

Leaching Kinetics of Crucial Metals from Bismutite Ore: Parameter Determination

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Abstract

This study investigated the leaching kinetics of the crucial metals-aluminum (Al), iron (Fe), and zirconium (Zr) from bismutite ore using sulfuric acid (H₂SO₄) as the lixiviant. The influences of acid concentration (0.1–3 mol/L), leaching time (5–120 min), and temperature (28–80 °C) on metal extraction efficiency were systematically evaluated. The results demonstrated that increased acid concentrations and higher temperatures significantly enhanced metal recovery. The optimal leaching conditions were 2.5 mol/L H₂SO₄, a solid-to-liquid ratio of 100 g/L, and a temperature of 80 °C for 120 minutes, yielding leaching efficiencies of 99.82% for Al, 85.4% for Fe, and 99.2% for Zr. Kinetic modelling via the shrinking core model indicated that the process is chemically controlled, with activation energies of 31.06, 38.02, and 42.79 kJ/mol for Al, Fe, and Zr, respectively. The enriched leachate produced under these conditions is suitable for the subsequent selective recovery of target metals. This work supports sustainable metallurgical practices by promoting efficient, multi-element extraction from complex ores.

1. Introduction

Bismuth known as a green and strategic metal is widely applied in high-precision and advanced fields such as medicine, the nuclear industry, electronics, defense, environmental management, and superconductivity due to its excellent thermal, optical and electrical properties [1-13]. The bismuth metal is generally not used alone but is combined with other elements to form high-purity materials with unique and superior properties. Owing to its low toxicity, bismuth is often used as a substitute for some toxic metals to achieve safety and environmental sustainability [4,5]. Bismuth occurs in a variety of ore deposit types, with the principal minerals including native bismuth (Bi), bismuthinite (Bi₂S₃), bismite (Bi₂O₃), bismutite (BiO₂)CO₃ and copper-bismuth ore (3Cu₂S·4Bi₂S₃) [14]. Among these, bismuthinite and bismite are the most significant. Bismuth ores in nature often coexist with metals such as lead, iron, aluminium, silicon, tin, and copper, making it challenging to mine bismuth as a distinct mineral [15]. Therefore, beneficial processes are necessary to obtain bismuth concentrates. Bismutite is a secondary bismuth carbonate hydroxide mineral that typically forms through the alteration or oxidation of primary bismuth minerals such as bismuthinite. It is an important supergene (oxidation-zone) mineral in bismuth-bearing ore deposits, is often associated with other secondary minerals such as cerussite, malachite, azurites, hematite, goethite, zinnwaldite and quartz. These deposits contain various essential metallic elements. Over the past few decades, bismuthinite and bismite ore have become the primary sources of commercially

exploited bismuth, while the by-products of the metallurgical processing of other metals as are secondary sources of bismuth [16-23].

Bismutite (Bi_2O_3) is one of the most important secondary bismuth minerals and has traditionally been regarded as a primary source for bismuth recovery. However, natural bismutite deposits rarely occur in a chemically pure form. Instead, they are often associated with substantial amounts of aluminium, iron, and zirconium, either incorporated as trace constituents within the crystal structure or occurring as part of the surrounding gangue matrix. The coexistence of these elements is particularly significant in hydrometallurgical processing, as their dissolution during leaching can introduce competing species into the pregnant solution, thereby influencing leaching kinetics, solution chemistry, and impurity management. In particular, aluminium and iron are known to form stable complexes under acidic conditions, while zirconium can exhibit strong resistance to dissolution, potentially leading to residue enrichment. Consequently, understanding the behaviour of these accompanying elements is essential for optimizing selective leaching conditions and ensuring efficient downstream purification of bismuth from bismutite ores. The selective extraction and recovery of these elements present a dual opportunity: mitigating their interference with bismuth purification and valorizing them as strategic co-products.

Aluminium, which is commonly sourced from aluminosilicate gangue, can be recovered under acidic or alkaline leaching regimes and reused in the production of aluminium-based alloys. Iron, often present as oxides or carbonates, can be solubilized and recovered as a raw material for pigments or ferroalloys. Zirconium, although present in minor quantities, is a high-value element with critical applications in nuclear and aerospace technologies. Its recovery from bismutite processing circuits not only enhances resource utilization but also contributes to the feedstock for advanced Al-Fe-Zr alloys. These alloys benefit from the unique properties of each element—the light weight and corrosion resistance of aluminium, the increase in iron strength and the refinement and thermal stabilization of zirconium's grain. Therefore, integrating multi-element recovery from bismutite ore aligns with modern metallurgical practices focused on sustainability, economic viability, and material performance. A range of processing techniques are available for the extraction of metals from bismuth ores, most notably pyrometallurgical and hydrometallurgical methods. Pyrometallurgical processing, which includes oxygen-enriched smelting and carbothermic reduction, is often limited by its high energy consumption and low lead recovery rates [3,24,25]. Conversely, hydrometallurgical methods have been extensively explored because of their comparatively lower environmental impact, superior metal selectivity, and higher extraction efficiency [3,26].

