Modeling and Box-Behnken design optimization of microwave treatment of sulphidic gold flotation tailing

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Abstract: As raw minerals become scarcer every passing day, the need for the recovery of mine tailings becomes essential. This research highlights the use of microwave energy as a green alternative to otherwise environmentally harmful methods of ore tailing recovery. The obtained results indicate that a 1.4 ppm Au and 3.5 % S sample floated with Aeroflot 208 and Aerophine 3418A increased the concentration of tailings over 18 % S and 4 ppm, Au, for recovery yield, resulting in 84 % and 80 % recovery, respectively. After microwave irradiation, 90 % of sulphur removal was reached under the optimum conditions of 50 minutes of irradiation using 1000 W for 4 g of the sample. Overall with 96.74 % correlation of the quadratic model using the Box-Behnken design and expressed coefficient R2 regression the model was proven to be suitable for heating and roasting processes of gold-bearing tailings.

Keywords: microwave heating, gold-bearing high sulfdie concentrate, bulk flotation, optimization, Box-Behnken experimental design

1. Introduction

It was around 6000-3000 B.C. where smelting of gold began around Mesopotamia. The technique most commonly used in extraction was by crushing, washing, and then applying heat enough to roast the material powdered (Adams, 2016). Today this is done by a process involving gas-solid reactions at high temperatures to purify the metal components, namely gold. This technique is effective when ore contains sulfur minerals and carbonaceous matter, however it is also a major contributor to air pollution.

As roasting is an exothermic process, it releases alarming rates of acidic and toxic compounds resulting in areas that are hundreds of meters wide and are largely still lifeless even after around 70 years since being of use (Wong et al., 1999). The temperature-sensitive process demands accurate monitoring and precise control, otherwise excessive heating leads to sintering to cause the closure of the pores and reduction in gold leaching, while a low-temperature roasting could be successive in liberating the gold particle as well as oxidizing the sulfur to the desired extent (Dougherty et al., 2020). It also requires high amounts of energy consumption making these traditional methods not economical for many countries.

For these reasons the development of an innovative, sustainable, and more efficient method is essential. In this study microwave energy is proposed as an alternative green method for thermal processes to be used in the mining industry, particularly in the heating and roasting processes as an inexpensive, less energy-consuming, sustainable pre-treatment method (Pickles, 2009; Benli and Adem, 2020; Lopez-Hortas, et al. 2022).

While conventional heating methods have the disadvantage of overheating and wasteful heating of insulators (such as gangue minerals), microwave energy has the advantage of selectively heating individual mineral phases within a mass, creating differential heating at the grain boundaries causing better liberation of valuable mineral grains. Thermal properties, including thermal conductivity and specific heat capacity, are also essential parameters that impact the heating rate in a heat transfer model to define thermal regions and evaluating the temperature distribution in the ore. During the heating time, the thermal conductivity will influence the diffusion of thermal energy from hot spots to cold
regions (Çengel, 2004). This selectivity is influenced by dielectric properties of ore and gangue minerals including gold, metals, sulfides, silicates, and carbonates as seen in Table 1.

Table 1. Dielectric and thermal properties of minerals present in typical gold ore

