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GOLD REMOVAL FROM GOLD-BEARING ORE USING ALPHA-CYCLODEXTRIN: RESPONSE SURFACE METHODOLOGY AND ARTIFICIAL NEURAL ANALYSIS NETWORK OPTIMIZATIONS

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ABSTRACT

This paper explores the use of alpha cyclodextrin (α-CD) for gold extraction from gold-bearing ore in the Democratic Republic of Congo (D.R.C). The research aims to identify the optimal gold removal conditions using Response Surface Methodology (RSM) and Artificial Neural Networks (ANN). Initially, ore samples were collected and processed to enhance leachability by reducing particle size. The leaching process employed a modified aqua regia with hydrobromic acid due to the strong molecular recognition between gold bromide ion and α-CD. Various leaching parameters, such as time, HBr concentration, pH, and stirring speed, were adjusted during experimentation. RSM yielded optimal values of 7.27 hours, 50 g/L, 1, and 200 rpm, resulting in 98.54% gold removal, while ANN produced slightly lower values of 7.5 hours, 50 g/L, 1, and 200 rpm, with 97.16% gold removal. For α-CD gold recovery, RSM and ANN optimization resulted in 40 minutes, 11.61 g/L, and pH 5, achieving 98.87% and 39.8 minutes, 10.71 g/L, and pH 5, with 99.98% gold removal, respectively. Validation tests supported these findings, indicating that RSM and ANN are successful methods for optimizing gold recovery from gold-bearing ore in the D.R.C.

I. INTRODUCTION

The concentration of the metal being extracted is a determining factor in the profitability of mining a specific ore, while the physical and chemical characteristics of gold affect the methods used for its processing. In the Democratic Republic of the Congo (D.R.C.), the basement is rich in many minerals which contain many metals in different proportions [1-2]. Gold is mainly found in the South Kivu and Ituri provinces, where artisanal mining is prevalent. Child labor is common in the industry and the D.R.C. government has ratified the International Labour Organization's Convention on the Worst Forms of Child Labour, and more needs to be done to eliminate child labor in the mining industry [3-4].

Gold mining is a complex process that involves exploration, processing, development, and extraction, among other steps. According to [5], gold is typically found in concentrated deposits, which makes mining more viable. The authors also note that there are three main methods of gold mining: open-pit mining, underground mining, and artisanal mining. Open-pit mining is used when the ore deposit extends deep into the ground, and it involves the removal of layers of overburden and ore. Underground mining, on the other hand, is used for deeper deposits and may involve the use of supports. As noted by [6], artisanal mining typically involves manual extraction and concentration of minerals, including gold, often using traditional methods. Mechanized artisanal mining, on the other hand, involves the use of machines like hand tools, motorized water pumps, and crushers, among others [7]. Once the gold has been extracted, it needs to be processed and refined. Other methods of concentrations are used to concentrate minerals which contain metals for further processes [8]. According to [9], the methods of gold concentration from the ore can include gravimetric methods, such as sluices, shaking tables, and centrifuges, which rely on the high density of gold to separate it from other minerals in the ore.

Gold mining has a significant impact on the global economy, and according to [10], the global gold mining industry produces about 2,500-3,000 tons of gold per year, with the largest producing countries being China, Australia, Russia, and the United States. This industry is a major contributor to the economy of many countries and the methods of mining gold have evolved over time, as noted by Wang and Forssberg [11]. However, gold mining also has both positive and negative impacts on the environment and society. On one hand, it can create jobs and economic growth, especially in developing countries [12]. On the other hand, it can lead to environmental degradation, including deforestation, pollution of water sources, and destruction of habitats [13]. Additionally, the social impacts of gold mining can be significant. Reports of human rights abuses, labor exploitation, and child labor in some mining communities have been documented [14], so that efforts are being made to address these issues through international regulations and corporate social responsibility initiatives, as noted by Murillo-Sánchez and Reyes-Santacruz [15].

According to a study by [16], gold extraction involves several stages, including leaching, extraction, refining, and electrolysis. The commonly used method for gold extraction is the cyanide leaching process, where a cyanide solution is used to dissolve the gold from the ore in tanks or heaps [17]. Gold is then recovered from the saturated leaching solution by adsorption onto activated carbon or resins [18]. The two main methods used for the recovery of gold from the cyanide solution are precipitation on zinc powder (cementation) and adsorption on activated carbon [18]. Cementation on zinc powder involves the precipitation of gold from the enriched cyanide solution onto zinc powder. The complete process involves solid-liquid separation after cyanidation, clarification of the gold solution, partial deaeration of the solution, addition of zinc powder and lead salt to improve gold precipitation, and recovery of the precipitated gold [17]. Adsorption on activated carbon is based on the property of activated carbon to absorb the gold in the cyanide solution. The carbon is prepared from natural hard carbon (fruit kernels, coconut shells) treated to develop its adsorption capacity and porosity. The gold solution is pumped to the adsorption columns, where activated carbon is added to adsorb the gold-cyanide complex [18]. After gold recovery, the refining stage is carried out to obtain the highest practical purity of gold. The process begins with washing the carbon to remove organic impurities, and then the gold adsorbed on the carbon is recovered by elution using a hot, caustic, aqueous cyanide solution. Impurities are removed by filtration through a bed of activated carbon. The regenerated carbon is returned to the adsorption circuit, where gold is recovered either by cementation on zinc or by electrowinning, which is the final stage of the gold extraction process involving the deposition of gold on the cathode after taking an electron to become metallic gold. The cathode is made of steel wool, and the anode is made of lead to facilitate oxygen release with minimal over potential [17].

Some authors have studied the thiourea process as an alternative to cyanide for the extraction of gold due to its environmental and health benefits, but the significant consumption of the reagent related to its oxidation in the solution is a disadvantage. To reduce this consumption, other authors investigated the mixture of thiourea with thiocyanate and iron sulfate [19-20]. However, recent developments aim to improve the process's cost-effectiveness and efficiency by reducing reagent consumption [21].

The [22] investigated the use of the chitosan and the manipuera processes, respectively. They have shown that chitosan can form complexes with gold after chlorination as shown in equation 1.

$$2Au + 2KClO_3 + 8HCl \rightarrow 2AuCl_4 + 2KCl + 4H_2O + O_2 \quad (1)$$

Aqua regia (AR) process consists of a mixture of hydrochloric acid (HCl) and nitric acid (HNO_3) in a 3/1 ratio that can dissolve gold in a matter of hours (equation 2). However, this process is highly corrosive and unstable and is not widely used at an industrial scale, limited to small and medium-sized processes, such as electrolytes in gold refining or recovering gold from electronic waste. Aqua regia can also dissolve other noble metals such as platinum and rhodium [23-24].