In recent years, considerable efforts have been dedicated to the development of leaching strategies for the selective recovery of bismuth and valuable elements from complex materials using both alkaline and acidic media. He *et al.*, [27] proposed an alkaline pressure oxidative leaching approach, wherein arsenic, antimony, and lead were effectively solubilized from bismuth-rich lead anode slime, while bismuth, along with noble metals such as gold and silver, was retained in the solid residue. In another study, a sodium thiosulfate system was employed to extract bismuth from lead sulfide concentrates; however, the process was ultimately discontinued because of its low leaching efficiency of only 47% for bismuth [28]. In comparison, acid leaching methods particularly those involving chloride media have demonstrated superior bismuth recovery, owing to the strong complexation between bismuth ions and chloride ligands [3–5]. Che *et al.* [3] developed an efficient method for extracting metallic bismuth from Pb-Bi slag through a combination of selective leaching and electrowinning. Under optimal conditions, bismuth was leached with a high efficiency of 99.2%, whereas lead dissolution was limited to 12.2%, ensuring selectivity. Shao *et al.*, [29] introduced an innovative extraction method that combines oxidative leaching with selective reduction. Under the optimized conditions of 40 minutes of leaching at 90 °C, with 150 g/L H_2SO_4 , 60 g/L Fe^{3+} , and a liquid-to-solid ratio of 6:1 mL/g, the leaching rates for tellurium and bismuth reached 95.61% and 95.77%, respectively. Kamali *et al.*, [30] introduced an effective approach for recovering bismuth from copper smelter flue dust, which contains a high concentration of iron. Initially, the dust underwent leaching with water and was diluted with sulfuric acid (0.6 M) to remove copper. Iron was subsequently separated from the leaching residue through magnetic separation at field strengths of 0.1 T and 0.4 T, with approximately 70% bismuth found in the non-magnetic portion. This non-magnetic fraction was then subjected to further leaching using a mixed solution of 1.4 M H_2SO_4 and 1 M NaCl, resulting in a bismuth recovery rate of over 98%. Bismuth was finally extracted from the solution via hydrolysis, forming bismuth oxychloride (BiOCl).

Owing to its low cost, wide availability, and strong leaching efficiency, sulfuric acid was employed in this study to increase the recovery of aluminium, iron, and zirconium from bismutite ore. The effects of acid concentration and leaching temperature on metal extraction efficiency were systematically investigated. The resulting leach solutions, which contain significant concentrations of aluminium, iron, and zirconium, have potential for direct application in the sustainable synthesis of multi-component alloy systems. This approach aligns with modern metallurgical practices emphasizing sustainability, cost-effectiveness, and improved material performance for advanced industrial applications.

2. Materials and Method

2.1 Materials

The bismutite ore used in this research was sourced from Ghana Village, Makarfi Local Government Area of Kaduna State, Nigeria. To prepare the sample for analysis, a series of processes, including crushing, optical sorting, millings, and sieving, was employed. The chemistry of the bismutite ore was defined by total dissolution via aqua-regia followed by solution analysis. For analysis of solutions, ICP-OES (ICP, Thermo-Fisher iCAP 7600 Radical ICP-OES) and atomic absorption spectroscopy (AAS, ZEEnit Model 700 P) was used. Dried residues were analyzed via Powder X-ray diffraction (PAN analytical PW 1830) with Cu K α radiation at a wavelength of ($\lambda = 1.5418 \text{ \AA}$) for phase analysis, an X-ray fluorescence (XRF) spectrometer analyzer using WD-XRF, Malvern Pan Analytical Model Zetium 4.0 kW for elemental composition, and a scanning electron microscope (Model: EVO 18, Carl Zeiss) with energy dispersive X-Ray analysis (SEM-EDAX) (Model: Element) for morphological analysis. The sulfuric acid (H₂SO₄) used in this research was of reagent grade. All the stock solutions and their further dilutions were made in deionized water. The key experimental parameters, such as concentrations of H₂SO₄ and reaction temperature, were optimized by the 3D colour map surface of the origin 8 software.

2.2 Leaching Procedures

Leaching experiments with sulfuric acid were conducted in a 500 mL Pyrex glass reactor equipped with a thermometer to control the reaction temperature and a stirrer to homogenize the solution. Pulverized ore (10 g/100mL) was added to the prepared solution at a predetermined lixiviant concentration and temperature. The samples were collected and analysed at 5, 10, 30, 60, and 120 min intervals to gain insights into the leaching kinetics. At the end of the leaching experiments, the withdrawn solution was filtered and analysed [3-5]. Analysis of the leach solutions was conducted via ICP-OES and the dried leach residues were analysed via X-ray diffraction (XRD) (with the Rietveld method, PAN analytical Highcore Plus software version 4.0) and X-ray fluorescence (XRF). Equation (1) was used to determine the yield of the elements present in leach solution.

$$A = \frac{(C_1 \times V)}{(m_1 \times w_1)} \times 100\% \quad (1)$$

Where m_1 (g) and w_1 (%) are the mass of the input materials and the composition of the element, respectively; C_1 and where V are the concentration of the element and the volume of the total leach solution, respectively.

