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Prediction and optimization studies for bioleaching of molybdenite concentrate using artificial neural networks and genetic algorithm

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ABSTRACT

This paper presents the application of an artificial neural network (ANN) in order to predict the effects of operational parameters on the dissolution of Cu, Mo and Re from molybdenite concentrate through meso-acidophilic bioleaching. The initial pH, solid concentration, inoculum percent and time (days) were used as inputs to the network. The outputs of the models included the percent of Cu, Mo and Re recovered. The development and training of a feed-forward back-propagation artificial neural network (BPNN) was used to model and predict their recoveries. 105 sets of data were used to develop the neural network architecture and train it. To reach the network with highest generalizability, the space of neural networks with different hidden layers (one up to three hidden layers) and with the varying number of neurons each layer were searched. As a result, it was found that (4-5-5-2-1); (4-7-5-2-1) and (4-7-1-1-1) arrangements could give the most accurate prediction for Cu, Mo and Re extraction respectively. The regression analysis of the models tested gave a good correlation coefficient of 0.99968, 0.99617 and 0.99768 respectively for Cu, Mo and Re recoveries. The results demonstrated that ANN has a good potential to predict Cu, Mo and Re recoveries. Also, genetic algorithm (GA) was used to find out the optimum levels of parameters in the best models defined by ANN. The maximum recovery of Cu, Mo and Re on the 30th day were nearly 73%, 2.8% and 27.17% respectively.

1. Introduction

There has been much interest in the development of bio-hydrometallurgical methods for the extraction of copper and other elements from sulfide minerals because they have many advantages over the more traditional pyrometallurgical techniques, which include reduced emissions to air, simplicity of operation, low cost and applicability to low-value ores or mineral resources that cannot be treated by conventional mining techniques (Brierley and Brierley, 2001; Akcil, 2004; D'Hugues and Spolaore, 2008; Anjum et al., 2010; Brierley, 2010; Castro et al., 2013). Bioleaching is an economical method for the recovery of metals which involves low investment and operation costs. Furthermore, it is more ecofriendly in comparison to other physico-chemical metal extraction processes (Ehrlich, 2004; Fu et al., 2008; Akcil and Deveci, 2010). The solubilization of metals by the application of microorganisms isolated from the mine sites and the subsequent recovery of metals from solution is referred to as bioleaching (Bosecker, 1997; Krebs et al., 1997; Panda et al., 2015; Rohwerder et al., 2003). In processes that operate at ambient temperatures of about 30–45 °C, the most predominant bacteria include iron- and sulfur-oxidizing *Acidithiobacillus ferrooxidans*, sulfur-oxidizing *Acidithiobacillus thiooxidans* and *Acidithiobacillus caldus*, and iron-oxidizing *Leptospirillum* spp. (*Leptospirillum ferriphilum* and *Leptospirillum ferrooxidans*) (Coram and Rawlings, 2002; Fouchera et al., 2003; Okibe et al., 2003; Rawlings, 2004, 2005). Recently, several bioleaching experiments have been carried out to evaluate the extraction of copper, molybdenum and rhenium from molybdenite concentrate. The results have indicated the successful removal of copper from Mo-concentrate within a short period of bioleaching using mesophilic and thermophilic (moderate and extreme) microorganisms; however, difficulties with low dissolution kinetics were observed for molybdenum and rhenium (Abdollahi et al., 2013a, 2013b, 2014, 2015). These experiments are essential in terms of

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expanding our knowledge and understanding such complex systems. In order to understand these systems better, we need to develop mathematical models as a simplified representation and abstraction of the real world entities.

Artificial neural networks (ANNs) are a new branch of intelligence science that is applied to understand the complex systems. Over the past 10 years, artificial neural networks (ANNs) and particularly feed-forward artificial neural networks (FANNs) have been extensively studied. In general, choosing an appropriate approach for modeling purposes highly depends on the complexity of the problem of interest and the available knowledge related to the problem. However, such considerable advantages such as the ability to approximate nearly any smooth and measurable function, making no prior assumptions regarding the distribution of data, the ability to model highly non-linear functions and the ability to accurately be generalized when presented with new, unseen data has led the MLP to find its way in a variety of applications (Gardner and Dorling, 1998; Hornik et al., 1989). The use of such networks can now be found for a number of predictions such as modeling of the greenhouse effect, simulating N_2O emissions from a temperate grassland ecosystem, modeling rare earth solvent extraction, bioleaching metals and coal microbial desulfurization (Jorjani et al., 2008a; Ahmadzadeh and Lundberg, 2013; Vasseghian et al., 2014). In fact, ANNs have been extensively applied in different branches of mineral processing such as flotation. Apart from that, it has also been used in studying some froth characteristics aimed at predicting the effect of changing flotation variables and flotation kinetics (Moolan et al., 1995; Labidi et al., 2007; Al Thyabat, 2008), optimization of froth flotation (Massinaei and Doostmohammadi, 2010) and modeling bubble surface area flux (Nakhaei et al., 2012). Moreover, application of ANNs has been the key step in the prediction of recovery and grade of flotation column concentrate (Ozbayoglu et al., 2008), estimation of coal Hardgrove Grindability Index (Jorjani et al., 2007) and organic and inorganic sulfur reduction from coal (Jorjani et al., 2008a, 2008b). It has been also used for ore sorting and classification (Singh and Rao, 2005). ANNs are used in dewatering (Jamsa-Jounela and Oja, 2000) in order to predict the performance of dewatering filters and are also applied in other sub branches of mineral processing and engineering.

Despite such an abundance of studies towards application of ANNs in conventional mineral processing techniques, not much work has been carried out in the area of hydrometallurgy and specially bio-hydrometallurgy. Some of the works that have been carried out are restricted to the metal bioleaching prediction in continuous processing of municipal sewage with *Acidithiobacillus ferrooxidans* (Laberge et al., 2000), expert control of the electrolytic process in zinc hydrometallurgy using ANNs (Wu et al., 2001), prediction of sulfur removal with *Acidithiobacillus* (Acharya et al., 2006), prediction of microbial desulfurization of coal (Jorjani et al., 2007) and prediction of the growth of indigenous *Acidithiobacillus thiooxidans* (Liu et al., 2008). It has been also used in modeling the performance of a biological Fe^{2+} oxidizing fluidized bed reactor (FBR) through popular neural network-back-propagation algorithm. Modeling the Fe^{3+} production in FBR and managing the regeneration of Fe^{3+} for heap leaching application have been investigated (Ozkaya et al., 2008). The prediction of Al_2O_3 leaching recovery in the Bayer's process is also determined through ANNs (Chehreh Chelgani and Jorjani, 2009). ANNs assists in describing the complex relationships between process parameters and predicting the performance of a fluidized bed bioreactor for Fe^{3+} regeneration and a gravity settler for precipitative iron removal with two different modeling methods namely the multiple regression modeling and artificial neural network (Nurmi et al., 2010). It is also used in the prediction of pre-oxidation efficiency of refractory gold concentrate by ozone in ferric sulfate solution (Li et al., 2011) and in the prediction of heavy metals recoveries (Zn, Cu, Ni, Pb, Cd and Cr) from dewatered metal plating sludge (with no sulfide or sulfate compounds) during a bioleaching process (Sari,2012). Prediction of ferric iron precipitation in a bioleaching process using partial least squares and artificial neural network (Golmohammadi et al., 2013) has gained much attention recently. In addition to that, optimization of iron bioleaching from contaminated kaolin clay through artificial neural network has been also carried out recently (Pazouki et al., 2012). However, artificial neural network approach has not yet been applied for modeling the process of bioleaching for Mo, Cu and Re recoveries from molybdenite concentrate. Accordingly, its background success in modeling mineral processing systems besides aforementioned advantages was cogent enough for us to apply such a prominent modeling approach in the present study and develop a mathematical model describing the bioleaching process of Mo, Cu and Re from molybdenite concentrate. The ultimate goal of mathematical modeling is to explain a system and the effect of its components on the behaviour of the system. Finally, this knowledge about the system's behaviour provides us with the ability to predict its behaviour in different situations and thereby have control on the system. In industrial systems, the goal of control is to gain the optimum performance. In bioleaching systems, especially, the goal is optimal extraction of element from economical point of view. Having the trained neural network as a mathematical model in hand, now we need to search for the optimum performance of the bioleaching system, which lies in the realm of optimization problems.

