

Prediction of yttrium, lanthanum, cerium, and neodymium leaching recovery from apatite concentrate using artificial neural networks

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(Received 2007-11-19)

Abstract: The assay and recovery of rare earth elements (REEs) in the leaching process is being determined using expensive analytical methods: inductively coupled plasma atomic emission spectroscopy (ICP-AES) and inductively coupled plasma mass spectroscopy (ICP-MS). A neural network model to predict the effects of operational variables on the lanthanum, cerium, yttrium, and neodymium recovery in the leaching of apatite concentrate is presented in this article. The effects of leaching time (10 to 40 min), pulp densities (30% to 50%), acid concentrations (20% to 60%), and agitation rates (100 to 200 r/min), were investigated and optimized on the recovery of REEs in the laboratory at a leaching temperature of 60°C. The obtained data in the laboratory optimization process were used for training and testing the neural network. The feed-forward artificial neural network with a 4-5-5-1 arrangement was capable of estimating the leaching recovery of REEs. The neural network predicted values were in good agreement with the experimental results. The correlations of $R=1$ in training stages, and $R=0.971$, 0.952 , 0.985 , and 0.98 in testing stages were a result of Ce, Nd, La, and Y recovery prediction respectively, and these values were usually acceptable. It was shown that the proposed neural network model accurately reproduced all the effects of the operation variables, and could be used in the simulation of a leaching plant for REEs.

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Key words: apatite; neural networks; rare earth elements; leaching; recovery

1. Introduction

Over the past 10 years, artificial neural networks (ANNs) and particularly feed-forward artificial neural networks (FANNs) have been extensively studied to present process models, and their use in industry has been rapidly growing [1]. The use of such networks can now be found for a number of predictions, such as modeling the greenhouse effect [2], simulation of N_2O emissions from a temperate grassland ecosystem [3], bioleaching of metals [4], coal microbial desulfurization [5], assessment of flotation experiments [6-7], and modeling of rare earth solvent extraction [8].

Rare earth elements (REEs) are the 15 lanthanides with atomic numbers 57 to 71, which are classified in two groups: the light or cerium subgroup comprising of the first seven elements with atomic numbers 57 to 63 and the heavy or yttrium subgroup, comprising of the elements with atomic numbers 64 to 71 as well as yttrium with atomic number 39 [9]. The diverse nuclear, metallurgical, chemical, catalytic, electrical, magnetic, and optical properties of the REE have led

to an ever increasing variety of applications. These uses range from mundane (lighter flints, glass polishing) to high-tech (phosphors, lasers, magnets, batteries, magnetic refrigeration) to futuristic (high-temperature superconductivity, safe storage, and transport of hydrogen for post-hydrocarbon economy) [10].

REEs are never found as a free metal in the earth's crust and their naturally occurring minerals consist of mixtures of various REEs and nonmetals [9]. REEs are found mainly in primary deposits associated with igneous intrusions and associated veins, dikes, and pegmatites, and secondary deposits of beach, dune, alluvial placers, and residual deposits. REEs bearing deposit minerals may occur as the main valuable component in well individualized deposits as well as potential byproducts derived from other minerals, such as apatite. Bastnasite, monazite, and xenotime are the three most economically significant minerals that contain essential or significant REEs. Other commercial sources of REEs are apatite, REE bearing clays, allanite, zircon, euxenite, and loparite [11].

The Chadormalu iron deposit is located in Yazd Province in the center of Iran, and the ore is being produced in an open pit operation. The feed of the plant consists of different minerals such as magnetite, hematite, martite, quartz, apatite, gypsum, biotite, and anhydrite. The plant concentrates are iron ores, hematite, and magnetite, and apatite is a byproduct [12].

The aims of the present work are the following.

(a) The assessment of lanthanum (La), cerium (Ce), yttrium (Y), and neodymium (Nd) leaching recoveries from Chadormalu apatite concentrate and possible variations with the change in acidity, solid-to-liquid ratio, agitation rate, and leaching time using the experimental data obtained at the laboratory level.