3. Results and Discussion

3.1 Raw Ore Characterization

The material purity of the raw ore determine via XRD analysis (Fig. 1) confirmed that the ore was primarily composed of bismutite (Bi_{4.00}C_{2.00}O_{10.00}: 96-900-1634), and zinnwaldite (KAl(FeLi)(Si₃Al)O₁₀F₂: 00-041-1482). The elemental constituents of the bismutite ore used in this study were examined via WD-XRF Malvern Pan Analytical Model Zetium 4.0 kW analysis and the results are shown in Table 1. However, the XRF analysis results confirmed that the major components of the minerals are Bi, Si, Al, Fe, and Zr.

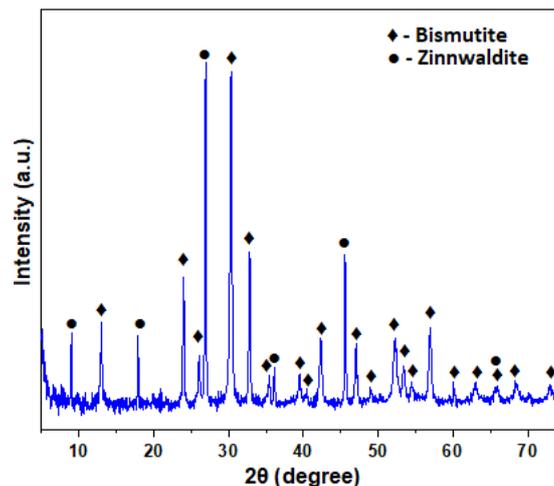


Fig. 1 X-ray diffraction of the raw ore

Table 1 Major content of the raw ore via XRF

Compound	Bi ₂ O ₃	Si ₂ O ₃	Al ₂ O ₃	ZrO ₂	Fe ₂ O ₃	ThO ₂	K ₂ O	CeO ₂	MgO	As ₂ O ₃
(Wt%)	58.34	15.17	10.25	5.109	6.36	0.283	2.19	0.33	0.21	0.06

3.2 Optimization of the Leaching Process

3.2.1 Influence of Acid Concentration

Sulfuric acid concentrations between 0.1 and 3.0 mol L⁻¹ were assessed under controlled conditions (particle size: 45 μm; solid-liquid ratio: 100 g L⁻¹; leaching time: 5–120 min; stirring rate: 400 rpm; temperature: 55 °C). As shown in Fig. 2, acid concentrations from 0.1 to 2.5 mol L⁻¹ facilitated rapid initial dissolution of aluminium, reaching 59.9% at 120 min, whereas the lowest concentration (0.1 mol L⁻¹) resulted in notably reduced extraction efficiency. Iron and zirconium displayed similar dissolution profiles, achieving final leaching efficiencies of 73.9% and 69.0%, respectively, with minimal variation after 120 min. An acid concentration of 2.5 mol L⁻¹ was determined to be optimal, balancing high recoveries of Al, Fe, and Zr with reduced reagent consumption.