Genetic algorithm optimization technique is a powerful tool that has been used in different mineral processing methods. Karr et al. (1997), Karr (1993), Karr and Weck (1996a,1996b) have used a combination of fuzzy logic and GA for applications in processes such as grinding, size separation in hydrocyclone and maximization of froth recovery in flotation circuits. Earlier reports made by Venter et al. (1997) describe about a GA-based approach to assemble various circuit types for learning classifier systems. While et al. (2004) have used an evolutional algorithm to optimize the performance of a cone crusher operating with a circulating load in a closed circuit, while Svedensten and Evertsson (2005) have developed a model for optimization of crushing plant by means of a genetic evolutionary algorithm. A multi-objective evolutionary algorithm to optimize comprehensive crushing and grinding plant has been developed by Huband et al. (2006). Also, Gupta et al. (2007) applied genetic algorithms to optimize coal preparation plants. In the recent years, GA was used by Ghobadi et al. (2011) to optimize the performance of the flotation circuits, while Pirouzan et al. (2014) used it for optimizing the flotation cells configuration. In the field of bio-hydrometallurgy, Jian et al. (2008) constructed a mathematical model for enargite bioleaching, following which optimization of model parameters using genetic algorithm was assessed. Similarly, the present work deals with the assessment of bioleaching of molybdenite concentrate for the extraction of copper, molybdenum and rhenium under varying pH conditions, solid concentration, inoculum percent and time. In this regard, the experimental data were obtained at a laboratory scale and used to model bioleaching process by means of artificial neural networks. Out of the broader and more dense sampling coverage of operational parameters by experimental data (105 samples of data), compared with statistical methods in which the goal is to reach the maximum recovery through minimum number of experiments, the model obtained by neural networks is more reliable and accurate. To our knowledge, this is the first time that ANNs have been used to predict Cu, Mo and Re recoveries in bioleaching tests using a mixed culture of meso-acidophilic microorganism consisting of *A. ferrooxidans*, *A. thiooxidans* and *L. ferrooxidans* species enriched from the Sarcheshmeh Copper Mine. Furthermore, the Genetic Algorithm optimization method was used to find the optimum levels of operational parameters (Inputs) in the trained neural network which leads to the maximum recoveries.

2. Materials and methods

2.1. Sample, chemical and mineralogical analyses

The mineral samples used in the experiments were obtained from

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Table 1
Results of experiments for molybdenite bioleaching (Cu, Mo and Re recoveries) in different operating conditions (70%, 15% and 15% numbers of the tests were used for training, testing and validation respectively).

Test no.	Parameters				Recovery %			Test no.	Parameters				Recovery %		
	Initial pH	Solid %	Inoculum %	Time (days)	Cu	Mo	Re		Initial pH	Solid %	Inoculum %	Time (days)	Cu	Mo	Re
1	1.6	3	5	0	0.00	0.00	0.00	54	2	9	15	20	43.68	0.81	2.24
2	1.6	3	5	5	37.80	0.35	1.98	55	2	9	15	25	44.12	0.86	2.67
3	1.6	3	5	10	40.59	0.51	8.43	56	2	9	15	30	44.72	1.04	4.23
4	1.6	3	5	15	42.89	1.34	9.35	57	1.46	6	10	0	0.00	0.00	0.00
5	1.6	3	5	20	42.89	2.26	10.45	58	1.46	6	10	5	37.24	0.88	0.69
6	1.6	3	5	25	44.82	2.27	11.54	59	1.46	6	10	10	43.44	1.36	4.99
7	1.6	3	5	30	45.84	2.47	19.10	60	1.46	6	10	15	44.42	1.61	5.70
8	2	3	5	0	0.00	0.00	0.00	61	1.46	6	10	20	44.80	1.67	6.11
9	2	3	5	5	45.40	0.40	3.71	62	1.46	6	10	25	45.00	1.86	6.81
10	2	3	5	10	46.04	0.71	6.66	63	1.46	6	10	30	45.63	2.00	9.52
11	2	3	5	15	48.58	1.49	7.51	64	2.14	6	10	0	0.00	0.00	0.00
12	2	3	5	20	49.10	1.99	8.28	65	2.14	6	10	5	40.65	0.19	1.91
13	2	3	5	25	49.35	2.22	8.75	66	2.14	6	10	10	40.75	0.36	2.67
14	2	3	5	30	50.70	2.56	14.61	67	2.14	6	10	15	41.87	0.83	3.21
15	1.6	9	5	0	0.00	0.00	0.00	68	2.14	6	10	20	42.93	1.06	3.48
16	1.6	9	5	5	40.53	0.34	1.18	69	2.14	6	10	25	43.44	1.11	4.00
17	1.6	9	5	10	42.35	0.69	2.65	70	2.14	6	10	30	44.23	1.39	7.22
18	1.6	9	5	15	43.84	0.87	3.04	71	1.8	0.95	10	0	0.00	0.00	0.00
19	1.6	9	5	20	44.95	0.91	3.24	72	1.8	0.95	10	5	49.00	0.00	5.02
20	1.6	9	5	25	47.24	0.99	3.61	73	1.8	0.95	10	10	53.90	0.03	0.12
21	1.6	9	5	30	47.90	1.15	6.37	74	1.8	0.95	10	15	58.29	0.04	0.60
22	2	9	5	0	0.00	0.00	0.00	75	1.8	0.95	10	20	60.58	0.14	2.35
23	2	9	5	5	42.05	0.33	1.37	76	1.8	0.95	10	25	63.80	0.18	2.93
24	2	9	5	10	44.84	0.67	2.16	77	1.8	0.95	10	30	66.77	0.59	10.01
25	2	9	5	15	45.41	0.67	2.47	78	1.8	11.05	10	0	0.00	0.00	0.00
26	2	9	5	20	47.38	0.75	2.64	79	1.8	11.05	10	5	38.47	0.17	1.11
27	2	9	5	25	49.01	0.82	3.06	80	1.8	11.05	10	10	40.11	0.47	1.59
28	2	9	5	30	49.60	1.01	5.92	81	1.8	11.05	10	15	40.54	0.51	1.86
29	1.6	3	15	0	0.00	0.00	0.00	82	1.8	11.05	10	20	41.98	0.57	1.98
30	1.6	3	15	5	47.29	0.17	2.87	83	1.8	11.05	10	25	44.09	0.60	2.33
31	1.6	3	15	10	48.03	0.48	5.46	84	1.8	11.05	10	30	44.66	0.78	3.72
32	1.6	3	15	15	48.81	1.05	6.66	85	1.8	6	1.59	0	0.00	0.00	0.00
33	1.6	3	15	20	49.46	1.66	7.36	86	1.8	6	1.59	5	40.82	0.23	1.32
34	1.6	3	15	25	51.21	1.66	8.29	87	1.8	6	1.59	10	43.92	0.68	4.01
35	1.6	3	15	30	52.52	1.97	15.36	88	1.8	6	1.59	15	44.73	1.15	4.65
36	2	3	15	0	0.00	0.00	0.00	89	1.8	6	1.59	20	45.65	1.17	4.90
37	2	3	15	5	43.53	0.01	3.71	90	1.8	6	1.59	25	46.06	1.29	5.63
38	2	3	15	10	45.87	0.30	4.68	91	1.8	6	1.59	30	46.77	1.49	10.06
39	2	3	15	15	46.61	0.81	6.25	92	1.8	6	18.41	0	0.00	0.00	0.00
40	2	3	15	20	47.79	1.32	6.56	93	1.8	6	18.41	5	39.79	0.42	2.19
41	2	3	15	25	48.07	1.34	7.43	94	1.8	6	18.41	10	42.38	0.83	2.20
42	2	3	15	30	49.47	1.71	14.03	95	1.8	6	18.41	15	43.13	1.13	2.70
43	1.6	9	15	0	0.00	0.00	0.00	96	1.8	6	18.41	20	43.91	1.17	2.90
44	1.6	9	15	5	37.24	0.23	1.04	97	1.8	6	18.41	25	44.89	1.30	3.63
45	1.6	9	15	10	39.56	0.47	1.98	98	1.8	6	18.41	30	45.72	1.61	5.83
46	1.6	9	15	15	41.15	0.73	2.36	99	1.8	6	10	0	0.00	0.00	0.00
47	1.6	9	15	20	41.17	0.83	2.57	100	1.8	6	10	5	33.42	0.25	1.95
48	1.6	9	15	25	41.39	0.89	2.84	101	1.8	6	10	10	38.61	0.66	3.16
49	1.6	9	15	30	41.95	1.06	4.72	102	1.8	6	10	15	42.28	0.99	3.43
50	2	9	15	0	0.00	0.00	0.00	103	1.8	6	10	20	42.55	0.99	3.66
51	2	9	15	5	39.51	0.47	1.14	104	1.8	6	10	25	43.54	1.12	4.27
52	2	9	15	10	39.67	0.69	1.60	105	1.8	6	10	30	46.92	1.39	5.46
53	2	9	15	15	42.51	0.81	2.04	–	–	–	–	–	–	–	–