$$Au + HNO_3 + 4HCl \rightarrow HAuCl_4 + NO + 2H_2O \quad (2)$$

It should be noted that for the purpose of dissolving gold, hydrobromic acid (HBr) is used instead of HCl in this paper to create a modified aqua regia (MAR). The equation 3 becomes equation 4 as follows:

$$Au + HNO_3 + 4\,HBr \rightarrow HAuBr_4 + NO + 2H_2O \quad (3)$$

Efforts have been made in recent years to replace mercury and cyanide with less toxic reagents, and advancements in technology have led to the development of more efficient and environmentally friendly methods. Out of the various alternatives, the cyclodextrin (CD) process has emerged as a noteworthy contender. As reported by [25], this process utilizes a cyclic sugar molecule known as α-CD, which demonstrates selectivity in extracting gold from the ore. Moreover, α-CD is a cyclic oligosaccharide that has been shown to be effective in gold recovery [26], it is biodegradable, non-toxic, and has a high affinity for gold ions, making it an attractive alternative to conventional gold recovery methods [27]. The efficiency of α-CD in gold recovery has been investigated in electronic waste [28], low-grade gold ores [29], and even in refractory gold ores [30]. Moreover, that α-CD efficiency in gold recovery is attributed to its ability to form inclusion complexes with gold ions [31]. The formation of inclusion complexes is based on the hydrophobic effect and the van der Waals forces between α-CD and gold ions [29]. This complexation process can lead to the removal of gold ions from the ore, resulting in high gold recovery rates. So that α-CD was found to be an effective extractant for gold recovery, and it offers several advantages over conventional extractants, such as cyanide.

To date, a number of studies have been carried out on the use of α-CD in gold recovery. For example, Yen et al. [32] investigated the effect of various factors, such as temperature, pH, and α-CD concentration, on the recovery of gold from electronic waste. They found that α-CD could effectively recover gold under a range of conditions. Similarly, Lee et al. [33] used α-CD to recover gold from waste printed circuit boards, and they reported a recovery rate of 98.5%. Despite its potential advantages, the use of α-CD in gold recovery is still in the early stages of development. Further research is needed to optimize the gold recovery process using α-CD and to understand the underlying mechanisms involved in the process.

The aim of this study is to investigate the efficiency of α-CD in modified aqua regia (MAR) gold recovery from gold-bearing ore and optimize the process using the simulation, optimization and prediction methods including Response Surface Methodology (RSM) and Artificial Neural Network (ANN), and then compare the results to find the optimum conditions. The ore will be characterized and series of experiments will be carried out. The significant parameters for leaching, neutralization and gold recovery processes such as time, pH, HBr and KOH concentrations ([HBr] and [KOH]), and α-CD concentration [α-CD], and their interactions will be also explored to determine the optimum conditions. After the experiments, the results will be analyses and the efficiency of the gold recovery process will then be evaluated by measuring the percentage of gold recovered from the ore. The findings of this study may have significant implications for the gold mining industry, particularly in the D.R.C., where the recovery of gold from low-grade ores is an important economic activity. Also, the use of α-CD as a lixiviant aims to reduce the environmental impact of gold mining and promote the application of green chemistry principles in the mining industry.

II. MATERIALS AND METHODS

II.1 MATERIAL COLLECTION

In this study, concentrated HBr and HNO_3 were used to form MAR (3/1 ratio) for gold leaching. α-CD at 99% was used as gold the extracting agent. Potassium hydroxide (KOH) was used for pH adjustment, and analytical-grade nitric acid (HNO_3) was used for gold analysis.

II.2 SAMPLE PREPARATION

Fifty (50) kg of gold-bearing ore was collected from 2 quarries located in South-Kivu province of the D.R.C. The homogenization of the sample was carried out using the cone and quartering method, and then divided into four representative parts using the quartering technique. A number of fragmentation operations were performed: primary and secondary crushing, a mineralogical analysis using transmitted and reflected light microscopy, and a chemical analysis using atomic adsorption spectroscopy/inductively coupled plasma (AAS/ICP). Moreover, 1 kg was used for AAS/ICP, and the remaining 49 kg gold ore sample was used for secondary crushing during 19.57 minutes in to get 80% passing 75 μm sieves.

III. GOLD RECOVERY STUDY

II.1 LEACHING PROCESS (LP)

The purpose of the process was to extract gold from a homogenized sample and 400 g of the sample was weighed and placed into a 1 L beaker for 5 min of contact time. Deionized water was added to the beaker to homogenize the pulp, and mechanical agitation was used to facilitate the process. HBr and HNO_3 were gradually added to the pulp while the pH and potential were adjusted as needed. The solid and liquid phases were then separated by filtration, and a certain amount of the filtrate was taken for chemical analysis. The cake was washed to eliminate any remaining leaching agent before being dried in an oven at a temperature of 105ºC for 24 hours. To ensure the precision in results, the leaching tests were duplicated and the average values were considered. Four factors were studied: time (x_1), HBr concentration (x_2), pH (x_3) and stirring speed (x_4), and a total of 47 tests were carried out by simultaneously varying the levels of each factor. Time, HBr concentration [HBr], pH, and stirring speed were varied in range of 1-7.5 hours, 50-200 g/L, 1-2, and 50-200 rpm, respectively.

II.2 NEUTRALIZATION PROCESS (NP)

The experimental procedure used for neutralization tests involved placing 50 mL of homogenized leachate in a 500 mL beaker, gradually adding a solution of KOH (0.1 M) to maintain the pH at required pH, stopping stirring once the set time was reached, separating the solid and liquid components through filtration, and finally analyzing 20 mL of the resulting filtrate to measure its chemical composition. The tests were triplicated and the average values were considered. Moreover, the following three factors with appropriate combination were studied: time (x_1), KOH concentration (x_2), and pH (x_3). Time, KOH concentration, and pH were varied in range of 1-10 hours, 84-224 g/L, and 5-6, respectively.

II.3 GOLD RECOVERY PROCESS USING α-CD (GP)

During the precipitation tests at room temperature (25°C), the process started with an amount of 50 mL of the filtrate which was taken into a 2 L beaker after ensuring the pH was at the desired level using KOH. α-CD was then gradually added while mechanically agitating the solution. Once the set time was reached, the recovered gold as well as the co-precipitates were removed by filtration, after what sodium bisulfite ($Na_2S_2O_5$) solution was used to disperse and reduce the complex α-Br in order isolate and precipitate the recovered gold metal. The liquid-liquid separation was carried out by decantation and a certain amount of the filtrate was collected for chemical analysis. Each recovery test was duplicated and the average value was considered. Time, α-CD concentration [α-CD], and pH were varied in range of 10-40 minutes, 5-20 g/L, and 5-6, respectively.

II.4 METHODS USED FOR PREDICTION, MODELING AND OPTIMIZATION OF STUDIED PARAMETERS

II.4.1 RSM METHOD

RSM known as full factorial method, was utilized to generate the experimental runs (using Minitab software and the analysis of variance (ANOVA)) varying the studied parameters [34-35]. In addition, the statistical technique of regression analysis was highlighted to determine a mathematical formula between the parameters and the response as shown in equation 4 [36-37].

$$y = f(x_1, x_2, x_3, x_4, \dots, x_n) \quad (4)$$

where y is the dependent variable (response) and x_i the independent variables (parameters).

As demonstrated by Murthy et al. [38], for a good fitness of the model, the coefficient of determination R^2 should be at least 0.80. The best precision of comparison of treatments was done by a value called coefficient of variation (CV) which should be low to minimize the dispersion around the mean value.