(b) To assess if it is possible, with the help of the experimental data resulting from the laboratory level, to predict the recoveries of Y, La, Ce, and Nd elements from the apatite concentrate, by means of neural networks.

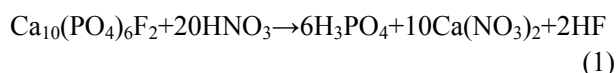
To the authors' knowledge, this is the first time that ANNs have been used to predict individual rare earth element's recovery in the leaching process. Because of the unequal prices of different rare earth elements, the prediction of the recovery of significant forms of REEs (Y, La, Ce, and Nd) from Chadormalu apatite concentrate could be helpful from the economical assessment point of view.

2. Background

2.1. Leaching of rare earth elements

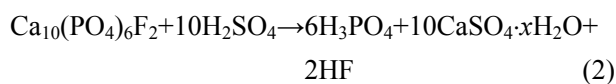
The recovery of a mixed rare earth oxide from apatite is based on the following.

(a) The total dissolution of the ore with nitric acid [13-21] as per the following reaction



When apatite is leached with HNO_3 , most of the rare earths substituted in the apatite lattice for calcium ions are dissolved.

(b) By the proposed treatment of the phosphogypsum byproduct from the conventional sulphuric acid route [22-23, 14-18] and the recovery of rare earth oxides from phosphoric acid sludge [24-25] as follows:



Some efforts were made to use hydrochloric acid as

a leaching agent for the recovery of REEs [14], but it does not operate on the industrial scale.

In the leaching process just mentioned, most of the REEs contribute to the gypsum byproduct. The nitric acid has been used for the recovery of REEs from gypsum.

The recovery of REEs from the phosphate fertilizer industry includes some disadvantages as follows [26]:

- Generation of a large amount of radioactive gypsum, which represents storage and environmental problems;
- Extensive material handling problems.

Although using nitric acid as a leaching agent is more expensive than using sulfuric acid, it solves the disposal problem because of gypsum [26]. According to these considerations, nitric acid has been used as a leaching agent to extract REEs from the apatite concentrate. In the pregnant liquors, the assay of Y, La, Ce, and Nd have been detected by inductively coupled plasma atomic emission spectroscopy (ICP-AES).

2.2. MATLAB software

MATLAB is a matrix based mathematical software package developed by Math Works Inc. with excellent computation and visualization abilities [27]. MATLAB operates as a powerful programming language and also has an interactive computational environment. The neural network toolbox is used to develop various neural networks and allows the user to quantitatively and graphically monitor the network training process and analyze the results [28].

3. Materials and methods

3.1. Sampling from apatite concentrate

In Chadormalu Concentrator Plant the gyratory crusher and auto genius (AG) mill comprise of the comminution system, the low intensity magnetic separator (LIMS) unit to produce magnetite concentrate and its tailings supply feed for high gradient magnetic separator (HGMS) and reverse flotation to produce the hematite concentrate. The apatite floatation unit produces apatite concentrate from the HGMS circuit tailings and the tailings of LIMS, HGMS, and apatite floatation circuits comprise of the total tail of the plant (Fig. 1).

The apatite concentrate sample was prepared in different working shifts of the plant, and representative samples were prepared by means of quartering and conning methods. The distribution of different forms of REEs in the sample is shown in Fig. 2; from this

figure it is evident that the most significant forms of REEs are Y, La, Ce, and Nd, which are the subject of the current study.

