Processing of Technogenic Gold-Bearing Raw Materials Using Preliminary Energy-Efficient Microwave Treatment

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ABSTRACT

This paper examines energy-efficient and environmentally friendly methods of processing technogenic gold-bearing raw materials. The main focus is on studying the kinetics of the thiourea leaching process after the preliminary oxidative roasting of gold-bearing materials obtained from technogenic waste from a gold extraction plant. This study compares traditional roasting in an electric furnace with roasting in a chemical reactor with microwave treatment. Preliminary oxidative microwave roasting was found to achieve a higher thiourea leaching reaction rate due to better exposure of refractory gold. Compared to traditional oxidative heating, roasting in a microwave-assisted chemical reactor reduces processing time and energy consumption while improving the degree of preparation of the raw materials for subsequent gold leaching and increasing gold recovery by 17.8%. This reduction in activation energy is due to preliminary oxidative roasting with microwave treatment (P 1.1 kW, frequency 2.45 GHz, 700 °C for 10 min), which results in an energy reduction of 14.716 kJ/mol versus 22.630 kJ/mol for traditional oxidative roasting in an electric furnace (750 °C for 60 min).

Keywords-gold; microwave; kinetics; thiourea; leaching

I. INTRODUCTION

Traditional gold ore processing technologies are characterized by gold losses at various stages [1], which leads to gold ending up in the tailing's storage facilities of gold extraction plants. This is due to the specific mineral composition of the ores, which affects processing technology and gold losses in tailings [2]. These facilities have grown in size but have not been developed due to the low profitability of processing [3]. Meanwhile, gold mineral reserves are declining, lasting for only 19-20 years, at the current level of consumption and annual production of approximately 3,000 tons [4]. The estimated global reserves of mineable gold in mineral deposits for 2024 are 59,000 tons [5]. Thus, the quantity and quality of gold ore are deteriorating. Gravity separation is a widely used and effective method for separating gold particles, which are usually larger than 0.074 mm or 200 mesh, from other minerals. In conventional gold extraction plants, gravity

separation is used to remove relatively large gold particles before flotation or cyanidation [6]. The deposits currently being developed are characterized by the persistence of raw materials and increasingly complex technological schemes for gold extraction [7]. These facts, along with the steady rise in gold prices [8], make improving the efficiency of gold processing [9] and processing raw materials with relatively low gold content extremely important. This includes technogenic deposits, such as various types of tailings ponds [10]. However, this process encounters problems related to hardness [11], fine dispersion, the impact of processing at a gold extraction plant, environmental influences during storage, and other factors that hinder gold extraction [12]. Considering this, technogenic waste can be divided into: flotation tailings [13], cyanide tailings from gold extraction plants [14], gravity concentration tailings [15], amalgamated concentration tailings [16], and aged tailings from sulfide and oxidized ores [17]. The method of processing technogenic gold-bearing raw materials depends on the composition of the material and the way mineral raw materials are processed, resulting in the formation of tailings [18]. One reason for gold losses is the presence of gold in sulfide minerals, which end up in tailings due to flotation. One solution involves re-flotation followed by cyanidation [19]. However, this approach is associated with high cyanide consumption and gold losses due to the toughness of sulfide minerals. It has been proposed that, prior to cyanidation, the concentrate undergoes oxidative roasting or further grinding to expose the gold [20], requiring increased energy costs [21]. One possible solution is roasting with microwave heating, which is the most economical and effective way to expose refractory rocks [22]. In addition to economic benefits, processing technogenic gold-bearing raw materials reduces environmental impact [23]. Using thiourea instead of cyanide can increase the environmental sustainability of waste processing while maintaining the efficiency of gold extraction [24]. Unlike cyanide-based leaching systems, modern monitoring technologies for cyanide detection in wastewater were proposed in [25], confirming the necessity of developing environmentally friendly alternatives, such as thiourea leaching. Authors in [26] showed the effectiveness of thiourea leaching from gold-bearing technogenic waste from various metallurgical industries, concluding that thiourea leaching from gold-enrichment tailings would also be effective in terms of gold extraction, resource costs, and environmental friendliness. This study aims to examine gold extraction processes from technogenic raw materials—tailings from the Maikain Gold Extraction Plant No. 1 in Kazakhstan—using microwave treatment. Over the course of its operation, the tailings storage facilities have collected up to 25 million tons of flotation tailings, cyanide tailings, and other types of waste from gold ore processing, posing a threat to the region's ecology. At the same time, due to imperfect processing technology, approximately 20–25 tons of gold were lost with the tailings [27], providing a research field for the composition and processing possibilities of these tailings, which can be important from an environmental and economic standpoint.