| Mineral     | Dielectric constant $\varepsilon'$ | Thermal conductivity W/m.K | Specific gravity SG | Specific heat capacity kJ/kg.K |
|-------------|------------------------------------|-----------------------------|---------------------|--------------------------------|
| **Silicates** |                                    |                             |                     |                                |
| Quartz      | 6.83                               | 7.6                         | 2.65                | 0.7                            |
| Sand        | 3.8                                | 1.2                         | 2.25                | 0.62                           |
| Microcline  | 6.92                               | 2.47                        | 2.560               | 0.8                            |
| Albite      | 6.02                               | 2.3                         | 2.620               | 0.85                           |
| Orthoclase  | 6.20                               | 2.2                         | 2.570               | 0.8                            |
| Mica        | 3-6                                | 0.71                        | 2.8 to 3.0          | 0.88                           |
| Talc        | 9.41                               | 2.92                        | 2.76                | 0.208                          |
| Biotite     | 9.28                               | 2.1                         | 3.2                 | N.A.                           |
| Muscovite   | 10                                 | 2.3 to 2.9                  | 2.77 to 2.88        | 0.207                          |
| Wollastonite| 6.17                               | 2.7                         | 2.909               | 0.1832                         |
| Olivine     | 6.77                               | 3.14                        | 3.2 to 4.4          | 0.79                           |
| Serpentine  | 11.48                              | 2.78                        | 2.5 to 2.6          | 1.09                           |
| **Sulfides** |                                    |                             |                     |                                |
| Pyrite      | 33.7 to 81                          | 38.9 to 41.4                | 5.016               | 0.5                            |
| Chalcopyrite| 33.7 to 81                          | 10.7                        | 4.1 to 4.3          | 0.54                           |
| Arsenopyrite| over 81                             | N.A.                        | 6.1                 | 0.43 (55°C)                    |
| Sphalerite  | 5.29                               | 26.7                        | 4.1                 | 0.45                           |
| Galena      | over 81                             | 2.0                         | 7.6                 | 0.21                           |
| Sulfur      | 8.45                               | 0.2                         | 2.1                 | 0.71                           |
| **Carbonates** |                                  |                             |                     |                                |
| Calcite     | 6.36                               | 3.8                         | 2.7                 | 0.793                          |
| Dolomite    | 8.45                               | 5.7 to 6.3                  | 2.9                 | 0.87                           |
| Carbon      | 3.0                                | 1.60                        | 2.3                 | 0.192                          |
| **Oxides**  |                                    |                             |                     |                                |
| Magnetite   | 33.7 to 81                          | 3.7 to 5.0                  | 4.9 to 5.2          | 0.78                           |
| Ilmenite    | 33.7 to 81                          | N.A.                        | 4.5 to 5.0          | 0.849                          |
| ** Metals** |                                    |                             |                     |                                |
| Gold (pure) | over 81                             | 317                         | 19.3                | 0.129                          |
| Silver (pure)| over 81                           | 410                         | 10.5                | 0.230                          |
| Copper (pure)| over 81                           | 385                         | 8.92                | 0.387                          |
| Iron (pure) | N.A.                               | 73                          | 7.86                | 0.448                          |
| Water       | 80.0                               | 0.58                        | 1.0                 | 4.182                          |
| Air         | 1.0                                | 0.025                       | 0.001               | 1.005                          |

Note: N.A. – not available. Data are from (read from Rosenholtz and Smith et al., 1936; Hemingway et al., 1981; Khesin and Eppelbaum, 1994; Popov et al., 1987; Bundschuh and Suárez, 2010)

1.1. Literature survey

Previous studies already show that microwave treatment is a sufficient pre-treatment method for various minerals. Microwave irradiation pretreatment was called a new type of clean energy for coals (Ma et al., 2020), increasing desulfurization of coal after adding magnetite enhanced magnetic separation of FeS2 (Uslu ve Atalay, 2004), grindability of high-ash coals (Sahoo et al., 2011), oxidized coal flotation (Xia et al., 2013), microwave-heated effects on phase transformation (Yang et al., 2016), preg-robbing (Wei Sung et al., 2020), magnetic separation of Fe and Ti (Kim et al., 2019), liberation of oolitic iron ore (Qin, et al., 2022), phase transformations of ilmenite ore (Myslyvchenko, et al. 2022), Cr
recovery (Peng et al., 2020), As removal (Ma et al. 2010), Cu recovery (Kingman et al., 2004), Mn ore (Li et al., 2020), the roasting of flash flotation concentrate (Amankwah and Ofori, 2020). The attention of researchers is primarily concentrated on liberation of coal and separation of pyrite, although a few processes have focus on refractory gold and gold tailings.