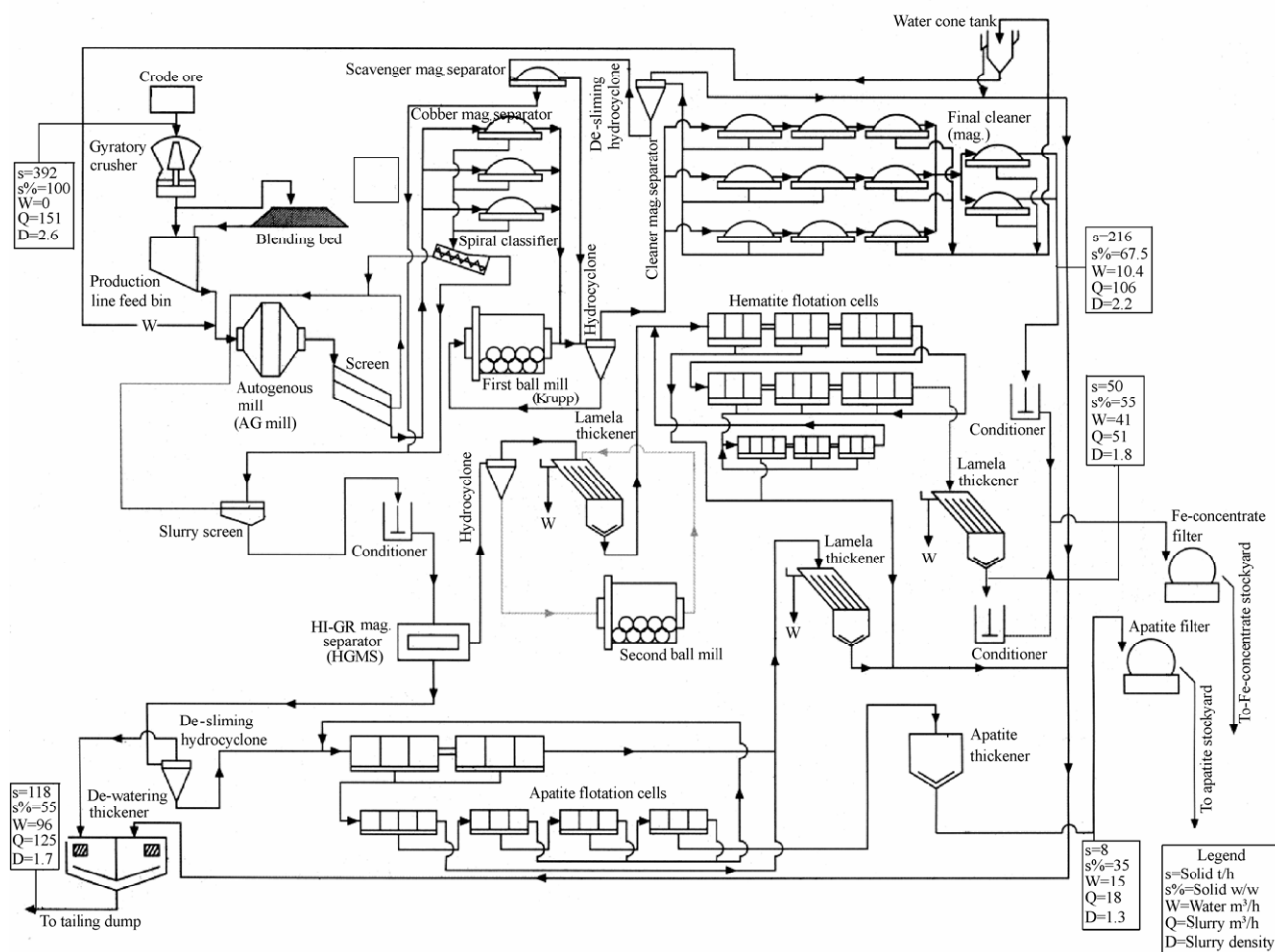


Fig. 1. Process flow diagram of Chadormalu Plant.

