated by China at an alarming 97%, as well as some derived industries such as refining, obtaining alloys, and, to a lesser extent, manufacturing new generation magnets. Currently, China has slowed down its production and very serious questions for the coming years [2].

According to the above, and due to the exploration and discovery of deposits with rare earths contents in the state of Hidalgo, Mexico [17, 18], that studies are proposed for the recovery of these precious and heavy metals, as well as rare earths, through the cationic exchange with natural absorbents such as non-metallic minerals, also occurring in the state of Hidalgo.

According to the abovementioned, the main goal of this work is to determine the cationic exchange capacity (CEC) of different non-metallic minerals, for the recovery of precious metals, rare earths elements, and heavy metals from leached liquors. In the same way, the determination of the CEC of these minerals could be of utility for their use in the treatment of waste water containing heavy metals. Although there are some works related to the study of the CEC of diatomite, there are not many studies related with the study of the CEC for the bentonite and phosphorite, leading to an important innovation to this work.

2. Materials and methods

In this section, the minerals and materials used to do the ion exchange for the recovery of heavy and precious metals, as well as rare earths elements, will be described.

2.1 Materials

The minerals used in this work were obtained in different regions of the State of Hidalgo, Mexico. They were collected in the grinding plants from open mines of diatomite, phosphorite, and bentonite. All the minerals were wholly characterized to describe the principal physicochemical characteristics that they have to carry out for the corresponding ion exchange. Basically, the characterization was performed preparing samples of each mineral (bentonite, phosphorite, and diatomite) and crushing and grinding them until obtain a range of particle size less than 100 μm .

2.2 Ion exchange methods

To execute the ion exchange, several experiments were executed using leaching liquors of a SEDEX-type mineral located in the State of Hidalgo, Mexico, containing precious metals and rare earths [17, 18], and previously characterized by ICP-OES. The average chemical composition having these liquors is shown in **Table 1**.

To carry out the ionic exchange with the natural minerals, a two-stage procedure was done as follows: 20 g of each mineral (bentonite, phosphorite, and diatomite), at a -400 mesh particle size, were weighted and then, were added to 500 ml volume of leaching solution containing the elements shown in **Table 1**. The experiments were executed at a stirring rate of 500 rpm, with a pH of 0.3, which was controlled adding NaOH constantly during all experiment, and the reaction time used was of 24 h. After ending the reaction time, solution was filtered and dried; then, both solids as residual liquors were characterized (ICP-OES, XRD, XRF, and SEM-EDS) to evaluate the capability of ion exchange of each mineral. The analysis done by ICP-OES was executed taking the final liquid after ion exchange, and then sample was diluted taking 1 ml of solution into 100 ml of distilled water having a 1/100

ICP analysis	
Element	Ppm
Au	45.93
Pd	1.92
Pt	0.005
Ce	81.79
Nd	56.97
La	51.89
Yb	33.35
Tb	25
Ge	9
Ga	2.5
Sm	1.75
Er	0.9
Eu	0.35

Table 1.

Chemical composition of the leaching liquors of the SEDEX mineral, done by ICP.

dilution factor. With this, all the elements fell in a range of concentration below 1 ppm for the case of Au, Ce, Nd, L, Tb, Ge, and Yb. For the rest of the elements, a direct sample was taken using standards between 1 and 5 ppm (without dilution).

2.3 Analytical methods

2.3.1 Characterization of minerals

The analytical methods used for the characterizations of the different mineral used to the ion exchange stage were characterized by dry granulometric analysis using a standard sieves (Tyler series); X-ray diffraction using an INEL equinox 2000 equipment was located at the Autonomous University of the State of Hidalgo (AUSH), and the spectra was obtained with a Cu $K_{\alpha 1}$ radiation of wave length of 1.50056 Å, a voltage of 30 kV, an intensity of 20 mA, and a sweep speed of 22 θ /min, which were then treated with the software MATH to identify the mineral species contained in each material.

2.3.2 Chemical analysis

Then, to determine the chemical composition of standard solutions and leached liquors (before and after ion exchange), an inductively coupled plasma spectrophotometer (ICP-OES) Perkin Elmer brand/model 2100 located at the AUSH was used. In the same way, scanning electron microscopy (SEM) was used to determine morphology and particle type of minerals, using a JEOL brand microscope model JMS6300, having an Energy Dispersive Spectrometer of X-ray (EDS) for chemical microanalysis and also located at the AUSH. And finally, X-ray fluorescence (XRF) was executed to determine the chemical composition of minerals before and after the ion exchange procedure, and for this analysis, a portable spectrophotometer of X-ray brand BRUKER, model S1 TITAN was used, also using the GeoChem calibration software (equipment located at the National Autonomous University of Mexico).

3. Results and discussion

3.1 Characterization of minerals

3.1.1 Bentonite before ion exchange experiments

Bentonite is a rock composed essentially of minerals from the group of smectites, regardless of any genetic connotation. Additionally, it has the ability to inflate and increase its volume by weight several times on contact with water, to form thixotropic gels when added in small quantities. Finally, it can be said that its main interchangeable cations are Na^+ , Ca^{2+} , K^+ , Mg^{2+} , Li^+ , and H^+ .

Table 2 shows the results obtained by ICP and FRX of the elements contained in the bentonite studied, of which silicon, aluminum, iron, sodium, and potassium are present in the majority among others.

Likewise, in **Figure 1**, the mineral species identified by X-ray diffraction are observed, in which the presence of majority of mineral phases, such as quartz,

Element (oxide)	ICP (wt.%)	FRX (wt.%)
Na_2O	2.66	0.92
MgO	2.71	2.76
Al_2O_3	14.10	14.92
SiO_2	73.10	73.41
K_2O	1.32	1.67
CaO	3.11	3.93
TiO_2	0.37	0.35
Fe_2O_3	2.63	2.60

Table 2.
 Chemical composition of natural bentonite.