As a statistical indicator, the Fisher's F-test ANOVA was used to outline the significance of the studied model after assessing the effects of the parameters as well as their interactions. [36] and [39] have shown that for a highly significant model, variable or interaction, the P-value must be lower than greater than 0.0001 in a 95% confidence intervals. Also, the significance was associated to a P-value between 0.0001 and 0.05, while the non-significance was associated to a P-value higher than 0.05.

The experimental design matrix for leaching process (LP) and gold recovery process using α-CD (GP) tests are presented in Table 1, while the corresponding matrix for neutralization process (NP) is shown in Table 2.

Table 1: Design matrix for LP and GR.

Run N°	**LP**				**GP**		
	Time, x_1 (h)	**[HBr], x_2 (g/L)**	**pH, x_3**	**Speed, x_4 (rpm)**	**Time, x_1 (min)**	**[α-CD], x_2 (g/L)**	**pH, x_3**
1	1.0	50	1	200	10	5	5.0
2	1.0	200	2	50	10	5	5.5
3	1.0	100	1	50	10	5	6.0
4	1.0	150	1	50	10	10	5.0
5	1.0	100	1	150	10	10	5.5
6	1.0	150	1	150	10	10	6.0
7	1.0	100	2	150	10	15	5.0
8	1.0	150	2	150	10	15	5.5
9	1.0	100	2	100	10	15	6.0
10	1.0	150	2	100	10	20	5.0
11	2.5	100	1	100	10	20	5.5
12	2.5	150	1	100	10	20	6.0
13	2.5	200	1	150	20	5	5.0
14	2.5	50	1	150	20	5	5.5
15	2.5	200	1	150	20	5	6.0
16	2.5	100	1	150	20	10	5.0
17	2.5	150	1	150	20	10	5.5
18	2.5	200	1	100	20	10	6.0
19	2.5	100	2	150	20	15	5.0
20	2.5	150	2	150	20	15	5.5
21	2.5	200	2	100	20	15	6.0
22	2.5	100	2	100	20	20	5.0
23	2.5	150	2	100	20	20	5.5
24	2.5	50	2	50	20	20	6.0
25	2.5	100	2	50	30	5	5.0
26	2.5	150	2	50	30	5	5.5
27	2.5	200	2	50	30	5	6.0
28	5.0	50	1	150	30	10	5.0
29	5.0	100	1	150	30	10	5.5
30	5.0	150	1	150	30	10	6.0
31	5.0	200	1	150	30	15	5.0
32	5.0	200	1	100	30	15	5.5
33	5.0	50	2	100	30	15	6.0
34	5.0	100	2	100	30	20	5.0
35	5.0	150	2	100	30	20	5.5
36	5.0	200	2	100	30	20	6.0
37	5.0	50	2	50	40	5	5.0
38	7.5	50	1	150	40	5	5.5
39	7.5	100	1	150	40	5	6.0
40	7.5	200	1	150	40	10	5.0
41	7.5	100	1	200	40	10	5.5
42	7.5	150	1	200	40	10	6.0
43	7.5	100	2	100	40	15	5.0
44	7.5	150	2	100	40	15	5.5
45	7.5	150	1	150	40	15	6.0
46	7.5	100	2	50	40	20	5.0
47	7.5	150	2	50	40	20	5.5
48	-	-	-	-	40	20	6.0

Source: Authors, (2023).

Table 2: Design matrix for NP.

Run N°	Time, x_1 (h)	[KOH], x_2 (g/L)	pH, x_3
1	1.0	84	5.0
2	1.0	84	5.5
3	1.0	84	6.0
4	1.0	112	5.0
5	1.0	112	5.5
6	1.0	112	6.0
7	1.0	168	5.0
8	1.0	168	6.0
9	2.5	84	5.0
10	2.5	84	5.5
11	2.5	84	6.0
12	2.5	112	5.0
13	2.5	112	5.5
14	2.5	112	6.0
15	2.5	168	5.0
16	2.5	168	5.5
17	2.5	168	6.0
18	2.5	224	5.5
19	2.5	224	6.0
20	5.0	84	5.0
21	5.0	84	5.5
22	5.0	84	6.0
23	10.0	112	5.0
24	10.0	112	5.5
25	10.0	112	6.0

Source: Authors, (2023).

A total of 47, 48, and 25 experimental runs were considered for LP, GR, and NP, respectively.

II.4.2 ANN METHOD

Known as promising method studied by many researchers [40-42], ANN as a statistical learning algorithms has been used in recent years in order to estimate by approximation a mathematical relationship between inputs and outputs of a specific process. Through information, the stimulation as well as the processing of ANN are performed within the nervous system.

The transfer functions between the input and hidden layers in one hand, and between the hidden and output layers on another hand, were performed using a hyperbolic tangent TANSIG (sigmoid) and a Purline, respectively, while the normalization of data of inputs and outputs was done by PREMNMX function [43-44]. In addition, the chosen learning algorithm was Levenberg-Marquardt. Beside the coefficient of determination (R^2), another estimator or predictor number called mean square error (MSE) was used to determine the performance of ANN. For a good performance, MSE value should be as small as possible and in the best case close to zero. Furthermore a good performance of the model corresponds to a high R^2 value and a lower MSE value which shows a good interaction or connection between the parameters of the model. Equations 5, 6 and 7 illustrate the sigmoid, MSE, and R^2, respectively.

$$logsig(x) = \frac{1}{1+e^{-x}} \tag{5}$$

$$MSE = \frac{1}{N} + \sum_{i=1}^{N}(t_i - o_i)^2 \tag{6}$$

$$R^2 = 1 - \frac{\sum(t_i - o_i)^2}{\sum(o_i - \bar{o})^2} \tag{7}$$

where n, t, o, and ō represent the number of samples, the target value, the output, ant the mean of output, respectively.

In this paper, the RSM and MATLAB were used to generate a matrix of the experimental responses. The choice of learning on multilayer perception network was 80 for network training, 5 for testing, and 5 for validation. The network was selected in feed-forward mode, consisting of input layer, hidden layer and output layer. Depending on the process (LP or GP), the numbers of neurons in input and output layers were specific, while the chosen number of neurons in the hidden layer was 10 due to the lowest mean square error resulted. Figure 1 illustrates the ANN architecture used for the experiments.