II. RESEARCH METHOD

The experiments were conducted at D. Serikbayev EKTU. The object of the study was a 1 kg flotation concentrate obtained from the tailings of the Maikain Gold Processing Plant No. 1, examining the mineralogical composition using an Olympus BX51 optical microscope with Mineral C7 software. The chemical and phase composition of the raw materials was analyzed using an ICP-MS 7500cx mass spectrometer from Agilent Technologies (USA) and an X'Pert PRO diffractometer (PANalytical), as shown in Figure 1. In order to determine how the mass and heat flow of the sample depend on temperature and heating time, a TGA/DSC 2 thermogravimetric analyzer (Mettler Toledo, Switzerland) was used, which provides quantitative information about changes in mass and thermal effects during heating. A pyrite flotation tailings concentrate was utilized as the sample, weighing approximately 80 mg. The sample was heated in an air atmosphere at a rate of 10 °C/min within the temperature range of 25 °C to 1200 °C. The concentrate was divided into two batches, each weighing 500 g. To compare the effectiveness of disclosure, one batch underwent oxidative roasting using a traditional method while the other underwent microwave treatment in a chemical reactor. Both batches were then subjected to thiourea leaching at different temperatures to determine the process's kinetic characteristics. A microwave chemical reactor (WBFY-201) was used for firing at temperatures of 600 °C for 5 min, 10 min, and 15 min, followed by 650 °C for 5 min, 10 min, and 15 min and 700 °C for 5 min, 10 min, and 15 min. The power was 1.1 kW at 2450 Hz, and the temperature was measured using a Benetech GM-1850 infrared thermometer (pyrometer) and a high-temperature laboratory electric furnace (SNOL 6.7/1300, manufactured by Utenos JSC, Lithuania). The furnace was used at temperatures of 650 °C for 30 min, 45 min, and 60 min, 700 °C for 30 min, 45 min, and 60 min and 750 °C for 30 min, 45 min and 60 min. Leaching was carried out with samples after oxidative firing in a furnace and in a microwave reactor.

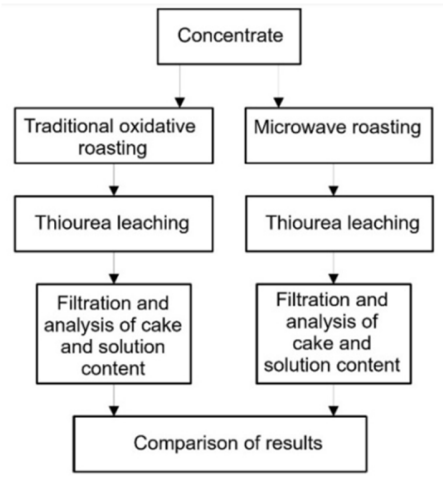

Fig. 1. Experimental scheme.

A 20 g/L aqueous solution of thiourea with an Fe^{3+} ion content of 6.0 g/L was utilized as the leaching reagent. Experiments were conducted at temperatures of 21 °C, 25 °C, 30 °C, and 35 °C, while the concentrate weight was 50 g, and the T/L ratio was 1:2 (solid to liquid). The material was leached for 30 min, 60 min, 90 min, and 120 min with stirring using a magnetic stirrer. The pH of the sample was 1.5 and samples

were taken every 15 min to study the process kinetics. All experiments were repeated twice, with the process parameters being selected during preliminary experiments. If the difference between the results was greater than 1%, the experiment was repeated. The activation energy of leaching was measured using the Arrhenius formula and the empirical graphs were pre-optimized using the least squares method in Excel. These graphs were then used to determine the leaching process duration that would ensure equal gold extraction into the solution at different thiourea leaching temperatures after preliminary microwave firing of the raw materials. To estimate the apparent activation energy, the present work determined the dependencies of the logarithm of the time required to achieve the same degree of gold extraction into the solution at different leaching temperatures on inverse temperature.