the Sarcheshmeh Copper Mine. Semi quantitative X-ray diffraction (SQXRD) technique was used to define mineralogical composition of the sample. It was found to contain about 89.8% molybdenite, 2% chalcopyrite and 2% pyrite. X-ray powder diffraction patterns were obtained using a Siemens D-500 diffractometer with Ni-filtered Cu–Ka radiation and a goniometer having speed of 1° 2θ/min. The chemical analyses showed that the molybdenite conc. contained 53.84% Mo, 1.56% Fe, 0.98% Cu and 550 ppm Re. The size distribution of the sample was 90 wt% less than 38 μm which was used for bioleaching tests. The results of the mineralogical studies revealed that molybdenite was the major phase of the Mo bearing mineral. Also, mineralogical study showed that molybdenite (MoS_2) was the main mineral phase and chalcopyrite ($CuFeS_2$) and pyrite (FeS_2) were the minor. It was also observed that chalcopyrite and pyrite could be distinguished in the molybdenite conc. as unwanted minerals. These minerals were liberated from the molybdenite in the 38 μm size fraction, however in some cases, copper bearing minerals and also pyrite were interlocked with molybdenite. Rhenium bearing minerals were neither detected by optical mineralogy nor instrumental techniques (XRD and SEM). Studies have shown that rhenium as elemental form is located within the molybdenite crystalline lattice (Abdollahi et al., 2013a, 2013b, 2014, 2015).

For better understanding of the presence of major and minor minerals in the molybdenite concentrate, scanning electron microscopy (SEM) and energy dispersive X-ray (EDAX) analyses (Philips XL 30 SEM) were conducted on original samples and treated solid residues obtained from the bioleaching experiments. The samples were air dried followed by mounting on stubs and coating with Au before

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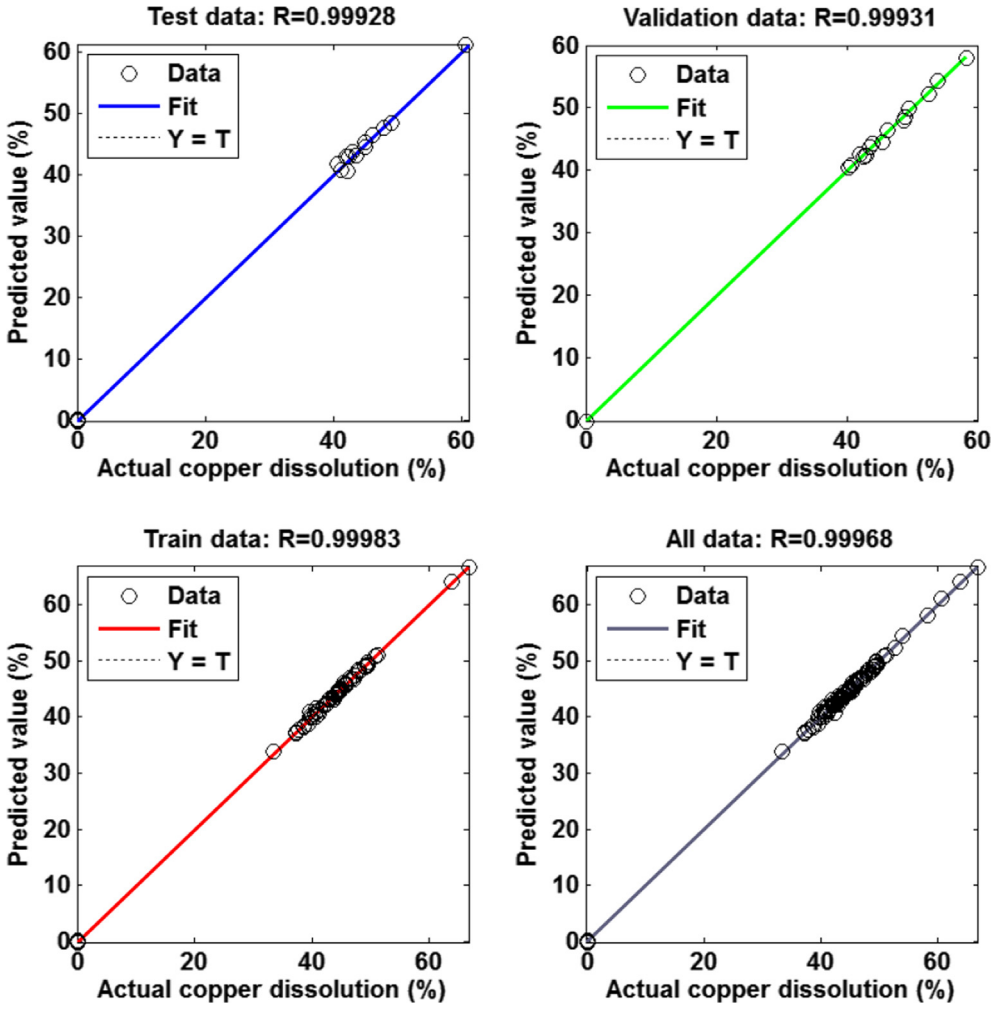


Fig. 1. The correlation results of Cu dissolution between the laboratory and predicted data in four data sets (Train data, Test data, Validation data and all data).

examination. The examination of internal structure of the samples, the distribution of mineral phases and the choice of areas for EDAX-analyses was carried out using a scanning electron microscope operating with back scattered electrons (BSE). The results also confirmed the mineralogical observation.

2.2. Culture media and microorganism

The microorganims used in the present experiment were a mixed meso-acidophilic bacterial consortium. The mixed culture comprised of iron and sulfur oxidizing bacteria (*Acidithiobacillus ferrooxidans, Acidithiobacillus thiooxidans,* and *Leptospirillum ferrooxidans*) that were originally enriched from mine drainage of the Sarcheshmeh Copper Mine, Kerman (Bakhtiari et al., 2008; Ahmadi et al., 2010). The cultures were originally grown in shake flasks (at 150 rpm, 32 °C) using chalcopyrite concentrate as an energy source. Due to potential toxicity of molybdenum, the cultures were subsequently adapted to grow with molybdenite as the sole energy source since adaptation is an unique biotechnological feature of these microorganims (Panda et al., 2013; Mishra et al., 2018). This involved successive enrichment steps for a period of 3 months with gradual increment in the molybdenite concentration (maximum up to 10% w/v), while decreasing the concentration of chalcopyrite. The cultures were repeatedly transferred into medium containing molybdenite concentrate to adapt the cultures. The mixed cultures were maintained on mineral salt medium (9 K) having sulfur and Fe^{2+} as the energy source and the pH was adjusted to 1.8 using sulfuric acid. The growth was monitored by measuring the redox potential and pH of the medium. The 9 K medium contained 3 g/L $(NH_4)_2SO_4$, 0.1 g/L KCl, 0.5 g/L K_2HPO_4, 0.5 g/L $MgSO_4{\cdot}7H_2O$, 0.01 g/L $Ca(NO_3)_2$ and 44.7 g/L $FeSO_4{\cdot}7H_2O$ (Silverman and Lundgren, 1959).

