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Sustainable Cobalt Leaching Using In-Situ CoO Reduction: A Microwave-Driven Solution for Complex Ores

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Abstract

This study presents a green cobalt extraction method from Zebesha ore in Zambia, combining carbothermic reduction with microwave-assisted leaching (MWL) and conventional leaching (CL). Ore mixed with 20 wt.% charcoal was roasted at 650 °C for 1.5 hours in air, reducing $Co_2O_3{\cdot}H_2O$ to CoO. A 1:1 mixture of roasted and unroasted ore was leached under MWL and CL conditions. The CoO in the roasted portion served as an in-situ reductant, converting Co^{3+} to Co^{2+} in the unroasted ore. MWL achieved 98.0% cobalt extraction in 30 minutes, compared to 96.5% in 180 minutes for CL. This method eliminates sodium metabisulphite, avoiding harmful sodium sulphate byproducts. The approach provides a sustainable, low-energy route for cobalt recovery from both primary ores and secondary resources such as spent lithium-ion batteries.

Keywords

Cobalt extraction; Microwave-assisted leaching; Conventional leaching; Carbothermic reduction roasting; In-situ reductant (CoO); Lithium-ion battery recycling; Green extraction technologies.

1. Introduction

Cobalt a transition metal is classified as a critical element with wide applications in catalysts, superalloys and rechargeable batteries where it improves the energy density, cycle life and stability [1], [2], [3]. The sharp growth of renewable energy storage and electric vehicles has resulted in increased cobalt demand, exerting pressure on global supply and the urgent need for sustainable extraction methods of cobalt from complex ores and recycling from spent alloys and lithium batteries [4], [5]. The Democratic Republic of Congo (DRC) and Zambia host most of the world's richest cobalt oxide ores occurring as complex heterogenite ($Co_2O_3{\cdot}H_2O$) typically associated with copper, iron, manganese and nickel [6], [7]. However, efficient extraction of cobalt from primary and secondary sources is challenging because it predominantly exists in trivalent (Co (III) state which is sparingly soluble the acid medium. To overcome this challenge, conventional leaching (CL) processes employ reductants such as sulphur dioxide, ferrous ion and sodium metabisulphite to convert Co^{3+} to soluble Co^{2+}[8], [9], [10]. While effective, this approach is associated with high reagent consumption and environmental challenges. For instance, $Na_2S_2O_5$ generates large amounts of sodium sulphate as a by-product, resulting in secondary pollution [11]. Carbothermic reduction of cobalt oxide ores has been reported to enhance the conversion Co (III) to Co (II) thus creating favourable leaching conditions for cobalt recovery [12], [13]. On the other hand, conventional methods are energy intensive and require extended leaching time to attain high cobalt recovery[5], [14]. Therefore, emerging technologies such as microwave assisted leaching (MWL) offer alternative pathways by exploiting volumetric and selective heating to accelerate leaching reaction kinetic, reduce energy consumption while improving metal recoveries [15], [16], [17]. Previous research studies have demonstrated that MWL can achieve higher or comparable extraction efficiency to CL within short periods of time, qualifying it as a potential green technology for critical metal recoveries.

This work investigates the integration of carbothermic reduction with Microwave-assisted leaching as a sustainable and efficient route for cobalt recovery from Zebesha complex cobalt ores. Ground ore mixed with charcoal was roasted at 650 °C to achieve partial reduction of $Co_2O_3 \cdot H_2O$ to CoO. A 50:50 blend of roasted and unroasted ore was then subjected to both MWL and CL, with the reduced ore acting as an in-situ reductant to dissolve cobalt without the addition of sodium metabisulphite. The combined approach achieved rapid and high extraction, demonstrating 98.0% cobalt recovery within 30 minutes under MWL compared to 96.5% in 180 minutes under CL. Beyond primary ores, this strategy provides a promising pathway for recycling cobalt from secondary resources such as spent lithium-ion batteries, aligning with the global push toward sustainable resource utilisation and circular economy principles.

2. Methodology and characterisation

Figure 1 gives a summary of the entire process from ore to grinding, carbothermic reduction, leaching, and characterization techniques.

