

PREDICTION, MODELING, AND OPTIMIZATION STUDIES OF ECO-FRIENDLY GOLD EXTRACTION USING ALPHA-CYCLODEXTRIN AND THE RSM AND CCD METHODS

Estudios de predicción, modelado y optimización de extracción ecológica de oro utilizando alfa-ciclodextrina y los métodos RSM y CCD

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ABSTRACT

In the Democratic Republic of the Congo, gold extraction from ore in South Kivu utilized alpha-cyclodextrin (α -CD) as an eco-friendly method to replace harmful methods. Analysis of variance and response surface methodology were employed for prediction, modeling, and optimization studies. Atomic absorption spectroscopy revealed a 0.06% gold content in a randomly taken ore sample. Leaching with modified aqua regia was optimized with the following parameters: 7.27 h, 50 g/L HBr concentration, pH 1, and 200 rpm stirring speed, resulting in 98.5% removal. Neutralization tests with potassium hydroxide (KOH) followed leaching to prepare the solution medium with appropriate pH for extraction test, by varying parameters such as time, KOH concentration, and pH. Following neutralization, extraction tests with α -CD were carried out and optimized with the following parameters: 40 min, α -CD concentration of 11.6166 g/L, and a pH of 5, resulting in a percent removal of 98.9%. α -CD's eco-friendly nature, cost-effectiveness, and high percent removal make it a promising and sustainable alternative for gold recovery, appealing to the mining industry.

Palabras clave: ANDEVA, metal valioso, agua regia modificada, optimización, solución ambiental.

RESUMEN

En Kivu del Sur, República Democrática del Congo, se utilizó alfa-ciclodextrina (α -CD) para la extracción de oro a partir de minerales, como método ecológico para reemplazar los métodos nocivos. Se emplearon el análisis de varianza y la metodología de superficie de respuesta para los estudios de predicción, modelado y optimización. La

espectroscopia de absorción atómica reveló un contenido de oro de 0.06 % en el mineral. La lixiviación con agua regia modificada se optimizó con los siguientes parámetros: 7.27 h, 50 g/L de concentración de HBr, pH 1 y velocidad de agitación de 200 rpm, lo que resultó en una eliminación del 98.5 %. Pruebas de neutralización con hidróxido de potasio (KOH) siguieron a la lixiviación, para preparar el medio con el pH adecuado para la prueba de extracción, variando parámetros como el tiempo, la concentración de KOH y el pH. Después de la neutralización se realizaron pruebas de extracción con α -CD y se optimizaron con los siguientes parámetros: 40 min, concentración de α -CD de 11.6166 g/L y pH de 5, resultando en un porcentaje de remoción de 98.9 %. La naturaleza ecológica, la rentabilidad y el alto porcentaje de eliminación de α -CD lo convierten en una alternativa prometedora y sostenible para la recuperación de oro, atractiva para la industria minera.

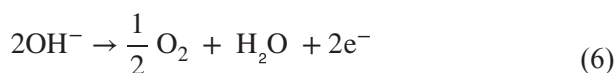
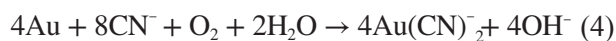
INTRODUCTION

Gold recovery from ores is a critical process in the mining industry, fueled by the escalating demand for this precious metal (Smith 2019). As a majority of metals to be extracted are contained in minerals, the mining sector has relentlessly pursued effective and sustainable gold recovery techniques (Mukunga 2019, Muanda et al. 2021). As the significance of gold in various industries, such as electronics, jewelry, and finance, continues to rise, the demand for this precious metal shows no signs of abating (Knosp et al. 2003, Oleszek-Listopad et al. 2015, Saradesh and Vinodkumar 2020). However, with depleting gold reserves and declining ore grades, the development of efficient and responsible extraction methods becomes imperative. Despite the low grades of gold in some of quarries, many processes are used for concentration, among them flotation, which is a gravimetric method based on density (Singh 2012, Muanda and Omalanga 2021a, b).

The leaching process is very important in gold recovery processes due to the dissolution of gold before the use of an organic extractant. The commonly used leaching solution is aqua regia (AR) which is a combination of hydrochloric and nitric acids (HCl and HNO₃, respectively), in a 3/1 ratio. AR is highly corrosive and can dissolve most metals (gold, copper, aluminum, platinum, and so on) or alloys (Grayson 2007, Bonggotgetsakul et al. 2016, Cyganowski et al. 2017). As an alternative to AR, modified aqua regia (MAR), where HNO₃ is replaced by hydrobromic acid (HBr), is used as a highly efficient gold dissolvent agent. Equations 1 and 2 show the dissolution of gold by AR and MAR, respectively. In addition, the molecular recognition between α -cyclodextrin and gold bromide is shown in equation 3.



Traditional cyanide-based extraction processes, where cyanide is used as an organic agent after leaching, have proven successful in recovering gold, but their detrimental effects on ecosystems and human health have spurred the need for safer alternatives (Bakatula-Nsimba et al. 2013, Acar 2016, Mertol et al. 2016, Xing and Lee 2019, Neag et al. 2020). The main equation of gold recovery by cyanation is known as the Elsner reaction (equation 1), while the other equations (4, 5, and 6) of the mechanism are written as follows (Deschênes 2005):



Nevertheless, the environmental impact and potential hazards of these processes have motivated researchers to seek safer and greener alternatives (Anthony et al. 2020, Karume et al. 2022). In recent years, alpha-cyclodextrin (α -CD), a cyclic oligoglucide, has emerged as a promising alternative for gold recovery due to its selective and environmentally friendly properties (Bhardwaj et al. 2020, Li et al. 2022). α -CD is composed of six glucose units and possesses unique molecular properties that have recently garnered attention in various industrial applications (Chang and Chen 2006). Its brute formula and molecular mass are C₃₆H₆₀O₃₀ and 972, respectively. Particularly noteworthy is its ability to selectively

form stable inclusion complexes, known as host-guest complexes, with hydrophobic molecules. This remarkable attribute has paved the way for exploring its potential in gold recovery processes. **Figure 1** shows the structure of α -CD where the value of n is equal to n .

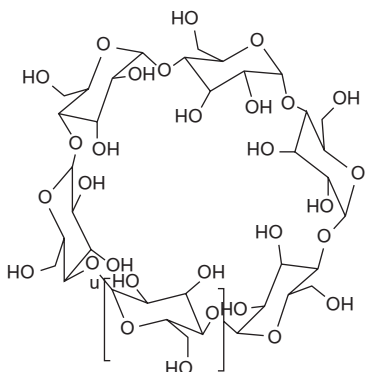


Fig. 1. Cyclodextrins representation (Karume et al. 2022).

Moreover, α -CD's potential as a gold recovery agent arises from its unique molecular structure, characterized by a hydrophilic exterior and a hydrophobic cavity (Jansook et al. 2017, Bezerra et al. 2020). This molecular arrangement empowers α -CD to selectively encapsulate gold ions, forming stable water-soluble inclusion complexes (Almagro and Pedreño 2020). This selectivity ensures that only gold is targeted during extraction, minimizing waste generation and maximizing gold yields.

Furthermore, the biodegradable nature and non-toxic properties of α -CD provide a significant advantage over cyanide, mitigating environmental risks associated with traditional extraction methods. These attributes make α -CD a compelling candidate for sustainable gold recovery practices. α -CD does not produce harmful byproducts, making it an eco-friendly choice for gold recovery. The abundance and low cost of α -CD make it a viable alternative to conventional methods.

The utilization of α -CD as an innovative gold recovery agent holds immense promise in transforming the mining industry. With its high selectivity for gold ions, eco-friendliness, and ease of handling, α -CD represents a significant step towards sustainable and responsible gold extraction practices. As research and development continue, the successful implementation of gold recovery processes using α -CD could mark a paradigm shift in the mining sector, offering

a greener and more efficient alternative to traditional cyanide-based methods.