In this research, the aim is to show the effectiveness of microwave energy on the reduction of sulphur content during thermal pretreatment of sulphidic concentrate from two-stage flotation process and to investigate the recovery potential of fine-grained flotation tailings. Response surface methodology, namely central composite design, 3-factor 3-level Box-Behnken experimental design were applied to model and optimize the influence of some parameters affecting microwave heating performance. Microwave irradiation power, microwave processing time, and sample mass were selected as the parameters in this optimization. Findings of the Box-Behnken experimental design and mineralogy of the sample were evaluated for the flotation plant tailing minerals.

2. Materials and methods

2.1. Materials

The gold ore sample used in this research was obtained from Turkey’s refractory gold ore in the western region of Turkey. The sample containing 1.4 ppm Au and 3.5 % S was crushed and milled through a primary jaw and roll crusher to all passing 2 mm. Before the flotation experiments, 1 kg of homogenized samples was ground in a laboratory size ball mill for 30 min to completely passing 106 µm, d$_{80}$ and d$_{70}$ passing of 53 µm and 38 µm, respectively. Flotation concentrates were firstly obtained from a two-stage flotation from a refractory gold ore flotation plant tailing in Turkey. Then, the obtained highly sulphidic flotation concentrate was exposed to microwave pretreatment.

2.2. Flotation experiments

In the flotation experiments, 1 kg of the ground sample pulped to 25 % solids were carried out at a laboratory-scale Denver flotation machine using a 2.5 L Denver cell. During two-stage rougher flotation, 200 g/t collector using the combination of Aero208 and Aerophine3418A (at a ratio of 1:1) collectors (Cytec series), 2000 g/t depressant (sodium silicate, Na$_2$SiO$_3$), and 50 g/t frother (methyl isobutyl carbinol, MIBC) were also added to the pulp at pH 4-4.5 and conditioned for 10 min and 5 min, respectively.

2.3. Microwave heating experiments

Microwave heating tests were conducted on 2-6 g of concentrate samples in a programmable domestic microwave oven (Arcelik MD554, Turkey) with the maximum output of 1200 W at 2.45 GHz. Ore samples were placed in 25 mL porcelain crucibles and later put into a 250 mL porcelain crucible filled with magnetite particles as shown in Fig. 1. Magnetite particles were used as a microwave absorber to increase the heat efficiency of the system. The crucibles were then placed on an alumina platform to serve as an insulator. The microwave heating experiments were performed in an open air atmosphere.

![Microwave heating system](image)

**Fig. 1.** Microwave heating systems are used in the experiments

The microwave furnace was operated at power levels of 360-1000 W. In-situ temperature changes of the ore sample were measured using a quartz glass thermometer up to 400 °C and for a higher range determined with a thermocouple. Following the heating process, the samples were taken out of the
oven, cooled rapidly in a desiccator at room temperature, separately ground, and stored at -18 ºC in polyethylene bags.

2.4. Characterization methods

The gold content of the samples was analyzed using the fire assay (cupellation) method by ALS Laboratory (İzmir). The elemental composition of the sample determined by Inductively coupled plasma mass spectrometry (ICP-MS). The mineralogical investigations were also characterized by polished and thin sections, and XRD diffraction patterns at Şişecam, R&D Center (İstanbul) using a Bruker XRF analyzer employing Rh X-ray tube and LIF 200 crystal. Thermal properties of the concentrate were performed on a thermogravimetric analyzer (DTGA)-TA-Q600 SDT instrument and universal analysis 2000 software environment. After microwave tests, the ignition loss of pre-treated samples was measured at 110 ºC in an oven (TEST, T420S, Turkey) and a digital balance of 0.001 g accuracy (Precisa, XB620, Switzerland). Finally, the Sulphur contents of the microwave test samples were analyzed by PC controlled ELTRA CS580 Elemental Analyzer.