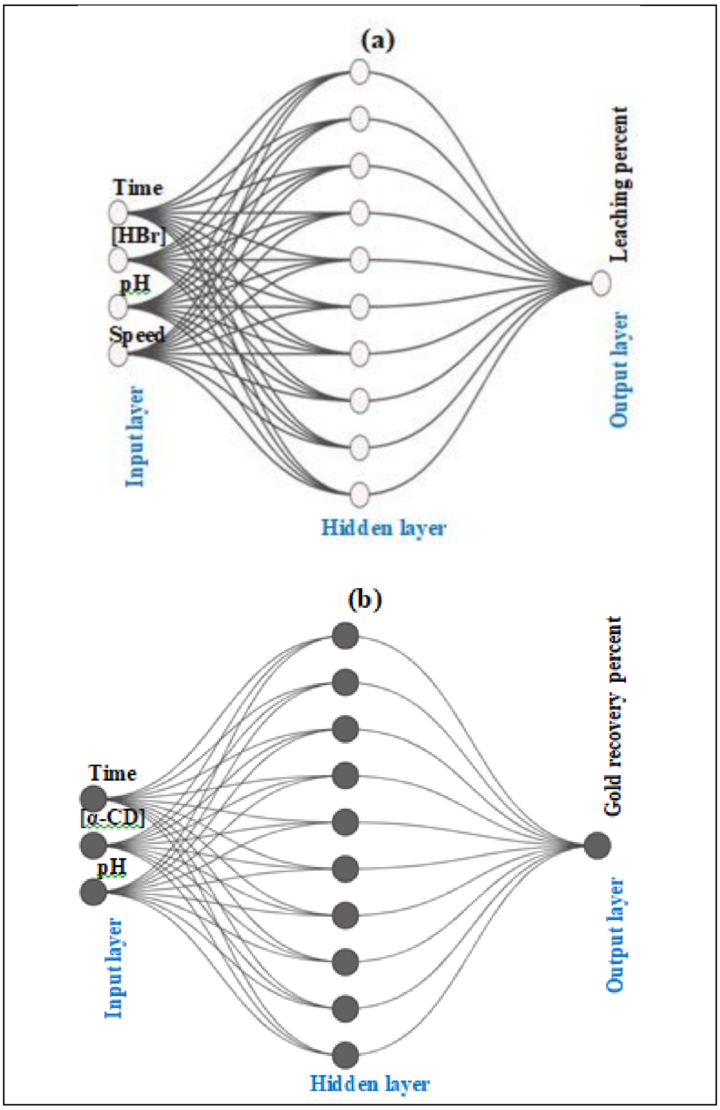

Figure 1: ANN architecture, (a): for LP and (b): for GP.
Source: Authors, (2023).

IV. RESULTS AND DISCUSSIONS

IV.1 RSM PREDICTION

The results of percent removals according to each process (LP, NP and GR) are investigated in this section. Note that these results were also obtained in another study conducted by the same authors of the present article taking into account central composite design (CCD), and the study has not yet been published. Table 3 presents the results of percent removals of the tests.

Table 3: Responses obtained for LP, NP and GR.

Run N°	Responses (%)		
	LP	NP	GR
1	0.001	54.4698	35.8545
2	67.879	65.1321	48.9586
3	33.927	73.7055	62.0582
4	70.399	40.9167	54.9625
5	36.432	51.6038	65.7851
6	72.815	60.2019	76.6032
7	36.432	18.4140	51.0053
8	56.121	37.7190	59.5463
9	20.617	70.0920	68.0830
10	57.042	77.7318	23.9828
11	48.654	83.3085	30.2424
12	68.595	67.7952	36.4973
13	86.060	75.4380	55.9201
14	26.361	81.0216	64.0012
15	86.060	67.7655	72.0779
16	49.990	75.4123	70.8062
17	69.890	80.9820	76.6058
18	84.806	81.4473	82.4011
19	35.271	87.0210	62.6271
20	55.170	79.0020	68.1253
21	70.206	81.5678	69.6590
22	34.054	82.1601	31.3830
23	53.995	86.4270	32.6146
24	9.061	78.7644	33.8518
25	32.773	69.0426	75.9856
26	52.755	-	79.0438
27	69.008	-	82.0976
28	73.270	-	86.6499
29	69.430	-	87.4266
30	61.855	-	88.1989
31	50.550	-	74.2500
32	49.104	-	72.7442
33	57.103	-	71.2351
34	53.301	-	38.7829
35	45.767	-	34.9965
36	34.504	-	31.2059
37	55.589	-	96.0508
38	83.120	-	94.0864
39	85.002	-	92.1173
40	11.107	-	98.8911
41	86.595	-	98.2474
42	51.502	-	93.9968
43	68.611	-	85.8708
44	33.604	-	79.3432
45	90.479	-	72.8130
46	66.944	-	46.1829
47	31.977	-	37.3737
48	-	-	28.5601

Source: Authors, (2023).

After obtaining the results of percent removals, the influences of studied parameters, as well as their interactions, on percent removals were evaluated through the generation of mathematical equations 8, 9, and 10 for LP, NP, and GR, respectively. The importance of the sign of the coefficient in the regression equation is in the fact that it can predict the nature of the studied parameters effects on the response. So that the positive sign of the coefficient implies the favorable increase of the response with the increase in parameters (variables) or their interactions, and vice versa. Moreover, the negative sign of the coefficient negative coefficients implies the decrease of the response with the increase in parameters (variables) or their interactions.

$$y = -91.3 + 31.74x_1 + 1.404x_2 - 4.0x_3 + 0.040x_4 - 0.465x_1^2 - 0.001773x_2^2 + 0.000097x_4^2 - 0.1860x_1x_2 - 0.96x_1x_3 - 0.0090x_1x_4 - 0.0740x_2x_3 - 0.000141x_2x_4 + 0.0139x_3x_4 \quad (8)$$

$$y = -128.60 + 14.3489x_1 - 52.627x_2 + 68.667x_3 - 1.71718x_1^2 + 3.0403x_2^2 - 4.1153x_3^2 + 14.9734x_1x_2 - 4.07045x_1x_3 - 0.0077x_2x_3 \quad (9)$$

$$y = -262.2 + 7.343x_1 + 16.209x_2 + 51.06x_3 - 0.001162x_1^2 - 0.45996x_2^2 - 0.953x_3^2 - 0.08385x_1x_2 - 0.9775x_1x_3 - 0.9306x_2x_3 \quad (10)$$

where y, x_1, x_2, x_3, and x_4 have already been defined in Table 1.

The leaching efficiency is directly proportional to the increase in time, [HBr], stirring speed, and inversely proportional to the pH. The findings support [45] study, which concluded that the presence of HNO_3 as an oxidizing agent aids in dissolving gold. Thus, the higher concentration of HBr leads to an increase in HNO_3 concentration, which effectively acts as an oxidant for trivalent gold ions. The trivalent gold ions are subsequently reduced by HBr, resulting in the formation of tetrabromidoaurate anions or tetrabromoauric acid ($AuBr_4^-$).

The results indicate that when the reaction time is shorter, even with varying concentrations of HBr, the contact between the gold particles and leachate is insufficient, leading to the consumption of other metals present in the solution by HBr. Also, an increase in HBr concentration is favorable for the formation and precipitation of silver bromide (AgBr), confirming the syudy of [46]. This, in turn, prevents the dissolution of silver from MAR and results in an increased leaching percentage.

The results indicate that increasing the time, [KOH], and pH leads to an increase in the neutralization percentage, with an optimal pH range of 5-6. This is due to the precipitation of other metals that typically accompany gold, such as iron, aluminum, copper, and cobalt, with increasing pH or [KOH]. The ionic forms of these metals react with hydroxide ions from KOH and subsequently precipitate out. Another factor is the progressive oxidation of Fe^{2+} to Fe^{3+} by oxygen in the air, which leads to precipitation in the form of $Fe(OH)_3$, starting at a pH of 3.5, as confirmed by [47]. Additionally, aluminum precipitation occurs within the pH range of 4 to 5.5. The precipitation processes for aluminum and iron ions are illustrated by equations 11 and 12, respectively.

$$Al^{3+} + OH^- \rightarrow Al(OH)_3 \quad (11)$$

$$Fe^{3+} + OH^- \rightarrow Fe(OH)_3 \quad (12)$$

According to that, it can be observed that the increase of [α-CD] leads to the increase of percent removal but not indefinitely. So that, [α-CD] reaches a certain value (11.62 g/L) after which the opposite phenomenon is observed. This may be attributed to the saturation of α-CD with solute at this value. Additionally, the recovery was observed from the first minute of reaction and with the increase of contact time, while at a pH range of 5-5.8 yields good results with at least 85% of gold recovered.