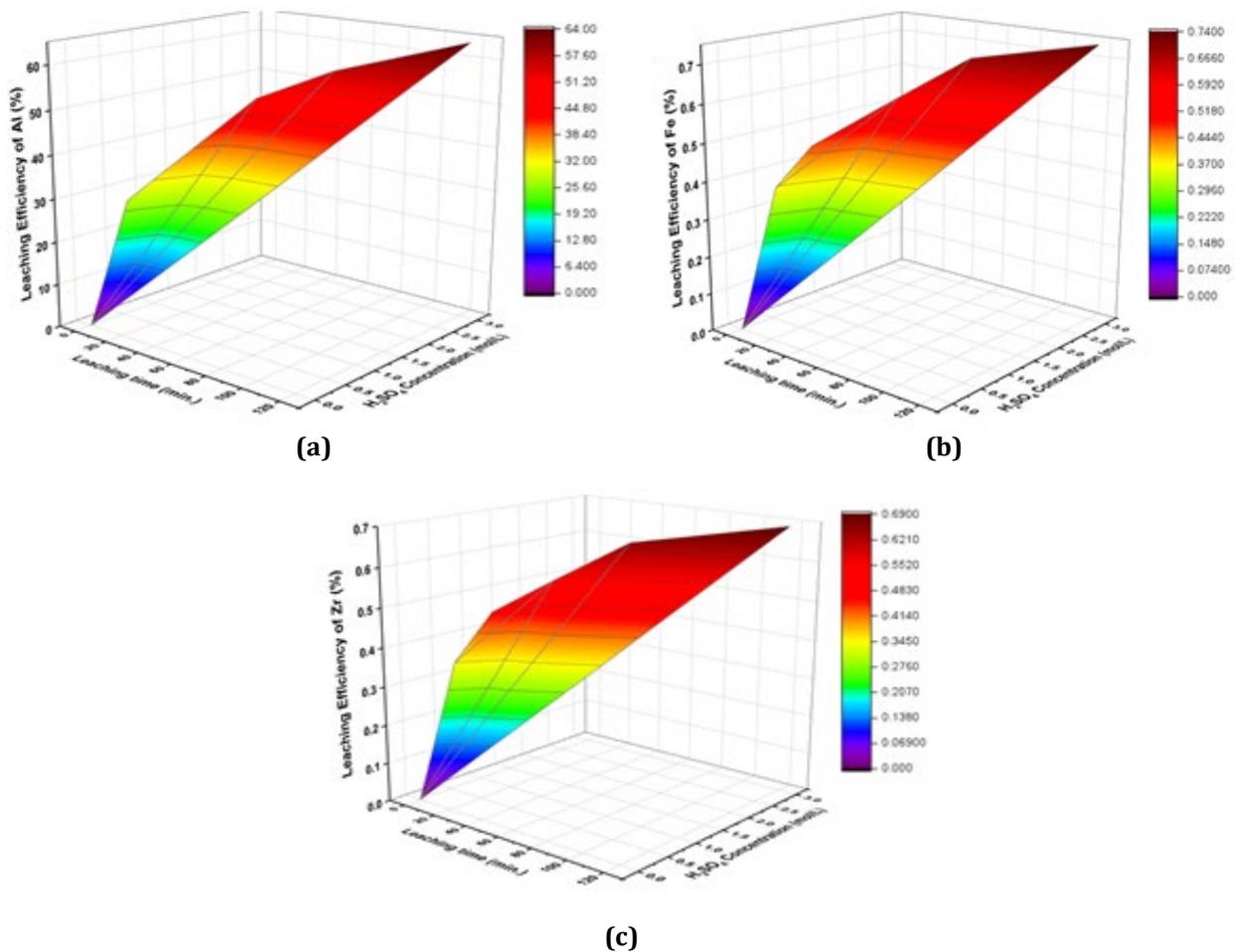


Fig. 2 Leaching efficiency of metals with different H₂SO₄ concentrations, and an S/L ratio of 100 g/L at 55 °C: (a) Al; (b) Fe; and (c) Zr. Experimental conditions: [H₂SO₄] = 0.1 – 3.0 mol/L; Time = 5-120 minutes; S/L ratio of 100 g/L; Reaction temperature = 55 °C; Particle size = 45 μm

3.2.2 Influence of the Reaction Temperature

The influence of the leaching temperature on the dissolution of Al, Fe, and Zr was assessed between 28 and 80 °C, with the H₂SO₄ concentration fixed at 2.5 mol L⁻¹ (Fig. 3). Elevated temperatures markedly enhanced metal recovery, with Fe and Zr approaching complete dissolution (99.82% and 99.2%, respectively) at 80 °C, while Al extraction reached 85.4%. The pronounced improvement in Fe and Zr recovery at higher temperatures suggests a strong temperature dependence of the leaching kinetics, likely due to increased reaction rates and enhanced

mass transfer at the solid–liquid interface. Accordingly, the optimum conditions for the leaching of crucial metals from bismutite ore were identified as 2.5 mol L⁻¹ H₂SO₄, a solid-to-liquid ratio of 100 g L⁻¹, 80 °C, and 120 min, resulting in high leaching efficiency.