2.5. Calculations: Box-Behnken experimental design

Box–Behnken experimental design and response surface methodology for modeling of microwave heating processes were preferred with the three levels and three variables since it offers a practical advantage in requiring a fewer number of runs. The total number of experiments was calculated according to Eq.1. The selected three important process variables are given in Table 2; sample mass (x1), microwave processing time (x2), and microwave power (x3). A total of 15 experimental runs was generated with 12 design points and 3 replications of center points.

\[
\text{Total number of experiments (N)} = 2 \left[ k (k - 1) \right] + n_c = 2 \left[ 3 (2) \right] + 3 = 15 \quad (1)
\]

where, \( k \) is the number of variables, and \( n_c \) is the number of center points.

| Independent variables | Levels of variables |
|-----------------------|---------------------|
|                       | Low | Middle | High |
| \( x_1 \): Sample Mass (g) | -1  | 0     | +1   |
| \( x_2 \): Processing Time (min) | 20  | 40    | 60   |
| \( x_3 \) Microwave Power (W) | 360 | 680   | 1000 |

To explain the optimization of the process parameters according to the removal amount of Sulphur during the process which is the dependent variable (Y), the second-order polynomial model is proposed to the predicted response using the following function.

\[
Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_1 x_2 + \beta_5 x_2 x_3 + \beta_6 x_1 x_3 + \beta_7 x_2 x_3 + \varepsilon \quad (2)
\]

in this model, \( \beta_0 \) as the intercept, and \( \beta_1 \) to \( \beta_3 \) as the linear regression coefficient, \( \beta_4 \) to \( \beta_7 \) as quadratic coefficients, \( \beta_7 \) to \( \beta_9 \) as cross-product coefficients, and \( \varepsilon \) is a random error that normally distributed with zero mean and constant variance. Interaction and the main effect of independent variables have been estimated from the experimental results (Minitab, 2019).

On the other hand, during the microwave heating studies, Sulphur removal percent \( (S_t) \) was calculated using the following equations:

\[
S_t = (1 - \frac{m_0 - m_t}{m_0}) \times 100 \quad (3)
\]

here, \( m_0 \) is an initial amount and \( m_t \) is a Sulphur content at a specific time.

Microwave power efficiency is related to a ratio of the reflected \((P_r)\) and transmitted \((P_t)\) amount of microwave power (Buffler, 1993), using the dielectric constant \((\varepsilon')\) of the minerals in Table 1 as follows;

\[
P_r = \left( \frac{\varepsilon' - 1}{\varepsilon' + 1} \right)^2 \quad (4)
\]

and,
\[ P_t = 1 - P_r \] (5)

3. Results and discussion

3.1. Characterization of flotation tailing

The gold content and elemental composition of the sample are given in Table 3. It was found that low-grade flotation tailing contains 2.6 ppm silver (Ag), 255 ppm arsenic (As), 41 ppm antimony (Sb). In Fig. 2a, the presence of quartz together with pyrite, sphalerite, and calcite proved in the XRD diffraction pattern of the sample. Muscovite which is a highly floatable mineral was also detected. Micro analyses were also made from dispersive spectroscopy images and elemental mapping (Fig. 2b) by scanning electron microscopy/energy dispersive spectroscopy (SEM-EDS) method.

| Microelements | ppm |
|---------------|-----|
| Au            | 1.37|
| Ag            | 2.6 |
| As            | 255 |
| Ba            | 80  |
| Ca            | 0.58|
| Cd            | 12.4|
| Co            | 11  |
| Cr            | 203 |
| Cu            | 105 |
| Mn            | 253 |
| Ni            | 27  |
| P             | 1660|
| Pb            | 41  |
| Sb            | 41  |
| Sr            | 808 |
| Ti            | 26  |
| V             | 106 |
| Zn            | 2470|
| Fe            | 3.81|
| S             | 3.47|

Table 3. ICP-MS analysis of the low-grade gold flotation tailing (ppm)

Fig. 2. Ore characteristics (a) XRD pattern for gold ore flotation tailing include silicates, pyrite, and galena (b) SEM-EDS results of the polished sample

3.2. Units bulk flotation without microwave pretreatment

A series of flotation tests were performed to obtain the sample that will be used for the microwave heating process from the complex and low grade of the ore. The results of two-step rough flotation carried out while varying the concentration of a combination of Aeroflot 208 and Aerophine 3418A collectors at the constant pH of 4 - 4.5, showed that the flotation recovery of gold increases to a concentration of total 100 g/t of collector, with decreasing selectivity, it then decreases considerably (Fig. 3). Total Sulphur content has been increased to about 19 % S and 84 % yield (Table 4), similar to Forrest et al. (2001), which shows the importance of not overdosing collector.
Physicochem. Probl. Miner. Process., 58(5), 2022, 149929