2.1 Materials

Complex cobalt ore samples were obtained from Zebesha Mine, Zambia. The ore was air-dried, crushed, and ground to a particle size of $P_{80} \approx 100$ µm. Analytical-grade sulphuric acid (H_2SO_4, 98%) was used as the lixiviant. Charcoal powder was used as the carbonaceous reductant. All reagents were of analytical grade, and deionised water was used for solution preparation.

2.2 Carbothermic Reduction

The ground ore was thoroughly mixed with 20 wt.% charcoal using a laboratory mixer. Approximately 100 g of the mixture was placed in ceramic crucibles and roasted in a muffle furnace at 650 °C for 1.5 h under an air atmosphere. The reduction process was designed to convert cobalt from the trivalent oxide state ($Co_2O_3 \cdot H_2O$) to divalent oxide (Co_3O_4). After roasting, the samples were cooled to room temperature, homogenised, and stored in sealed containers prior to leaching experiments.

2.3 Leaching Procedure

Roasted ore was blended with unroasted ore in equal proportions (50 wt.% of each) to provide an *in-situ* reductant during leaching. Leaching experiments were carried out using two different methods: conventional leaching (CL) and Microwave-assisted leaching (MWL). In both cases, a solid-to-liquid ratio of 25 % (w/w) was maintained:

- **Conventional Leaching (CL):** 50 g of ore blend was leached in 200 mL H_2SO_4 solution at 60 °C under mechanical stirring (300 rpm). Samples of the leachate were withdrawn at regular intervals (30, 60, 120, and 180 min), filtered, and analysed for cobalt concentration.
- **Microwave-Assisted Leaching (MWL):** 50 g of ore blend was mixed with 200 mL H_2SO_4 solution and leached in a microwave reactor operating at 100 W and 60 °C. Aliquots of solution were withdrawn at 5, 15, and 30 min, filtered, and analysed.

2.4 Analytical Methods

Cobalt concentrations in the leachates were determined using Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP-OES, PerkinElmer Optima model). Mineralogical changes before and after reduction roasting were examined by X-ray diffraction (XRD, PANalytical Aeris) using Cu Kα radiation. Phase identification was carried out using HighScore Plus software. The extent of cobalt extraction (%) was calculated according to Equation (1):

$$Leaching\ efficiency = \frac{M_{solution}}{M_{solution}+M_{residue}} \times 100 \quad (1)$$

where $M_{residue}$ and $M_{solution}$ is the mass of metals in residue solids and leach solution, respectively.

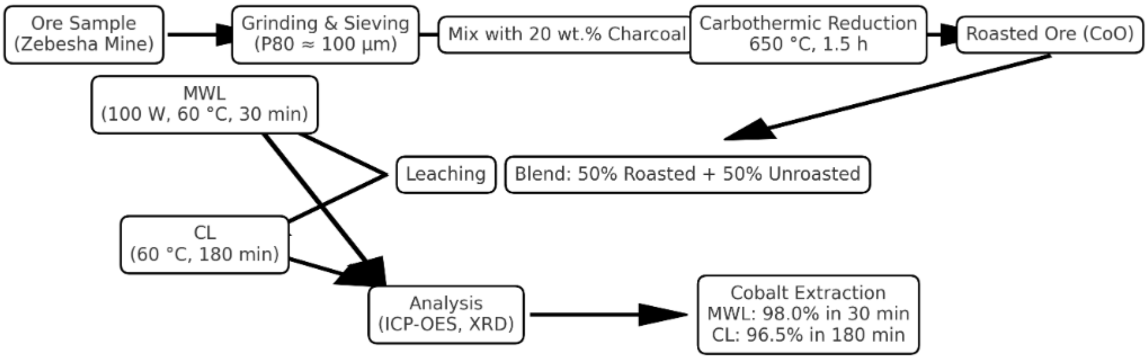

Figure 1 Process flow diagram for carbothermic reduction and leaching of Zebesha ore. The roasted ore acts as an in-situ reductant in the blended feed.