III. RESULTS AND DISCUSSION

The mineralogical analysis data show the presence of 69.71% pyrite and 18.34% chalcopyrite. Additionally, sphalerite and other minerals are present in the concentrate, as displayed in Table I.

TABLE I. MINERALOGICAL ANALYSIS DATA FOR CONCENTRATE OBTAINED FROM TAILINGS AT THE MAIKAIN GOLD PROCESSING PLANT NO. 1

Minerals by mass (%)	Minerals by mass (%)
Pyrite	69.71
Chalcopyrite	18.34
Sphalerite	12.7
Others	0.75

Figure II shows grains of metallic copper and free grains of pyrite (Py), chalcopyrite (Chp), bornite (Bo), sphalerite (Spl), and galena (Gn).

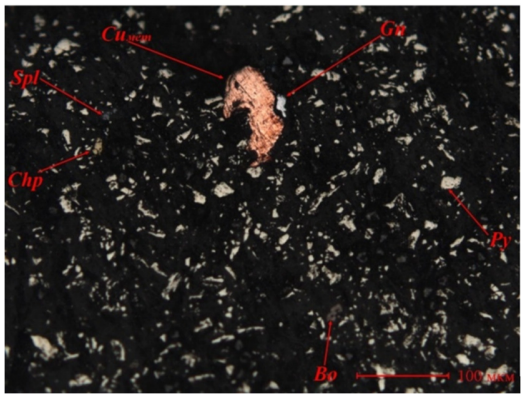

Fig. 2. Single grain of metallic copper, free grains of pyrite, chalcopyrite, bornite, sphalerite and galena.

Rational analysis revealed that 82.34% of the gold is bound to sulfide minerals, 68.21% of which is bound to pyrite, while 14.13% is bound to other sulfide minerals. Therefore, exposing pyrite and other sulfide materials can significantly increase the amount of gold extracted. Table II depicts the chemical analysis of the concentrate obtained from the tailings of the Maikain Gold Extraction Plant No. 1, having 17.7% iron, 7.1% copper, 17.2% zinc, and other elements. The gold content is 18.1 g/t.

TABLE II. CHEMICAL ANALYSIS DATA (MASS, %)

Elements	Fe	Cu	Zn	Al	Au (g/t)	Ag (g/t)	$S_{общ}$
Mass, %	17.7	7.1	17.2	5.1	18.1	20.8	33.2

Thermogravimetric Analysis (TGA) was performed on a concentrate obtained from the tailings of the Maikain Gold Extraction Plant No. 1, in order to determine the thermal behavior in an air atmosphere when heated to 1200 °C. The obtained TGA curves, shown in Figure 3, present the stages of mass loss.