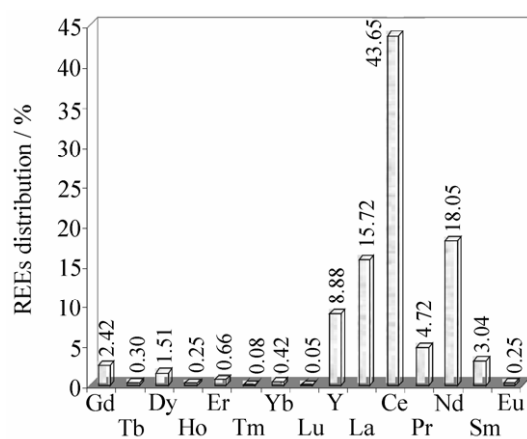


Fig. 2. Distribution of different REEs in apatite concentrate.

The representative sample with the particle size 80% less than 50 μm (d_{80} :50 μm) was used for leaching studies without further size reduction.

3.2. Leaching studies

The leaching experiments were carried out using nitric acid with concentrations of 20vol%, 35vol%,

50vol%, and 60vol%; reaction times of 10, 20, 30, and 40 min; particle size (d_{80}) of 50 μm ; solid to liquid ratios of 30wt%, 40wt%, and 50wt%; agitation rates of 100, 150, and 200 r/min; and the temperature was fixed at 60°C. Following the reaction, the reactor was cooled and filtered to recover the pregnant liquor. The filtrate was washed with diluted nitric acid and hot water thrice, and the pregnant liquor was analyzed for REEs by ICP-AES. The results are shown in Table 1.

In the first stage of the experiment, nine leaching tests were conducted, three different acidities (20vol%, 35vol%, and 50vol%) were used for three solid-to-liquid ratios (30wt%, 40wt%, and 50wt%), and temperature, leaching time, agitation rate, and particle size were 60°C, 10 min, 150 r/min, and <50 μm respectively (test No.1 to 9 in Table 1). The temperature did not influence the leaching efficiency of rare earths, but a higher temperature was advantageous for the volatilization of S and F [17]. As a result, from these stages in the experiment, the recoveries of REEs decrease with an increase in solid-to-liquid ratio and increases with an increase in acidity.

To assess whether or not the recoveries of REEs are enhanced by increasing the acidity to more than 50vol%, nitric acid of 60vol% was used in the second stage of the experiment. To investigate the effect of leaching time and agitation rate on the recoveries of REEs, eight leaching tests were performed, in which four different leaching time (10, 20, 30, and 40 min) were used for two different agitation rates (100, 200 r/min), and the solid to liquid ratio and acidity were kept at 30wt% and 60vol%, respectively (test No.10 to 17 in Table 1). The results illustrate that once the agitation rate was kept at 100 r/min with an increase in leaching time from 10 to 20 min, the recoveries of La, Ce, Nd, and Y increased from 58%, 48%, 55%, and

59% to 67%, 56%, 68%, and 67%, respectively. After the mentioned time, the recoveries of REEs were not changed considerably. Also at the agitation rate of 200 r/min, the recoveries of REEs increased with an increase in leaching time from 10 to 30 min, and did not change substantially after 30 min. Subsequently, the conditions of acidity 60vol%, solid-to-liquid ratio 30wt%, leaching time 30 min, agitation rate 200 r/min, and temperature 60°C were selected as the optimum operational conditions for the extraction of REEs. Under these conditions the recoveries of La, Ce, Nd, and Y were achieved at 74%, 59%, 72%, and 73%, respectively.

Table 1. Results of the experiments for REE recovery under different operational conditions

Test No.	Time / min	(Solid/Liquid) / wt%	Acid concentra- tion / %	Agitation rate / (r·min ⁻¹)	Lanthanum recovery / %	Cerium re- covery / %	Neodymium recovery / %	Yttrium recovery / %
1	10	30	20	150	0.4	7.5	8.6	0.2
2	10	40	20	150	1.3	1.5	2.3	1.2
3	10	50	20	150	0.0	0.5	0.5	0.4
4	10	30	35	150	13.2	21.8	23.0	18.7
5	10	40	35	150	13.2	11.4	17.3	17.5
6	10	50	35	150	26.0	5.0	0.3	21.1
7	10	30	50	150	26.4	27.6	40.3	50.3
8	10	40	50	150	19.8	19.4	28.8	28.1
9	10	50	50	150	4.0	8.4	6.3	2.3
10	10	30	60	100	58.0	48.0	55.0	59.0
11	20	30	60	100	67.0	56.0	68.0	67.0
12	30	30	60	100	67.4	57.0	70.0	67.9
13	40	30	60	100	69.7	58.8	70.7	68.4
14	10	30	60	200	60.0	49.0	58.0	58.0
15	20	30	60	200	67.0	52.0	66.0	64.0
16	30	30	60	200	74.0	59.0	72.0	73.0
17	40	30	60	200	76.9	61.0	72.9	74.3