Furthermore, the yield of recovery is low at low values of pH and time, but increases as these parameters values increase.

Gold recovery is a rapid process that commences within the first minute of the reaction between the extractant solution (α-CD) and the gold-containing solution ($KAuBr_4$). With time, $AuBr_4^-$ ions become encapsulated within the second sphere cavity formed between the primary OH faces of repeated face-to-face α-CD pairs. These pairs are stabilized by hydrogen bonding interactions, leading to an increase in recovery percentage, as demonstrated by [48]. On another hand, K^+, α-CD, and $AuBr_4^-$ molecules recognize each other, involving aureate anion $[AuBr_4]^-$ and hexa-aqua cation $[K(OH_2)_6]^+$ interacting non-covalently. This results in rapid co-precipitation of α-CD and KAuBr4 in water, eventually forming a 1:2 ratio complex (needlelike) $\{[K(OH_2)_6][AuBr_4]\subset(\alpha\text{-CD})_2\}_n$, as concluded by Liu and Wang [39].

As α-CD concentration increases, recovery percentage also increases up to a maximum value before decreasing. Beyond this point, encapsulation ceases, and needlelike complex formation no longer occurs due to the absence of $KAuBr_4$ solution. The optimal ratio for complex formation is 1:2, where $[AuBr_4]/[\alpha\text{-CD}]=1/2$. Performance decreases when the ratio falls below or exceeds this threshold. The neutralization of accompanying metals of gold during neutralization step plays an important role in increasing percent removal in gold recovery process.

ANOVA was investigated and some indicator values were found to analyse the model performance, such as R^2 and adjusted R^2 values, P-values, degree of freedom (df), and so on. The confidence level was 95%. Tables 4 and 5 show the results of ANOVA, and according to the results, it is obvious that: (1) for LP, the model, the time, as well as the time-[HBr] interaction are highly significant. In addition, the pH is significant and the remaining parameters ([HBr] and speed) are non-significant. (2) for GP, the model and all the parameters are highly significant.

Table 4: Analysis of variance for LP percentage.

Source	**df**	**Sum of Square**	**Mean Square**	**F-Value**	**P-Value Prob>F**	**Observations**
Model	4	3750.4	937.6	13.88	<0.0001	HS
x_1	1	792.3	792.3	11.73	<0.0001	HS
x_2	1	141.0	141.0	2.09	0.158	NS
x_3	1	909.9	909.9	13.47	0.001	S
x_4	1	24.3	24.3	0.36	0.553	NS
x_1^2	1	131.5	131.5	1.95	0.172	NS
x_2^2	1	631.7	631.7	9.35	0.004	S
x_4^2	1	0.8	0.8	0.01	0.913	NS
x_1x_2	1	14451.2	14451.2	213.89	<0.0001	HS
x_1x_3	1	21.8	21.8	0.32	0.574	NS
x_1x_4	1	17.3	17.3	0.26	0.616	NS
x_2x_3	1	61.4	61.4	0.91	0.347	NS
x_2x_4	1	1.8	1.8	0.03	0.873	NS
x_3x_4	1	1.5	1.5	0.02	0.882	NS
Error	33	2229.6	67.6			
Total	46	22365.2				

Note: F: function of Fishers, df: degrees of freedom, P-value: level of significance, NS: not significant; S: significant; HS: highly significant; CV=10.7259%; R^2=0.9003; Adj. R^2=0.7825.

Source: Authors, (2023).

Table 5: Analysis of variance for GR percentage.

Source	**df**	**Sum of Square**	**Mean Square**	**F-Value**	**P-Value Prob>F**	**Observations**
Model	3	14016.9	4672.28	13195.92	<0.0001	HS
x_1	1	4441.1	4441.10	12542.97	<0.0001	HS
x_2	1	9412.6	9412.63	26584.06	<0.0001	HS
x_3	1	163.1	163.13	460.73	<0.0001	HS
x_1^2	1	0.6	0.65	1.83	0.184	NS
x_2^2	1	6346.9	6346.87	17925.45	<0.0001	HS
x_3^2	1	0.6	0.60	1.71	0.199	NS
x_1x_2	1	1318.2	1318.17	3722.90	<0.0001	HS
x_1x_3	1	955.6	955.60	2698.90	<0.0001	HS
x_2x_3	1	216.5	216.51	611.50	<0.0001	HS
Error	38	13.5	0.35			
Total	47	22868.7				

Note: F: function of Fishers, df: degrees of freedom, P-value: level of significance, NS: not significant; S: significant; HS: highly significant; CV=0.82515%; R^2=0.9994; Adj. R^2=0.9993.

Source: Authors, (2023).

The results of Tables 4 and 5 indicate that the R^2 values of 0.9003 and 0.9994 for LP and GP respectively were highly significant confirming a good reliability, reproducibility and precision of the models. The CVs of 10.7259% and 0.82515% for leaching and gold recovery respectively were also obtained. In addition, the obtained P-value <0.0001 for both models confirm that the good prediction of the gold removal varying the parameters studied.

The optimization conditions were found for better gold removal in both LP and GR. From the results, the conditions were (1) time of 7.27 hours, [HBr] of 50 g/L, pH of 1 and stirring speed of 200 rpm for leaching percent of 98.540% and desirability of 1, (2) time of 40 minutes, [α-CD] of 11.6166 g/L and pH of 5 for gold recovery percent of 98.8736% and desirability of 0.99978. Under these conditions, the concentration of gold increased from 592.99 ppm to 3,372 ppm in the filtrate after LP, while it increased from 3,372 ppm to 11,944 ppm in the organic phase after GR.

IV.2 ANN PREDICTION

According to Table 3, the prediction of the response was done using ANN, and 75%-15%-15% learning choice was done for network training, testing and validation, respectively. The illustration of the observed mean square error (MSE) against the epochs for both LP and GP is given in Figure 2. According to [49], MSE also give information on the degree of correlation between the target and the predicted responses of the network.