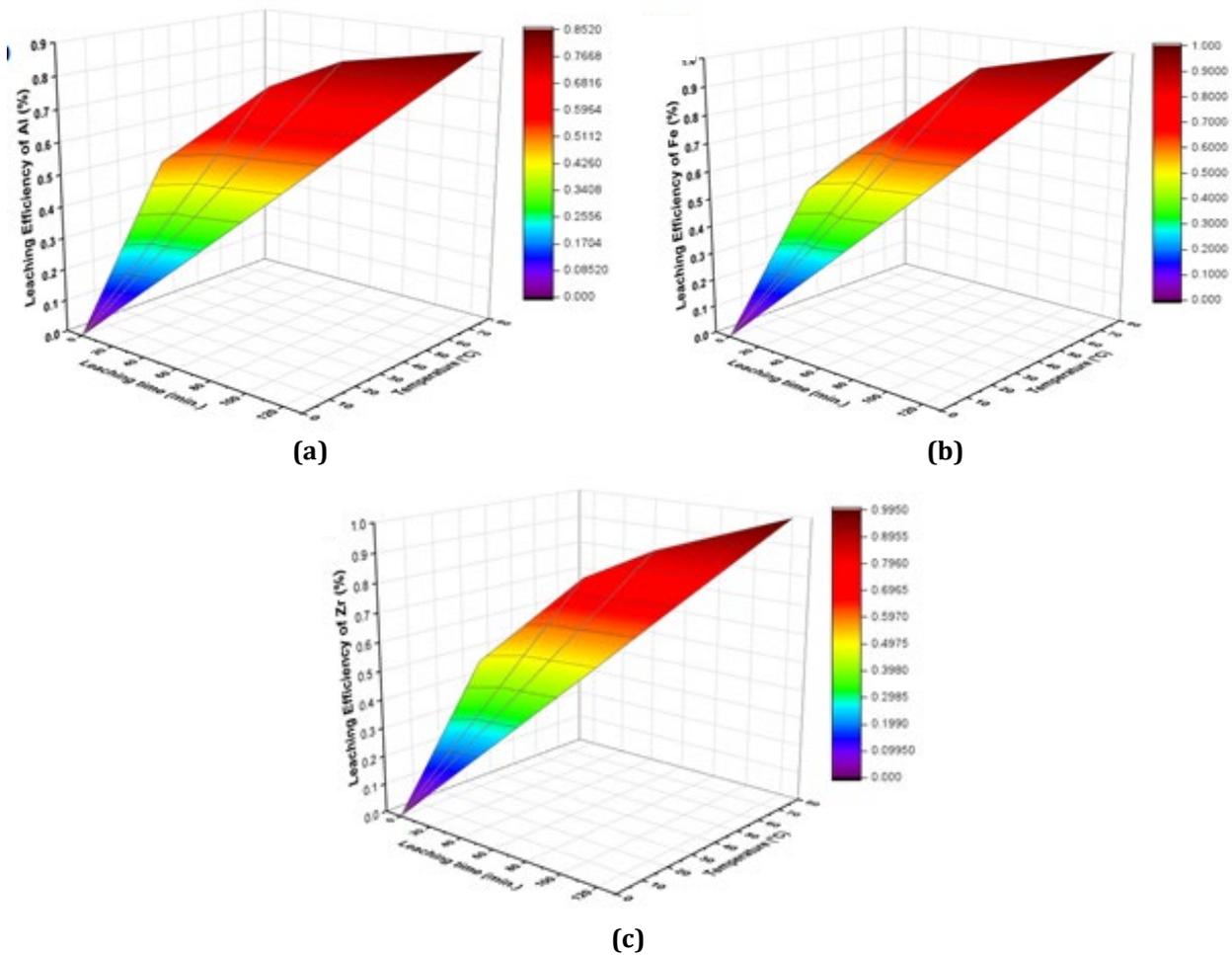


Fig. 3 Leaching efficiency of metals at different temperatures with 2.5 mol/L H₂SO₄ and an S/L ratio of 100 g/L: (a) Al; (b) Fe; and (c) Zr. Experimental conditions: [H₂SO₄] = 2.5 mol/L; Time = 5-120 minutes; S/L ratio of 100 g/L; Reaction temperature = 28-80 °C; Particle size = 45 μm

3.3 Kinetic Analysis of the Leaching Process

3.3.1 Kinetic Analysis

The leaching kinetics of bismutite ore in sulfuric acid were interpreted via the shrinking core model (SCM), a framework widely applied to describe solid–liquid heterogeneous reactions. In this model, soluble reactants diffuse through a product layer to the unreacted core, with the overall rate controlled by the slowest step in the sequence—the rate-determining step. Given the morphological characteristics of the ore and the observed leaching behaviour, the process is consistent with an unreacted shrinking core mechanism. The SCM, which typically assumes spherical particles, was used to analyse the kinetic data, enabling quantitative evaluation of the rate-controlling phenomena. The leaching trends of Al, Fe, and Zr under the determined optimal leaching conditions were fitted to the SCM rate equations (Eqs. 2-4) to establish the controlling step and associated kinetic parameters [31,32].

$$1 - (1 - \alpha)^{1/3} = kt \quad \text{Chemical control} \quad (2)$$

$$1 - \frac{2}{3}\alpha - (1 - \alpha)^{2/3} = kt \quad \text{Diffusion control} \quad (3)$$

$$\frac{1}{3} - \ln(1 - \alpha) \left[1 - (1 - \alpha)^{\frac{1}{3}} \right] = kt \tag{4}$$

Mixed control

Where k = the specific rate constant (min^{-1}); t = the leaching time (min.); and α = the fraction of bismutite ore dissolved.

The leaching data obtained at various reaction temperatures were evaluated via the proposed kinetic models to determine their suitability. Eqs (3) and (4) yielded low coefficients of determination (R^2), indicating limited agreement with the experimental observations and suggesting that these models do not adequately capture the dissolution behaviour of the system. In contrast, Eq. (2) produced a substantially higher R^2 value, reflecting a strong correlation between the predicted and experimental values. This superior fit implies that the leaching process is more accurately described by the rate equation associated with this model, which is consistent with a reaction mechanism dominated by surface chemical control. The plot of $1 - (1 - \alpha)^{1/3}$ versus leaching time at different temperatures (Fig. 4) further supports this interpretation. The apparent activation energy (E_a) was subsequently calculated via the Arrhenius equation (Eq. (5)), providing quantitative insight into the temperature dependence of the leaching rate constant.

$$\ln k = \ln A - E_a/RT \tag{5}$$

Where K is the rate constant of the chemical reaction, T is the temperature, A is the frequency factor, E_a is the activation energy, and R is the universal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$). To experimentally determine E_a , the Arrhenius plot involves plotting $\ln K$ against $1/T$. The resulting linear slope from this plot is denoted as $-E_a/R$.

