Development of a Predictive Model for
the Recovery of Rare Earth Elements from
the Leaching Process of Chilean Ores

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Abstract

In this paper, it was developed a statistical model for the recovery of Rare Earth Elements in a leaching process, from Chilean ores, using the Neural Networks technique. The complexity for the elaboration of predictive and mathematical models for the conventional leaching process, besides the difficulty of obtaining rare earth elements from low-grade minerals; represent a big challenge for the development of theoretical studies. Recovery of the elements of interest were modeled. In this case, Lanthanum, Cerium and Iron. The latter, because it represents the largest amount of impurities present in the leaching solution. Using the most important data (Place of origin, initial acid dose, temperature, solid/liquid ratio, initial ore mass, lanthanum, cerium and iron law), 3 different neural networks were developed. In this way, it is possible to affirm that this methodology of artificial neural networks can be used to determine the degree of recovery of a species of interest, according to the most important variables of the process.

Keywords: Neural network, Rare Earth Elements, Hydrometallurgy

1 Introduction

Rare Earth Elements (REE) are a group that includes 17 elements, which are the lanthanides, scandium (Sc) and yttrium (Y). These elements are usually divided into two subgroups: light rare earth elements, among them: cerium (Ce), lanthanum (La), praseodymium (Pr), neodymium (Nd), samarium (Sm) and Europium (Eu),
and the group of yttrium or "heavy rare earth elements", consisting of yttrium (Y), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), Erbium (Er), Thulium (Tm), Ytterbium (Yb) and Lutetium (Lu). Rare earth elements are called "rare" due to some historical reasons. Previously, it was thought that only a few rare minerals could be found and isolated. Also, because they behaved as a single chemical unit and their separation was a difficult and expensive task. (Al-Nafai, 2015) [2]

Their unique physical and chemical properties have made them essential in technological equipment (for example, magnets, catalysts, batteries, etc.). A potential risk of REE supply, environmental concerns and economic benefits promotes research on the processing of secondary minerals, such as mine tailings or electronic waste, in the US. Although significant research has been conducted on resource extraction and a variety of technologies have been developed or proposed, most of them are still under research or only suitable for some specific secondary resources. The efficiency and selectivity of the extraction of REE, together with the cost and engineering during the design of the process, require a substantial optimization before its subsequent commercialization. At the same time, the understanding of the interactions between rare earth minerals in a low concentration and leaching, as well as the behavior of the selective leaching of different phases of rare earths in remains of magnets and other technological waste, is still not enough. In order to optimize the possibilities of extracting REE from secondary resources, it is necessary to continue improving the predominant leaching technologies currently in use in the production of REE (Peelman, et al., 2012) [10] (Gupta & Krishnamurthy, 2005) [6]

Fig. 1: Main applications of REE (Survey, 2011) [11]
Mineral processing of REE, usually involves two types of operations: physical and chemical processing. Physical processing separates the REE minerals from the gangue, in order to produce a concentrate of those species. Chemical processing includes extraction and separation processes. Thus, the concentrate is converted into an REE compound. The latter is a final or intermediate product for the subsequent production of individual REEs or other compounds. The REE extraction process uses one or more reagents to break down the minerals and recover them in solution. The REE separation process uses solvent extraction, ion exchange or chemical precipitation to produce mixed REE oxides.

The varieties of REE extraction methods use many types of reagents. These are mainly inorganic acids, alkalis, electrolytes and chlorine gas. Commonly used acids include sulfuric acid (H\textsubscript{2}SO\textsubscript{4}), hydrochloric acid (HCl) and nitric acid (HNO\textsubscript{3}). Sodium hydroxide (NaOH) and sodium carbonate (Na\textsubscript{2}CO\textsubscript{3}) are the most common alkalis. Among the most common electrolytes are ammonium sulfate ((NH\textsubscript{4})\textsubscript{2}SO\textsubscript{4}), ammonium chloride (NH\textsubscript{4}Cl), and sodium chloride (NaCl). HCl, HNO\textsubscript{3} and H\textsubscript{2}SO\textsubscript{4} are commonly used to extract REE from silicate ore, such as gadolinite, eudialite and alanite. The alkalis and H\textsubscript{2}SO\textsubscript{4} are mainly used to leach REE from phosphate minerals, such as monazite and xenotimium. Carbonatite minerals, such as bastnasite, are treated using H\textsubscript{2}SO\textsubscript{4} or alkalis. HNO\textsubscript{3} is mainly used to leach eudialite and apatite. The extraction of REE from ionic absorption clay deposits uses electrolytic solutions. Chlorine gas (Cl\textsubscript{2}) is exclusively used in the REE chlorination process, which can treat most minerals.