This scientific paper seeks to introduce and explore the application of α -CD in gold recovery and its potential to revolutionize traditional gold extraction processes. Its primary objective is to provide a comprehensive overview of the use of α -CD in gold recovery. A thorough examination of its molecular interactions with gold ions and its potential to replace conventional cyanide-based methods will be undertaken to shed light on the benefits, challenges, and prospects of implementing α -CD as a sustainable and environmentally friendly solution for gold extraction. The secondary objective is to investigate the optimization efficiency and scalability of gold recovery processes using response surface methodology (RSM), envisioning a future where this greener and more efficient alternative could revolutionize the industry. Moreover, the paper will present the current state of research and development in the field of α -CD-mediated gold recovery, providing insights into ongoing advancements and the current landscape of this innovative approach. The exploration will also extend to investigating the challenges and potential barriers to the widespread adoption of α -CD in the mining industry, which will help identify areas for improvement and further research.

By advancing the state of knowledge on α -CD's application in gold recovery, this paper contributes to the ongoing efforts to promote responsible and sustainable mining practices while meeting the global demand for this valuable metal. It also provides insights that will inspire further exploration and application of α -CD as an environmentally friendly and effective agent for gold recovery in the mining industry.

METHODOLOGY

Sampling

Gold-bearing ore was collected from two quarries in South-Kivu, Democratic Republic of the Congo, resulting in a total of 50 kg from each site. The sample was homogenized using the cone and crown method. Then, another technique (quartering), was used to make the sample more representative. The final sample was safely conserved for further steps. The initial step of the crushing process involved breaking down the large collected sample of gold ore (50 kg) into more manageable sizes. This was achieved by using a sturdy sledgehammer to reduce the ore into smaller pieces, which were suitable for further

crushing. Before proceeding with the complete sample, a smaller portion (1 kg) was set aside for assaying purposes. This selected portion underwent analysis through atomic absorption spectroscopy/inductively coupled plasma (AAS/ICP) to estimate the concentration of gold. Once the primary crushing and initial assay were completed, the remaining 49 kg of the sample were ready for further processing. The sample underwent another round of crushing, first using a laboratory jaw followed by a laboratory cylindrical crusher. To ensure consistent results, this crushing process was carefully timed for approximately 19.58 min, so that 80% of the material passed through a 75 μm sieve. For the sample characterization with AAS/ICP, a high graphite atomization (graphite furnace HGA 700) was employed. Calibration curves

were plotted using solutions in an argon current (20, 40, 60, and 80 ng Au/cm³), with the use of a nickel matrix modifier. The analysis was conducted using a platform called temperature-stabilized platform (STPF) associated with the graphite furnace, which yielded good signals (Petrović et al. 2001).

Materials used

In this study, only analytical grade chemical reagents were utilized, and all the stock solutions were prepared using distilled water.

Recovery process

Figure 2 illustrates the recovery process employed for sampling the quarries of South-Kivu province, utilizing α -CD. Several tests were conducted

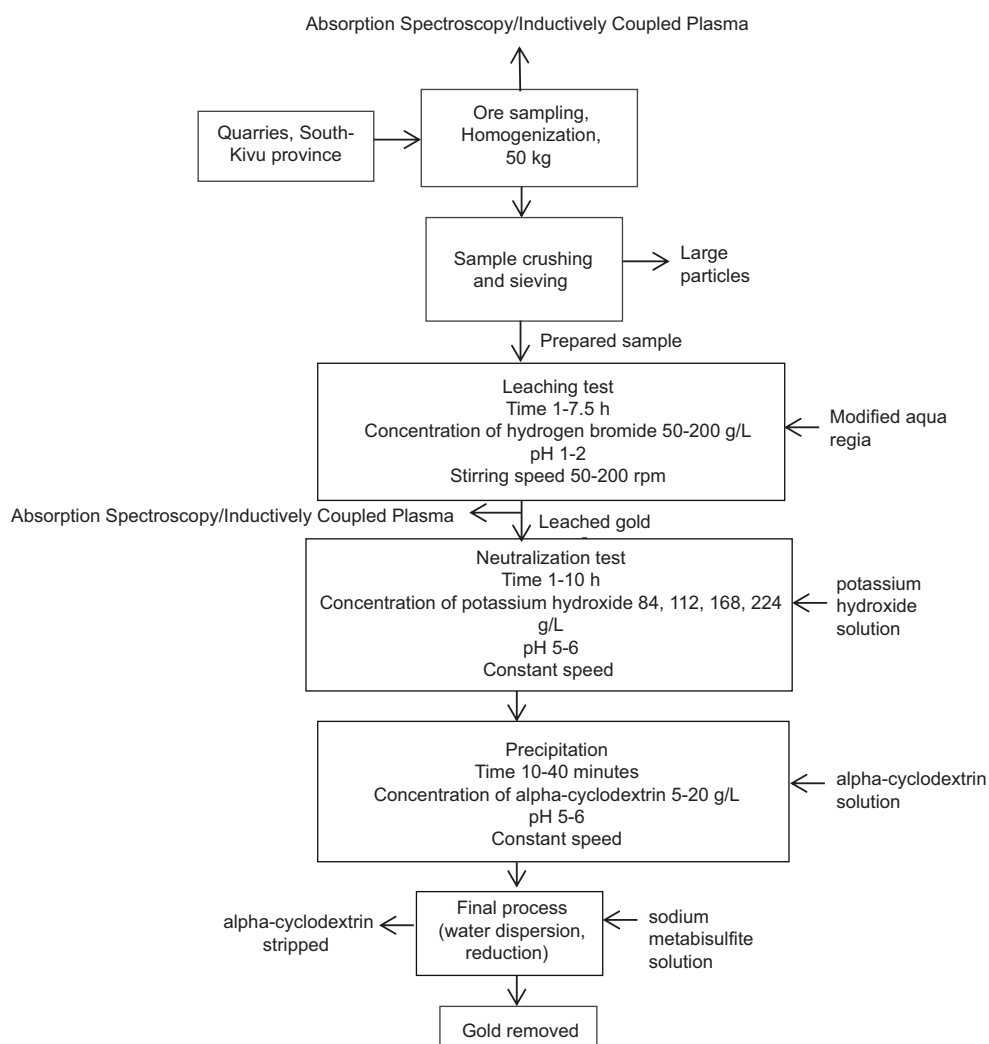


Fig. 2. Gold recovery full process.

to maximize gold recovery from the ore sample, including leaching tests, neutralization tests, and gold recovery tests using α -CD. To optimize the recovery process, various physicochemical parameters were studied. To analyze the impacts of these parameters and the design, Minitab and OriginPro software were utilized.

Leaching test (LT)

During the LT, a total amount of 400 g of the sample (gold ore) was mixed with 800 mL of a specific leaching solution (MAR) in a 2 L beaker. The mixture was allowed to react for 5 min with continuous stirring at a specific speed. After 24 h of digestion in MAR, the gold ore concentrate (HAuBr_4), called digestate or digested product, was obtained and separated by filtration. The valuable metal-containing filtrate was collected for further experiments. The remaining solid material, known as the cake, was washed and dried in an oven at 105 °C for 24 h.

To determine the leaching percentage, AAS/ICP analysis was performed to measure the final gold concentration after leaching. Each LT was duplicated and the average value was retained for calculations. The leaching percentage or recovery (%) was calculated using the following formula:

$$\text{Recovery (\%)} = \frac{P_1 T_1 - P_2 T_2}{P_1 T_1} \times 100 \quad (7)$$

where P_1 , P_2 , T_1 , and T_2 are the mass of the ore before leaching, the mass of dry solid obtained after leaching, and the concentration of gold before and after leaching, respectively.