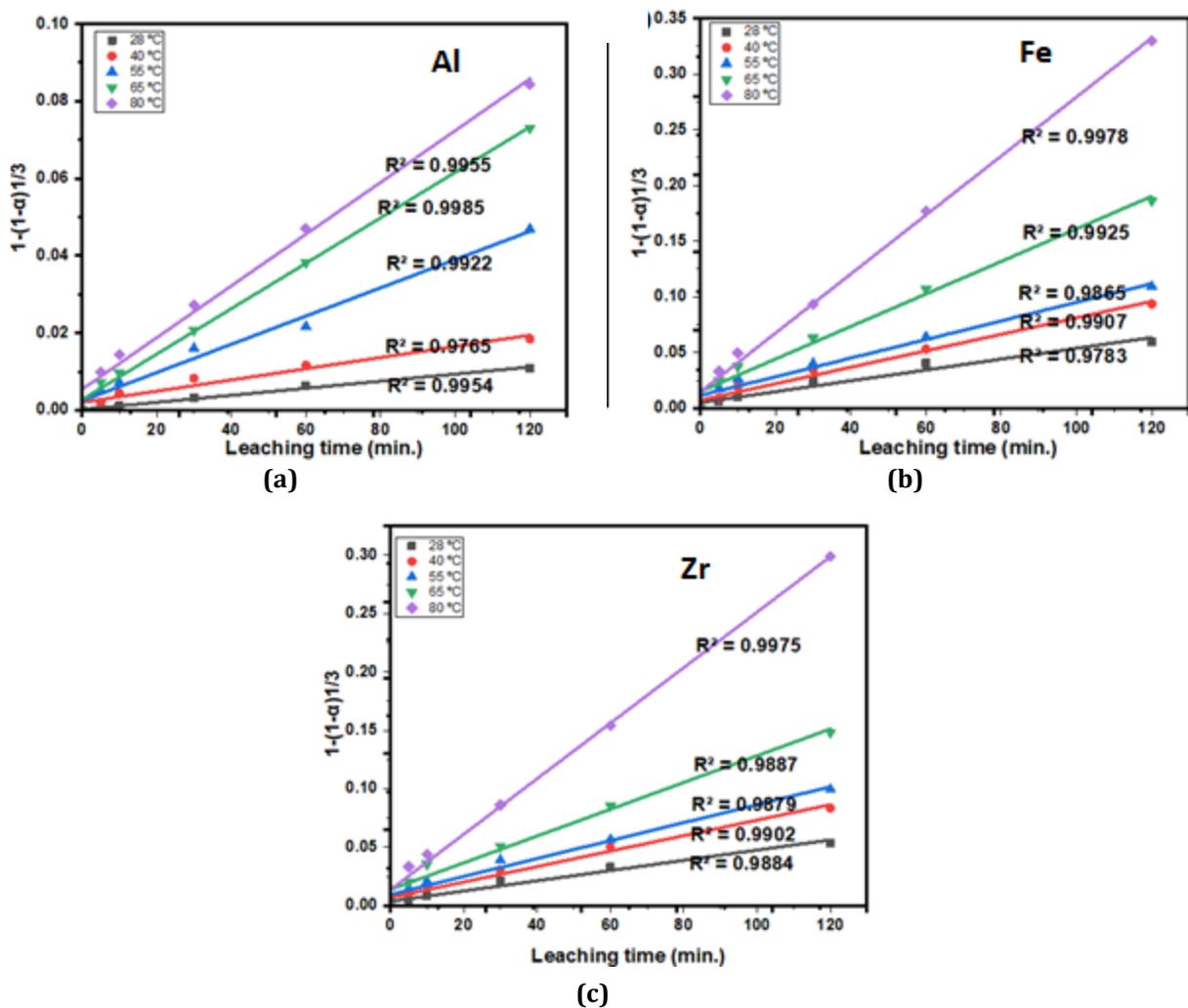


Fig. 4 Fitting of the leaching kinetics data by the shrinking core model; (a) Al; (b) Fe; and (c) Zr. Experimental conditions: Same as Fig. 2

The leaching kinetics of Al, Fe, and Zr were examined at various temperatures via the shrinking core model (SCM), with analysis focused on the first 120 min of leaching in $2.5 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ (Fig. 4). Bismuth data were excluded because of their significantly slower dissolution in acidic media, with efficiencies below 15%, likely attributed to strong Bi^{3+} -chloride complexation, which enhances solubility in chloride-rich environments but limits dissolution under the present conditions. For Al, Fe, and Zr, the chemical reaction control model yielded excellent agreement with the experimental data, with coefficients of determination (R^2) above 0.97 (Fig. 4a-c), indicating that surface chemical control is the predominant rate-limiting mechanism. This conclusion is further supported by the activation energy values derived from the Arrhenius analysis, which fall within the range typically associated with chemically controlled leaching processes in hydrometallurgical systems.