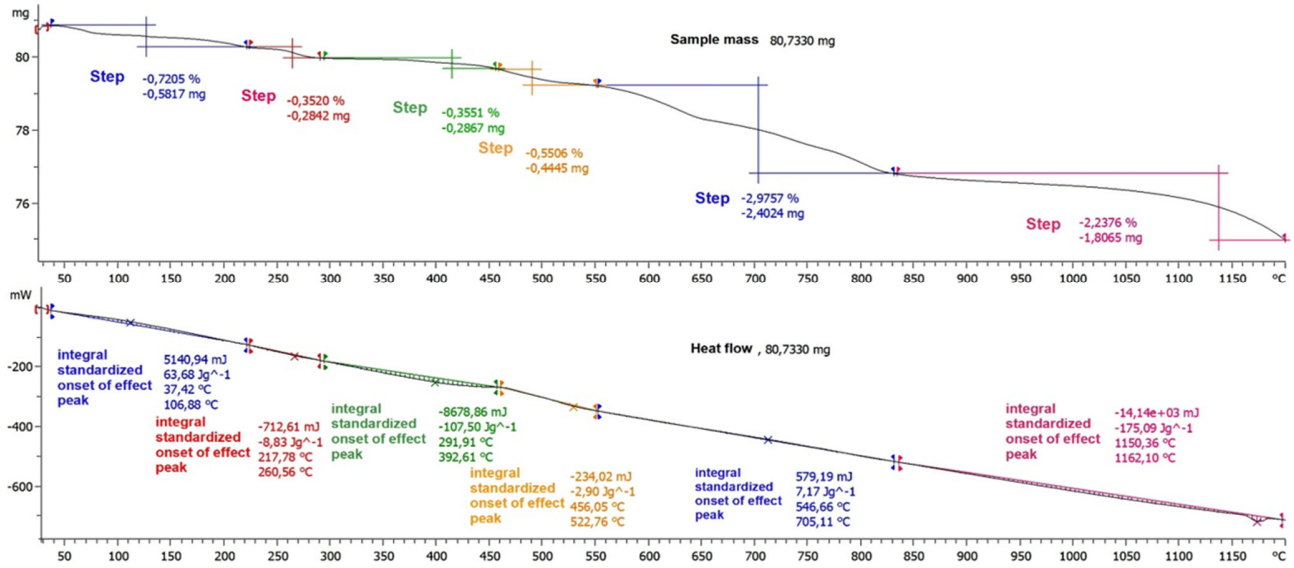

Fig. 3. Thermogravimetric (TGA) curves of concentrate obtained from tailings of the Maikain gold extraction plant No. 1.

The initial weight loss below 150 °C is due to moisture removal, while a slight weight decrease in the 150–300 °C range is due to the burning off of flotation reagent residues. Mass loss between 300 °C and 550 °C is associated with the oxidation of pyrite (FeS_2), releasing sulfur dioxide (SO_2), according to:

$$4FeS_2 + 11O_2 \rightarrow 2Fe_2O_3 + 8SO_2 \uparrow \quad (1)$$

The greatest weight loss occurs above 550 °C and up to a temperature of approximately 800 °C, indicating the most intense phase of the oxidation process and the formation of iron oxides (Fe_2O_3, Fe_3O_4). Beyond that, the curves stabilize. Therefore, heating above 800 °C is unnecessary. This finding aligns with the findings in [28], suggesting that pyrite decomposes at temperatures ranging from 750 to 820 °C.

A. Results of Firing and Leaching

Table III illustrates the results of the phase and chemical analyses performed after heating using the traditional method in a muffle furnace.

TABLE III. RESULTS OF PHASE AND CHEMICAL ANALYSIS AFTER HEATING USING THE TRADITIONAL METHOD

T (°C)			
	650 °C	700 °C	750 °C
t (min)	FeS_2(mass. %)		
35	39.3	12.32	3.81
45	22.4	16.18	2.24
60	18.7	5.20	1.21

The most effective parameter for reducing the amount of pyrite was a heating temperature of 750 °C for 60 min. The amount of pyrite was reduced to 1.21%. For the leaching stage, samples were taken from materials fired for 60 min at temperatures of 650 °C, 700 °C, and 750 °C. Table IV presents the results of phase and chemical analysis after heating in a microwave reactor.

TABLE IV. RESULTS OF PHASE AND CHEMICAL ANALYSIS AFTER HEATING IN A MICROWAVE REACTOR

T (°C)			
	600 °C	650 °C	700 °C
t (min)	FeS_2(mass. %)		
5	54.33	36.83	15.09
10	50.73	19.37	2.59
15	40.48	2.1	0.24

Heating at 700 °C for 15 min reduced the amount of pyrite to a maximum of 0.24%. For the leaching stage, samples that had undergone microwave treatment at 600 °C, 650 °C, and 700 °C for 15 min each were taken. The kinetic characteristics of the thiourea leaching process for samples heated under various conditions, were determined. Figure 4 shows the leaching graphs for samples fired for 60 min using the traditional method at temperatures of 650 °C, 700 °C, and 750 °C. The maximum gold extraction occurs with add extraction in both cases. Figure 5 portrays the leaching graphs for samples calcined in a microwave reactor at 600 °C for 15 min, 650 °C for 15 min, and 700 °C for 10 min. The maximum gold extraction from samples pretreated in a microwave reactor at 700 °C was 98.4%. Samples heated using the traditional method yielded 80.6% gold extraction at a leaching temperature of 35 °C. These values and their processing results were then used to estimate the apparent activation energy of the concentrate's interaction with an aqueous thiourea solution [29]. In order to achieve this, the dependence of the logarithm of the time required for the same degree of gold extraction into the solution at different leaching temperatures on the inverse temperature, was determined. Table V shows the duration of thiourea leaching of the concentrate to ensure the specified gold extraction into the solution at different temperatures for a sample that underwent preliminary roasting for 60 min at 750 °C using the traditional method. Figure 6 illustrates the dependence of the logarithm of the time required for the same degree of gold extraction into solution at different leaching temperatures on the inverse temperature, with preliminary calcination of samples using the traditional method, at 650 °C, 700 °C, and 750 °C. Table VI presents the duration of thiourea leaching of the concentrate to ensure the specified gold extraction into the solution at different temperatures for a sample that underwent preliminary roasting for 15 min at 750 °C using the traditional method.