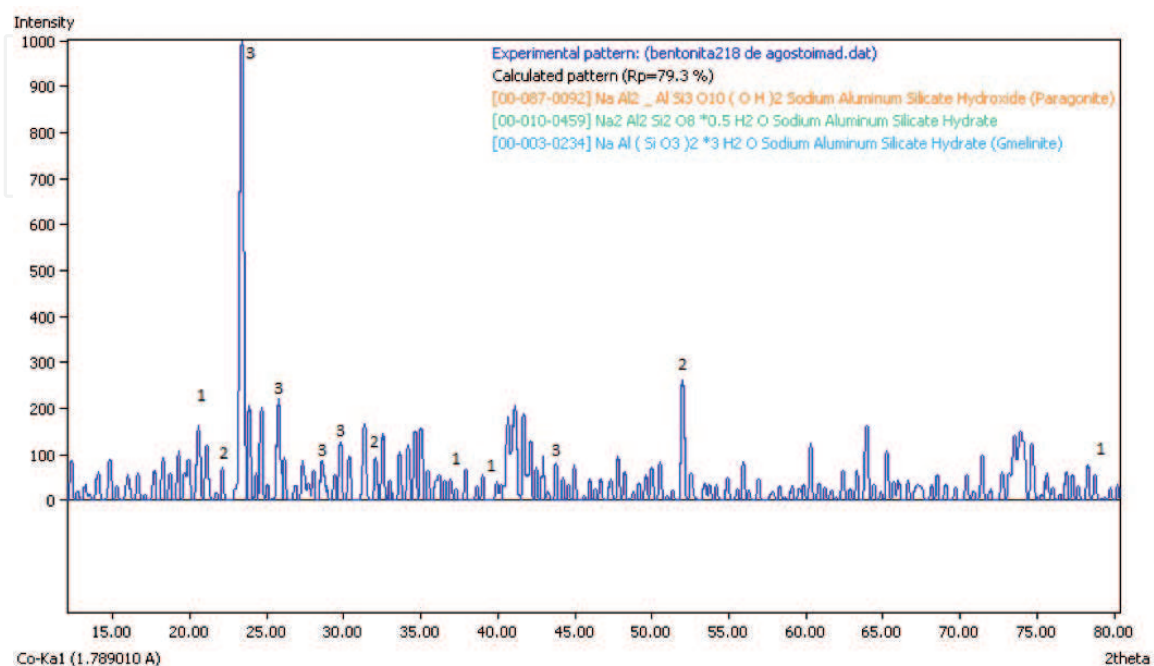


Figure 1.
 X-ray diffractogram of bentonite.

anorthoclase, orthoclase, albite, and berlinite, can be seen, as well as sodium silicoaluminates (paragonite and gmelinite).

On the other hand, **Figure 2** shows an image of a bentonite particle at -400 mesh, analyzed by SEM-EDS, where the semiquantitative and point composition can be observed, showing the presence of elements such as silicon, aluminum, sodium, magnesium, potassium, and iron, which are characteristic in the bentonite (**Figure 2a**); likewise, the bentonite particle is shown in detail, having a lot of porosity, which is essentially one of the most important physical characteristics for the ion exchange that could be carried out, besides the particle size, offering a large surface and contact area, **Figure 2b**.

3.1.2 Bentonite after ion exchange experiments

The results obtained after ion exchange experiments are presented as chemical composition of liquors before and after the procedure; **Table 3** shows the chemical composition of these liquors obtained by ICP, showing also the efficiency of ion exchange for each element. It can be noticed that for all elements present in original leaching liquor, the efficiency is over 90%.

After the ion exchange, the solution was filtered and the solid residue of the bentonite was dried and analyzed by XRD. The mineral species identified by X-ray diffraction (**Figure 3**), are majority mineral phases, such as quartz, anorthoclase, orthoclase, albite, berlinite, and silico-aluminates, typical of bentonite. Likewise, signs indicating the presence of rare earths and precious metals are noted, which corroborates that the exchange of these elements was made to the natural absorbent.

Finally, **Figure 4** shows the image of a bentonite particle after performing the ion exchange with the leaching liquors of a SEDEX-type mineral, where the semiquantitative composition and the distribution of the elements are shown by X-ray mapping, observing that, indeed, the aforementioned elements were absorbed in the bentonite ore.

3.1.3 Phosphorite before ion exchange experiments

Phosphorites are sedimentary rocks that contain at least 20% P_2O_5 in the form of cryptocrystalline fluorapatite, apatite, or some other minerals containing

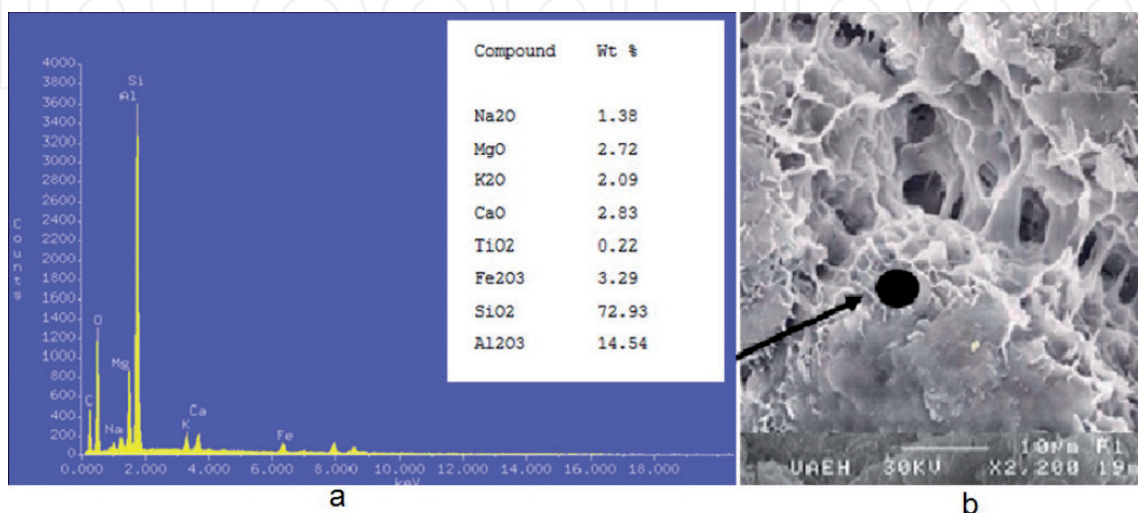


Figure 2. SEM analysis of bentonite: (a) point microanalysis by SEM-EDS and (b) general image (SEM-SE).