Acid roasting is an important process of REE ores decomposition. It is classified in low roasting temperature (<300ºC) or high roasting temperature (> 300ºC). The low temperature process, previously used in the 1970s to treat low-grade REE concentrates, requires long and complex processes to remove impurities from the leaching solution. To inhibit the generation of soluble impurities, the high-temperature acid roasting process was developed in the 1980s to treat high-grade REE minerals.

Currently, most of the REE extraction operations using acid roasting prefer the high temperature process. This process is relatively simple, but generates dangerous exhaust gases such as hydrogen fluoride (HF), sulfur dioxide (SO\textsubscript{2}), sulfur trioxide (SO\textsubscript{3}) and silicon tetrafluoride (SiF\textsubscript{4}). Normally, a water purification is used to capture most of the exhaust gases. An acid mixture containing HF, H\textsubscript{2}SO\textsubscript{4} and H\textsubscript{2}SiF\textsubscript{6} can be recovered in the initial waste. A second purification, using a diluted solution of sodium carbonate, allows to purify the exhaust gases before being released. A general toasting and leaching process for bastnasite and monazite is shown in Figure 2 (left). This consists of milling, mixing with H\textsubscript{2}SO\textsubscript{4}, roasting, leaching and solid-liquid separation. The REE mineral is usually reduced to less than 100 mm (150 mesh) before being mixed with concentrated acid. A rotary kiln is used for acid roasting. A filter or thickener can be used to separate the leaching solution from the waste. The higher the acid/mineral ratio, the more REE and thorium dissolves. Lower ratios allow a more selective dissolution of the ore. At
temperatures above 300°C, the rate of decomposition rises, but the leaching of thorium is also reduced due to the formation of the insoluble compound ThP$_2$O$_7$. This process removes thorium to the waste at some rare earth processing plants. The REE, thorium and uranium are converted into soluble sulfates during acid roasting with H$_2$SO$_4$. The main reactions include:

$$2\text{RECO}_3\text{F} + 3\text{H}_2\text{SO}_4 = \text{RE}_2(\text{SO}_4)_3 + 2\text{HF} + 2\text{H}_2\text{O}$$  \hspace{1cm} (1)

$$2\text{REPO}_4 + 3\text{H}_2\text{SO}_4 = \text{RE}_2(\text{SO}_4)_3 + 2\text{H}_3\text{PO}_4$$  \hspace{1cm} (2)

$$\text{ThO}_2 + 2\text{H}_2\text{SO}_4 = \text{Th}(\text{SO}_4)_2 + 2\text{H}_2\text{O}$$  \hspace{1cm} (3)

$$2\text{U}_3\text{O}_8 + 6\text{O}_2 + 6\text{H}_2\text{SO}_4 = 6\text{UO}_2\text{SO}_4 + 6\text{H}_2\text{O}$$  \hspace{1cm} (4)

$$\text{RE}_2\text{O}_3 + 3\text{H}_2\text{SO}_4 = \text{RE}_2(\text{SO}_4)_3 + 3\text{H}_2\text{O}$$  \hspace{1cm} (5)

Most side reactions include:

$$\text{CaF}_2 + \text{H}_2\text{SO}_4 = \text{CaSO}_4 + 2\text{HF}$$  \hspace{1cm} (6)

$$\text{Fe}_2\text{O}_3 + 3\text{H}_2\text{SO}_4 = \text{Fe}_2(\text{SO}_4)_3 + 3\text{H}_2\text{O}$$  \hspace{1cm} (7)

$$\text{SiO}_2 + 2\text{H}_2\text{SO}_4 = \text{H}_2\text{SiO}_3 + \text{H}_2\text{O} + 2\text{SO}_3$$  \hspace{1cm} (8)

$$4\text{HF} + \text{SiO}_2 = \text{SiF}_4 + 2\text{H}_2\text{O}$$  \hspace{1cm} (9)

$$\text{H}_2\text{SiO}_3 = \text{SiO}_2 + \text{H}_2\text{O}$$  \hspace{1cm} (10)

$$2\text{H}_3\text{PO}_4 = \text{H}_4\text{P}_2\text{O}_7 + \text{H}_2\text{O}$$  \hspace{1cm} (11)

$$\text{Th}(\text{SO}_4)_2 + \text{H}_4\text{P}_2\text{O}_7 = \text{ThP}_2\text{O}_7 + 2\text{H}_2\text{SO}_4$$  \hspace{1cm} (12)