3.3. Artificial neural networks

Artificial neural networks are powerful tools that can be used to model and investigate various highly complex and nonlinear phenomena. The artificially intelligent neural network (NN) approach is an alternative statistical prediction method inspired by studies on the human nerve and brain system. Intelligence is embedded into a neural network by teaching and training them by using a series of examples and patterns. The information acquired through the training is retained and represented by a set of connection weights within the neural network structures. The nature of neural network memory leads to efficient responses (*i.e.*, giving answers) when presented with previously unseen inputs. The advantage of the neural network approach over the conventional approach is that the problem is directly modeled and has a toler-

ance for even noisy data. Unlike multiple linear or nonlinear regression techniques, which require a pre-defined empirical model, neural networks can identify and learn the correlative patterns among the input and corresponding output values once a training data set is provided [29-30].

Neural networks are well-suited for many applications such as control problems, diagnostics, mapping, stock market prediction, optimization, sensor data fusion, and pattern recognition [31]. An ANN is composed of several elementary, interconnected processing elements known as perceptrons that work together in a parallel way. These networks are capable of reproducing a process from training examples (neuro-computing approach) rather than forming a coded algorithm, which simulates the process on the basis of a mathematical model (programmed computing ap-

proach) [29]. After the training process of the existing data set, neural networks have the capability of predicting values within and outside the range of the data set (*i.e.*, interpolation and extrapolation). Perceptron can be viewed as a single-output black box computing element with multiple inputs. The output is obtained by processing the weighted sum of the inputs with a transfer function called the activation function. For the purpose of the present application, a multilayer perceptrons neural network (MLP NN) structure has been trained with a back-propagation (BP) training procedure. The training continues until the error between the prediction and the actual data is minimized [31].

For developing a nonlinear ANN model of a system, feed-forward architecture namely MLP is most commonly used. This network usually consists of a hierarchical structure of three layers described as input, hidden, and output layers, comprising of I , J , and K , and L number of the processing nodes, respectively (Fig. 3).

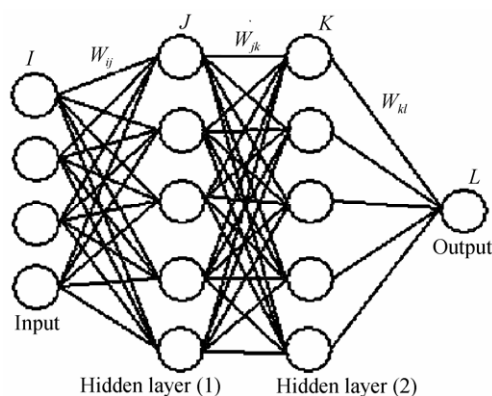


Fig. 3. Architecture of the feed-forward neural networks.

Table 2. Statistical analysis of the recovery value prediction and generalization performance of ANN

Element	Performance of ANN models in training step		Performance of ANN models in testing step
	Correlation coefficient	MSE*	Correlation coefficient
Cerium	1.00	2.36×10^{-30}	0.971
Neodymium	1.00	1.82×10^{-27}	0.952
Lanthanum	1.00	1.25×10^{-25}	0.985
Yttrium	1.00	3.78×10^{-31}	0.980

Note: MSE—the mean square error.