Element	Content before ion exchange (ppm)	Content after ion exchange (ppm)	Efficiency of the ion exchange (%)
Au	45.93	0.072	99.84
Ce	81.79	0.000	100.00
La	51.89	0.000	100.00
Nd	56.97	0.020	99.96
Pd	1.92	0.108	94.37
Yb	33.35	0.009	99.97
Ge	9	0	100
Gd	1.4	0.111	92.07
Tb	0.25	0	100
Sm	1.75	0	100
Er	0.9	0	100
Eu	0.35	0.011	96.86
Pt	0.005	0	100

Table 3.
 Results of the ion exchange done using bentonite (ICP).

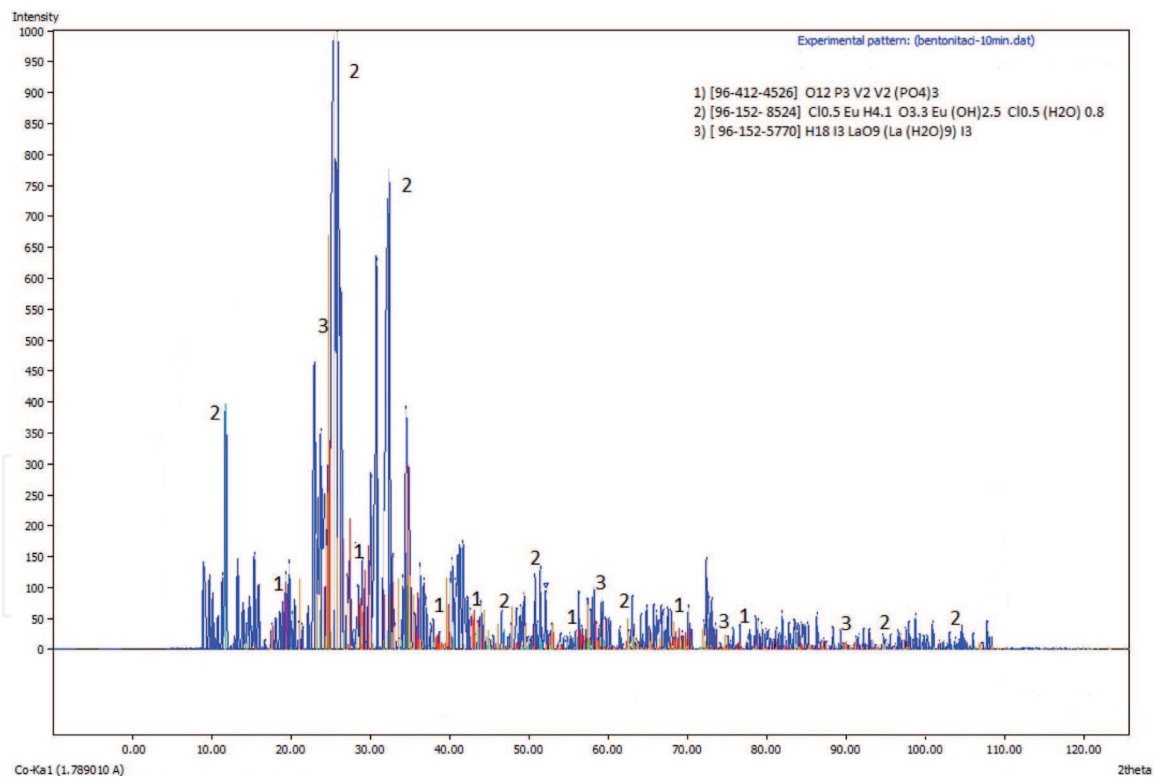


Figure 3.
 X-ray diffractogram of bentonite after ion exchange.

phosphorus. They usually occur in layers, and it can also be present in the form of crusts, spherulites, and nodules in sedimentary horizons. Rocks with less than 10% phosphate receive the adjective of phosphates. Its main interchangeable cations are: Ca^{2+} , K^+ , and Mg^{2+} .

Before carrying out the ion exchange experiments, the phosphorite was characterized to know its composition and compare it at the end of the ion exchange

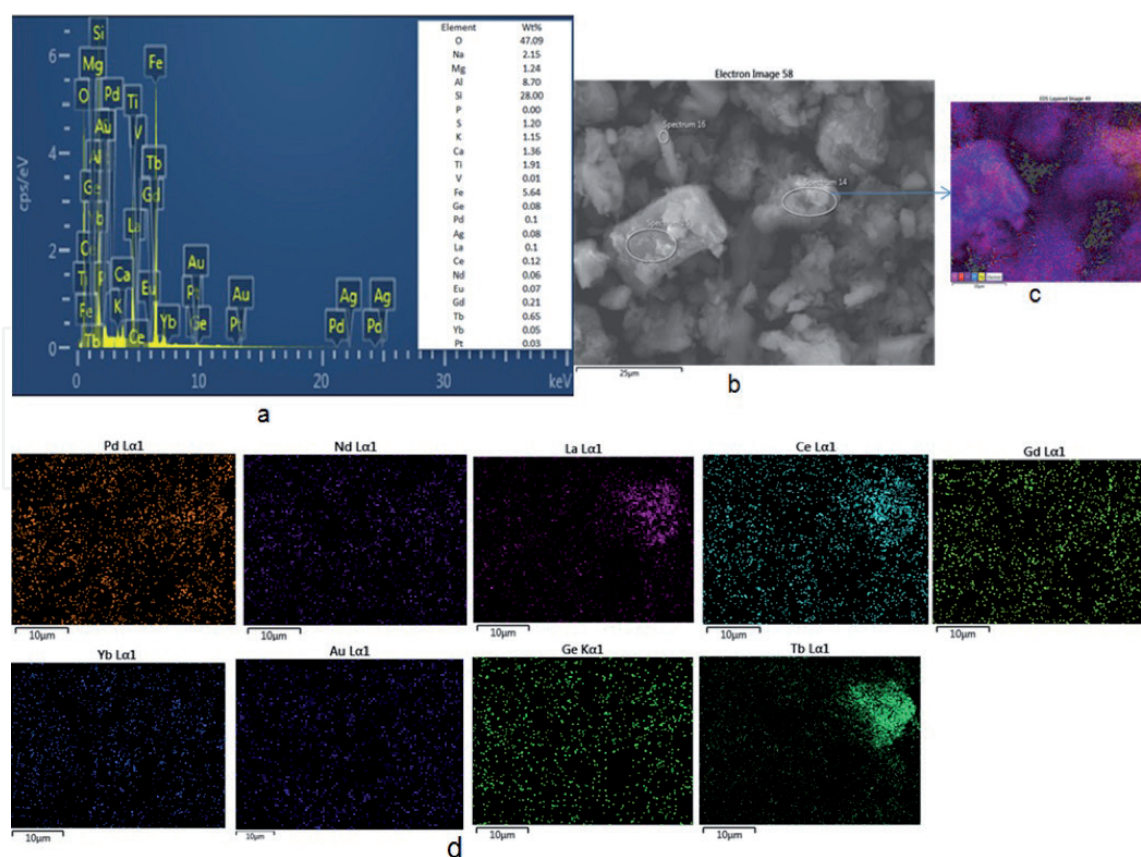


Figure 4. Photomicrographs of bentonite after ion exchange: (a) general microanalysis SEM-EDS; (b) general image, 2000 \times , SEM-SE; (c) point image where the microanalysis was taken; and (d) distribution mapping of rare earth elements and precious metals in the bentonite.