The study investigated four factors: time, HBr concentration ($[\text{HBr}]$), pH, and stirring speed with symbols x_1 , x_2 , x_3 , and x_4 , respectively. As shown in **table I**, each factor was studied at three levels, resulting in a full factorial design with a total of 47 LTs. The laboratory setup for the LT is shown in **figure 3**.

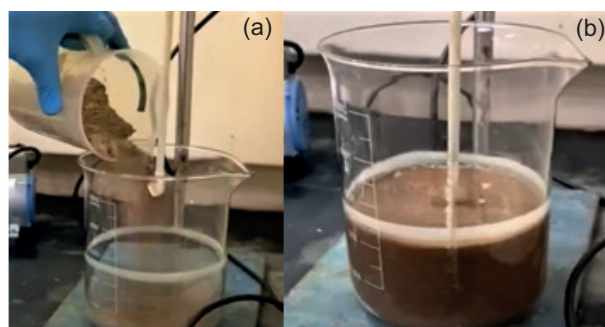


Fig. 3. Lab scale (a) charging, (b) leaching.

Neutralization test (NT)

The NTs were performed using 50 mL solutions from leaching added to a 500 mL beaker. Potassium hydroxide (KOH) was used (1.0 M) as pH regulator. After neutralization, filtration was performed to eliminate any base metals or insoluble silver bromide present in the solution. To determine the optimal neutralization value, each test was conducted three times, and the average value was taken into consideration. The study focused on three factors: Time, KOH concentration ($[\text{KOH}]$), and pH coded x_1 , x_2 , x_3 , which were appropriately combined for experimentation.

The main purpose of neutralization was to determine the necessary time for removing impurities that could potentially contaminate the concentrate following leaching. **Table II** presents the variables, codes, and symbols utilized in the full factorial design.

Gold recovery tests with α -CD (GT- α)

The neutralized solution (KAuBr_4) was divided into 50 mL aliquots, which were placed in a 2 L beaker. α -CD was gradually added to the solution at the required amount while keeping the room temperature in mind. From the first minute of the reaction, a complex named potassium tetrabromoaurate

TABLE I. PARAMETERS USED FOR THE LEACHING TESTS.

| Parameters | Time (h) | | Hydrogen bromide concentration (g/l) | | pH | | Stirring speed (rpm) | |
|----------------|----------|------|--------------------------------------|------|-------|------|----------------------|------|
| Symbols | x_1 | | x_2 | | x_3 | | x_4 | |
| | Level | Code | Level | Code | Level | Code | Level | Code |
| Code and level | 1 | 1 | 50 | 1 | 1 | 1 | 50 | 1 |
| | 2.5 | 2 | 100 | 2 | 2 | 2 | 100 | 2 |
| | 5 | 3 | 150 | 3 | | | 150 | 3 |
| | 7.5 | 4 | 200 | 4 | | | 200 | 4 |

TABLE II. PARAMETERS USED FOR THE NEUTRALIZATION TEST.

| Parameters | Time (h) | | Potassium hydroxide concentration (g/L) | | pH | |
|----------------|----------|------|---|------|-------|------|
| Symbols | x_1 | | x_2 | | x_3 | |
| | Level | Code | Level | Code | Level | Code |
| Code and level | 1 | 1 | 84 | 1 | 5 | 1 |
| | 2.5 | 2 | 112 | 2 | 5.5 | 2 |
| | 5 | 3 | 168 | 3 | 6 | 3 |
| | 10 | 4 | 224 | 4 | | |

alpha-cyclodextrin (recovered gold α -Br) was formed and facilitated the formation of the co-precipitates that were then removed by filtration. Three parameters were examined in this section: time, α -CD concentration [α -CD], and pH coded x_1 , x_2 , and x_3 , respectively, with their appropriate combinations. The parameters and the ranges are presented in **table III**.

TABLE III. PARAMETERS USED FOR GOLD RECOVERY TESTS WITH ALPHA-CYCLODEXTRIN.

| Parameters | Time (min) | | Alpha-cyclodextrin concentration (g/L) | | pH | |
|----------------|------------|------|--|------|-------|------|
| Symbols | x_1 | | x_2 | | x_3 | |
| | Level | Code | Level | Code | Level | Code |
| Code and level | 10 | 1 | 5 | 1 | 5 | 1 |
| | 20 | 2 | 10 | 2 | 5.5 | 2 |
| | 30 | 3 | 15 | 3 | 6 | 3 |
| | 40 | 4 | 20 | 4 | | |

After filtration, the α -Br complex was dispersed in water and reduced with sodium bisulfite ($\text{Na}_2\text{S}_2\text{O}_5$). This reduction resulted in the formation of recovered gold metal as a precipitate, followed by the liberation of the encapsulated gold from the early formed inclusion complex. The recovered gold metal was collected through decantation of the aqueous solution.

After recovering the gold, it underwent characterization using AAS. Each GT- α was duplicated, and the average value was considered.

Prediction, modeling, and optimization

The prediction of the percent removal in LTs, NTs, and GT- α was done using Minitab software, resulting in the determination of a mathematical equation between the dependent variable (response)

and the independent variables (studied parameters). Furthermore, analysis of variance (ANOVA) was used to evaluate the statistical parameters influencing the performance of the process. An important factor (P-value) was used to assess the effects of the studied parameters and their interactions. For a P-value greater than 0.05, the variable or interaction was identified as non-significant with 95% confidence intervals. On the other hand, for a P-value less than 0.0001 the variable or interaction was identified as highly significant (Liu and Wang 2007).

In addition, modeling and optimization were performed using RSM, while central composite rotatable design (CCRD) was used to establish second-order models (Muanda and Omalanga 2021b). The general form of the relationship between the response and the parameters (regression equation) was considered as follows:

$$y = f(x_1, x_2, x_3, \dots, x_i) \quad (8)$$

where y is the dependent variable and x_i are the independent variables.

Other important factors, namely the coefficient of determination (R^2) and the coefficient of variation (CV), were also evaluated. R^2 was used to evaluate the model's fitness and how it could vary, while CV was used to evaluate the degree of precision of treatment comparison. According to Liu and Wang (2007), a good fit of a model is associated with a R^2 value equal to at least 0.80. A high CV value corresponds to a substantial variation from the average value, while a low CV value corresponds to a low degree of dispersion (Murthy et al. 2000).

OriginPro software was used to perform regression equations for LT, NT, and GT- α , resulting in the plotting of two-dimensional (2D) contour plots and three-dimensional (3D) response surfaces. The information provided by those plots was used to interpret the results obtained for both the variables and their interactions. The plotting was carried out by considering only two variables for the LT, NT, and GT- α models. In contrast, the remaining variables were kept at the center level.

RESULTS AND DISCUSSION

Characterization of the sample

The sample analyses were done at the chemical and metallurgical laboratory of the Société Générale de Surveillance (General Society of Surveillance, SGS), using a Perkin Elmer 1100 B AAS, where the gold solution sample was charged into the tube by

TABLE IV. CHEMICAL COMPOSITION OF GOLD-BEARING ORE (SGS).

| Metal | Co | Au | Ag | Fe | Al | Cu | Mg | Si |
|-------------|------|-------|---------|-------|------|------|------|-------|
| Content (%) | 0.39 | 0.059 | 0.00014 | 22.35 | 3.95 | 0.21 | 1.75 | 17.65 |

SGS: laboratory of the Société Générale de Surveillance.

an autosampler AS-70. The results of the AAS/ICP sample analysis are displayed in **table IV**, revealing the presence of other metals associated with gold.