3. Results

3.1. Phase Transformations During Carbothermic Reduction

X-ray diffraction (XRD) analysis (**Figure 2**) confirmed that the as-received Zebesha ore contained cobalt predominantly in the trivalent oxide form ($Co_2O_3 \cdot H_2O$) along with gangue minerals such as quartz. After carbothermic reduction at 650 °C for 1.5 h with 20 wt.% charcoal, Co^{3+} was successfully reduced to the divalent state, forming CoO as the dominant cobalt-bearing phase. This phase transformation provided enhanced leachability during subsequent hydrometallurgical processing.

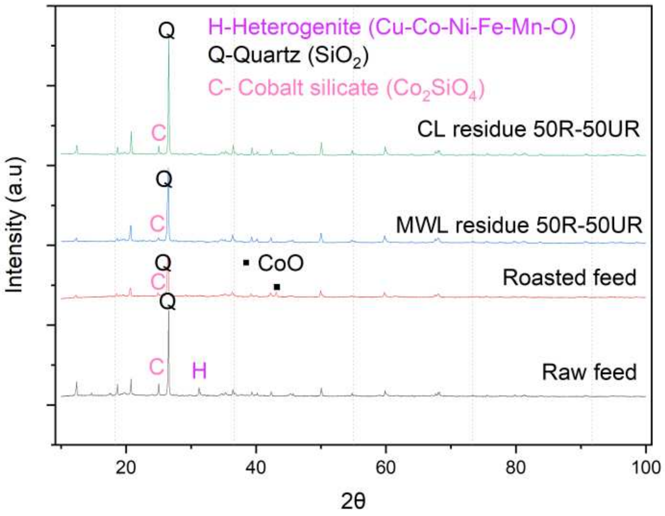

Figure 2 The XRD patterns of the raw feed, roasted feed, and leach residues (MWL and CL)

Figure 2 shows the XRD patterns of the raw feed, roasted feed, and leach residues (MWL and CL) reveal the phase transformations occurring during cobalt extraction. The raw feed is dominated by heterogenite (H, Cu–Co–Ni–Fe–Mn–O) with reflections at 2θ ≈ 32° and 36°, quartz (Q, SiO_2) at 2θ ≈ 20.8° and 26.6°, and cobalt silicate (C, Co_2SiO_4) at 2θ ≈ 23° and 35°. After carbothermic roasting, new peaks of CoO appear at 2θ ≈ 36.5° and 42.4°, confirming the reduction of Co^{3+} to Co^{2+}, while quartz and cobalt silicate remain unchanged. In the MWL residue of the 50% roasted–50% unroasted blend, quartz (26.6°) and cobalt silicate (23°, 35°) are the major phases, with only weak traces of CoO, indicating significant cobalt dissolution under microwave-assisted leaching. The CL residue shows a similar phase composition but retains slightly stronger CoO peaks compared to MWL, highlighting the higher efficiency of microwave-assisted leaching in extracting cobalt from the partially roasted ore.

3.2. Cobalt Extraction by Microwave-Assisted and Conventional Leaching

The blended ore (50% roasted + 50% unroasted) was subjected to both conventional leaching (CL) and microwave-assisted leaching (MWL). **Figure 3** illustrates the cobalt extraction profiles as a function of leaching time. MWL demonstrated significantly faster kinetics, achieving 98.0% cobalt extraction within 30 minutes, compared to 96.5% extraction in 180 minutes under CL conditions.