Element	ICP (wt.%)	XRF (wt.%)
Na ₂ O	0	0.16
Al ₂ O ₃	2.43	2.40
P ₂ O ₅	36.03	36.08
K ₂ O	0.15	0.21
CaO	59.99	59.95
TiO ₂	0.48	0.31
Fe ₂ O ₃	0.92	0.89

Table 4. Average chemical composition of phosphorite before ion exchange.

experiments, and thus to evaluate its exchange capacity for the elements present in the leaching liquors. **Table 4** shows the results obtained by ICP and XRF of the chemical composition for the phosphorite, where the average contents of 36% of P₂O₅ are shown, as well as the majority of aluminum, calcium, and minor elements such as oxides of sodium, potassium, titanium, and iron.

On the other hand, the mineral species identified by X-ray diffraction (**Figure 5**) are mainly mineral phases of calcium and phosphorus.

Finally, in **Figure 6**, an image of phosphorite particles at –400 mesh can be seen, which was analyzed by SEM-EDS, and here, also the presence of major elements of phosphorus, aluminum, and calcium oxides, as well as minor elements of sodium, titanium and iron, can be observed, as is shown in **Figure 6a**; similarly, the photomicrographs of the phosphorite particle are shown in **Figure 6b**.

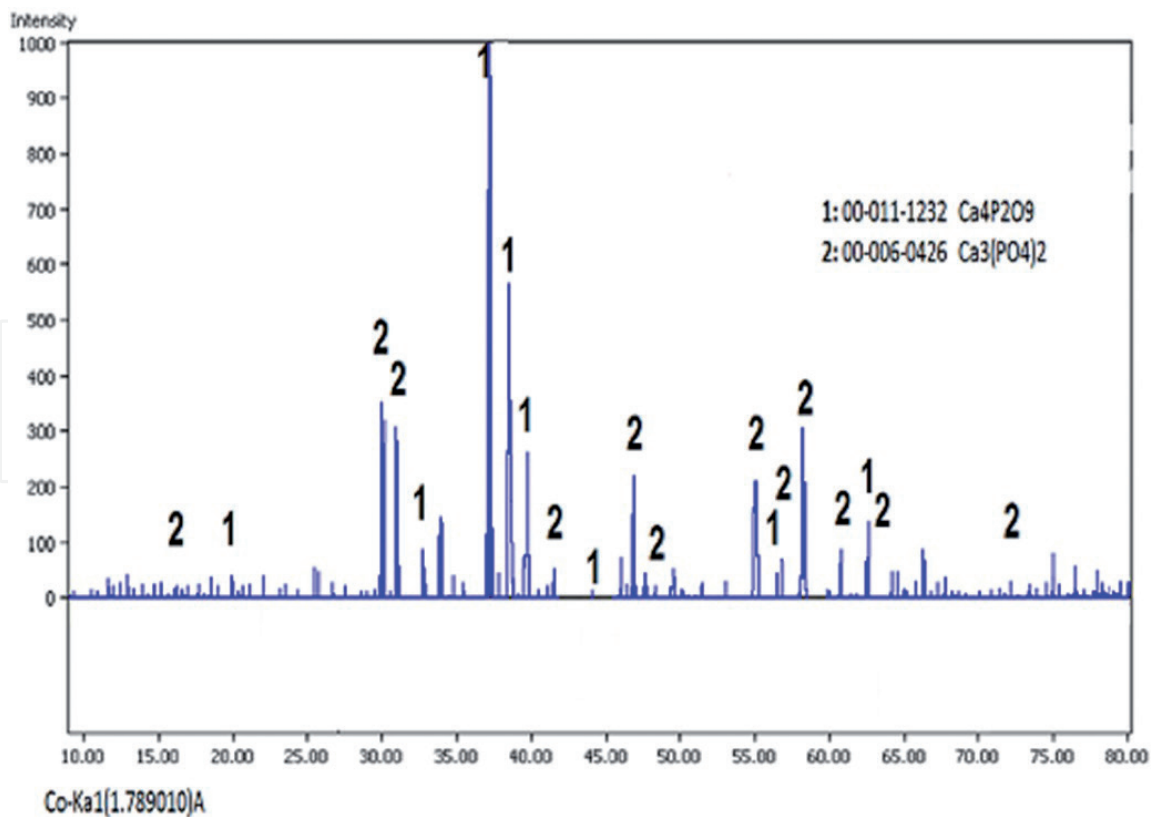


Figure 5.
 X-ray diffractogram of phosphorite, before ion exchange.

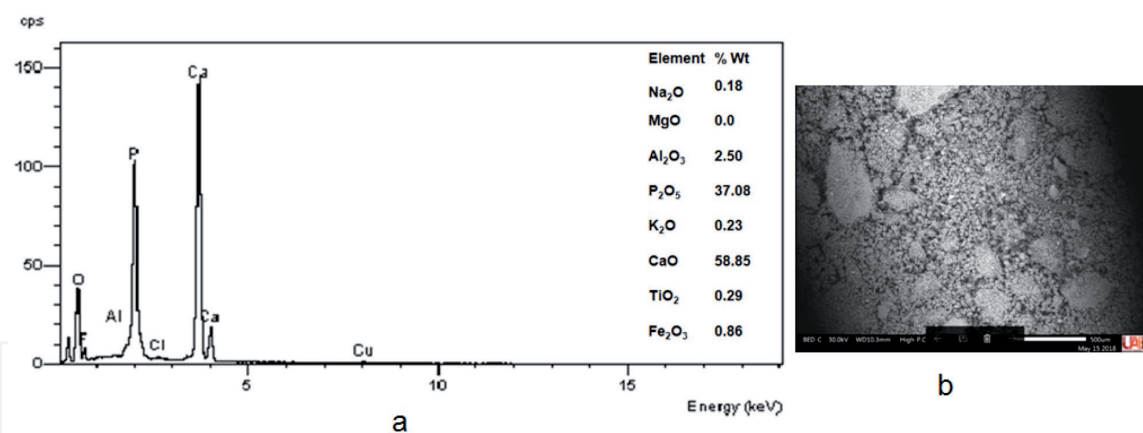


Figure 6.
 Photomicrographs of the phosphorite -400 meshes, (a) SEM-EDS microanalysis and (b) general image, 2200×, SEM-SE.