Leaching test (LT)

All LTs were done at room temperature, and the final solutions were analyzed in the laboratory to measure the percent removal. The results of the LT percent removal are presented in **table V**, from which results equation 9 was obtained by regression analysis for leaching percent removal:

$$y = -91.3 + 31.74x_1 + 1.404x_2 - 4.0x_3 + 0.040x_4 - 0.465x_1^2 - 0.001773x_2^2 + 0.000097x_4^2 - 0.1860x_1x_2 - 0.96x_1x_3 - 0.0090x_1x_4 - 0.0740x_2x_3 - 0.000141x_2x_4 + 0.0139x_3x_4 \quad (9)$$

where y , x_1 , x_2 , x_3 and x_4 represent the leaching percentage, the time, the [HBr], the pH, and the stirring speed, respectively.

Notably, equation 9 reveals that x_1 exhibits the most significant positive impact on increasing leaching percentage compared to x_2 and x_4 . Conversely, x_3 exerts a negative effect on the percentage and is also significant for the model. This suggests that an acidic medium proves to be the most desirable for the leaching process. Another crucial factor to consider is the consumption of MAR (presumably a reagent) by other metals present in the ore. When the overall concentration of MAR increases, the leaching efficiency improves as it facilitates the dissolution of more gold. In summary, the acidic medium (associated with x_3) and higher MAR concentration play pivotal roles in enhancing the leaching process and its efficiency for gold extraction. On the other hand, x_1 has the most substantial positive impact among the variables analyzed.

Table VI presents the ANOVA results, which indicate that the interactions x_1x_3 and x_2x_3 have significant importance for the model. Despite this, the overall findings were satisfactory owing to the high R^2 value ($R^2 = 0.9003$) and the adjusted determination coefficient (Adj. $R^2 = 0.7825$).

Figure 4 depicts the 2D contour plots and 3D response surface, showcasing the highly significant model with a P-value lower than 0.0001. The low

TABLE V. RESULTS OF PERCENT REMOVAL FOR LEACHING TESTS.

| Run N ₀ | Code level of variable | | | | Percent removal (%) |
|-----------------------|------------------------|-------|-------|-------|------------------------|
| | x_1 | x_2 | x_3 | x_4 | |
| 1 | 1 | 1 | 1 | 4 | 0.001 |
| 2 | 1 | 4 | 2 | 1 | 67.9 |
| 3 | 1 | 2 | 1 | 1 | 33.9 |
| 4 | 1 | 3 | 1 | 1 | 70.4 |
| 5 | 1 | 2 | 1 | 3 | 36.4 |
| 6 | 1 | 3 | 1 | 3 | 72.8 |
| 7 | 1 | 2 | 2 | 3 | 36.4 |
| 8 | 1 | 3 | 2 | 3 | 56.1 |
| 9 | 1 | 2 | 2 | 2 | 20.6 |
| 10 | 1 | 3 | 2 | 2 | 57.0 |
| 11 | 2 | 2 | 1 | 2 | 48.7 |
| 12 | 2 | 3 | 1 | 2 | 68.6 |
| 13 | 2 | 4 | 1 | 3 | 86.1 |
| 14 | 2 | 1 | 1 | 3 | 26.4 |
| 15 | 2 | 4 | 1 | 3 | 86.1 |
| 16 | 2 | 2 | 1 | 3 | 50.0 |
| 17 | 2 | 3 | 1 | 3 | 69.9 |
| 18 | 2 | 4 | 1 | 2 | 84.8 |
| 19 | 2 | 2 | 2 | 3 | 35.3 |
| 20 | 2 | 3 | 2 | 3 | 55.2 |
| 21 | 2 | 4 | 2 | 2 | 70.2 |
| 22 | 2 | 2 | 2 | 2 | 34.1 |
| 23 | 2 | 3 | 2 | 2 | 54.0 |
| 24 | 2 | 1 | 2 | 1 | 9.1 |
| 25 | 2 | 2 | 2 | 1 | 32.8 |
| 26 | 2 | 3 | 2 | 1 | 52.8 |
| 27 | 2 | 4 | 2 | 1 | 69.0 |
| 28 | 3 | 1 | 1 | 3 | 73.3 |
| 29 | 3 | 2 | 1 | 3 | 69.4 |
| 30 | 3 | 3 | 1 | 3 | 61.9 |
| 31 | 3 | 4 | 1 | 3 | 50.6 |
| 32 | 3 | 4 | 1 | 2 | 49.1 |
| 33 | 3 | 1 | 2 | 2 | 57.1 |
| 34 | 3 | 2 | 2 | 2 | 53.3 |
| 35 | 3 | 3 | 2 | 2 | 45.8 |
| 36 | 3 | 4 | 2 | 2 | 34.5 |
| 37 | 3 | 1 | 2 | 1 | 55.6 |
| 38 | 4 | 1 | 1 | 3 | 83.1 |
| 39 | 4 | 2 | 1 | 3 | 85.0 |
| 40 | 4 | 4 | 1 | 3 | 11.1 |
| 41 | 4 | 2 | 1 | 4 | 86.6 |
| 42 | 4 | 3 | 1 | 4 | 51.5 |
| 43 | 4 | 2 | 2 | 2 | 68.6 |
| 44 | 4 | 3 | 2 | 2 | 33.6 |
| 45 | 4 | 3 | 1 | 3 | 90.5 |
| 46 | 4 | 2 | 2 | 1 | 67.0 |
| 47 | 4 | 3 | 2 | 1 | 32.0 |

TABLE VI. ANOVA OF LEACHING TESTS.*

| Source | df | Sum of squares | Mean square | F-value | P-value Prob > F | Remarks |
|-----------|----|----------------|-------------|---------|---------------------|---------|
| 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 |
| $x_1 x_2$ | 1 | 14451.2 | 14451.2 | 213.89 | <0.0001 | HS |
| $x_1 x_3$ | 1 | 21.8 | 21.8 | 0.32 | 0.574 | NS |
| $x_1 x_4$ | 1 | 17.3 | 17.3 | 0.26 | 0.616 | NS |
| $x_2 x_3$ | 1 | 61.4 | 61.4 | 0.91 | 0.347 | NS |
| $x_2 x_4$ | 1 | 1.8 | 1.8 | 0.03 | 0.873 | NS |
| $x_3 x_4$ | 1 | 1.5 | 1.5 | 0.02 | 0.882 | NS |
| Error | 33 | 2229.6 | 67.6 | | | |
| Total | 46 | 22365.2 | | | | |

*CV = 10.7%; $R^2 = 0.9003$; Adj. $R^2 = 0.7825$.

F: Fisher's function; df: degrees of freedom; P-value: level of significance; NS: not significant; S: significant; HS: highly significant.

value of the coefficient of variation further confirmed the precision and reliability of the experiments.

The leaching percent removal of gold is directly influenced by two main factors: the reaction time and the [HBr]. Simultaneously, it is inversely affected by the pH level. The significance of the reaction time during LT is evident from the data presented in (figure 4a, b) underscoring the pivotal role of time in achieving successful leaching, with a substantial positive effect on gold leaching. At approximately 5 h of reaction time, the leaching percentage reaches its peak value at 79%, confirming the direct correlation between time and gold recovery.

The interaction between reaction time and [HBr] is also noteworthy. The red surface area in the graph represents the most favorable conditions for leaching, resulting in a larger recovery of gold. Figure 4a, c, reveals that along the low time zone, the yield is minimally affected by [HBr] due to insufficient contact between the gold particles and the leachate, as well as competition with other metals in the solution. As the presence of an oxidizing agent is crucial for gold dissolution, it has been observed that HNO_3 effectively contributes to the formation of trivalent gold ions, which were then reduced by HBr. The reduction involved the formation of tetrabromide aurate anions (AuBr_4^-).