2.3. Bioleaching experiments

The bioleaching experiments were carried out in 250 mL Erlenmeyer flasks under shaking conditions in an orbital incubator shaker (Innova 4200 model, New Brunswick Scientific Company, USA) maintained at a temperature of 32 ± 1 °C and a rotation speed of 150 rpm. The oxidation-reduction potential (ORP) was measured with an WTW model 323 Eh meter having a Pt combination redox electrode with an Ag/AgCl reference electrode. The pH of the medium was adjusted to 1.8 by adding sulfuric acid. The changes in pH were monitored with a Metrohm model 827 pH meter having gel-filled combination pH probe with an Ag/AgCl reference electrode. The efficiency of the bioleaching process was examined by measuring the concentration of Cu, Mo, and Re in the solution over time. The bacterial activity/growth was monitored by Fe^{2+} iron oxidation and measurement of ORP values. In addition to Fe^{2+} oxidation, the bacterial growth was also monitored through cell count method using a Neubauer counter having a depth of 0.02 mm and area of 1/400 mm^2, placed under the light microscope (Ziess Biological Microscope). The adapted bacteria sub-cultured in the 9 K medium were used in the bioleaching experiments with an initial cell density of 0.8×10^7 cells/mL. After 30 days of bioleaching, the highest cell counts were observed in the range of 8–9 $\times 10^7$ cells/mL.

For the bioleaching tests, the effect of variation in four parameters

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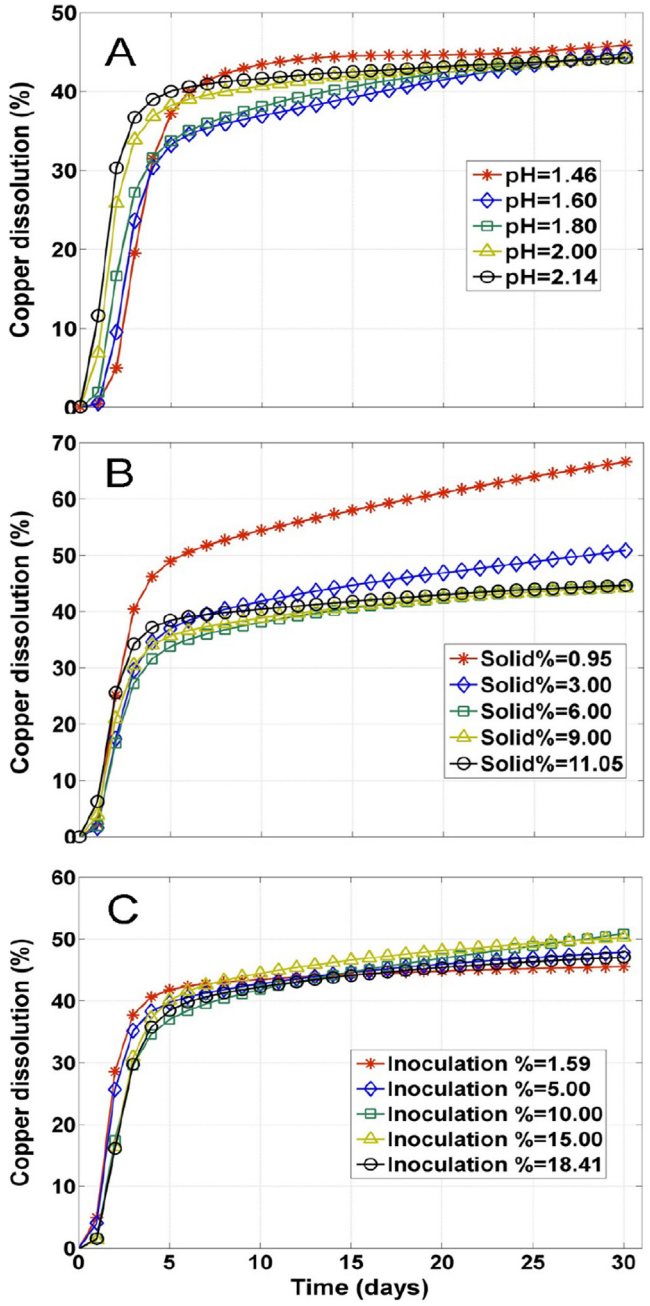


Fig. 2. The predicted copper dissolution versus time of bioleaching in terms of changes of the (A) initial pH (B) solid concentration and (C) inoculum percent.

such as initial pH, solid concentration, inoculum percent and time on Cu, Mo and Re extraction from the concentrate were assessed. In general, these parameters are one of the important aspects when optimization studies for bioleaching are considered (Panda et al., 2017). For every bioleaching test, 5 g $FeSO_4{\cdot}7H_2O$ along with 1 g sulfur were added as an energy source to 100 mL media solution in each flask. Any water loss due to evaporation, during bioleaching, was compensated by adding required volume of distilled water. In a total, 105 sets of data were obtained. The results of copper, molybdenum and rhenium recoveries along with the operating conditions are presented in Table 1.

2.4. Sampling procedure for chemical assay

The bioleaching study was carried out for a total period of 30 days. Samples were periodically withdrawn at regular intervals of 5 days each and were analyzed for Cu, Mo and Re recovery using inductively coupled plasma-atomic emission spectroscopy (ICP-AES). To calculate the fraction of Cu, Mo and Re leached, the following equation was used (Dehghan et al., 2009):

$$X_i = \frac{\left(V_0 - \sum_{i=1}^{i-1} v_i\right) C_i + \sum_{i=1}^{i-1} v_i C_i}{M\left(\frac{C_M}{100}\right)} \quad (1)$$

where X_i is the Cu, Mo or Re extraction corresponding to sample i, V_0 is the initial volume of the leaching solution in the reactor (mL), v_i is the volume of sample i withdrawn from the reactor (mL), C_i is the Cu, Mo or Re is the concentration in sample i (mg/L), M is the initial mass of the molybdenite concentrate in grams added into the reactor and C_M is the Cu, Mo or Re percentage in the molybdenite concentrate. It is to be noted that the experiments were carried out in duplicates, some were randomly repeated a third time, and the results were averaged.

2.5. Modeling and optimization methods (ANN and GA)

A total of 105 sets of data were used for modeling the bioleaching process in order to predict the Cu, Mo and Re extraction by ANN (feed-forward back-propagation ANN model). The influence of four parameters including the initial pH, solid concentration, inoculum percent and time on the bioleaching process were assessed. The normalized experimental data (Eq. (S2)) were used for modeling the bioleaching process via different arrangements of artificial neural networks. The reason for developing different arrangements as possible models is to opt the network with the highest generalizability and this procedure is explained in more details in the following paragraphs. The output of the neural network is the prediction of the recovery of three elements including copper, molybdenum and rhenium. It is worth mentioning that the process of modeling is performed for each element separately.

One of the key steps in modeling problems using ANNs is determining the architecture or arrangements of the network which includes determining the number of hidden layers, the number of neurons per layer and the type of transfer function within each neuron. The number of hidden layers should be determined so that the network has a trade-off between fitness to data and generalization power. Unfortunately, at present time there is no general guideline to determine the optimal number of the layers and neurons in a straightforward way. As a result, like majority of studies, we adopted trial and error procedure to find the optimal network for each element. In this regard, the space of neural networks with different architectures were searched to find the architecture with acceptable measures as generalizability power.

In each trial, the shell of created network with initial random weights should be trained by the data or in other words, the network should learn the pattern governing the data. Commonly, in MLP networks the learning process which is equivalent to minimization of an objective function is performed using back-propagation algorithm. In this formulation, the objective function is a measure of modeling error given the network's weights. However, the non-linear mapping between the input and output in ANNs leads to non-convex objective functions which might have multiple local minima. In this situation, the back-propagation algorithms have the potential to be trapped in local minima. In such problems, application of global search algorithms like GA, Particle Swarm, etc. could be fruitful. Yet, using these algorithm are excessively time consuming and computationally burdensome. Considering these two extremes of limitations, we innovated a middle strategy taking advantage of both global search and gradient-based algorithms. In our innovation, we start the learning process with several different random weights and perform the minimization using back-propagation. At the end, the solution which satisfies our criteria for generalizability power of the network is opted as prototype of a specific architecture to be compared against prototypes of other architectures.