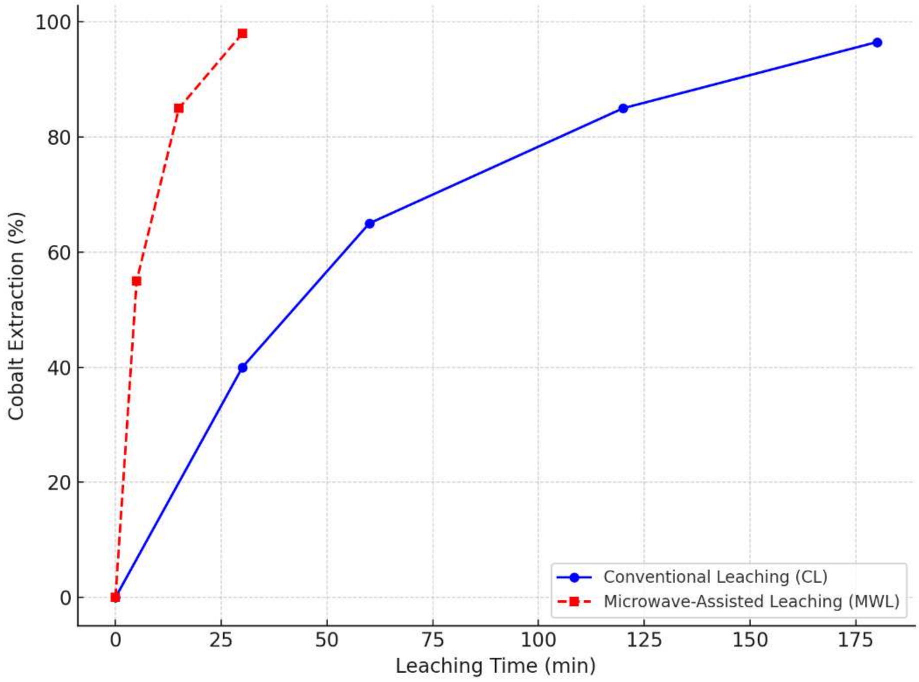

Figure 3 MWL shows higher cobalt extraction (98.0% in 30 min) compared to CL (96.5% in 180 min) demonstrating faster leaching kinetics and elimination of external reductants in cobalt extraction.

3.3. Mechanism of leaching

Various studies have demonstrated that microwave energy is effective in accelerating leaching rates of minerals [18]. However, the mechanism for high metal recoveries remains controversial and vague [17]. Mingos and Baghurst attributed the accelerated rates and recoveries in microwave systems to the higher boiling point of slurries than in conventional systems which result in improved temperature of the reactive layer and improves the surface energy [19]. Particularly, in cobalt extraction the higher metal recoveries in MWL may arise from several synergistic effects of microwaves selectively coupling with polar and conductive phases such as CoO, water and acidic ions which results in volumetric heating and generation of hot spots. This effect induces micro-fracturing, defect formation and enhances porosity in the mineral matrix thereby facilitating fast reagent ingression and reduction in energy barriers [16], [20]. Furthermore, microwave irradiation accelerates the electron transfer reactions, this lowers the activation energy for reductive dissolution as evidenced in the unpublished work by Siame et al where the activation energy in MWL was 38kJ/mol compared to 108kJ/mol. For conventional leaching. In our work, when CoO is present as an in-situ reductant, the Co^{2+} absorbs and interacts with microwave energy and effectively reduces the Co^{3+} to Co^{2+}, this results in higher cobalt recovery in microwave leaching than in conventional leaching. Microwave enhanced redox kinetics have been demonstrated in other oxide systems including lithium-cobalt-nickel-oxide cathodes [21], [22]. Several recent studies have consistently reported appreciably lower activation energies in MWL than conventional leaching further confirming the role of MW radiation in accelerating dissolution [16] [23]. Collectively, these effects render CoO a more efficient reductant under microwave conditions, accounting for the higher cobalt recoveries achieved in MWL.

3.4. Elimination of External Reductants

The roasted ore served as an in-situ reductant, facilitating the reduction of Co^{3+} in the unroasted fraction to Co^{2+}. This eliminated the need for sodium metabisulphite ($Na_2S_2O_5$), a commonly used but environmentally problematic reductant that generates sodium sulphate residues. The approach therefore reduces chemical consumption and mitigates secondary waste generation.

4. Implications for Sustainability

The integration of carbothermic reduction and MWL offers a green processing pathway for cobalt recovery. Compared to CL, MWL reduced leaching time by 83% and avoided the use of sodium metabisulphite. The method is applicable not only to primary ores but also to secondary resources such as spent lithium-ion batteries, contributing to circular economy and sustainable critical metal recovery.

References

[1] T. E. Graedel and A. Miatto, ‘U.S. Cobalt: A Cycle of Diverse and Important Uses’, *Resour. Conserv. Recycl.*, vol. 184, p. 106441, Sep. 2022, doi: 10.1016/j.resconrec.2022.106441.