Table 3. Preprocessing parameters for ANN

Variable	Mean	Stand deviation
Log (Time)	2.67	0.55
Acidity / %	46.76	15.81
Rotation rate / (r·min ⁻¹)	150.00	35.36
Solid to liquid / %	35.29	7.99

A total of 17 sets of data were used in the present study, from which 11 sets were used for training and six sets for testing the network. The training process

In this study, neural networks were used to predict the relationships between operational variables such as leaching time, agitation rate, acidity, and pulp density, as the input sets, with Ce, Y, Nd, and La recoveries, as the output sets (Table 2). Time in the logarithmic basis had a better correlation with rare earth element recovery than the normal form; therefore time was used in the logarithmic basis and the other variables were used in the normal form (Table 3). The MLP architecture ANN model has been developed by considering two hidden layers with five neurons, one input layer and one output layer with four neurons and one neuron respectively, and with training using the back-propagation algorithm, for Ce, Y, Nd, and La recovery predictions (Fig. 3).

4. Results and discussion

Neural network training can be made more efficient by certain pre-processing steps. In the present work, all the inputs (before feeding to the network) and the output data in the training phase preprocess the network training set by normalizing the inputs and targets so that they have means of zero and standard deviations of 1:

$$Np = (Ap - \text{mean}Ap) / \text{std}Ap \quad (3)$$

where Ap is the actual parameter, $\text{mean}Ap$ is the mean of actual parameters, $\text{std}Ap$ is the standard deviation of the actual parameter, and Np is the normalized parameter (input) [32]. The mean and standard deviation for preprocessing the input and output variables are shown in Table 3.

was stopped after 11, 54, 32, and 15 epochs for the prediction of Ce, Nd, La, and Y recoveries, respectively. The correlation coefficient (R) value for the training set of all elements was 1 (Figs. 4-7). The performance function that was used was the mean square error (MSE). The average square error between the network predicted outputs and the target outputs are shown in Table 3.

The R values for the testing sets were 0.971, 0.952, 0.985, and 0.98 for the recoveries of Ce, Nd, La, and

Y, respectively (Figs. 8-11).

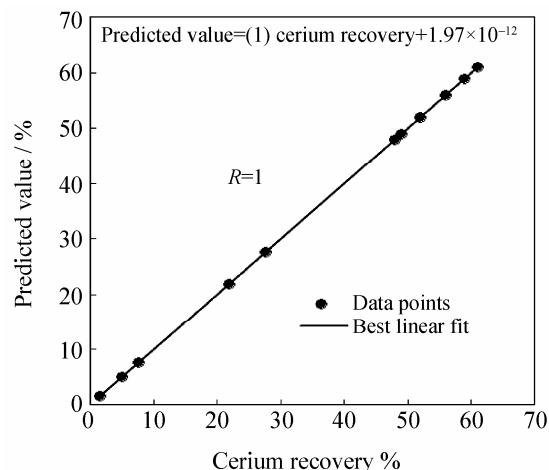


Fig. 4. Recovery value predicted by neural network in the training process *versus* those actual measured in laboratory for cerium.

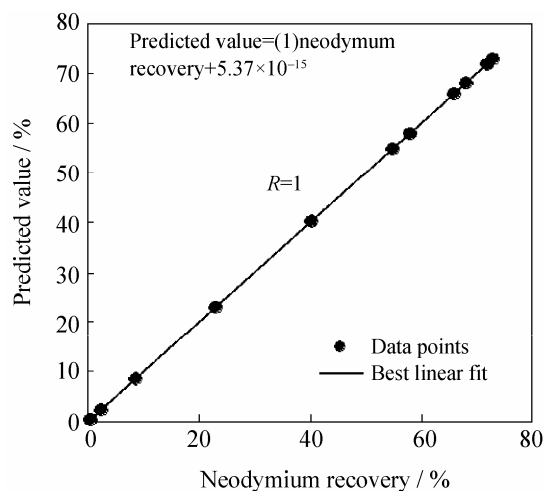


Fig. 5. Recovery value predicted by neural network in the training process *versus* those actual measured in laboratory for neodymium.