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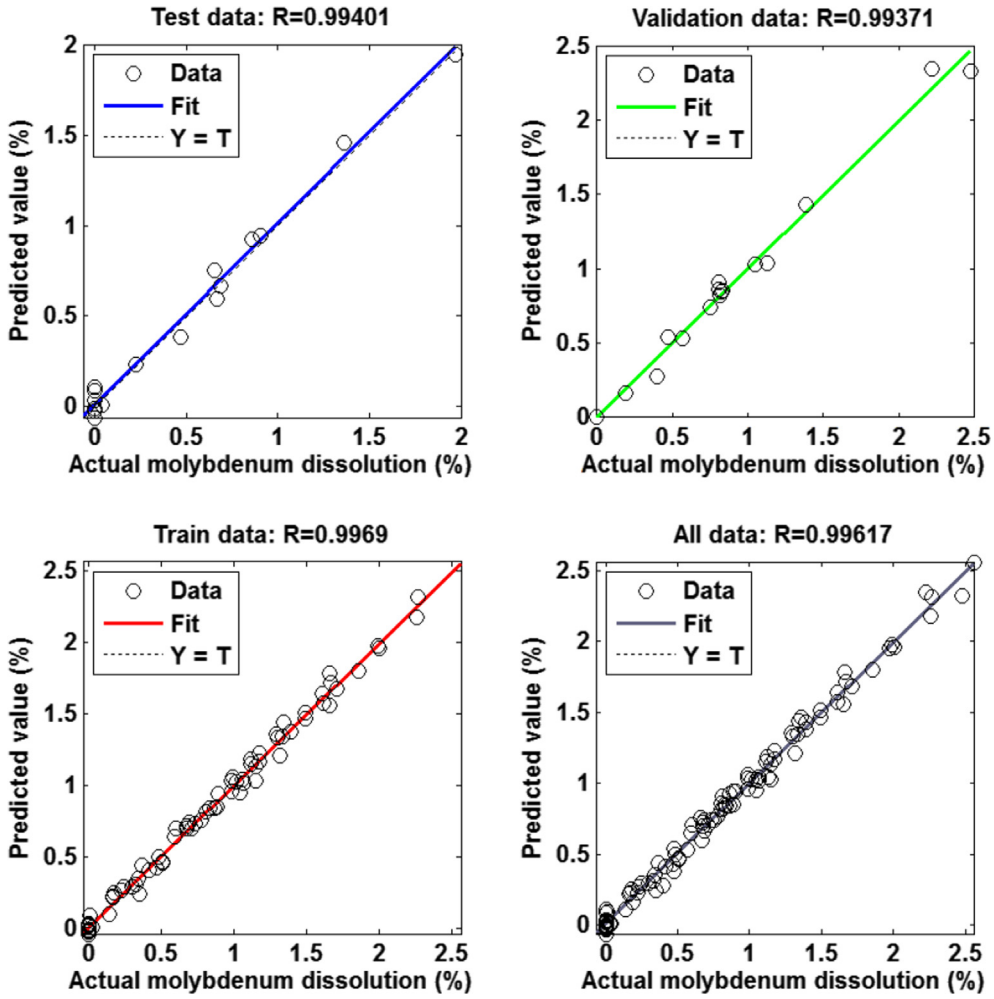


Fig. 3. The correlation results of Mo dissolution between the laboratory and predicted data in four data sets (Train data, Test data, Validation data and all data).

In this way, out of initiating learning process from different points in objective function, it is less possible for an architecture's prototype to correspond to a local minima. To guarantee the generalizability of a network, the common technique of cross-validation is applied during learning process to prevent overfitting phenomenon (Fig. S2). To achieve this goal, a fraction of data is set aside as Validation and Test data and is not included in the training process. During the training process, if overfitting of the data starts to occur, the prediction error for validation data set would start to grow steadily. After observing such an increase for a predefined number of successive epoches, the training process is stopped and the network's weights and biases is restored to the point where the increase started. Likewise, the prediction error for test data set is a measure to opt a network as the prototype of networks with the same architecture. From this perspective, the network corresponding to the lowest test data set error is selected as the architecture prototype.

After this brief introduction, three types of neural networks (one hidden layer, two hidden layers and three hidden layers) were designed. The number of neurons in the neural network with one hidden layer was varied from one up to 40. The number of neurons in the neural network with two hidden layers was varied from one up to 20 in each layer. Also, the number of neurons in the neural network with three hidden layers was varied from one up to 10 in each layer. To prevent being trapped in local minima, 200 network shells with the same architecture but different initial random weights were created and trained by the data. The network with the lowest error for test data sets among all the networks (with different hidden layers) was selected as efficient network or the network with highest generalizability. In the present study, the training, testing and validation subsets were selected to be 70%, 15% and 15% of all the data respectively.

All the network design and training were implemented in MATLAB environment with particular use of the Neural Network Toolbox. Training was performed using the Levenberg–Marquardt optimization algorithm. As mentioned earlier, a normalization step is included in the modeling procedure before learning step.

After modeling the bioleaching process with neural network approach, the genetic algorithm method was applied to find the optimum level of input parameters at which bioleaching process yields a maximum extraction of the three above mentioned elements. To do so, the designed neural networks in the previous section were used as a mathematical model between the inputs (operational parameters) and the output (dissolution value). So this model can be imported in a genetic algorithm to evaluate the performance of the combinations of different operational parameters. It is important to note that in MATLAB software, GA tries to find the global minimum while our goal is to find a combination of the operational parameters in order to maximize dissolution. In order to solve this problem, the following equations have been defined as objective functions for Cu, Re and Mo, respectively.

$$Y_{Cu} = \frac{1}{d_{Cu}}, \tag{2}$$

$$Y_{Mo} = \frac{1}{d_{Mo}}, \tag{3}$$

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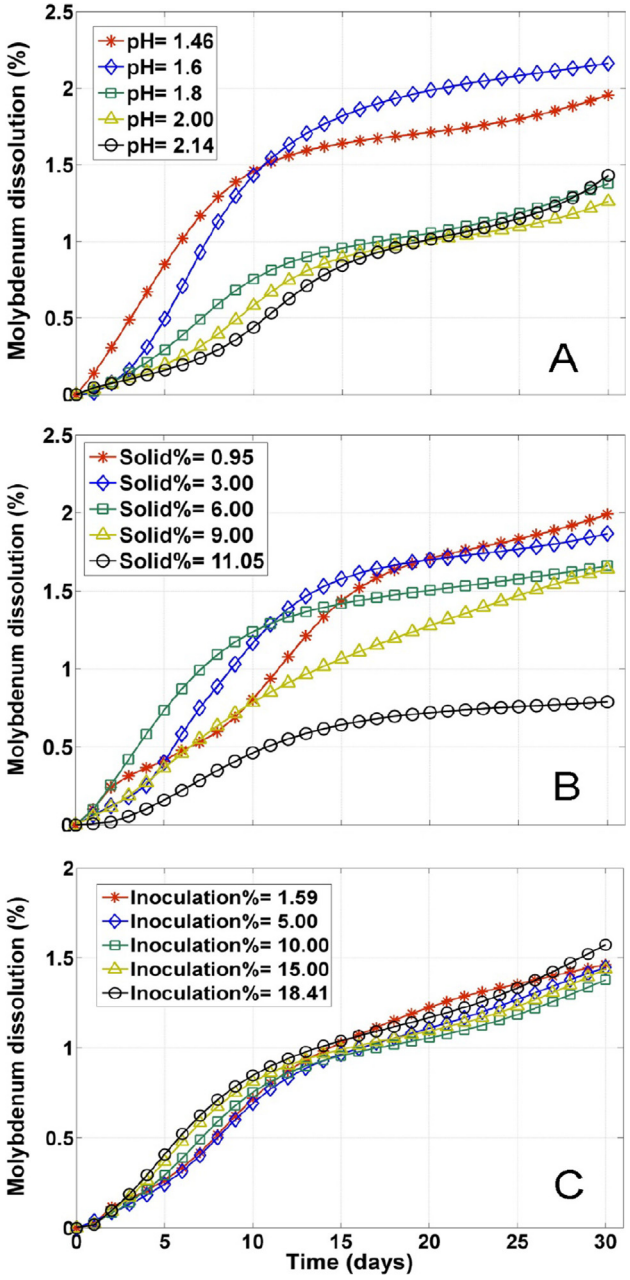


Fig. 4. The predicted molybdenum dissolution rate versus time of bioleaching in terms of changes of the (A) initial pH (B) solid concentration and (C) inoculum percent.

$$Y_{Re} = \frac{1}{d_{Re}}, \quad (4)$$

where Y_{Cu}, Y_{Mo} and Y_{Re} are fitness values, in terms of GA, corresponding to Cu, Re and Mo, respectively while d_{Cu}, d_{Mo} and d_{Re} represent dissolution values for Cu, Mo and Re, respectively.