The analysis of the time-pH interaction revealed that reducing the medium's acidity significantly decreased the leaching yield, regardless of the reaction

time. Additionally, shorter contact times resulted in lower gold recovery, confirming the findings from the time-[HBr] interaction. The desired red zone was most evident within the time range of 3-7 h and pH of 1-1.345.

Furthermore, the [HBr]-pH interaction indicated an inverse relationship between [HBr] and pH. The highest yield was achieved when the pH was approximately 1 and the [HBr] ranged between 120-200 g/L. Figure 4d, e, f demonstrates that the leaching percentage of metals remained relatively unchanged at varying stirring speeds, suggesting that the leaching reaction was not influenced by mass transfer control. In addition, in the stirring speed range of 150-200 rpm, the red zone became apparent in the [HBr]-speed, time-speed, and pH-speed interactions. These observations suggest the existence of an optimal stirring speed range for LT.

Overall, data from figure 4a, c indicate that around 80% of gold recovery was achievable when the [HBr] ranged from 60 to 90 g/L, highlighting the positive impact of increased [HBr] on the production of AuBr_4^- ions. On the other hand, the increase in leaching percentage was ascribed to the presence of silver bromide compound (AgBr), which impeded the dissolution of silver from the aqua regia (Park and Fray 2009). Table VII displays the results of the optimization process aimed at enhancing LT efficiency while minimizing the studied parameters.

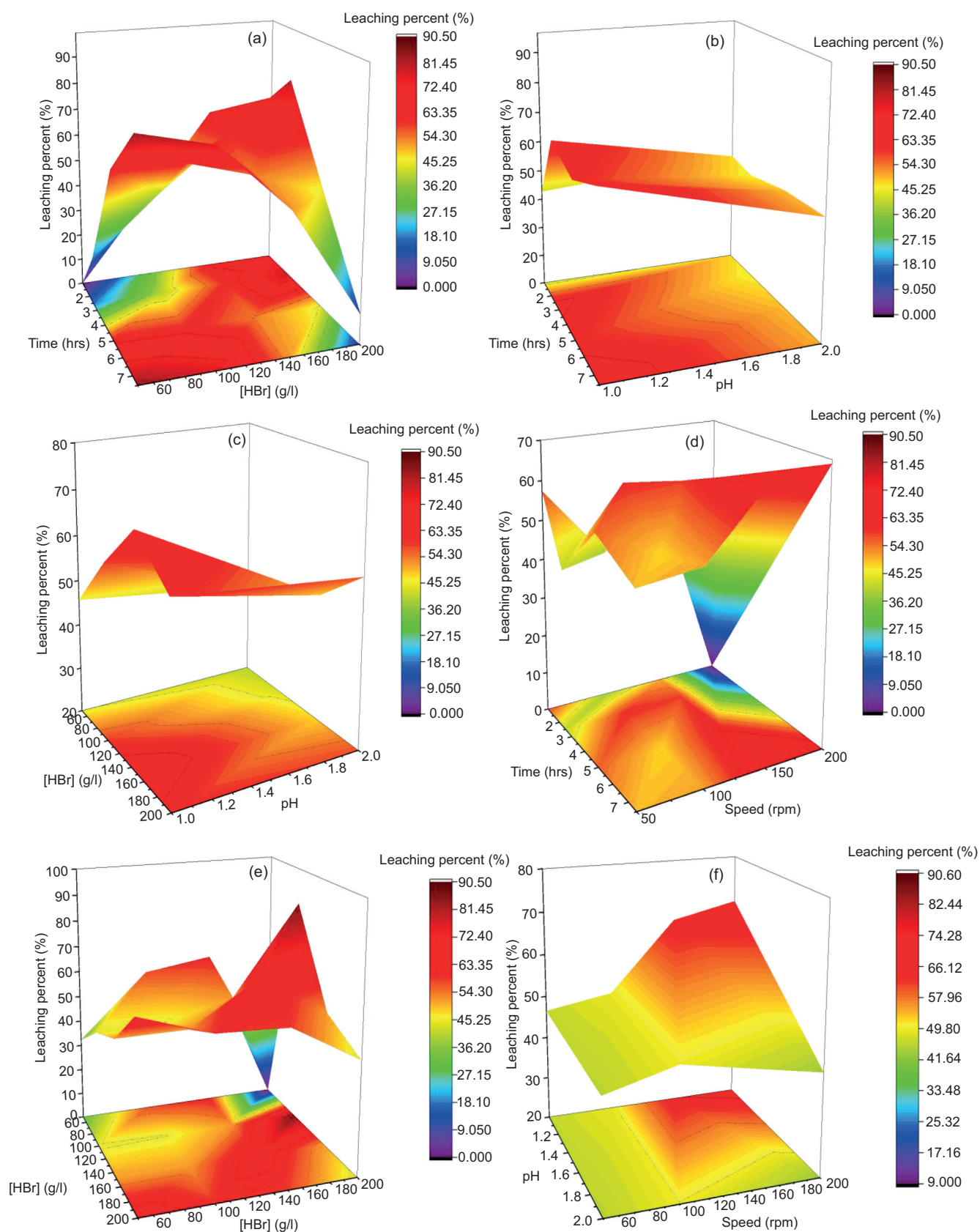


Fig. 4. Response surface plots for leaching tests holding other parameters at the center level: (a) time- hydrogen bromide concentration; (b) time-pH; (c) hydrogen bromide concentration-pH; (d) time-speed; (e) hydrogen bromide concentration-speed; (f) pH-speed.

TABLE VII. OPTIMIZATION FOR LEACHING TESTS.

| Case | Target | Time (h) | Hydrogen bromide concentration (g/L) | pH | Stirring speed (rpm) | Leaching percent (%) | Desirability |
|------------------|---------|----------|--------------------------------------|----|----------------------|----------------------|--------------|
| Leaching percent | Maximum | 7.27 | 50 | 1 | 200 | 98.540 | 1.00 |
| Variables | Minimum | | | | | | |

After analyzing the data presented in **table VII**, a significant finding emerged, and the optimized conditions for LT were a time of 7.27 h, an [HBr] of 50 g/L, a pH value of 1, and a stirring speed of 200 rpm. The outcome showed a 98.5% leaching percentage with a high probability of 95%. These conditions were chosen for subsequent experiments on account of their potential. The LT revealed that the gold concentration obtained in the filtrate was impressive (3372 ppm). These findings further reinforce the effectiveness of the selected leaching conditions and highlight the promise of this approach for extracting gold from the ore.

Neutralization test (NT)

During the experiments, all tests were conducted at room temperature. **Table VIII** presents the results derived from the model established using Minitab software. The regression analysis yielded an equation to calculate the neutralization percentage as follows:

$$y = -128.60 + 14.3489x_1 - 52.627x_2 + 68.667x_3 - 1.71718x_1^2 + 3.0403x_2^2 - 4.1153x_3^2 + 14.9734x_1x_2 - 4.07045x_1x_3 - 0.0077x_2x_3 \quad (10)$$

where y , x_1 , x_2 , and x_3 represent the neutralization percentage, the time, the [KOH], and the pH, respectively.

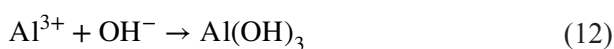
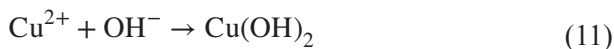
It is evident from equation 10 that the neutralization percentage is mainly dependent on x_3 (pH) and x_1 (time). An analysis of 2D contour plots and the 3D response surface is shown in **figure 5**.