Fig. 3. Effect of collector concentration on flotation recovery, using a combination of Aerofloat 208 and Aerophine 3418A

Table 4. Percent of flotation recovery results based on the concentration of collector combination

| Collector Concentration (g/t) | Products | Amount | Content | Recovery |
|------------------------------|----------|--------|---------|----------|
|                              |          |        | %       | Au, ppm  | S, %    | Au, %   | S, %    |
| 50+50                        | Concentrate | 24 | 4.34 | 18.5 | 82.4 | 84.0 |
|                              | Tailing   | 76 | 0.32 | 0.7  | 17.7 | 16.0 |
|                              | Feed      | 100 | 1.37 | 3.9  | 100.0 | 100.0 |
| 100+100                      | Concentrate | 27 | 4.12 | 19.2 | 82.0 | 78.8 |
|                              | Tailing   | 73 | 0.34 | 0.9  | 18.0 | 21.2 |
|                              | Feed      | 100 | 1.37 | 3.9  | 100.0 | 100.0 |
| 200+200                      | Concentrate | 23 | 4.77 | 20.1 | 79.1 | 81.7 |
|                              | Tailing   | 77 | 0.37 | 0.8  | 20.9 | 18.3 |
|                              | Feed      | 100 | 1.37 | 3.9  | 100.0 | 100.0 |

Fig. 4 presents the compositional changes of the gold ore concentrate during roasting by thermogravimetric analysis (TGA) and derived thermogravimetric analysis (DTGA). When the sample was heated from room temperature to 850 °C, the mass-loss rate increased rapidly and reached 12.1 %. The mass losses below 400 °C correspond to dewatering and removal of organics from the sample. The major mass loss of over 10 % between 450 °C - 700 °C for the oxidation of sulfide mineral pyrite to hematite and sulfur dioxide according to the following main combustion reaction during the heating process;

\[ 4\text{FeS}_2 + 11\text{O}_2 \leftrightarrow 2\text{Fe}_2\text{O}_3 + 8\text{SO}_2 \]  

Similar to pyrite, other main reactions belong to sulfur minerals like arsenopyrite in the refractory ore as follows;

\[ 2\text{FeAsS} + 5\text{O}_2 \leftrightarrow \text{Fe}_2\text{O}_3 + \text{As}_2\text{O}_3 + 2\text{SO}_2 \]  

Hu et al., (2017) studied in the same temperature range and obtained high arsenic sulfide concentration with relative content of about 10 % of sulfide, and the mass loss reached 27.5 %. Double refractory gold ore was heated from 420 °C to 520 °C, 1.7 % of mass loss from DTGA was almost the same as the theoretical mass loss (Nanthakumar et al., 2007). Comparison to these refractory ores, in our case 18 % of sulfide content flotation concentrate was heated up between 420 °C and 476 °C, and 4.5 % of mass loss were obtained from the DTGA curve corresponding to near its theoretical mass loss. In addition to sulfide decomposition, the TGA curve of the refractory gold flotation concentrate has a 1.5 % minor mass loss for the combustion of carbonaceous matter (given in Eq. 4) between 530 °C to 630 °C. The decomposition of the dolomite to magnesium oxide and calcium oxide (from 690 °C to 775 °C).
and then, the calcite decomposition occurs over the temperature range of about 775 - 830 °C. About 0.3 % and 0.5 % of mass loss for the case of dolomite and calcite decomposition were obtained as follows:

\[
\begin{align*}
C + O_2 & \leftrightarrow CO_2 \quad (8) \\
CaCO_3 & \leftrightarrow CaO + CO_2 \quad (9) \\
CaCO_3\cdot MgCO_3 & \leftrightarrow CaO + MgO + 2CO_2 \quad (10)
\end{align*}
\]