3. Results and discussion

The modeling and optimization results obtained from artificial neural network (ANN) and genetic algorithm (GA) approaches are presented as follows:

3.1. Modeling and optimization of copper dissolution

The neural network with (4-5-5-2-1) arrangement was selected as the optimum arrangement with mean square error of 0.0956 for training data, 0.4976 for testing data, and 0.2080 for the validation data. The correlation results between laboratory and predicted data in four data sets (Train data, Test data, Validation data and all data) are presented in Fig. 1. The correlation coefficients (R^2) of 0.99983, 0.99928, 0.99931 and 0.99968 for training, testing, validation and all data respectively, are indicative of good job of modeling the underlying process by the network.

The results of predicted copper dissolution from molybdenite concentrate during bioleaching are presented in Fig. 2. The dissolution of copper versus time of bioleaching during the 30 days period has been shown in Fig. 2A. It was obtained in terms of pH changes from 1.46 to 2.14, which involved five steps. In the graph shown in Fig. 2A, 6% and 10% were selected as the median for the other two parameters i.e. solid concentration and inoculum percent respectively. In addition to that, the copper dissolution rate plotted against the time of treatment has been shown in Fig. 2B, which was obtained in terms of changes in the solid concentration. The solid concentration was varied from 0.95% to 11.05% in five levels. The other two factors i.e. initial pH and inoculum percent are in median values of 1.8 and 10% respectively. Also in Fig. 2C, the inoculum percent was changed from 1.59% to 18.41% in five levels. Apart from that, the other two parameters i.e. initial pH and solid concentration are also in median values of 1.8 and 6 percent respectively. The results show that the changes in initial pH and inoculum percent have little effect on the copper dissolution during the bioleaching process. Furthermore, increasing the solid concentration decrease the rate of bioleaching. The maximum copper extraction of approximately 66% was achieved with a solid concentration 0.95%. The results indicated that the bioleaching phenomenon occured mostly within the first 10 days and thereafter it remained nearly constant. One of the reasons could be jarosite formation during the bioleaching test because of the highly oxidative environment and the accumulation of insoluble Fe^{3+} iron during the process. The GA found that the optimum copper dissolution is reached when operational parameters are set to be 1.46, 0.95 and 12.44 for the initial pH, solid concentration and inoculum percent respectively. For this combination of parameters, the copper dissolution reached nearly 73% following 30 days of incubation.

3.2. Modeling and optimization of molybdenum dissolution

The neural network with (4-7-5-2-1) arrangement was selected as the optimum arrangement with the lowest mean square error of 0.0026, 0.0041 and 0.0051 for training data, testing data and validation data respectively. The correlation results of the laboratory and predicted data in the four data sets (Train data, Test data, Validation data and all data) are shown in Fig. 3. The correlation coefficients (R^2) of 0.9969, 0.99401, 0.99371 and 0.99617 were obtained for training data, testing data, validation data and all data; respectively.

Fig. 4 shows the results of the predicted molybdenum dissolution from molybdenite concentrate through bioleaching. In Fig. 4A, molybdenum dissolution (%) has been plotted against the time of bioleaching during 30 days period, which was obtained in terms of changes in pH from 1.46 to 2.14 in five steps. In this graph, the other two parameters i.e. the solid and inoculum percent are in median value of 6% and 10% respectively. As seen in Fig. 4, the molybdenum dissolution increased with decrease in the initial pH during bioleaching test and the maximum dissolution of 2% was achieved after 30 days at a pH of 1.46 and 1.6. Fig. 4B shows the molybdenum dissolution rate versus the time of bioleaching, which was obtained in terms of changes in solid concentration. The solid concentration was varied from 0.95% to 11.05% in five levels. The other two factors i.e. the initial pH and inoculum percent were 1.46 and 15% respectively. It is observed that increasing the solid concentration from 0.95% to 11.05% reduced the dissolution rate of molybdenum (2% in 0.95 percent of the solid and 0.8% in 11.05 percent of the solid). In Fig. 4C, the inoculum percent was varied from 1.59% to 18.41% in five steps. Also the other two

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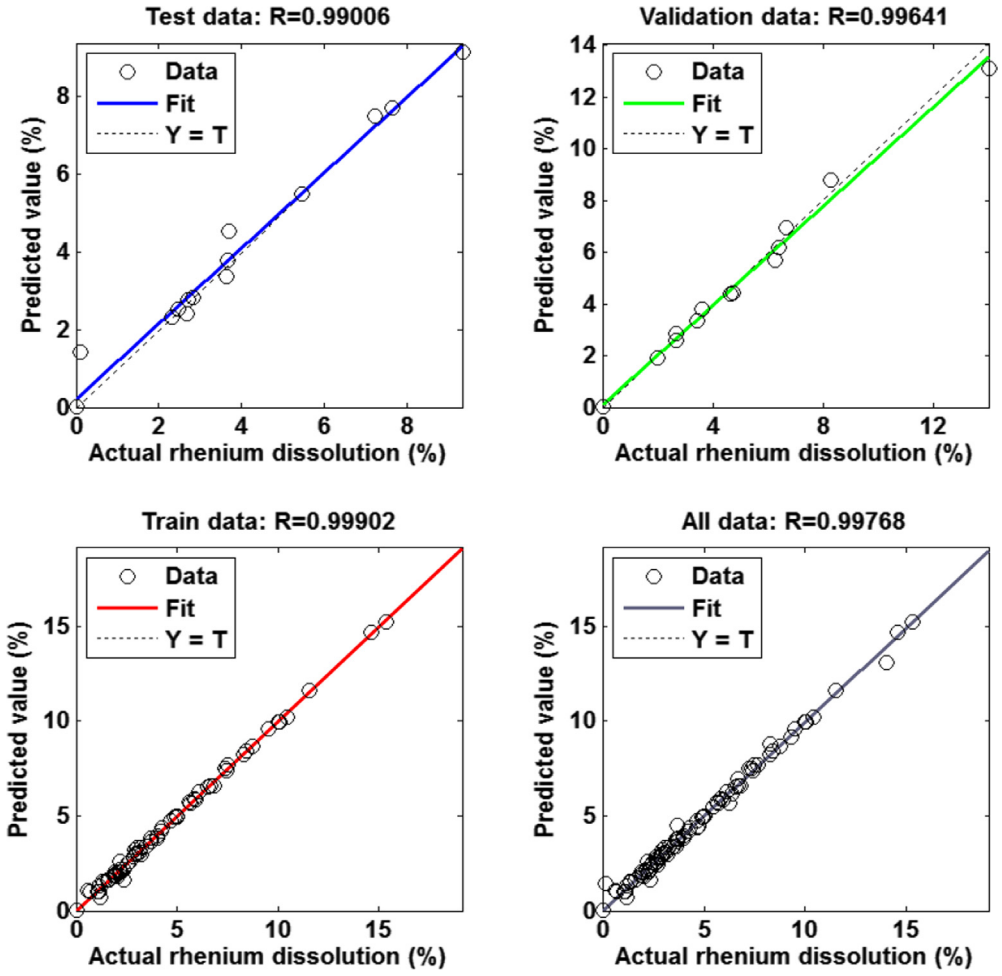


Fig. 5. The correlation results of Re dissolution between the laboratory and predicted data in four states (Train data, Test data, Validation data and all data).

parameters i.e. the initial pH and solid concentration were in median values of 1.8 and 6 percent respectively. The results obtained confirmed that changes in inoculum percent have a little effect on the molybdenum dissolution. The optimum parameters were defined using genetic algorithm method using an initial pH of 1.46, solid concentration of 1.09 and an inoculum percent of 18.41. In these conditions, the maximum dissolution of molybdenum reached 2.83% following 30 days of treatment.