3.1.4 Phosphorite after ion exchange experiments

After carrying out the ion exchange, using phosphorite as an absorbent mineral, the solution was filtered and analyzed by ICP, comparing the results with the initial composition of the leaching liquors. The results are shown in **Table 5**, where a comparison is made between the original leaching liquors before and after the exchange, as well as the efficiency of the cation exchange of this mineral; it is observed that in all cases, efficiencies are greater than 99% of the elements exchanged, which can determine that phosphorite is a good natural absorbent for rare earths and precious metals.

Likewise, the solids obtained after the ion exchange were separated and dried to be later analyzed by XRD. The mineral species identified by X-ray diffraction (**Figure 7**) are majority phases such as quartz, anorthoclase, orthoclase, albite,

berlinite, and silicoaluminates. Similarly, the presence of rare earths, such as lanthanum and cerium, and precious metals that were all adsorbed by this mineral are noted.

Element	Content before ion exchange (ppm)	Content after ion exchange (ppm)	Efficiency of the ion exchange (%)
Au	45.93	0	100
Ce	81.79	0.037	99.95
La	51.89	0.017	99.97
Nd	56.97	0.02	99.96
Pd	1.92	0	100
Yb	33.35	0.001	99.99
Ge	9	0	100
Gd	1.4	0.008	99.43
Tb	0.25	0	100
Sm	1.75	0	100
Er	0.9	0	100
Eu	0.35	0	100
Pt	0.005	0	100

Table 5.
Results of the ion exchange done using phosphorite (ICP).

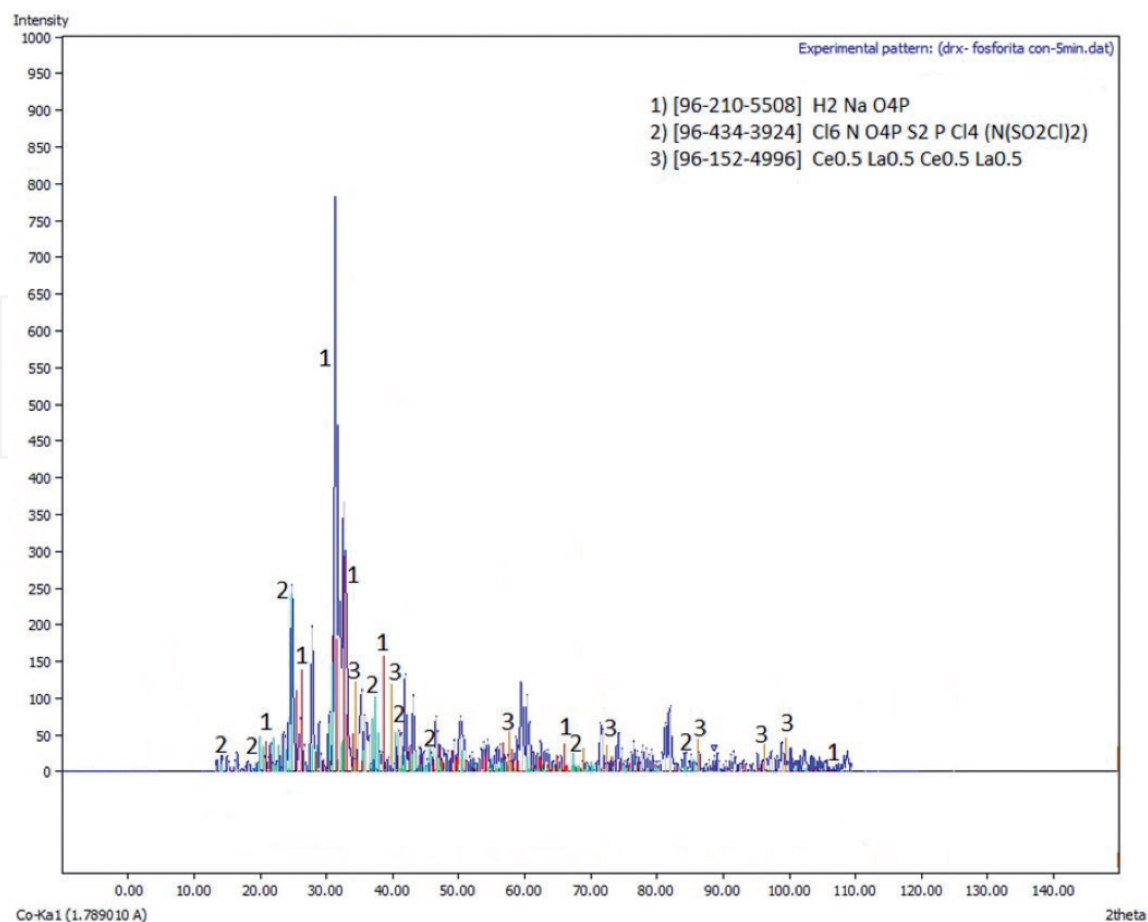


Figure 7.
X-ray diffractogram of phosphorite material after ion exchange.

Finally, **Figure 8** shows the image of phosphorite particles after performing the ion exchange with the leaching liquors of a SEDEX-type mineral, where the semiquantitative point composition and the X-ray mapping of the elements are shown. This verifies that effectively the aforementioned elements were absorbed in the mineral.