In the time range of 2.00-2.50 and pH range of 5.05-6, **figure 5** indicates a prominent red zone (representing approximately an 87% yield). As the pH approaches values between 5.5 and 6, this red zone intensifies further. This observation can be attributed to the presence of other metals alongside gold in the ore, which tend to precipitate as the pH or [KOH] increases. Upon analyzing the data presented in **table IV**, it is evident that the ore contains various metals, including aluminum, cobalt, iron, copper,

TABLE VIII. RESULTS OF PERCENT REMOVAL FOR NEUTRALIZATION TESTS.

| Run No | Code level of variable | | | Percent removal (%) |
|--------|------------------------|-------|-------|---------------------|
| | x_1 | x_2 | x_3 | |
| 1 | 1 | 1 | 1 | 54.5 |
| 2 | 1 | 1 | 2 | 65.1 |
| 3 | 1 | 1 | 3 | 73.7 |
| 4 | 1 | 2 | 1 | 40.9 |
| 5 | 1 | 2 | 2 | 51.6 |
| 6 | 1 | 2 | 3 | 60.2 |
| 7 | 1 | 3 | 1 | 18.4 |
| 8 | 1 | 3 | 3 | 37.7 |
| 9 | 2 | 1 | 1 | 70.1 |
| 10 | 2 | 1 | 2 | 77.7 |
| 11 | 2 | 1 | 3 | 83.3 |
| 12 | 2 | 2 | 1 | 67.8 |
| 13 | 2 | 2 | 2 | 75.4 |
| 14 | 2 | 2 | 3 | 81.0 |
| 15 | 2 | 3 | 1 | 67.8 |
| 16 | 2 | 3 | 2 | 75.4 |
| 17 | 2 | 3 | 3 | 81.0 |
| 18 | 2 | 4 | 2 | 81.4 |
| 19 | 2 | 4 | 3 | 87.0 |
| 20 | 3 | 1 | 1 | 79.0 |
| 21 | 3 | 1 | 2 | 81.6 |
| 22 | 3 | 1 | 3 | 82.2 |
| 23 | 4 | 2 | 1 | 86.4 |
| 24 | 4 | 2 | 2 | 78.8 |
| 25 | 4 | 2 | 3 | 69.0 |

and others. During LT using MAR, HNO₃, and HBr, some of these metals are in dissolved form and exist in the solution as ions. However, when neutralization occurs with KOH, these metals are subjected to precipitation, following the chemical equations 11, 12, and 13, ultimately leading to an increase in the overall yield.



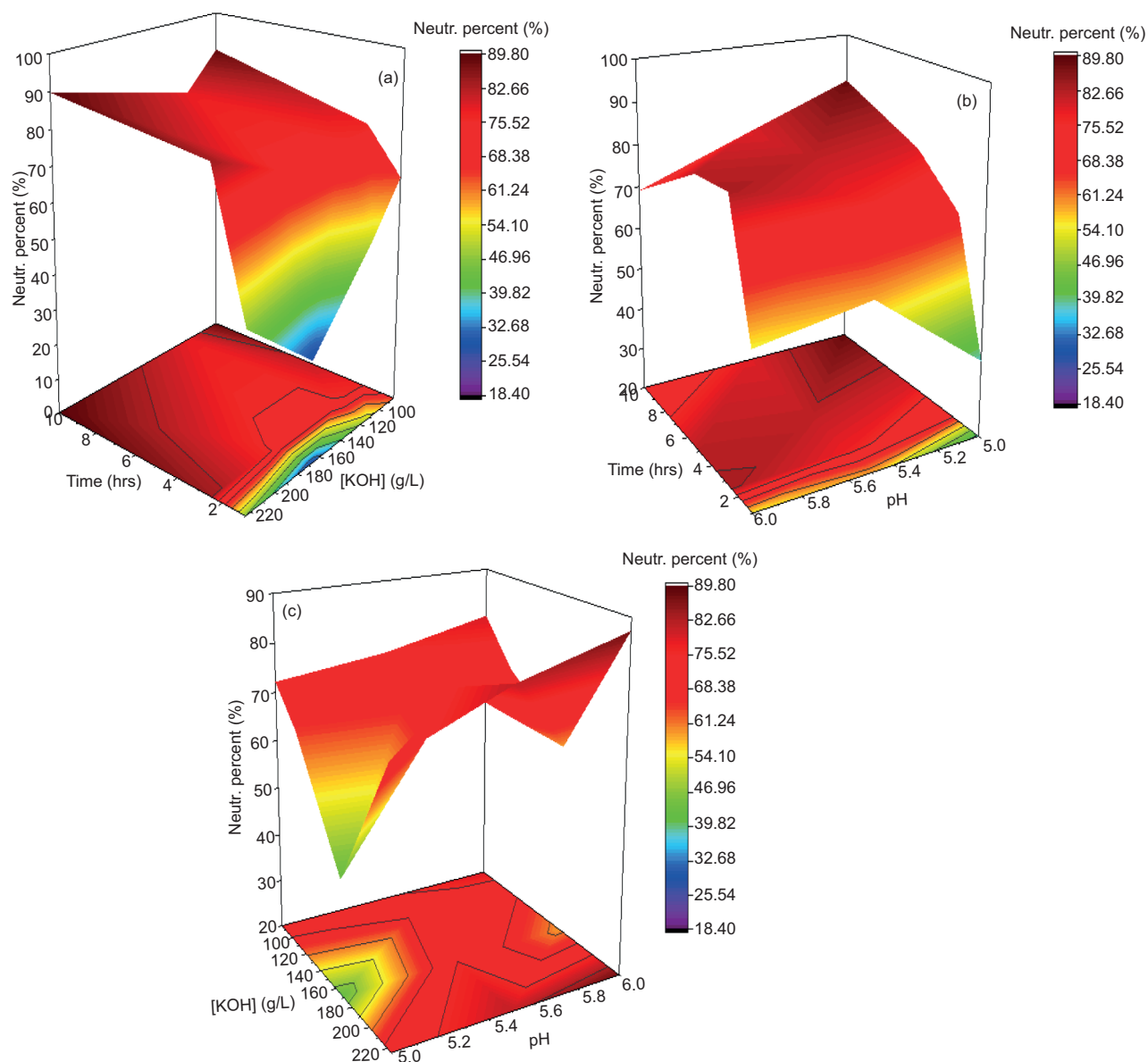
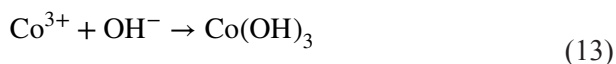


Fig. 5. Response surface plots for neutralization tests holding other parameters at the center level: (a) time-potassium hydroxide concentration; (b) time-pH; (c) potassium hydroxide concentration-pH.



Starting at pH 3.5, the precipitation of iron occurred due to the progressive oxidation of Fe^{2+} to Fe^{3+} caused by the presence of oxygen in the air. As demonstrated by Wei et al. (2005), aluminum precipitation occurred within the pH range of 4 to 5.5, while for copper, the precipitation began at pH 4, reaching completion at pH 6.

Gold recovery test with α -CD (GT- α)

This investigation focused on a specific range of conditions, considering room temperature and a pH range of 5-6. The time and $[\alpha\text{-CD}]$ varied within the ranges of 10-40 min and 5-20 g/L, respectively. To illustrate the outcomes of the extraction process using α -CD, **figure 6** presents the neutralized solution containing gold in two distinct states: (a) before the separation and (b) during the GT- α . Moreover, **table IX** presents the results of percent removal related to GT- α .

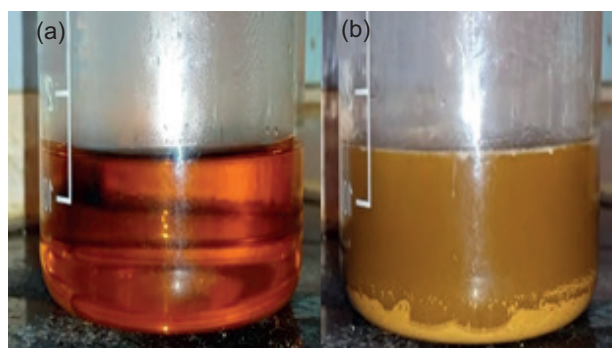


Fig. 6. GT- α . (a) Before adding alpha-cyclodextrin, (b) after adding alpha-cyclodextrin.