At the Saskatchewan Research Council, numerous acid roasting tests have been performed. Normally, the acid/concentrate mass ratio is between 1/1 and 2/1, depending on the degree of REE of the mineral and the gangue associated with it. When the REE law is high, the consumption of acid is relatively low. The consumption of acid will increase if the content of carbonate, fluoride and/or iron minerals is high. Similarly, acid consumption is affected by roasting time and temperature. A higher roasting temperature and shorter times allow reducing the consumption of acid, maintaining the same decomposition efficiency. However, a higher roasting temperature can decrease the recovery of REE by formation of soluble sulfates. The solubility of REE sulfates will also decrease with an increase in the leaching temperature. Therefore, aqueous leaching takes place at room temperature. In addition to the minerals of bastnasite and monazite, acid roasting and aqueous leaching can also be used to process xenotimium, aeschinite, and REE silicates.
Acid leaching by dilute HCl dissolves calcium carbonate and concentrated HCl to decompose bastnasite. The main reaction of the latter type is shown by equation (13):

\[ 3RECO_3F + 6HCl = RE_2Cl_3 + REF_3 + 3H_2O + 3CO_2 \]  

REE fluorides remain in the waste after the decomposition of HCl. After an initial solid-liquid separation, NaOH is used to convert them into REE hydroxides, according to equation (14)

\[ REF_3 + 3NaOH = REOH + 3NaF \]  

After a second solid-liquid separation, the hydroxides of REE are dissolved by the excess of HCl. This process is shown in Figure 3 (right). Concentrated HCl breaks down alanite, cerite and gadolinite. The reaction between the HCl solution and gadolinite is expressed in equation (15)

\[ RE_2FeBe_2(SiO_4)_2O_2 + 12HCl = 2RECl_3 + FeCl_2 + 2BeCl_2 + 2SiO_2 + 6H_2O \]  

Hydrochloric acid can also leach oxides, carbonates and other rare earth intermediates in subsequent stages.

![Diagram](image)

**Figure 2:** (Left.) General process of roasting and acid leaching for bastnasite and monazite, using H_2SO_4. (Right) General leaching process using HCl for bastnasite.
The agitation leaching and its variables are shown schematically in figure 3:

Figure 3: (Left) General structure for agitation leaching reactor (Xingyang, 2015) [12], (Right) Main operating parameters of the continuously stirred leach reactor (CSTR) (Mathworks, 2017) [8]

Agitation leaching is an important extraction process in REE hydrometallurgy. Many processes use leaching methods to extract REE, such as leaching with acids, alkalis and/or salt reagents. These methods typically use agitation tanks, for example, to leach REE by the process of acid roasting of bastnasite with sulfuric acid. The agitation tank has many advantages as leaching equipment (Liu, et al., 2015) [7]: (a) It provides beneficial hydrodynamic conditions for mass transfer; (b) It can accelerate the chemical reaction effectively; (c) Reduce air pollution to protect the environment, because the stirred tank is an airtight container. The operation study of a reactor involves the coupling of several mathematical models, such as the turbulence model for the leaching solution, free body balances for the movement of suspended particles, and finally the occurrence of a chemical reaction for the release of the species of interest from the mineral. Due to the large amount of information that is necessary to consider for the recovery of REE in any leaching process, a predictive model based on the neural network will be developed, which will allow establishing a correlation between the main input data for the leaching and the recovery of the species of interest.


Neural networks are composed of simple elements that work in parallel. The elements are inspired by the biological nervous systems. As in nature, the network is determined to a large extent by the connections between elements. It is possible to train a neural network to perform a particular function by adjusting the values of connections (weights) between elements. The common neural networks are adjusted, or trained, so that a particular input leads to a specific target output. Such a situation is shown in Figure 4. The network is adjusted, based on a comparison of the output and the target, until the network output matches the destination.
Typically many of these input/target pairs are used, a concept known as supervised learning, to form a network.