3.1.5 Diatomite before ion exchange experiments

Diatomite is a siliceous sedimentary rock of biogenic origin, composed of fossilized skeletons of diatomite frustules. It is composed by sedimentary accumulation

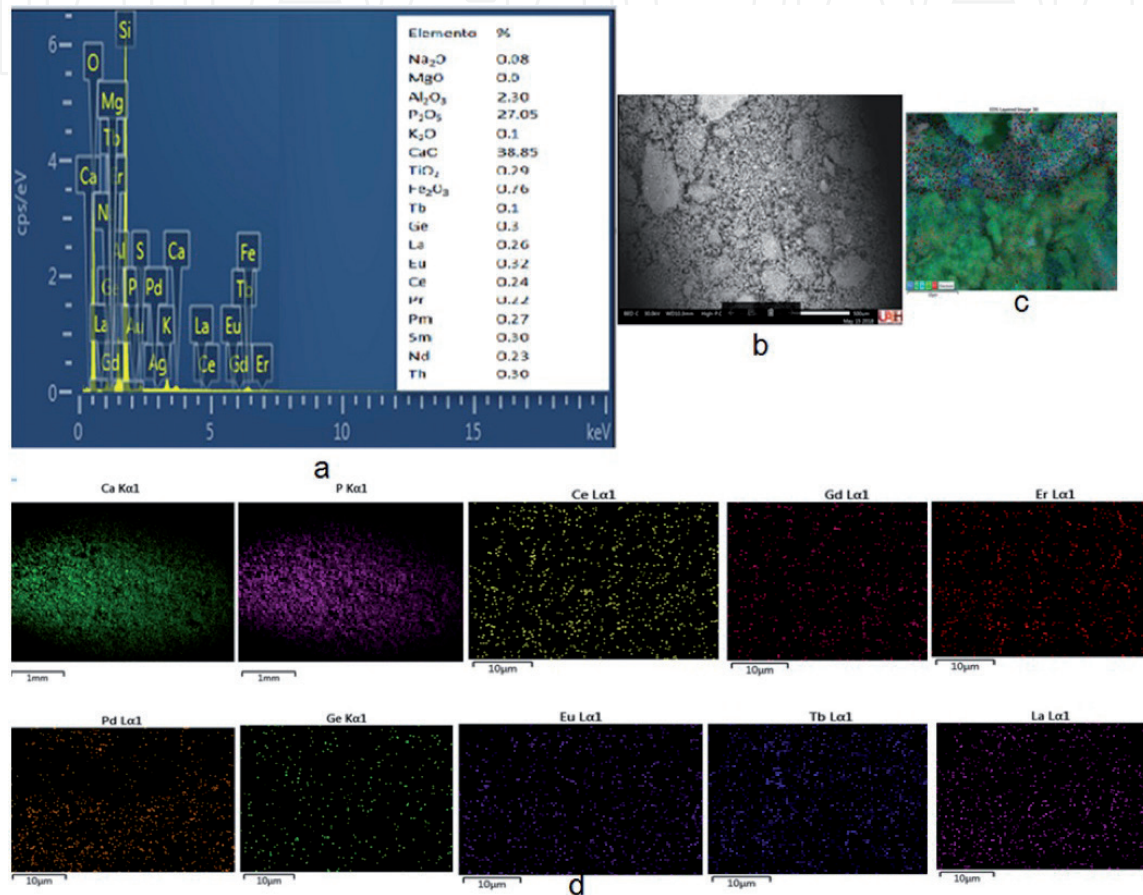


Figure 8. Photomicrographs of the phosphorite after ion exchange: (a) SEM-EDS microanalysis; (b) general image, 2000×, SEM; (c) point image where the microanalysis was taken; and (d) distribution mapping of rare earth elements and precious metals in the phosphorite.

Element	ICP (wt.%)	XRF (wt.%)
Na ₂ O	0.61	0.923
MgO	1.79	1.87
Al ₂ O ₃	11.63	7.77
SiO ₂	76.00	77.36
K ₂ O	2.41	1.2
CaO	0.85	1.04
TiO ₂	0.50	0.348
Fe ₂ O ₃	1.95	2.35

Table 6. Average chemical composition of diatomite before ion exchange.

to form large deposits with a sufficient thickness to have a commercial potential. Its main interchangeable cations are Na^+ , Ca^{2+} , K^+ , Mg^{2+} , and Si^{2+} .

Firstly, before carrying out the ion exchange experiments, the diatomite mineral was characterized to evaluate, at the end, its exchange capacity. **Table 6** summarizes the results obtained by ICP and XRF of the original elements contained in the diatomite, presenting average contents of 76.68% of silicon, as well as the majority contents of alumina, hematite, potassium oxide, magnesium oxide, calcium oxide, and minor elements such as sodium oxide and titanium.

Likewise, the mineral species present in the diatomite were identified by X-ray diffraction (**Figure 9**), observing the presence of majority mineral phases such as quartz, albite and berlinite.

Finally, in **Figure 10**, an image of a diatomite particle at -400 mesh is shown, in which an analysis was carried out by SEM-EDS. The presence of major elements such as silicon, aluminum, sodium, magnesium, potassium, and iron, are characteristic of the diatomite. Likewise, a photomicrograph of the diatomite particle can be seen in **Figure 10b** where the characteristics properties of the diatoms of the material can be observed.

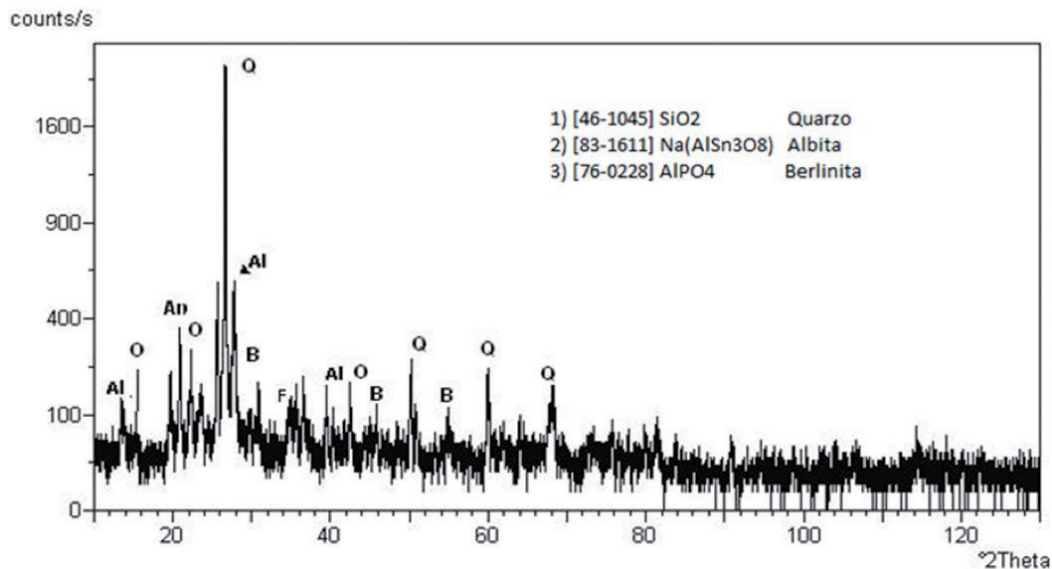


Figure 9.
X-ray diffractogram of diatomite material before ion exchange.