3.3. Modeling and optimization of rhenium dissolution

The neural network with (4-7-1-1-1) arrangement was selected as the optimum arrangement with the lowest mean square error of 0.0286 for training data, 0.1678 for testing data and 0.1145 for the validation data. The correlation results of the laboratory and predicted data in the four data sets (Train data, Test data, Validation data and all data) are presented in Fig. 5. The correlation coefficients (R^2) of 0.99902, 0.99006, 0.99641 and 0.99768 were obtained for training data, testing data, validation data and all data, respectively.

The results of the predicted rhenium dissolution from the molybdenite concentrate during bioleaching are presented in Fig. 6. In Fig. 6A, the rhenium dissolution (%) have been plotted against the time of bioleaching during 30 days, which was obtained in terms of pH variation from 1.46 to 2.14 in five steps. Meanwhile the other two parameters i.e. solid and inoculum percent were in median values of 6% and 10% respectively. As seen from Fig. 6, the dissolution of rhenium increased with the decrease in initial pH during bioleaching process and under these circumstances, the maximum dissolution of approximately 9.5% was achieved at pH 1.46 after a period of 30 days. The rhenium dissolution rate versus the time of bioleaching, which was obtained in terms of changes in solid concentration has been given in Fig. 6B. The solid concentration was varied from 0.95% to 11.05% in five levels. The other two parameters i.e. the initial pH and inoculum percent were 1.8 and 10% respectively. By increasing the solid concentration from 0.95% to 11.05%, the dissolution rate of rhenium decreased (18% in 0.95 percent of the solid and 4% in 11.05 percent of the solid). The inoculum percent was changed from 1.59% to 18.41% in five steps which has been shown in Fig. 6C. Also the other two parameters i.e. initial pH and solid concentration were in median values of 1.8 and 6% respectively. The results demonstrated that high amount of inoculum percent had positive effects on the rhenium dissolution. Furthermore, the optimum parameters obtained using genetic algorithm were an initial pH of1.46, solid concentration of 0.95 and inoculum percent of 18.41. Under these conditions, the rhenium dissolution reached 27.17% after 30 days.

Dissolution of rhenium had an upward trend in the last days of the process. This is because the dissolution of rhenium is strongly dependent on the dissolution of molybdenite. As the molybdenite dissolved, the rhenium also enters the soluble phase. According to the literature reviews and our experimental results, molybdenite is a highly refractory mineral and it takes a long time to start molybdenite dissolution in the bioleaching process. Molybdenite dissolution in the last days of the process (25th–30th) has increased and, as a result, the rhenium dissolution from the molybdenite lattice has occurred more rapidly. Another reason for the higher dissolution of molybdenite and rhenium in the last days of treatment is the more adaptation of microorganisms and their higher growth which had a positive effect on the dissolution

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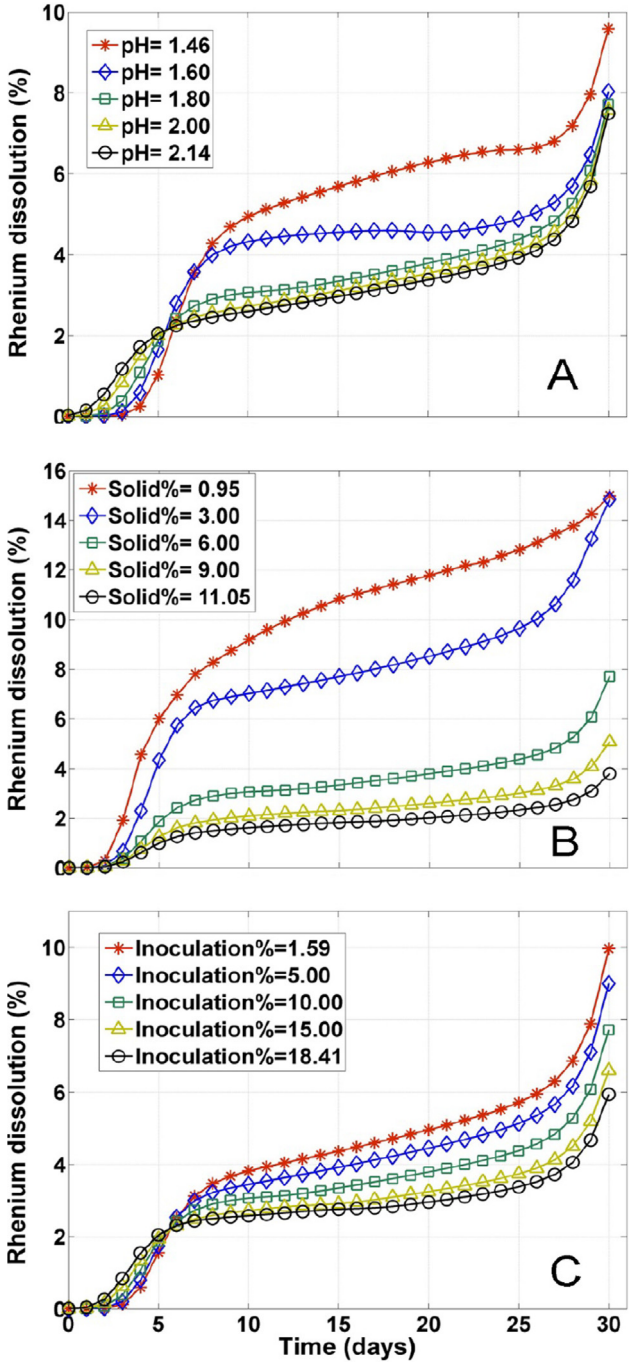


Fig. 6. The predicted rhenium dissolution versus time of bioleaching in terms of changes of the (A) initial pH (B) solid concentration and (C) inoculum percent.

efficiency.

The dissolution of Mo and Re followed a similar response to experimental conditions (Fig. 7). The data in the scatter plot (Fig. 7) could be described with a linear regression model ($R^2 \sim 0.7$). It is necessary to note that in the absence of some data that seemed as anomalies (maybe because of error) in the graph, the correlation coefficient increased upto 0.9 which was statistically acceptable. Based on the Eh-pH diagrams, dissolved molybdenum occurred as MoO_4^{2-} (molybdate) and rhenium as ReO_4^- (perrhenate) as their oxyanion chemistry was similar in acid solutions (Brookings, 1988; Icenhower et al., 2008).

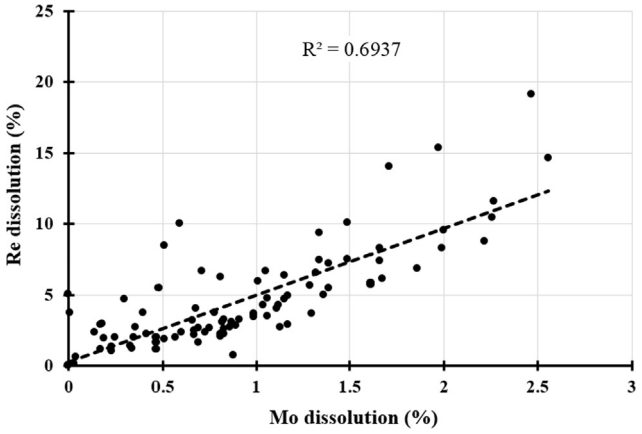


Fig. 7. Scatter plot of relative dissolution of Re and Mo in the mixed mesophilic bioleaching experiments (32 °C). All time course data were used to construct the plot.

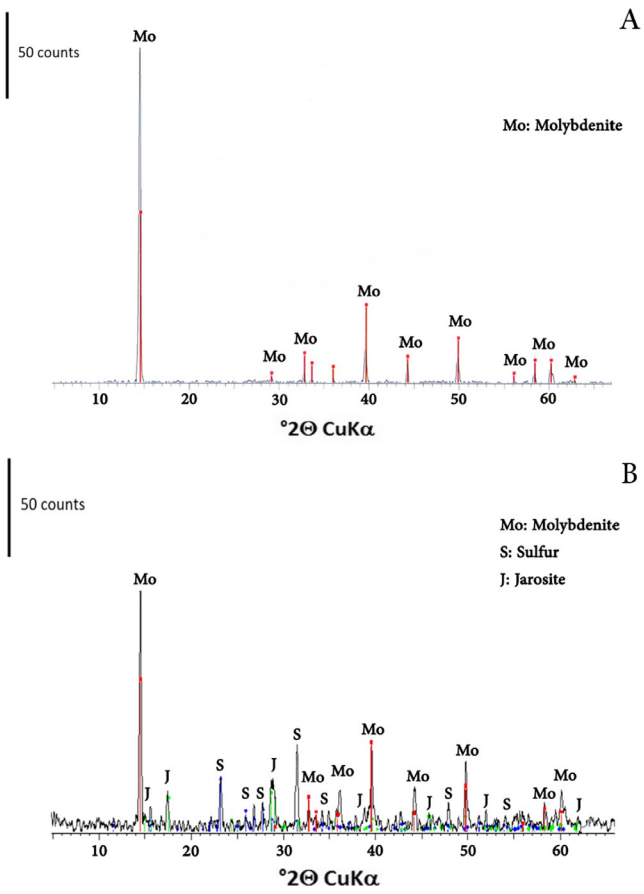


Fig. 8. XRD patterns of the molybdenite sample (A) original and (B) bioleached solid residue.