By conducting regression analysis on the gold recovery data presented in **table IX**, the following equation was derived:

$$y = -262.2 + 7.343x_1 + 16.209x_2 + 51.06x_3 - 0.001162x_1^2 - 0.45996x_2^2 - 0.953x_3^2 - 0.08385x_1x_2 - 0.9775x_1x_3 - 0.9306x_2x_3 \quad (14)$$

where y , x_1 , x_2 , and x_3 , represent the gold recovery percentage, the time, the $[\alpha\text{-CD}]$, and the pH, respectively. ANOVA results are presented in **table X**.

In **figure 7**, the results of 2D contour plots and the 3D response surface reveal the significance of all three parameters on the gold recovery percentage. The experiments exhibit good reliability and precision, as confirmed by the CV value (0.83%). Regarding the reaction process, upon adding the $\alpha\text{-CD}$ solution to the KAuBr_4 solution, a rapid reaction occurs, forming a shiny pale brown suspension from the first minute. This observation aligns with findings from Anthony et al. (2020), who propose a series of two coordination spheres for transition metals during the reaction. In the first coordination sphere, direct bonds link molecules and metal, alongside hydrogen bonds to ligands. On the other hand, in the second coordination sphere, the connection arises from the attachment between molecules in the first coordination sphere and the ions within the charged complexes bonded to the first coordination.

Significantly, the metal complexes' reactivity and chemical properties are more influenced by the first coordination sphere rather than the second. In a recent study by Karume et al. (2022), it was shown that during the GT- α process, AuBr_4^- ions become encapsulated within the cavity of the second coordination sphere. This cavity is formed between the

TABLE IX. RESULTS OF PERCENT REMOVAL FOR GOLD RECOVERY TESTS WITH ALPHA-CYCLODEXTRIN.

| Run N_0 | Code level of variable | | | Percent removal (%) |
|--------------|------------------------|-------|-------|------------------------|
| | x_1 | x_2 | x_3 | |
| 1 | 10 | 5.0 | 5.0 | 35.9 |
| 2 | 10 | 5.0 | 5.5 | 49.0 |
| 3 | 10 | 5.0 | 6.0 | 62.1 |
| 4 | 10 | 10 | 5.0 | 55.0 |
| 5 | 10 | 10 | 5.5 | 65.8 |
| 6 | 10 | 10 | 6.0 | 76.6 |
| 7 | 10 | 15 | 5.0 | 51.0 |
| 8 | 10 | 15 | 5.5 | 59.5 |
| 9 | 10 | 15 | 6.0 | 68.1 |
| 10 | 10 | 20 | 5.0 | 24.0 |
| 11 | 10 | 20 | 5.5 | 30.2 |
| 12 | 10 | 20 | 6.0 | 36.5 |
| 13 | 20 | 5.0 | 5.0 | 55.9 |
| 14 | 20 | 5.0 | 5.5 | 64.0 |
| 15 | 20 | 5.0 | 6.0 | 72.1 |
| 16 | 20 | 10 | 5.0 | 70.8 |
| 17 | 20 | 10 | 5.5 | 76.6 |
| 18 | 20 | 10 | 6.0 | 82.4 |
| 19 | 20 | 15 | 5.0 | 62.6 |
| 20 | 20 | 15 | 5.5 | 68.1 |
| 21 | 20 | 15 | 6.0 | 69.7 |
| 22 | 20 | 20 | 5.0 | 31.4 |
| 23 | 20 | 20 | 5.5 | 32.6 |
| 24 | 20 | 20 | 6.0 | 33.9 |
| 25 | 30 | 5.0 | 5.0 | 76.0 |
| 26 | 30 | 5.0 | 5.5 | 79.0 |
| 27 | 30 | 5.0 | 6.0 | 82.1 |
| 28 | 30 | 10 | 5.0 | 86.6 |
| 29 | 30 | 10 | 5.5 | 87.4 |
| 30 | 30 | 10 | 6.0 | 88.2 |
| 31 | 30 | 15 | 5.0 | 74.3 |
| 32 | 30 | 15 | 5.5 | 72.7 |
| 33 | 30 | 15 | 6.0 | 71.2 |
| 34 | 30 | 20 | 5.0 | 38.8 |
| 35 | 30 | 20 | 5.5 | 35.0 |
| 36 | 30 | 20 | 6.0 | 31.2 |
| 37 | 40 | 5.0 | 5.0 | 96.1 |
| 38 | 40 | 5.0 | 5.5 | 94.1 |
| 39 | 40 | 5.0 | 6.0 | 92.1 |
| 40 | 40 | 10 | 5.0 | 99.0 |
| 41 | 40 | 10 | 5.5 | 98.2 |
| 42 | 40 | 10 | 6.0 | 94.0 |
| 43 | 40 | 15 | 5.0 | 85.9 |
| 44 | 40 | 15 | 5.5 | 79.3 |
| 45 | 40 | 15 | 6.0 | 72.8 |
| 46 | 40 | 20 | 5.0 | 46.2 |
| 47 | 40 | 20 | 5.5 | 37.4 |
| 48 | 40 | 20 | 6.0 | 28.6 |

primary OH faces of the repeated face-to-face $\alpha\text{-CD}$ pairs, which are stabilized by hydrogen bonding interactions. Thus, the experimental results, coupled

TABLE X. DATA OF ANOVA OF GOLD RECOVERY TESTS WITH ALPHA-CYCLODEXTRIN.*

| Source | df | Sum of squares | Mean square | F-value | P-value Prob > F | Remarks |
|-----------|----|----------------|-------------|----------|---------------------|---------|
| 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 |
| $x_1 x_2$ | 1 | 1318.2 | 1318.17 | 3722.90 | <0.0001 | HS |
| $x_1 x_3$ | 1 | 955.6 | 955.60 | 2698.90 | <0.0001 | HS |
| $x_2 x_3$ | 1 | 216.5 | 216.51 | 611.50 | <0.0001 | HS |
| Error | 38 | 13.5 | 0.35 | | | |
| Total | 47 | 22868.7 | | | | |

*CV = 10.7%; $R^2 = 0.9003$; Adj. $R^2 = 0.7825$.

F: Fisher's function; df: degrees of freedom; P-value: level of significance; NS: not significant; S: significant; HS: highly significant.

with the coordination sphere insights, confirm the significance and reliability of the model for gold recovery.

In this unique chemical dance, K^+ molecules, α -CD, and $AuBr_4^-$ ions engage in a synchronized recognition and coordination, leading to the facilitation of a specific second-sphere interaction involving the aureate anion $[AuBr_4]^-$ and hexa-aqua cation $[K(OH_2)_6]^+$ in a non-covalent manner. Following the findings of Liu et al. (2014), the next step involves the rapid co-precipitation of α -CD and $KAuBr_4$ in water, eventually forming a needle-like 1:2 ratio complex $\{[K(OH_2)_6][AuBr_4] \subset (\alpha\text{-CD})_2\}_n$. After filtering the resulting co-precipitate, it undergoes reduction with $Na_2S_2O_2$ to free the enclosed gold from the inclusion complex.

The recovered gold is then characterized using AAS to determine the percentage of gold obtained. Interestingly, as observed from **figure 7b, d**, time directly influences the gold recovery percentage. Notably, gold recovery initiates from the first minute of the reaction, but it takes approximately 28.5 min to reach the dark red zone, where a yield of at least 80% is achieved. **Figure 7b, f** reveals that the percentage yield within the range of 80-89% is associated with $[\alpha\text{-CD}]$ s ranging from 5.75 to 14 g/L. Nevertheless, attaining yields of at least 98% and aligning with the dark red zone requires a reduction in the $[\alpha\text{-CD}]$ range to 5.75-10.21 g/L.