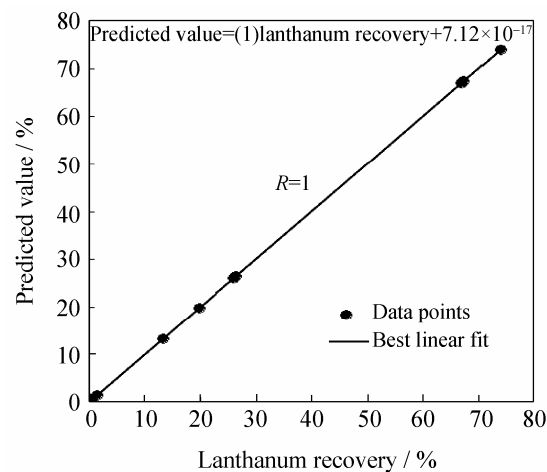


Fig. 6. Recovery value predicted by neural network in the training process *versus* those actual measured in laboratory for lanthanum.

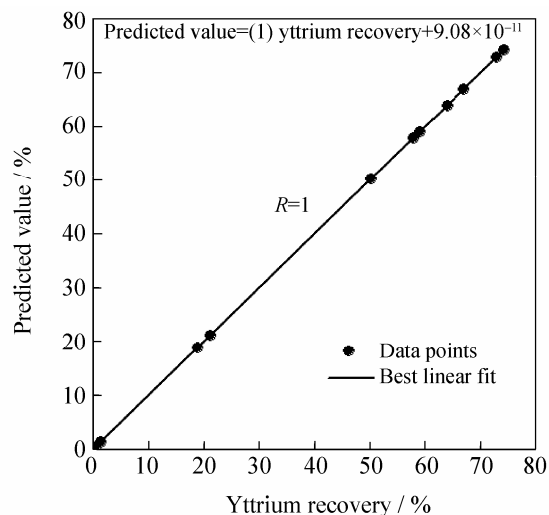


Fig. 7. Recovery value predicted by neural network in the training process *versus* those actual measured in laboratory for yttrium.

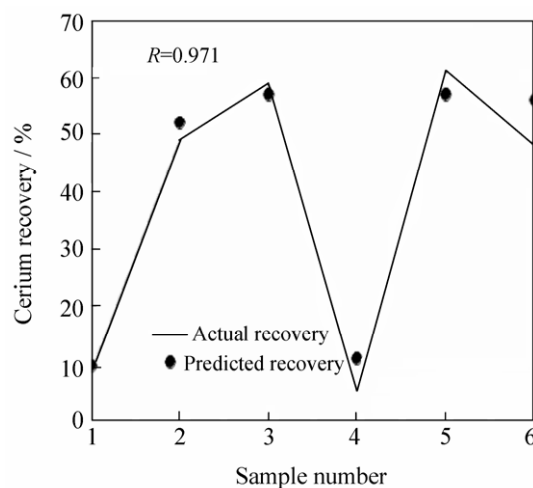


Fig. 8. Comparison of actual recovery with those estimated by ANN in the test process for cerium.

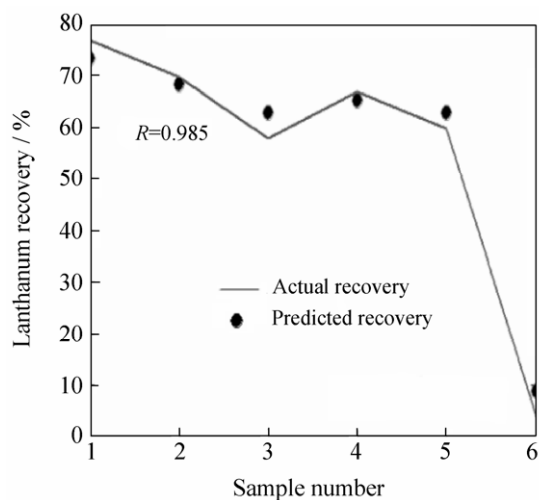


Fig. 9. Comparison of actual recovery with those estimated by ANN in the test process for lanthanum.