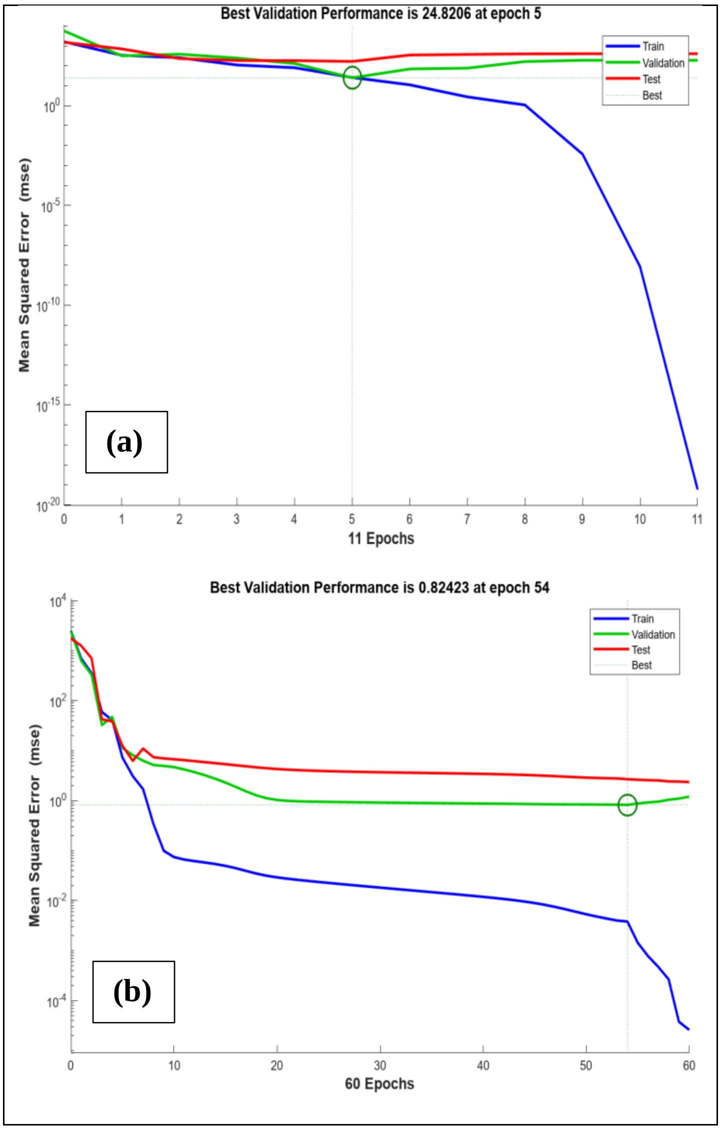

Figure 2: Performance of training, validation and testing (a) for LP, (b) for GR.
Source: Authors, (2023).

For LP (figure 2.a), it can be observed that for validation and testing there are steady decreases in MSE up to epoch 5, from where there is certain equilibrium up to epoch 11. In addition there is a regular decrease in MSE for training up to epoch 8 and then a sharp decrease is observed up to epoch 11. The best value of MSE is 24.8206 at epoch 5 involving the best performance and suggesting that the learning stage of the network at its best level is obtained after the fifth iterations. For GP (figure 2.b), MSE declines for training, validation and testing up to a certain equilibrium around epoch 9. Therefore, after 54 iterations, the best learning stage of the network is attained at MSE value of 0.8242 confirming a good correlation between gold recovery and the target. The error histograms of both LP and GP are presented in Figure 3 (a and b). It is seen that the errors are almost symmetrically distributed, so that they have a non-significant effect on the studied model. Furthermore, it is obvious that for GP, the mean value of the instances is closed to 5, which confirms the good performance of the model.

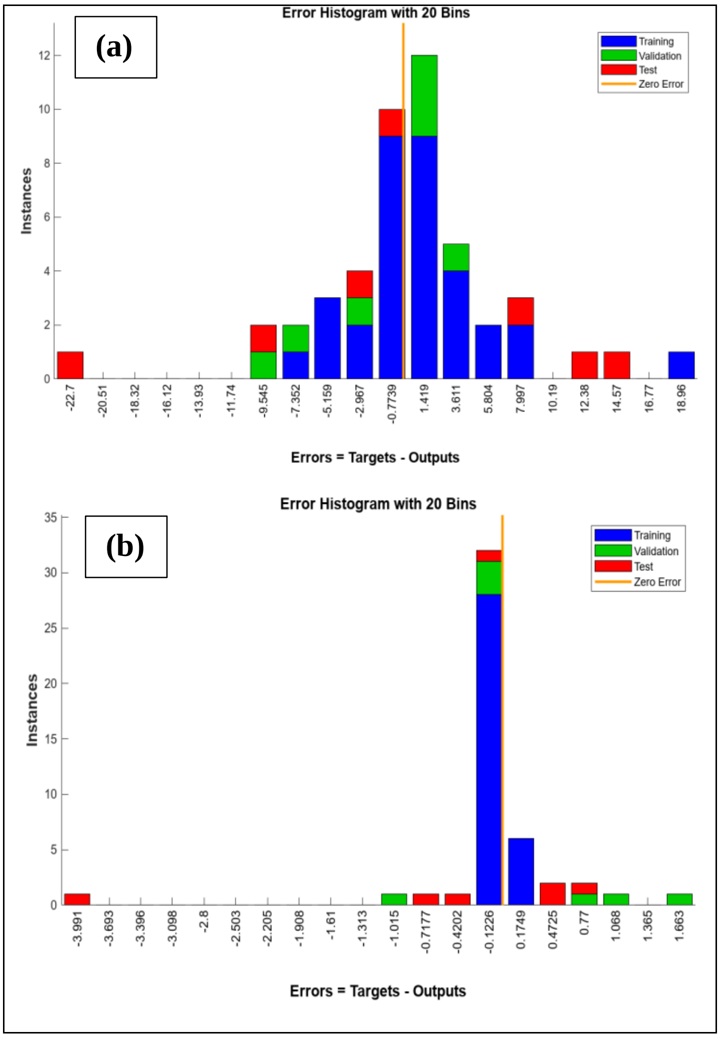

Figure 3: Error histograms (a) for LP, (b) for GP.
Source: Authors, (2023).

Figure 4 and 5 show the network performance related LP and GP, respectively. Considering the LP, it is obvious that the R^2 values for training, validation, testing and overall are respectively 0.99027, 0.90934, 0.71337, and 0.9392. According to the GP, the R^2 values of 0.99992, 0.99712, 0.99289, and 0.99952 were obtained for training, validation, testing and overall respectively.