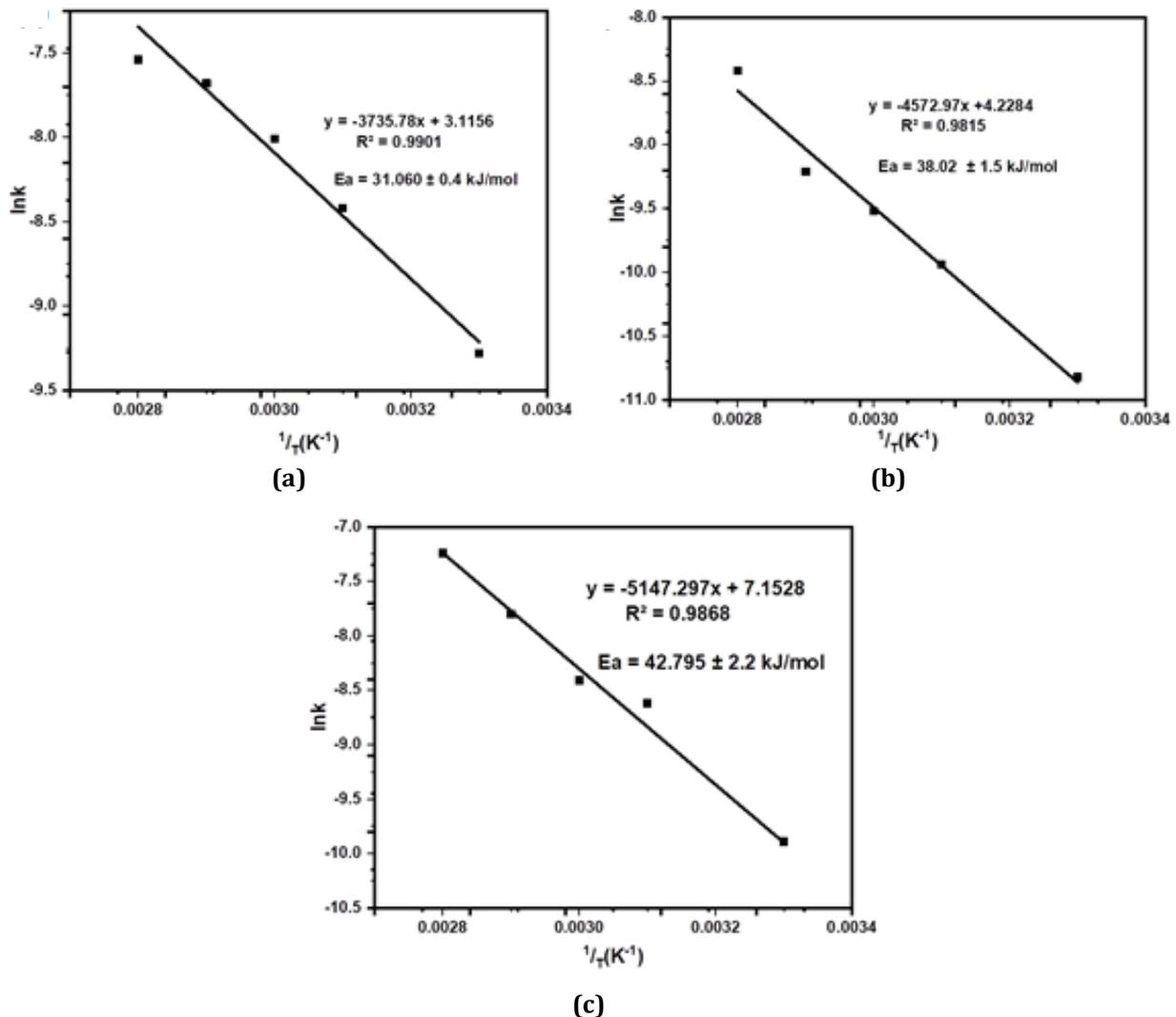


Fig. 5 Arrhenius plot; (a) Al; (b) Fe; and (c) Zr. Experimental conditions: Same as Fig. 3

The rate constants (k) obtained from the best-fit kinetic model (Eq. (1)) at different reaction temperatures were used to construct an Arrhenius plot of $\ln k$ versus $1/T$ (K^{-1}). Figure 5a-c shows the apparent E_a values associated with the metal leaching process across different temperature ranges. Specifically, the apparent E_a values were determined to be approximately $31.06 \text{ kJ mol}^{-1}$ for Al, $38.02 \text{ kJ mol}^{-1}$ for Fe, and $42.79 \text{ kJ mol}^{-1}$ for Zr (Fig. 5). In hydrometallurgical systems, activation energies below approximately 20 kJ mol^{-1} are typically associated with diffusion-controlled processes, whereas values in the range of $20\text{--}80 \text{ kJ mol}^{-1}$ generally indicate surface chemical reaction control. The obtained E_a values lie within the latter range, supporting the conclusion that the dissolution of Al, Fe, and Zr from bismutite ore under the investigated conditions is governed predominantly by a chemically-controlled reaction at the solid-liquid interface. This is consistent with the superior fit of the kinetic data to Eq. (1), which corresponds to the surface chemical control regime of the shrinking core model.