![General scheme of operation of a neural network](image1)

**Figure 4:** General scheme of operation of a neural network (Barber, 2007) [3]

The neuron, the basic unit of neural networks, and its genetic structure are shown in Figure 5:

![Main neuron components](image2)

**Figure 5:** Main neuron components (Barber, 2007) [3]

The perceptron consisting of weights, the sum processor, an activation function and an adjustable threshold processor, called bias. The entries ($x^1$, $x^2$, $x^3$..$x^m$) and the connection weights ($w^1$, $w^2$, $w^3$..$w^m$) in figure 5 are typically real, positive and negative values. If the characteristic of some $x^i$ tends to cause increases in the perceptron, the $w^i$ weight will be positive; If the characteristic $x^i$ inhibits the perceptron, the weight $w^i$ will be negative. Next, the basic concepts to apply in the tool for the construction of neural networks, belonging to MATLAB, are shown.

**Training (train).** Training a network consists of repeatedly using inputs to a perceptron and changing the weights and biases, according to the error. The perceptron will eventually find weight and bias values that solve the problem. Each displacement through all training and destination input vectors is called a step. As the training occurs, the new weight ($w^i$) and bias ($b$) values should be checked by calculating the network output for each input vector, to see if all the objectives are met.

**Learning rules.** It is a procedure to modify the weights and biases of a network. It can also refer to as a training algorithm. The rules of learning are divided into two
broad categories: supervised and unsupervised learning. In supervised learning, the learning rule is provided with a set of examples (the initial data set) of the appropriate network behavior, where the initial data is the input to the network, and the goal is the correct output (destination) correspondent. As the inputs are applied to the network, the outputs are compared with the targets. The learning rule adjust the weights and biases of the network to move the outputs of the network, closer to the targets. The learning rule used falls into this category of supervised learning.

**Gradient descent with momentum (traingdm).** The least squared error (LMS) algorithm is an example of supervised training, in which the learning rule is provided by a set of examples of desired network behavior: \((p_1,t_1), (p_2,t_2), \ldots (p_Q,t_Q)\). Here, \(p_Q\) is a net input, and \(t_Q\) it is the corresponding destination output (target). As each input is applied to the network, the network output (output) is compared to the target. The error is the difference between the destination output and the network output. To minimize the average of the sum of these errors, the following function is used:

\[
mse = \frac{1}{Q} \sum_{k=1}^{Q} e(k)^2 = \frac{1}{Q} \sum_{k=1}^{Q} (t(k) - p(k))^2
\]

Where \(mse\) is the mean square error.

The LMS algorithm adjusts the weights and biases of the linear network, in order to minimize the mean square error. This index is a quadratic function. Thus, it will have a minimum, depending on the characteristics of the input vectors. Specifically, the characteristics of the input vectors determine whether or not a single solution exists.

**The performance of a trained network is measured in the sets of linear regression graphs of training, validation and testing, by means of linear regression analysis between the response of the network (network_outputs) and the targets.**

For a perfect fit (outputs exactly equal to the targets), the slope would be 1, and the intersection would be 0. In the example of figure 6, the numbers are very close. In addition, graphics provide correlation values (R) between network responses and objectives.

Figure 6: Comparative linear adjustment between the proposed network and the targets to be obtained (Milivojevic, et al., 2012) [9] (Duric, et al., 2009) [5]
4 Results and discussions

The input data will be the same for the 3 proposed networks, while the output data will be the recovery of La, Ce and Fe for the leaching experiments.

- The type of network used will be advance and retro propagation (feed-forward backpropagation)
- The training function for the network will be of the Levenberg-Marquardt type (trainlm)
- The performance of the network will be measured through the average square error (MSE) function
- The properties of the first layer will be taken with a number of neurons of 10, and the transfer function for these will be TANSIN, since it provides the least mean square error in the validation tests.
- Finally, the linear regression curves that compare the results obtained from the use of the network (output) with the real output data (target) will also be shown.