In this work it was observed that the recoveries of individual REEs could be predicted satisfactorily by

using the ANN model.

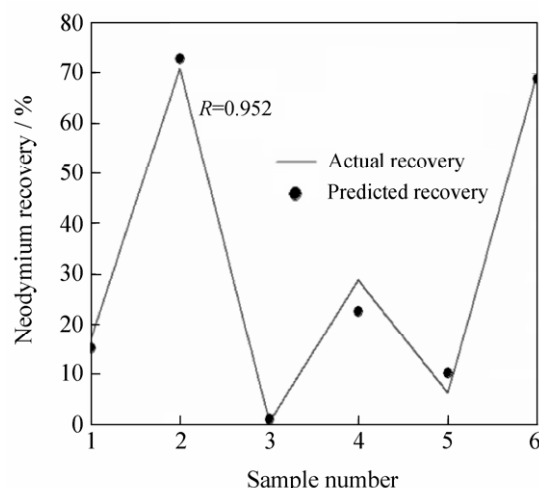


Fig. 10. Comparison of actual recovery with those estimated by ANN in the test process for neodymium.

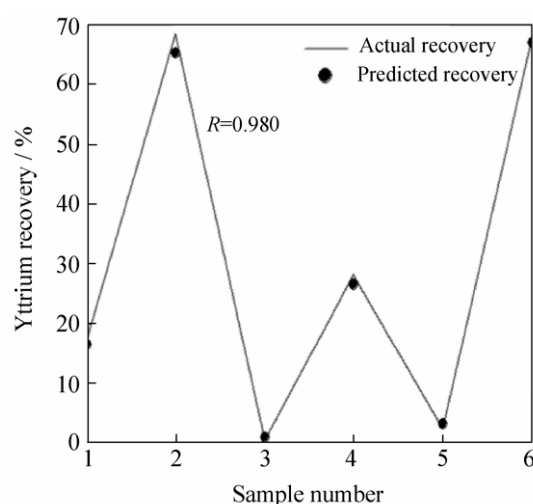


Fig. 11. Comparison of actual recovery with those estimated by ANN in the test process for Yttrium.

5. Conclusions

(1) The operational variables of acidity 60vol%, solid to liquid ratio 30wt%, leaching time 30 min, agitation rate 200 r/min, and temperature 60°C were obtained as the optimum conditions for the extraction of REEs. The recoveries of La, Ce, Nd, and Y were achieved at 74%, 59%, 72%, and 73%, respectively, which correspond to the mentioned optimized points.

(2) The produced data, in the laboratory optimization process, were used for simulating by means of ANN. Feed-forward artificial neural networks with 4-5-5-1 arrangements were used for estimating REE recoveries. In the testing process, the correlation coefficients of 0.971, 0.952, 0.985, and 0.98 were achieved for the prediction of Ce, Nd, La, and Y recoveries, respectively, which were quite satisfactory.

(3) These studies on REE leaching recovery predic-

tion, using ANN, can be helpful to the following applications. (a) In the REE leaching industry, the REEs in pregnant liquors are being analyzed using expensive analytical methods, ICP-AES and ICP-MS, which are not always available and/or affordable; therefore, the prediction of REE recovery corresponding to different leaching operational variables can be attractive from an economical point of view and for saving the expenses of REE analyzing in the operating life of the REE leaching plant. (b) Different rare earth elements have different prices, and the prediction of their recovery, separately, can help in the evaluation and determination of the best operational variables to achieve the maximum revenue.

Acknowledgments

The authors would like to thank Mr. Iraj Hurmazdi, mineral processing expert, for his valuable scientific comments during the first stage of investigation, and Mrs. Salehi, the employee of the Geological Survey of Iran, for her kind assistance during this research.

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