Also, considering the interaction between $[\alpha\text{-CD}]$ and pH, a yield of at least 86% is observed when the pH and $[\alpha\text{-CD}]$ are maintained at 5.65 and 8 g/L,

respectively. On the other hand, analyzing the interaction between pH and time, a yield of at least 80% is achieved when the pH and time are maintained at 5-5.8 and 35 min, respectively. This observation is explained by the efficient elimination of a few accompanying gold metals through NT.

In **figure 7**, the results indicate that the recovery percentage increases with an increase in $[\alpha\text{-CD}]$ up to a certain maximum value, after which it decreases. This behavior can be attributed to the requirement of a perfect molecular recognition match within the formed complex, which needs a specific ratio of 1:2 between $[AuBr_4]$ and $[\alpha\text{-CD}]$. Deviations from this ratio result in a decrease in the percentage of recovery. To achieve better removal of gold, the value of $[\alpha\text{-CD}]$ should ideally be around twice that of $[AuBr_4]$. However, the time variation closely mirrors the recovery percentage changes. Additionally, within the chosen pH range (5-6), pH has minimal influence, confirming effective neutralization. The RSM method was employed to predict optimization conditions, and the results are detailed in **table XI**.

The success of GT- α has been confirmed through analysis of the obtained organic phase and raffinate by AAS. Under specific conditions (a time of 40 min, $[\alpha\text{-CD}]$ of 11.6166 g/L, and pH of 5), there is a 95% degree of confidence that the gold recovery will be 98.9% with a desirability of 0.99978. The results also reveal that the concentrations of gold in the organic phase and raffinate were 11 945 ppm and 306 ppm, respectively.

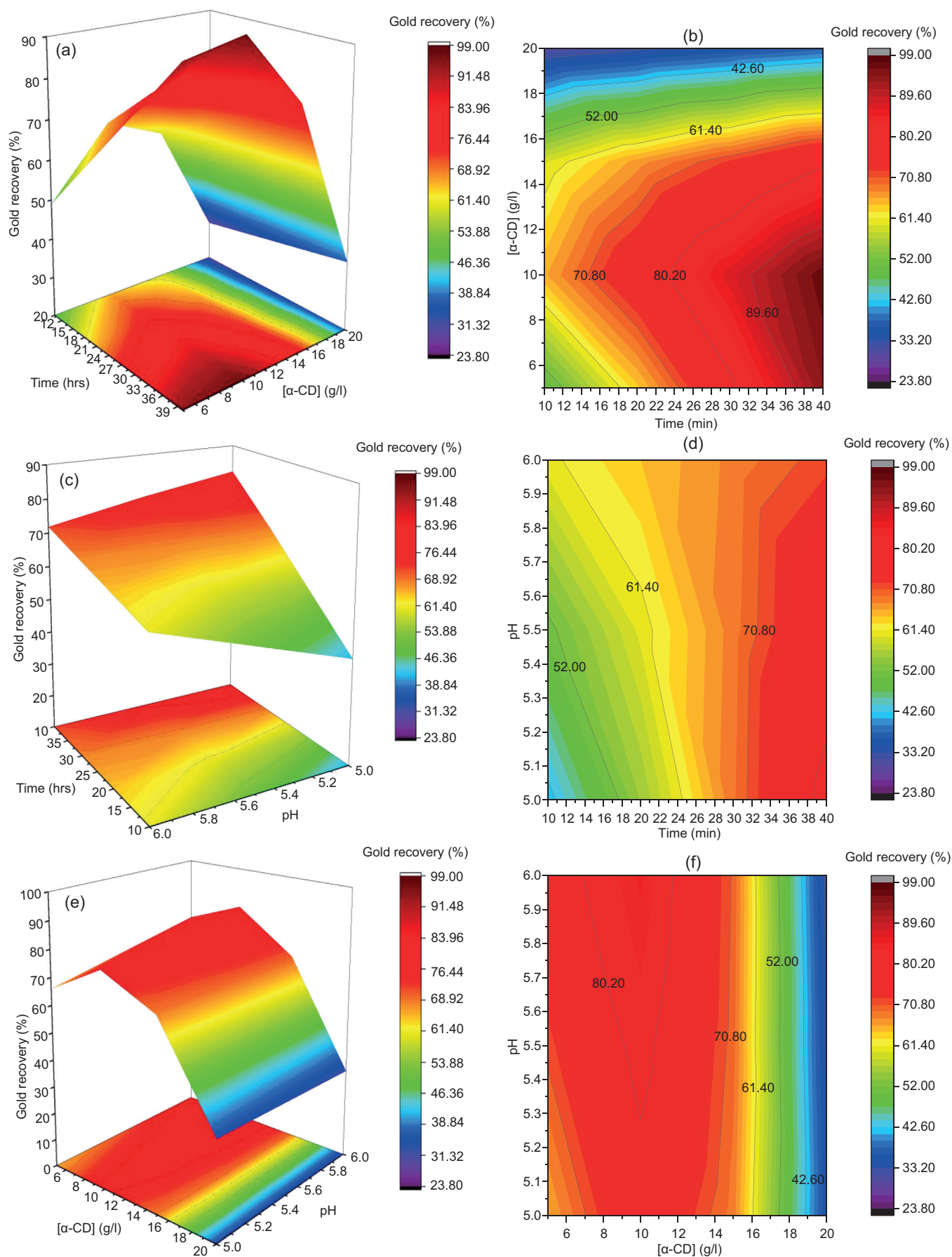


Fig. 7. Response surface plots for gold recovery tests with alpha-cyclodextrin holding other parameters at the center level: (a, b) time-alpha-cyclodextrin concentration; (c, d) time-pH; (e, f) alpha-cyclodextrin concentration-pH.

TABLE XI. OPTIMIZATION FOR GOLD RECOVERY TESTS WITH ALPHA-CYCLODEXTRIN.

| Case | Target | Time (min) | Alpha-cyclodextrin concentration (g/L) | pH | Gold recovery (%) | Desirability |
|---------------|---------|------------|--|----|-------------------|--------------|
| Gold recovery | Maximum | 40 | 11.6166 | 5 | 98.87 | 0.99978 |
| Variables | Minimum | | | | | |

CONCLUSION

In this study, the use of eco-friendly alpha-cyclodextrin (α -CD) as an alternative method for gold extraction from ore in South Kivu, Democratic Republic of the Congo, was investigated. The results demonstrated that α -CD offers a sustainable and promising approach to replace harmful gold extraction methods. To optimize the gold removal process, ANOVA and RSM were employed. Initially, a randomly taken ore sample contained 0.06% of gold using AAS.

The leaching process was then optimized using MAR with specific parameters: 7.27 h, 50 g/L [HBr], pH 1, and 200 rpm stirring speed, resulting in an impressive 98.5% gold removal. An optimal extraction medium was achieved for subsequent tests by conducting neutralization tests using KOH to prepare the extraction solution with the appropriate pH. The variation of parameters such as time, KOH concentration, and pH were crucial in reaching this achievement. Extraction tests with α -CD were performed and optimized with the following parameters: extraction time of 40 min, α -CD concentration of 11.6 g/l, and pH of 5, resulting in a remarkable gold removal of 98.9%.

Lastly, in the leaching test, an impressive gold content of 3372 ppm was observed in the filtrate, further validating the efficacy of the α -CD extraction process, while in the gold test, the concentrations of gold in the organic phase and raffinate were measured at 11 945 ppm and 306 ppm, respectively.

Overall, this study showcases the potential of α -CD as a greener and more efficient method for gold extraction, offering a significant step forward in environmentally responsible mining practices.

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