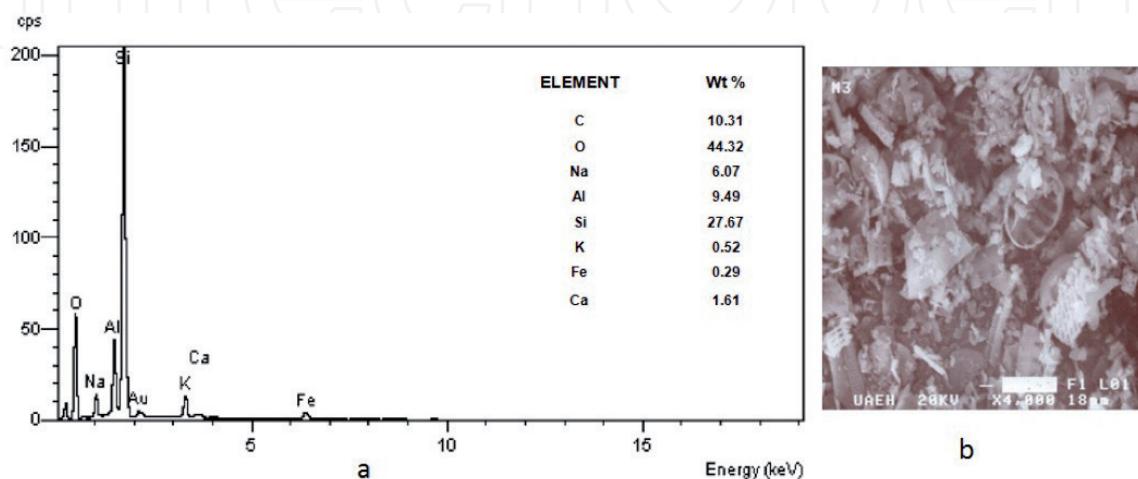


Figure 10.
Photomicrographs of the diatomite -400 meshes, (a) SEM-EDS microanalysis and (b) general image, $4000\times$, SEM-SE.

3.1.6 Diatomite after ion exchange experiments

After carrying out the ion exchange, the diatomite mineral was characterized to know the elements exchanged and thus calculate the ion exchange capacity of this mineral. The results are shown in **Table 7**, where a comparison is made between the original leaching liquors and after the exchange through ICP analysis. Likewise, it shows the % of efficiency of the cation exchange and as a result can be seen that

Element	Content before ion exchange (ppm)	Content after ion exchange (ppm)	Efficiency of the ion exchange (%)
Au	45.93	0	100
Ce	81.79	0.003	99.996
La	51.89	0.004	99.99
Nd	56.97	0.008	99.99
Pd	1.92	0	100
Yb	33.35	0.001	99.997
Ge	9.0	0	100
Gd	1.4	0.03	97.86
Tb	0.25	0	100
Sm	1.75	0	100
Er	0.9	0	100
Eu	0.35	0.003	99.14
Pt	0.005	0	100

Table 7.
 Results of the ion exchange done using diatomite (ICP).

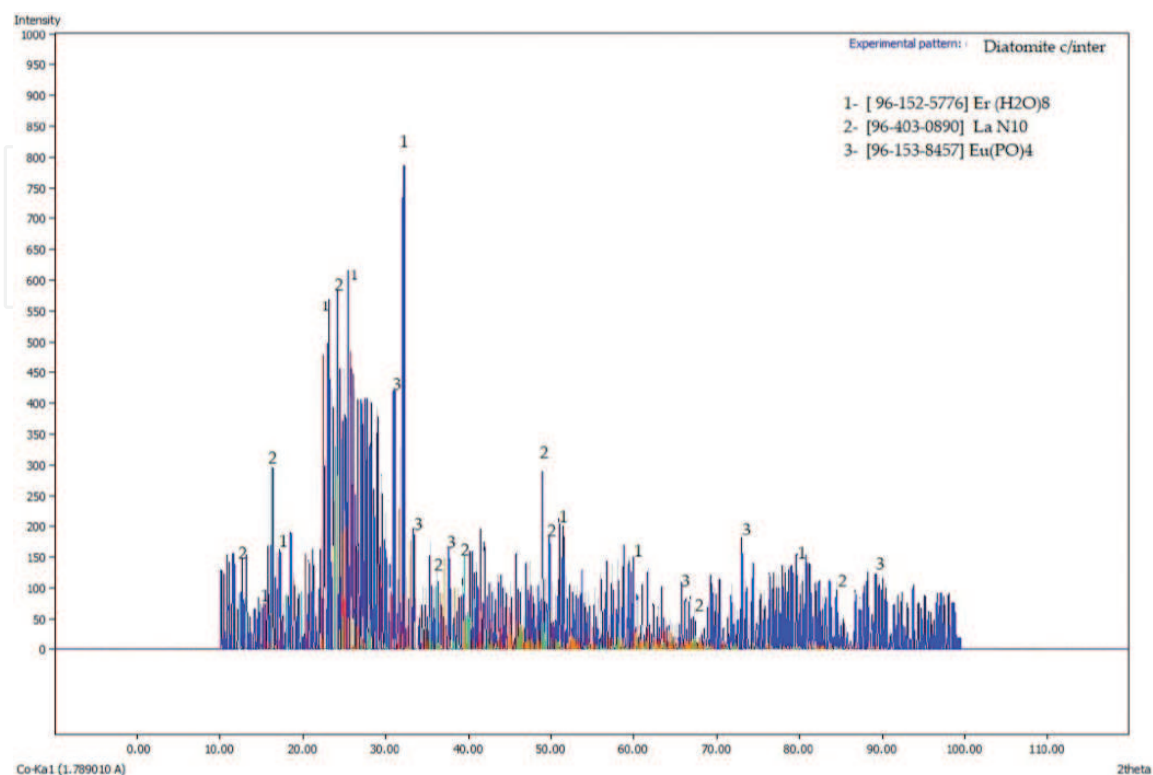


Figure 11.
 X-ray diffractogram of diatomite material, after ion exchange.