3.4. Analytical characterization: XRD & SEM studies

Following bioleaching of the molybdenite concentrate, the solid residues obtained from the leaching experiments were analyzed using XRD and its results revealed the presence of a considerable amount of jarosite and elemental sulfur. Occurrences of higher amount of jarosite and sulfur formation might be ascribed to the addition of Fe^{2+} and sulfur in the medium that lead to higher oxidation of the same by the microorganims with an increase in pH. Apart from that, sulfide mineral dissolution and release of iron and sulfur into the solution also partially

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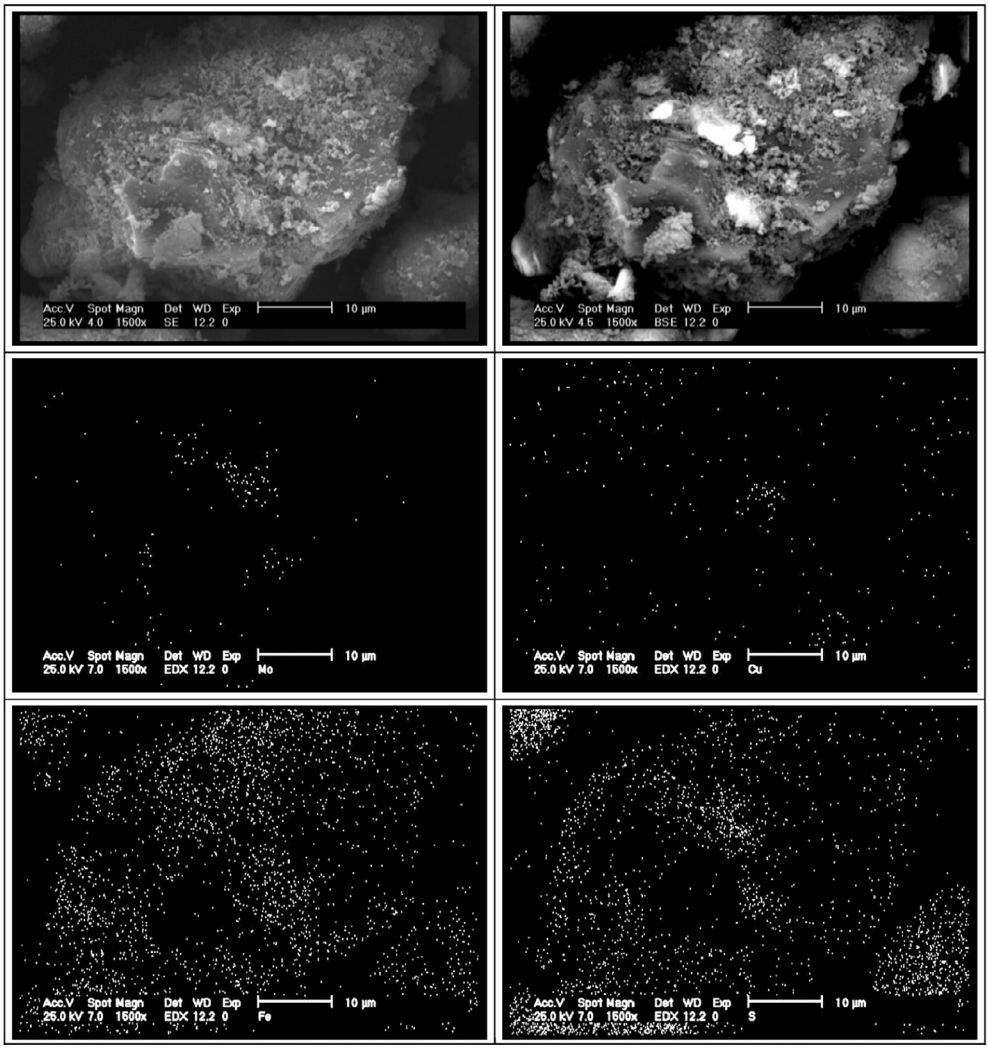


Fig. 9. SEM image of the molybdenite sample and EDAX analysis showing jarosite particles and sulfur on its surface.

contributes to the formation of jarosite and elemental sulfur. Jarosite and sulfur formation have negative effects on many applications, especially in the area of bio-hydrometallurgy. Jarosite and sulfur create kinetic barriers due to the minor diffusion of reactants and products through the precipitation zone (Dutrizac, 1999; Daoud and Karamanev, 2006; Panda et al., 2013; Sasaki et al., 2006, 2009). As a result, the jarosite and elemental sulfur produced during the process might have covered the surface of the minerals leading to low dissolution kinetics of the minerals (Diffusion control in the process). The XRD graph of the sample treated with mixed consortia of meso-acidophilic bacteria is shown in Fig. 8. Furthermore, the SEM images from solid residues confirmed the presence of jarosite and sulfur which were formed on the molybdenite surface. X-ray mapping revealed that most parts of particles were covered with S and Fe bearing components which has been shown in Fig. 9. Results showed that a highly oxidative environment and also the presence of Fe^{3+} formed during the process triggered the production of basic ferric hydroxy sulfates such as jarosite. Jarosite and sulfur formation had negative effects on the bioleaching process especially during molybdenum extraction. Jarosite and sulfur create kinetic barriers due to amino diffusion of reactants and products through the precipitation zone.

4. Conclusions

The application of feed-forward back-propagation ANN for modeling the effects of operational parameters on the dissolution of Cu, Mo and Re from a molybdenite concentrate using meso-acidophilic bioleaching approach was studied. The predicted values obtained by neural network corresponded closely to the experimental results and quite satisfactory correlations (more that 99%) for training, testing and validation stages of Cu, Mo and Re recoveries were achieved. The developed neural network models accurately reproduced all the effects of operational variables and could be used in modeling of the bioleaching of molybdenite concentrate for extraction of Cu, Mo and Re. The most appropriate architectures for Cu, Mo and Re were 4-5-5-2-1, 4-7-5-2-1 and 4-7-1-1-1; respectively, which were trained using the Levenberg–Marquardt algorithm. The results have shown good potential of ANN for predicting the Cu, Mo and Re dissolution from the Mo-concentrate in the meso-acidophilic bioleaching tests. Thus, the ANN model developed in this study can provide fast and reliable predictions of Cu, Mo and Re dissolution. Furthermore, genetic algorithm was used to find the optimum combination of the operational parameters (Input) yielding the maximum recoveries by exploring through the neural network which developed at the previous step. In this regard, GA found the maximum Cu, Mo and Re recoveries to be obtained on 30th day with as high as values of 72.99%, 2.83% and 27.17%; respectively. In comparison to statistical methods such as RSM and Taghouchi, the key advantage of optimal combination of operational parameters obtained by proposed method of modeling via ANN and then optimizing by GA is that these value are more reliable to be the optimum operational

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parameters. Such a remarkable reliability stems from the broader and denser sampling coverage of operational parameters by experimental data. This coverage lets the ANN model to provide reliable knowledge about a broad range of operational parameters. As a result, the optimum point found in this model is more reliable. However, the situation is different in statistical approaches. The basic idea of these approaches is to reach optimal performance of the process through minimum number of experiments. Being blind about a broad range of operational parameters, such an estimated optimal performance might be far from the true optimum point. Also, the jarosite formation during the bioleaching test was confirmed by XRD and SEM analyses which caused the reduction of bioleaching progress.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.mineng.2018.10.008.

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