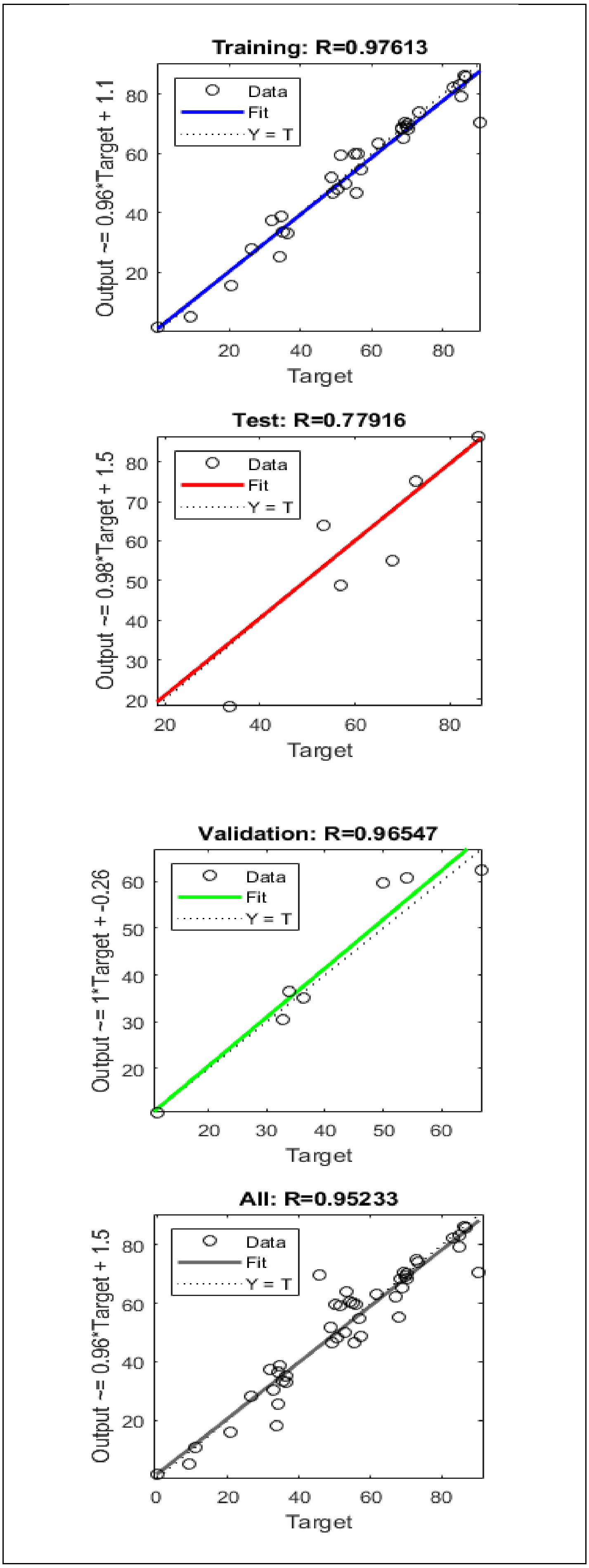

Figure 4: Four-in-one plot of network performance of LP.
Source: Authors, (2023).

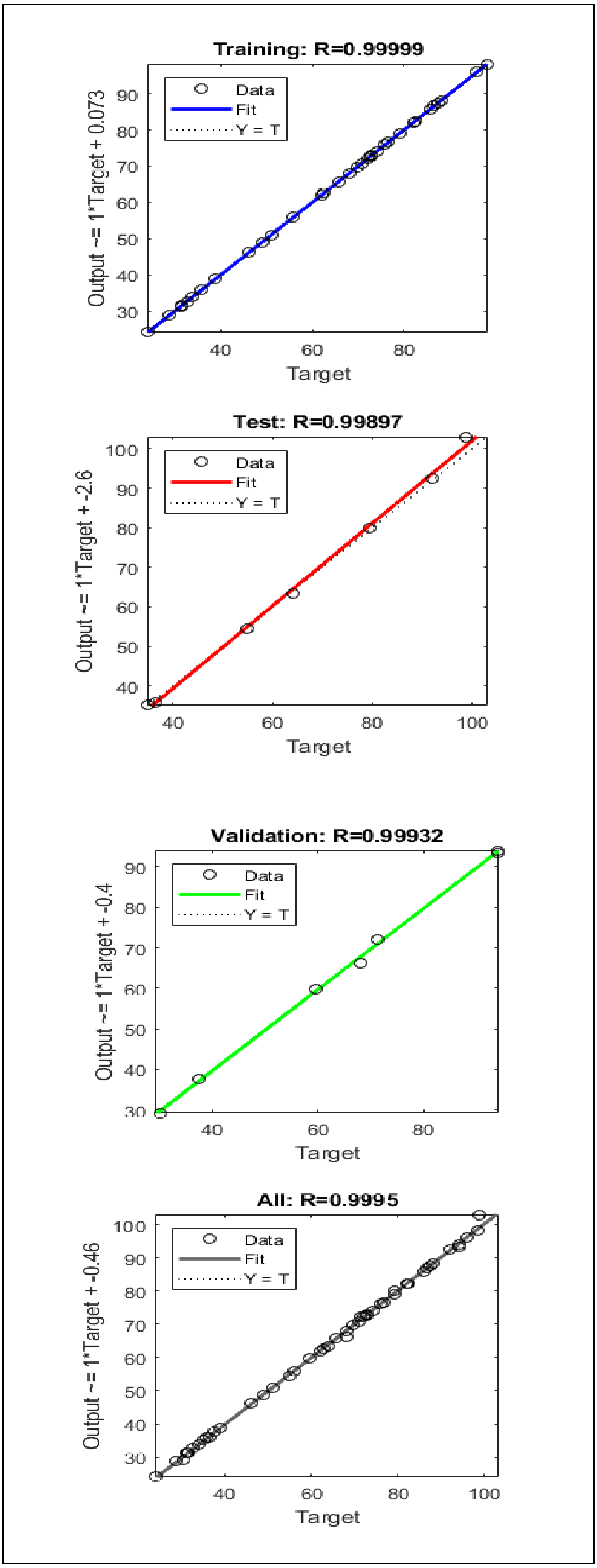

Figure 5: Four-in-one plot of network performance of GP.
Source: Authors, (2023).

From these results, it is obvious that due to high values of R^2 which is >90%, the fitting performance of the neural network was highly significant although the observed low value of R^2 (0.71916) in testing stage for LP. The low observed value of R^2 was due to the non-sufficient data to test the network and a low learning choice (15%), but the overall performance of the network was not affected. The equations 13 and 14 show the relationship between the overall output and the target (All) for the determination of the overall predicted network model.

$$Overall\ output = 0.96 * Target + 1.5 \tag{13}$$

$$Overall\ output = 1 * Target - 0.46 \tag{14}$$

IV.3 COMPARISON OF RSM AND ANN, AND OPTIMIZATIONS

The use of the equations 13 and 14 allowed evaluating the performance of the models, followed by the comparison between the values of the experimental data, the predicted ANN and RSM values, either for of LP and GP, respectively. The results are presented in Table 6.

Table 6: Experimental data, RSM and ANN results for LP and GP.