3.3.2 Residues Characterization

The leaching residues were analysed via X-ray diffraction (XRD) to determine mineralogical changes, and their elemental composition was quantified via X-ray fluorescence (XRF). As shown in Fig. 6, the XRD patterns revealed notable alterations compared with those of raw ore (Fig. 1). Peaks attributable to zinnwaldite, present in the untreated sample, were no longer detected after leaching, whereas bismutite-related phases persisted, appearing as romarinate ($\text{Bi}_2(\text{H}_2\text{O})_2(\text{SO}_4)_2(\text{OH})_2$; 01-076-1103), and eulytine ($\text{Bi}_4(\text{SO}_4)_3$; 01-080-1596). This suggests that the applied leaching conditions effectively extracted the target metals while retaining bismuth- and silicon-bearing compounds in the solid residue. XRF analysis (Table 2) supported these observations, revealing the complete removal of Fe and Zr from the solid phase and a pronounced reduction in the Al content. These compositional shifts are consistent with the high leaching efficiencies of 99.82% for Fe, 99.2% for Zr, and 85.4% for Al—confirming that Fe and Zr dissolution reached completion under the selected optimal conditions, whereas Al extraction remained partially limited.

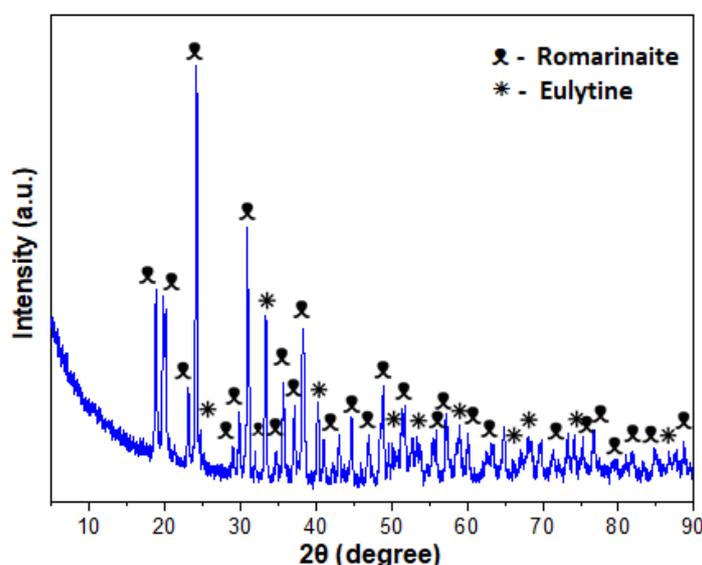


Fig. 6 XRD patterns of leaching residues with 2.5 mol/L H_2SO_4 and an S/L ratio of 10 g/100 mL at 80 °C for 120 min

Table 2 Elemental composition of the leach residues

Compound	Bi_2O_3	Si_2O_3	SO_3	Al_2O_3	ZrO_2	Fe_2O_3	ThO_2	K_2O	CeO_2	MgO	As_2O_3
(Wt%)	46.89	18.17	27.15	1.11	0.045	0.011	0.283	1.19	0.23	0.05	0.03

4. Conclusion

The leaching behaviour of aluminium, iron, and zirconium from bismutite ore was systematically investigated via sulphuric acid. The optimal leaching conditions (2.5 mol L⁻¹ H_2SO_4 , 100 g L⁻¹ solid-to-liquid ratio, 80 °C, 120 min) resulted in nearly complete dissolution of Fe (99.82%) and Zr (99.2%), with Al achieving a moderate recovery of 85.4%. Kinetic modelling demonstrated that the dissolution of all three metals followed the shrinking core model, with surface chemical reaction control as the dominant rate-limiting step, a conclusion further supported by activation energy values characteristic of chemically controlled leaching. XRD and XRF analyses of the leach residues confirmed the removal of Fe- and Zr-bearing phases, whereas bismuth- and silicon-rich compounds persisted in the solid fraction. These mineralogical results are consistent with the high leaching efficiencies and selective phase dissolution observed. Overall, this study demonstrates the effectiveness of H_2SO_4 in promoting the selective recovery of critical metals from bismutite ore, while generating a multi-metallic leachate suitable for downstream selective separation and purification. This approach offers a promising hydrometallurgical route that aligns with modern metallurgical priorities of process efficiency, resource valorization, and sustainable material production.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

Conceptualization, methodology, analysis data, writing-original draft: Ayo F. Balogun; conceptualization, methodology, supervision, writing-review and editing: Alafara A. Baba. The authors have approved the final version.

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