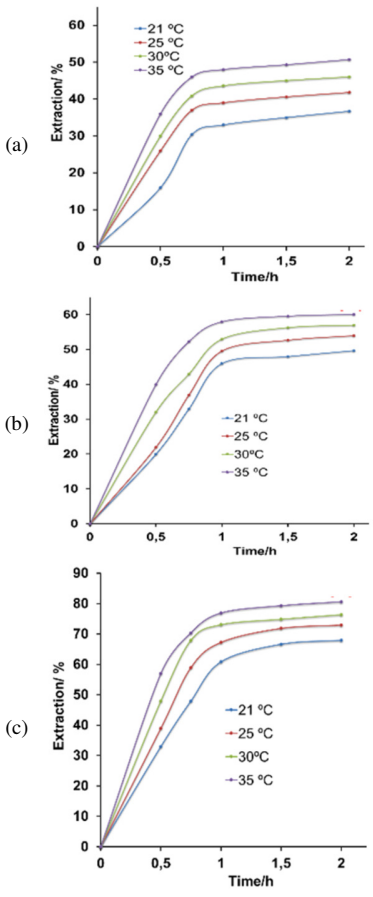

Fig. 4. Graphs showing the dependence of gold extraction on the duration and temperature of the leaching process for samples calcined using the traditional method for 60 minutes at temperatures of: (a) 650 °C, (b) 700 °C, and (c) 750 °C.

Figure 7 displays the relationship between the logarithm of the time required for gold extraction into solution at different leaching temperatures and the reverse temperature during the preliminary treatment of samples in a microwave reactor at 600 °C, 650 °C, and 700 °C. The angular coefficients of the straight lines $lg\ \tau = f(1000/T)$ are related to the apparent activation energy by the ratio $d(lg\ \tau)/d(1000/T) = E_a/(2.3R)$.

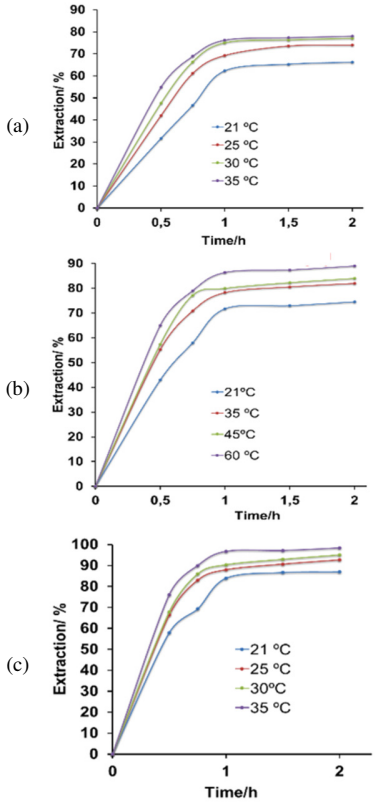

Fig. 5. Graphs showing the dependence of gold recovery on the duration and temperature of the gold leaching process from samples pretreated in a microwave reactor at temperatures of: (a) 600 °C, (b) 650 °C, and (c) 700 °C.