Fig. 4. TGA/DTGA curves of gold-bearing high sulfide concentrate

3.3. Modeling studies of the microwave heating process

The observed experimental results of the removal percent of Sulphur content during the microwave heating process of refractory gold flotation concentrate were fitted with a quadratic regression equation (Eq. 11) of total amount, microwave energy application time, and microwave energy power. Therefore, the response (Y, removal %) was expressed as a function of the sample mass (x₁), microwave processing time (x₂), and microwave irradiation power (x₃) for any coded level as below:

\[
Y = -106.016 + 30.535 x_1 + 0.556 x_2 + 0.083 x_3 - 0.765 x_1^2 + 0.028 x_2^2 + 4.22x10^{-5} x_3^2 - 0.447 x_1 x_2 - 0.009 x_1 x_3 - 1.93x10^4 + x_2 x_3 \quad (11)
\]

Table 5 presents the experimental and predicted response of the model for the 15 runs. The Sulphur removal efficiency of flotation concentrate increases while ore amount (x₁) increases up to 4 g as shown in Fig. 5. This initial evolution of S removal is due to an increase in the ratio of microwave absorber minerals such as pyrite, arsenopyrite, and galena in refractory ore to the surface area/ bulk volume of the porcelain crucible. For the sample amounts higher than 4 g, the observed decrease of removal efficiency indicates that the efficient penetration of the microwave through the material is entirely lower by thermal conduction of the ore restricts the internal temperature distribution in the bulk ore. The MW time (x₂) and power (x₃) have a positive effect due to the competition of microwave penetration and enough thermal conduction heat transfer. Fig. 6 shows high temperature also causes the color differences between the raw concentrate and microwave treated concentrate.

The correlation of the coefficient R² of the regression quadratic model was 97.4 %, suggesting that the relationship between the dependent and independent variables could be described well using this model. The relationship of the predicted and experimental Sulphur removal percent by microwave oxidation is determined that a strong linear relationship between the predicted values and the actual values. Our results showed that the established three-factor Box-Behnken model could predicate the removal percent of Sulphur by microwave energy pretreatment and linear regression equation and interaction (Table 6) are significant.

Fig. 5. Influence of the process factors [ore amount (x₁, g), MW process time (x₂, min), and MW power (x₃, MW)] on the mean value of sulphur removal efficiency
Fig. 6. Color changes of refractory gold flotation concentrate on the amount of ore

Table 5. Three-factor Box-Behnken experimental design and observed response

| Rank | Coded Levels of parameters | Actual | Response | Sulfur Removal, % |
|------|---------------------------|--------|----------|-------------------|
|      | $x_1$ | $x_2$ | $x_3$ | Mass (g) | Time (min) | Power (W) | Experimental, % | Predicted, % |
| 1    | 0     | 0     | 0     | 4     | 40     | 680     | 44.17          | 43.75          |
| 2    | 0     | 1     | 1     | 4     | 60     | 1000    | 98.08          | 99.01          |
| 3    | 1     | 1     | 0     | 6     | 60     | 680     | 58.42          | 51.80          |
| 4    | 0     | -1    | -1    | 4     | 20     | 360     | 18.17          | 10.35          |
| 5    | -1    | -1    | 0     | 2     | 20     | 680     | 9.79           | 16.41          |
| 6    | 0     | 1     | -1    | 4     | 60     | 360     | 51.10          | 48.61          |
| 7    | -1    | 0     | 1     | 2     | 40     | 1000    | 90.46          | 81.35          |
| 8    | 1     | 0     | 1     | 6     | 40     | 1000    | 69.64          | 68.44          |
| 9    | 0     | 0     | 0     | 4     | 40     | 680     | 42.92          | 43.75          |
| 10   | -1    | 0     | -1    | 2     | 40     | 360     | 7.88           | 9.08           |
| 11   | 1     | 0     | -1    | 6     | 40     | 360     | 12.08          | 21.19          |
| 12   | 0     | 0     | 0     | 4     | 40     | 680     | 44.17          | 43.75          |
| 13   | 0     | -1    | 1     | 4     | 20     | 1000    | 70.98          | 72.58          |
| 14   | 1     | -1    | 0     | 6     | 20     | 680     | 69.64          | 68.44          |
| 15   | -1    | 1     | 0     | 2     | 60     | 680     | 86.65          | 87.94          |