All developed networks will have the following structure:

![Custom Neural Network (view)](image)

Figure 7: Structure of the developed neural network

In figure 7, it is possible to see that the structure of the neural network used is composed of 10 neurons with the transfer function "TANSIG", while in the output layer a single neuron with the transfer function, "PURELIN" will be used. The criterion of choice for this configuration was the value obtained by the performance function chosen, in this case, MSE (Mean Square Error), since the addition of more neurons does not show significant improvements. The use of the transfer functions "LOGSIG" and "PURELIN" for the hidden layer, yielded MSE values higher than 10, regardless of the number of neurons used, so their use was discarded.

The neural networks obtained will predict the recovery of the species of interest for this work: La, Ce and Fe. The first 2 species are important due to their high economic value, while iron acts as a pollutant that has a behavior in solution similar to the previous ones, and decreases the global recovery of rare earths in leaching.

Table 1 shows the results obtained for the leaching experiences developed:
TABLE I: Parameters used and results obtained in each leaching test

<table>
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<tr>
<th>Place of origin</th>
<th>Initial acid/ Kg Metric ton</th>
<th>$T/ ^{\circ}C$</th>
<th>S/L rate</th>
<th>Initial mass/g</th>
<th>$La_{law}/%$</th>
<th>$Ce_{law}/%$</th>
<th>$Fe_{law}/%$</th>
<th>$La_{recovery}/%$</th>
<th>$Ce_{recovery}/%$</th>
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*The numbers 1, 2 and 3 represent the provinces of Sierra Aspera, Veracruz and Cerro Carmen, III region of Chile, respectively.

The criterion adopted to obtain the definitive neural network was the minimum performance value for the 3 stages of the development of the network: Training, validation and tests. Figure 8 shows that, although the error decreases continuously in the training period, it tends to increase for the data analyzed in the validation and testing processes.

Because the MSE value increases over the course of cycles, each test is considered a failed test, so the program stops after the maximum number of errors allowed for each sample.

Figure 8 shows the characteristics of the neural network obtained for the recovery of Lanthanum from the leaching tests carried out.
The linear regression graphs allow comparing the relationship between the results predicted by the network and the real values of the recovery of La. If these values are correlated with each other, that is, the results obtained by the network are similar to the real ones, the correlation parameter that relates Output (network output) to Target (actual value of the parameter to be studied) yields values close to 1. Figure 9 (left) shows that in the training, validation and test of the results, the linear correlation values "R" are higher than 0.99, which indicates that the network is an effective predictor for recovery of Lanthanum in the leaching tests. Finally, figure 9 (right) allows us to conclude that in most of the results obtained by the network, the values of the recoveries show a similar trend.
The biggest differences in some of the data are due to the inability of the network to progress with validation.

**Neural network to determine the recovery of Cerium:**

![Neural Network Diagram](image)

Figure 10: Results obtained for the leaching of Cerium in leaching.

![Validation Graph](image)

Figure 11: Validation of the results obtained by the neural network with respect to the real values obtained, for the recovery of Cerium.

As in the previous case, the neural network to determine the recovery of Cerium was developed using the same input data, but replacing the output by the respective recoveries of this species of interest. The graphs obtained from figure 10 show that the trials were stopped due to the low correlation values obtained in the data corresponding to the validation section, so that only 4 cycles were developed, even though the performance function reached was similar to that obtained in the previous case.
Neural network to determine the recovery of Iron:

Figure 12: Results obtained for the recovery of Iron in leaching

As iron is associated with the minerals bearing Lanthanum and Cerium, it is not possible to carry out leaching with selective type parameters to recover only those species of interest, as shown by the global recovery charts in table 1, with respect to the value of the networks. It is for this reason that, in order to recover La and Ce from these minerals, the dissolution of this impurity is strictly necessary. Therefore, in order to concentrate Lanthanum and Cerium, a purification step is necessary to obtain REE concentrates.

Figure 13: Validation of the results obtained by the neural network with respect to the real values obtained.
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Conclusions

The main conclusions of this work are the following:

1. The use of the neural network technique allows establishing correlations between data series, as well as quantifying variables of all kinds, such as the origin of the mineral, a goal that is difficult to achieve through other techniques.

2. Based on the construction of networks, it is possible to establish correlations to obtain a series of different results, as in this case, obtain 3 types of different recoveries from the same database.

3. Despite being forced to stop the process at few epochs, the networks were able to accurately obtain results similar to the real ones.

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