TABLE V. DURATION OF THIOUREA LEACHING OF CONCENTRATE ENSURING THE SPECIFIED EXTRACTION OF GOLD INTO SOLUTION AT VARIOUS LEACHING TEMPERATURES, LASTING 60 MIN USING THE TRADITIONAL METHOD AT A TEMPERATURE OF 750 °C

T		***1/T***	***τ***	***τ***	***lg τ***
°C	K	K^{-1}	h	min	min
Extraction into a 15% solution					
21	294	0.0034	0.08282	4.96943	0.6963069
25	298	0.0033	0.06894	4.13650	0.6166339
30	303	0.0033	0.06036	3.62178	0.5589222
35	308	0.0032	0.05386	3.23163	0.5094226
Extraction into a 30 % solution					
21	294	0.0034	0.33129	19.8777	1.2983669
25	298	0.0033	0.27576	16.5460	1.2186939
30	303	0.0033	0.24145	14.4871	1.1609822
35	308	0.0032	0.21544	12.9265	1.1114826
Extraction into a 45% solution					
21	294	0.0034	0.74541	44.7249	1.6505494
25	298	0.0033	0.62047	37.2285	1.5708764
30	303	0.0033	0.54326	32.5960	1.5131647
35	308	0.0032	0.48474	29.0847	1.4636651

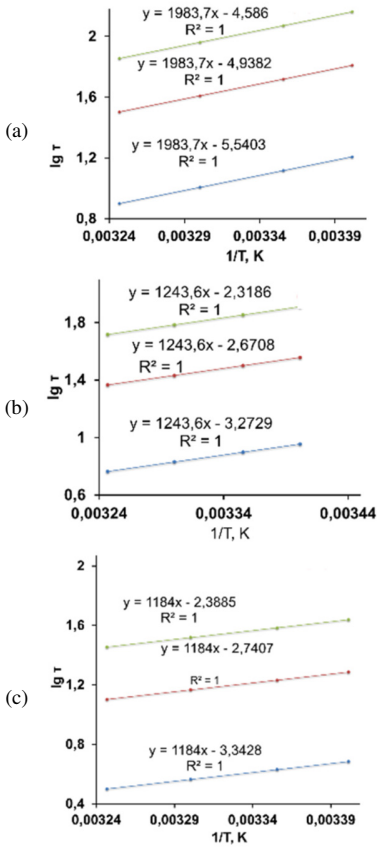

Fig. 6. Dependence of the logarithm of the leaching process duration on its inverse temperature during preliminary firing of samples using the traditional method at temperatures of: (a) 650 °C, (b) 700 °C, and (c) 750 °C.

TABLE VI. DURATION OF THIOUREA LEACHING OF CONCENTRATE ENSURING THE SPECIFIED GOLD EXTRACTION INTO SOLUTION AT VARIOUS LEACHING TEMPERATURES, LASTING 15 MIN IN A MICROWAVE REACTOR AT A TEMPERATURE OF 700 °C

T		***1/T***	***τ***	***τ***	***lg τ***
°C	K	K^{-1}	h	min	min
Extraction into a 15% solution					
21	294	0.0034	0.0468	2.808	0.4484959
25	298	0.0033	0.0406	2.438	0.3870642
30	303	0.0033	0.0385	2.311	0.3639127
35	308	0.0032	0.0349	2.098	0.3218225
Extraction into a 30% solution					
21	294	0.0034	0.1872	11.234	1.050556
25	298	0.0033	0,1625	9.752	0.989124
30	303	0.0033	0.1541	9.246	0.965973
35	308	0.0032	0.13987	8.39233	0.923883
Extraction into a 45% solution					
21	294	0.0034	0.42129	25.2777	1.40273840
25	298	0.0033	0.36572	21.9435	1.34130679
30	303	0.0033	0.34674	20.8044	1.31815526
35	308	0.0032	0.31471	18.8827	1.27606508

The apparent activation energy of the interaction between the concentrate and the aqueous solution of thiourea after preliminary calcination of the samples using the traditional method at temperatures of 650 °C, 700 °C, and 750 °C was 37.914 kJ/mol, 23.768 kJ/mol, and 22.630 kJ/mol, respectively