Fig. 7. Parity plot for predicted and experimental data for Sulphur reduction from gold flotation concentrate

Table 6. Analysis of variance (ANOVA) of the response surface model to predict the removal % of sulphur

| Source of variation | Degree of freedom | Sum of squares | Adjusted mean square | F ratio | P-value |
|---------------------|-------------------|----------------|----------------------|---------|---------|
| Regression          | 9                 | 11723.6        | 1302.62              | 16.47   | 0.003*  |
| Linear              | 3                 | 9704           | 3234.66              | 40.9    | 0.001*  |
| Quadratic           | 3                 | 579.5          | 193.18               | 2.44    | 0.18    |
| Interaction         | 3                 | 1440.1         | 480.02               | 6.07    | 0.04**  |

*P < 0.01, **P < 0.05
3.3. Three-dimensional response surface plots of the parameters

The relation between the variable and response was further elucidated using a Pareto chart (Fig. 8a) and a three-dimensional response surface (Fig. 8b-d). Microwave power appears to have the greatest impact of 60 % (probability, p < 0.001), after which microwave process time also seems to be an effective parameter with 20 % and p < 0.001. Whereas, the lowest effect was seen in the mass with a p-value of 0.952. The microwave application time should be increased as the sample amount increases. According to the Pareto diagram the effect of mass is much less among others, the applications in a short time, the amount of the sample should be kept low. Even so, when applied microwave under 30 minutes, the removal S remains below 40 % efficiency.

Fig. 8c also confirms the effect of application time and power on the removal efficiency of Sulphur from the gold ore flotation concentrate. It has been observed in Fig. 8d that the increase in mass does not have a significant effect by increasing the microwave power applied, the efficiency was still below 25 %, and the excessive energy use of low-mass works with high applications in high power applications causes an increase in energy consumption, which will lead to waste of resources. Since the presence of microwave-retaining minerals is related to the effectiveness of microwave energy transfer depending on the time applied to refractory gold ore at 800 W (Ma et al., 2010), by supporting our experimental findings, they demonstrated the benefits of applying microwave energy for 80 min higher than our findings.

After having studied the effect of independent variables on the response, the optimum values of the selected variables were obtained by solving the quadratic regression model, as well as by analyzing the response surface plots. Hence, all the variables were decided in range and the optimum formulation was the one that gives a higher percentage of Sulphur removal in microwave heating processing efficiency. A diagram of optimization obtained with maximum desirability (d=1), given by Minitab 16. Optimum microwave heating process parameters for $x_1$, $x_2$, and $x_3$ were found to be 4 g sample, 50 min microwave irradiation time, and 1000 W microwave irradiation power with a prediction of 90 % for Sulphur removal.

3.4. Evaluating the power efficiency of the microwave compared to conventional heating

Fig. 9 shows how important ore mineralogy is when microwave heating is applied, which only heats the precious ore instead of gangue. $P_r$ and $P_l$ values are calculated using the dielectric constant ($\varepsilon'$) of the minerals (seen in Table 1) according to Eqs. 4 and 5, respectively. Microwave irradiation sensitive
minerals meaning for which green microwave processing is effective are shown in the graph with a smiley face. This is important to energy savings, carbon footprint, and the economy of the process comparing conventional heating processing like roasting.