Run N°	LP			GP		
	Exp. removal percent (%)	RSM pred. removal percent (%)	ANN pred. removal percent (%)	Exp. removal percent (%)	RSM pred. removal percent (%)	ANN pred. removal percent (%)
1	0.001	0.000	1.340	35.854	35.839	35.912
2	67.879	74.92	55.075	48.958	49.156	48.882
3	33.927	33.45	36.468	62.058	61.997	62.073
4	70.399	68.15	70.243	54.962	54.926	54.415
5	36.432	38.49	35.148	65.785	65.917	65.784
6	72.815	72.48	75.068	76.603	76.432	76.571
7	36.432	28.22	33.122	51.005	51.016	51.010
8	56.121	58.51	59.805	59.546	59.681	59.736
9	20.617	24.77	15.754	68.083	67.869	68.076
10	57.042	55.41	54.890	23.982	24.108	24.001
11	48.654	50.21	52.012	30.242	30.446	29.279
12	68.595	70.6	68.333	36.497	36.308	35.752
13	86.060	83.51	86.489	55.920	55.851	55.858
14	26.361	23.4	27.991	64.001	64.28	63.474
15	86.060	83.51	86.489	72.077	72.234	72.069
16	49.990	52.3	59.698	70.806	70.746	70.761
17	69.890	72.34	69.192	76.605	76.849	76.644
18	84.806	82.13	83.406	82.401	82.476	82.293
19	35.271	40.6	33.652	62.627	62.644	62.748
20	55.170	56.94	59.902	68.125	66.42	66.314
21	70.206	62.33	68.286	69.659	69.721	69.698
22	34.054	37.82	25.398	31.383	31.543	31.392
23	53.995	54.51	60.745	32.614	32.993	32.601
24	9.061	9.61	4.877	33.851	33.967	33.884
25	32.773	35.52	30.384	75.985	75.63	75.999
26	52.755	52.56	49.977	79.043	79.172	79.023
27	69.008	60.74	65.405	82.097	82.238	82.087
28	73.270	65.02	73.986	86.649	86.333	86.707
29	69.430	70.67	70.506	87.426	87.549	87.409
30	61.855	67.45	63.272	88.198	88.288	88.220
31	50.550	55.37	48.190	74.250	74.039	74.111
32	49.104	55.11	46.592	72.744	72.928	72.763
33	57.103	52.62	48.859	71.235	71.34	72.167
34	53.301	54.92	63.839	38.782	38.746	38.873
35	45.767	48.35	69.564	34.996	35.308	35.077
36	34.504	32.93	38.874	31.205	31.394	31.275
37	55.589	51.09	46.736	96.050	95.177	96.050
38	83.120	100.84	82.487	94.086	93.832	93.302
39	85.002	83.23	79.439	92.117	92.009	92.439
40	11.107	21.43	10.633	98.891	101.688	103.031
41	86.595	83.57	85.950	98.247	98.016	98.266
42	51.502	56.74	59.365	93.996	93.867	94.018
43	68.611	66.21	68.600	85.870	85.201	85.876
44	33.604	36.39	18.328	79.343	79.202	80.022
45	90.479	56.76	70.423	72.813	72.727	72.900
46	66.944	66.15	62.483	46.182	45.716	46.242
47	31.977	36.68	37.430	37.373	37.391	37.578
48	-	-	-	28.560	28.589	28.743

Source: Authors, (2023).

According to the results of Table 6, the values of R^2 related to LP between experimental data and predicted RSM results on one hand, and between experimental data and predicted ANN results on another hand, were calculated using Excel and the results were 0.9488 and 0.9423, respectively. In addition, concerning GP the value of R^2 for the relationship between the experimental data and predicted RSM results was 0.9997, while the R^2 associated to the relationship between the experimental data and predicted ANN results was 0.9995. Those results presented very high and significant performance of fitting for LP and GP, either in RSM and ANN. The optimization results of percent removal of LP and GP using RSM are carried out in Table 7, and based on the maximization of the equations 8 and 10. Concerning the optimization using ANN, the prediction equations 13 and 14 were used to develop a genetic-algorithm (GA) in MATLAB optimization tool.

Table 7: Optimization of gold recovery.

		Time (hr)	[HBr] (g/L)	pH	Speed (rpm)	Percent removal (%)	Validation experiment1 (%)	Validation experiment2 (%)
LP	RSM	7.27	50	1	200	98.54	97.58	98.22
	ANN	7.5	50	1	200	97.16	96.58	97.01
		Time (min)	**[α-CD] (g/L)**	**pH**	**Percent removal (%)**	**Validation experiment1 (%)**	**Validation experiment2 (%)**	
GP	RSM	40	11.61	5	98.87	98.45	97.87	
	ANN	39.8	10.71	5	99.98	99.95	99.26	

Source: Authors, (2023).

The best percent removals for LP using RSM were predicted at the following conditions: time=7.27 hours, [HBr]=50 g/L, pH=1 and stirring speed=200 rpm, with a removal efficiency of 98.54%. By using ANN-GA, the optimal conditions were closed (time of 7.5 hours, [HBr] of 50 g/L, pH of 1 and stirring speed of 200 rpm) to those obtained with RSM with a percent removal of 97.16%. Moreover, it was found that in GP the optimal gold percent removal using RSM was 98.87% with a contact time of 40 minutes, a [α-CD] of 11.61 g/L and a pH of 5. The use of ANN-GA method revealed the highest percent removal of 99.98% with time, [α-CD] and pH, of 39.80 minutes, 10.71 g/L, and 5.15, respectively. Therefore the results of comparison confirmed the best performance of both RSM and ANN in gold removal during LP and GP. However, Table 7 reveals a quite difference in LP between RSM and ANN-GA maximum percent removal (1.38%), while the difference between ANN-GA and RSM in GP is equal to 1.11%.

V. CONCLUSIONS

This paper demonstrates the successful use of alpha cyclodextrin (α-CD) for gold extraction from gold-bearing ore in the Democratic Republic of Congo (D.R.C). Through the implementation of Response Surface Methodology (RSM) and Artificial Neural Networks (ANN), the study identified the optimal gold removal conditions. Ore samples were initially processed to enhance leachability, and a modified aqua regia with hydrobromic acid was employed for the leaching process, leveraging the strong molecular recognition between gold bromide ion and α-CD. The experimentation involved adjusting various leaching parameters such as time, HBr concentration, pH, and stirring speed. RSM yielded optimal values of 7.27 hours, 50 g/L, pH 1, and 200 rpm, resulting in an impressive 98.54% gold removal. Meanwhile, ANN produced slightly lower values of 7.5 hours, 50 g/L, pH 1, and 200 rpm, with 97.16% gold removal. These results highlight the effectiveness of both RSM and ANN in optimizing gold extraction from the ore. Additionally, for α-CD gold recovery, RSM and ANN optimization resulted in 40 minutes, 11.61 g/L, and pH 5, achieving 98.87% and 39.8 minutes, 10.71 g/L, and pH 5, with 99.98% gold removal, respectively. These findings were further validated, reinforcing the success of RSM and ANN as methods for maximizing gold recovery from gold-bearing ore in the D.R.C. Overall, the use of α-CD in combination with RSM and ANN presents a promising approach for sustainable and efficient gold extraction processes in resource-rich regions like the D.R.C., potentially opening new avenues for environmentally friendly mining practices and enhanced economic opportunities.

VI. AUTHOR'S CONTRIBUTION

Conceptualization: Meschack Mukunga Muanda and Pele Pascal Daniel Omalanga.
Methodology: Meschack Mukunga Muanda and Pele Pascal Daniel Omalanga.
Investigation: Meschack Mukunga Muanda, Pele Pascal Daniel Omalanga, Vanessa Mwambaie Mitonga and Michée Ngoy Ilunga.
Discussion of results: Meschack Mukunga Muanda, Pele Pascal Daniel Omalanga, Vanessa Mwambaie Mitonga and Michée Ngoy Ilunga.
Writing – Original Draft: Meschack Mukunga Muanda and Michée Ngoy Ilunga.
Writing – Review and Editing: Meschack Mukunga Muanda and Michée Ngoy Ilunga.
Supervision: Meschack Mukunga Muanda and Pele Pascal Daniel Omalanga.
Approval of the final text: Meschack Mukunga Muanda, Pele Pascal Daniel Omalanga, Vanessa Mwambaie Mitonga and Michée Ngoy Ilunga.

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