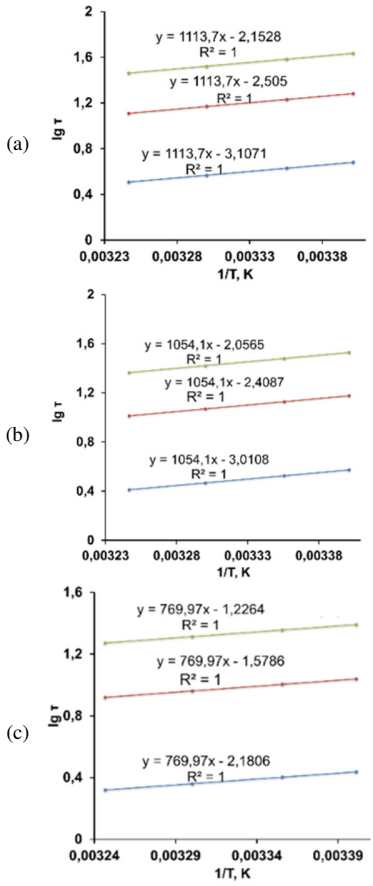

Fig. 7. Dependence of the logarithm of the leaching process duration on its inverse temperature during preliminary treatment in a microwave reactor of samples at temperatures of: (a) 600 °C, (b) 650 °C, and (c) 700 °C.

The apparent activation energy of the interaction between the concentrate and the aqueous thiourea solution after the samples were treated in a microwave reactor at temperatures of 700 °C, 650 °C, and 600 °C was 14.716 kJ/mol, 20.146 kJ/mol, and 21.285 kJ/mol, respectively. The corresponding apparent activation energy values are 8-20 kJ/mol for the diffusion mode and 40-300 kJ/mol for the kinetic mode [30]. Thus, the thiourea leaching process of the concentrate after calcination in a muffle furnace and treatment in a microwave reactor at 600 °C and 650 °C proceeds in a mixed mode. Thiourea leaching of the concentrate after preliminary treatment of the sample in a microwave reactor at 700 °C proceeds in the diffusion region. In all cases, it can be seen that, at the same temperature, the activation energy of the process is lower during treatment in a microwave reactor than during traditional heating. This phenomenon is explained by microwaves' direct effect on metal inclusions in minerals, leading to better raw material opening, explaining the decrease in activation energy.

B. Calculation of Processing Costs

The use of microwave processing appears advantageous, as it results in a 17.8% increase in gold yield and a reduction in time and energy costs of approximately 53% compared to sequential processing sequences. Table VII presents the key process indicators in USD. It is observed that using intensification reduces the cost of processing 50 g of concentrate by USD 0.16. In terms of 1 ton, the savings will be USD 3,200. The estimated reduction in emissions is approximately 1.07 kg of CO_2 per 50 g of substance [31]. This equates to a reduction in emissions of over 21.4 tons of CO_2 per ton of raw material.

TABLE VII. KEY PROCESS INDICATORS IN USD

Indicator	Traditional firing	Microwave reactor	Savings
Energy consumption	2.5 kW·h	1.2 kW·h	1.3 kW·h
Cost of electricity	0.12 USD/ kW·h	0.12 USD/ kW·h	
Energy consumption	0.3 USD	0.14 USD	0.16 USD
Energy savings of 50 g			0.16 USD
Savings on 1 ton of raw materials			≈3.2 USD/tons

Authors in [32] examined the effectiveness of microwave heating versus traditional heating methods. This study however, differs in terms of processing speed, temperature, the impact of microwave radiation on activation energy, and the estimation of general process costs. Authors in [33] showed that optimal sulfur oxidation of 67.28% can be achieved at a temperature of 404 °C. In this paper, oxidation of over 99%, was achieved at 700 °C.

IV. CONCLUSIONS

Using process intensification technologies significantly reduces the cost of processing technogenic raw materials. This approach is economical and environmentally friendly, and it reduces the carbon footprint. It has been shown that processing gold-bearing technogenic samples in a microwave reactor provides more efficient extraction of finely dispersed gold. Compared to traditional roasting, this method significantly reduces process duration and energy costs while improving gold recovery rates. Kinetic analysis of thiourea leaching confirmed that preliminary microwave treatment accelerates gold dissolution reactions by reducing the process's activation energy. Therefore, using microwave technologies to process technogenic gold-bearing raw materials is promising because it increases the energy efficiency, environmental friendliness, and profitability of non-ferrous metal extraction processes.

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