Fig. 9. Percentage of effective usage of microwave power ($P_r/P_t$) for common refractory gold ore minerals present in a typical sulfide flotation concentrate

The average power per unit volume converted into heat as the sum of $P_r$ and $P_t$, based on the conversation of energy (1st law of thermodynamics). In our case, very fine free gold grains (4 ppm) and silver (2.6 ppm) are associated with pyrite (34.29 wt %) and arsenopyrite (0.45 wt %), chalcopyrite (1.18 wt %), galena (4.43 wt %), hygroscopic moisture (3 wt %) and neglected non-sensitive minerals like quartz, feldspar, and sphalerite, etc. Whereas, general total transferred heat:

$$Q_{total} = Q_{crucible} + Q_{magnetite} + Q_{moisture} + Q_{quartz} + Q_{feldspar} + Q_{muscovite} + Q_{pyrite}$$
$$+ Q_{galena} + Q_{arsenopyrite} + Q_{gold} + Q_{silver} + Q_{iron} + \cdots = m \, C_p \, \Delta T$$

where, $m$ is the total mass of the sample, $\Delta T$ is the temperature differences ($\Delta T = T_{max} - T_{min}$) where:

$$Q_{total} = \frac{\partial}{\partial t} \left( \rho \, C_p \, T \right) \quad \text{or a special case, } Q_{total} = \rho \, C_p \, \Delta T \quad \text{at steady-state}$$

where $t$ is the time (s), $\rho$ is the density ($kg/m^3$), $c_p$ is the specific heat capacity (kJ/kg.K). Then, considering the above minerals and their contents in gold-bearing flotation concentrate, the effective heat capacitance ($\rho \, C_p$)$_T$ can be calculated at a constant temperature as given;

$$\rho \, C_p = \left( \frac{\partial q}{\partial T} \right)_p = \sum_{i=1}^{n} \rho_i \, C_{p,i} = 236.3 \, \frac{kJ}{m^3 K}$$

under unsteady-state conditions, the total transferred heat is equal to the power absorbed per unit volume ($P_V$) can be calculated with the heating rate (d$T$/dt). As it is seen from Fig. 10 (b), it is the slope of each curve between the $T$ vs. $t$ graph.

$$\rho \, C_p \, dT = P_V \, dt \quad \Rightarrow \quad \frac{dT}{dt} = \frac{P_V}{\rho \, C_p} = P_V = \rho \, C_p \, \frac{dT}{dt}$$

Fig. 10. Effect of microwave heating on the temperature of the concentrate
Next utilizing Eqs. 13-15, the heat power dissipated in the concentrate was obtained as seen in Table 7. Finally, the overall efficiency of microwave energy conversion into thermal energy (Eff, %) was calculated with a ratio of power dissipated ($P_v$) to the main power ($P_m$) of the oven between 360-1000 W. The calculated efficiency was increased to 37% for the optimum microwave energy for the gold-bearing concentrate. The calculated efficiency was increased by 37% for approaching the performance to optimum microwave energy for the gold-bearing concentrate.

| $P_m$, W | slope | $P_v$ | Eff, % |
|----------|-------|-------|--------|
| 1000     | 11.5  | 2717.4| 36.8   |
| 680      | 9.1   | 2150.3| 31.6   |
| 360      | 6.3   | 1488.6| 24.2   |

4. Conclusions

From the successful combination of bulk flotation and microwave treatment, the following conclusions could be found:

1. From the results obtained, microwave technology applied on heating and roasting processes of gold-bearing sulfidic tailings, worked efficiently, rapidly, and compared to its counterparts more sustainably. In a 4 g sample, just 50 min of microwave irradiation time using 1000 W resulted in 90 % of Sulphur removal.
2. The combination of Aeroflot 208 and Aerophine 3418A successfully was used for the increasing concentration of ore over 18 % S and 4 ppm Au for flotation recovery yield, resulting in 84 % and 80 % recovery, respectively.
3. The effectiveness of microwave energy application for the recovery of fine gold grains from waste tailings was found to be dependent on the type of minerals.
4. The quadratic model using the Box-Behnken design expressed coefficient $R^2$ regression with 96.74 % correlation suggesting the effectiveness of the applied model.
5. The Box-Behnken design was efficiently applied for the heating and roasting process for the treatment of ore wastes and tailings under microwave irradiation.
6. The overall efficiency of microwave energy conversion into thermal energy was calculated as 37% (for 1000W) for the optimum microwave energy for the gold-bearing concentrate. This proves that microwave energy is a great alternative to traditional methods for being economically obtained, quickly processed with maximum results.

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