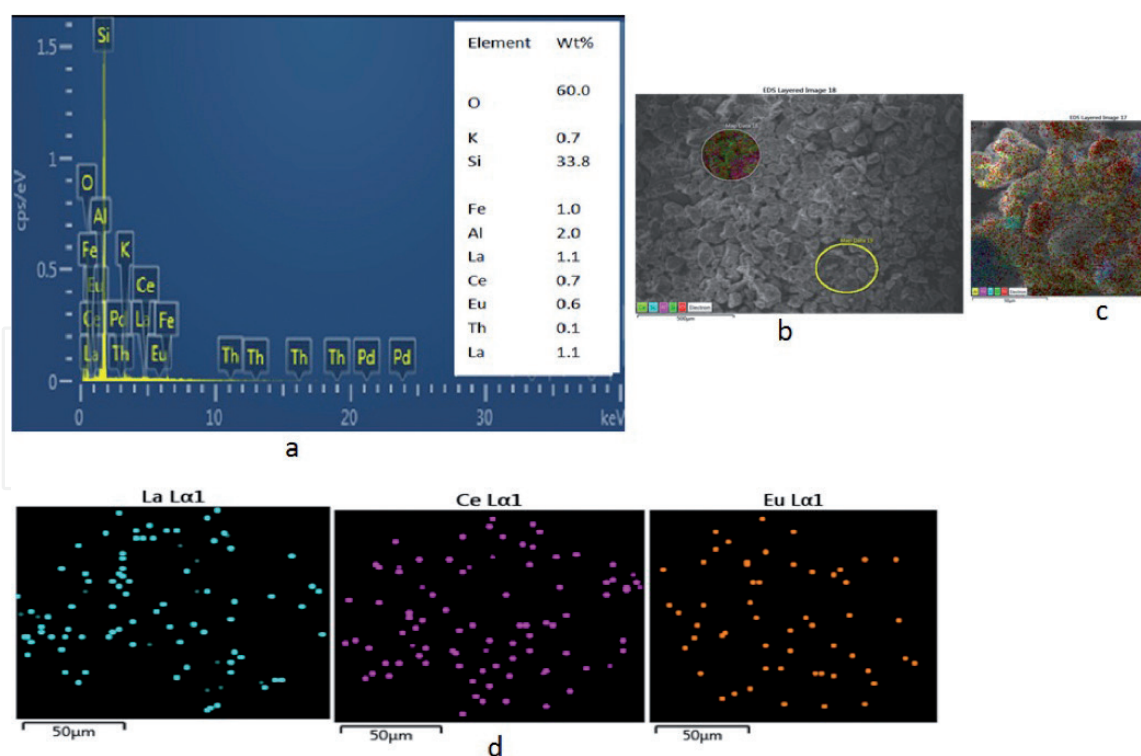


Figure 12. Photomicrograph of the diatomite –400 meshes, (a) SEM-EDS microanalysis, (b and c) general image, SEM-SE, and (d) mappings.

diatomite shows an exchange efficiency for the precious metals of 100%, as well as the Pd, Ge, Tb, Sm, and Er. The other elements show an efficiency superior to 99%, not so for the Gd, since its exchange efficiency is 97.86, which corroborates that the diatomite has an exchange efficiency for these types of metals. On the other hand, the author also has to demonstrate the efficiency of cationic exchange of diatomite, getting 96.46% of efficiency for the removal of As^{3+} , 95.44% for Ni^{2+} , and 98.80% of Pb^{2+} [13].

Likewise, the mineral species were identified by X-ray diffraction (**Figure 11**), observing the presence of majority mineral phases such as quartz, anorthoclase, orthoclase, albite and berlinite; as well as the presence of rare earths such as Er, La, and Eu.

Finally, **Figure 12** shows the image of a diatomite particle after ion exchange with the leaching liquors of a SEDEX type mineral, where can be observed the semiquantitative point composition and the distribution of elements by X-ray mapping, can be concluded that the elements present in the leaching liquors were effectively absorbed into the diatomite particles.

4. Conclusions

The preliminary results in the cation exchange of heavy metals, precious metals, and rare earths, through the use of non-metallic porous minerals, show a good efficiency since for most of the cases, recoveries over 99% were obtained, except for some elements; in the case of bentonite, for Pd, only 94.37%, for Gd, only 92.09%, and for Eu, only 96.87% absorption were obtained, and in the case of the phosphorite, the lowest value was for Gd with a 99.43% efficiency; in the case of diatomite, Gd presented a 97.86% efficiency in the exchange (**Table 8**). Therefore, it can be concluded that these natural absorbers can be used with a high efficiency for the exchange of these metals, noting also that for most of the cases, Gd presented recoveries above 90% and for the case of the precious metals, it was above 99% for all the minerals used.

Element	Bentonite % in CEC	Phosphorite % in CEC	Diatomite % in CEC
Au	99.84	100	100
Ce	100.00	99.95	99.996
La	100.00	99.97	99.99
Nd	99.96	99.96	99.99
Pd	94.37	100	100
Yb	99.97	99.99	99.99
Ge	100	100	100
Gd	92.07	99.43	97.86
Tb	100	100	100
Sm	100	100	100
Er	100	100	100
Eu	96.86	100	99.14
Pt	100	100	100

Table 8.
Cation exchange capacity (CEC), per element, of the mineral used for the removal of precious metals, heavy metals and rare earth elements.

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Conflict of interest

The authors declare no conflict of interest.

Notes

This chapter not only shows unpublished preliminary results obtained by the authors where they have evaluated the capacity of ion exchange for some natural minerals found in the state of Hidalgo Mexico, but also exhibits an exhalative sedimentary mineral found by the same authors in the north of the state of Hidalgo, where ore mineral has important contents of precious metals and some rare earths elements.

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