[2] N. M. Dawood and A. M. Salim, ‘A Review on Characterization, Classifications, and Applications of Super Alloys’, *J. Univ. Babylon Eng. Sci.*, pp. 53–62, Apr. 2021.

[3] Z. Liu *et al.*, ‘Toward high-performance lithium-oxygen batteries with cobalt-based transition metal oxide catalysts: Advanced strategies and mechanical insights’, *InfoMat*, vol. 4, no. 4, p. e12260, 2022, doi: 10.1002/inf2.12260.

[4] C. Earl, I. H. Shah, S. Cook, and C. R. Cheeseman, ‘Environmental Sustainability and Supply Resilience of Cobalt’, *Sustainability*, vol. 14, no. 7, Art. no. 7, Jan. 2022, doi: 10.3390/su14074124.

[5] M. Chandra, D. Yu, Q. Tian, and X. Guo, ‘Recovery of Cobalt from Secondary Resources: A Comprehensive Review’, *Miner. Process. Extr. Metall. Rev.*, vol. 43, no. 6, pp. 679–700, Aug. 2022, doi: 10.1080/08827508.2021.1916927.

[6] D. Selley *et al.*, ‘A New Look at the Geology of the Zambian Copperbelt’, in *One Hundredth Anniversary Volume*, J. W. Hedenquist, J. F. H. Thompson, R. J. Goldfarb, and J. P. Richards, Eds., Society of Economic Geologists, 2005, p. 0. doi: 10.5382/AV100.29.

[7] T. Putter, S. Decrée, célestin banza, and B. Nemery, *Mining the Katanga (DRC) Copperbelt: geological aspects and impacts on public health and the environment – towards a holistic approach.* 2011.

[8] M. D. Mwema Mpoyo, M. &. Kafumbila K., ‘Use of sulphur dioxide as reducing agent in cobalt leaching at Shituru hydrometallurgical plant’, *J. South. Afr. Inst. Min. Metall.*, vol. 102, no. 1, pp. 1–4, Jan. 2002, doi: 10.10520/AJA0038223X_2755.

[9] P. Bwando and T. Kanz, ‘Laboratory Column Leaching of Cobalt using Raffinate and Sodium Metabisulfite’.

[10] N. Vieceli, C. A. Nogueira, C. Guimarães, M. F. C. Pereira, F. O. Durão, and F. Margarido, ‘Hydrometallurgical recycling of lithium-ion batteries by reductive leaching with sodium metabisulphite’, *Waste Manag.*, vol. 71, pp. 350–361, Jan. 2018, doi: 10.1016/j.wasman.2017.09.032.

[11] F. D. Uahengo, Y. R. Hara, and O. Bazhko, ‘Leaching a complex copper–cobalt oxide ore from Zebesha Mine in Zambia, a novel method’, *Miner. Process. Extr. Metall.*, vol. 133, no. 3, pp. 119–127, Sep. 2024, doi: 10.1177/25726641241276737.

[12] Y. Wang, X. Chang, M. Chen, W. Qin, and J. Han, ‘Effective extraction of nickel and cobalt from sintered nickel alloy via reduction roasting and leaching’, *Miner. Eng.*, vol. 203, p. 108336, Nov. 2023, doi: 10.1016/j.mineng.2023.108336.

[13] S. Ilyas, R. R. Srivastava, H. Kim, N. Ilyas, and R. Sattar, ‘Extraction of nickel and cobalt from a laterite ore using the carbothermic reduction roasting-ammoniacal leaching process’, *Sep. Purif. Technol.*, vol. 232, p. 115971, Feb. 2020, doi: 10.1016/j.seppur.2019.115971.

[14] S. Stanković, S. Stopić, M. Sokić, B. Marković, and B. Friedrich, ‘Review of the past, present, and future of the hydrometallurgical production of nickel and cobalt from lateritic ores’, *Metall. Mater. Eng.*, vol. 26, no. 2, pp. 199–208, 2020, doi: 10.30544/513.

[15] O. S. Akinyele *et al.*, *Application of Microwave Technology on Spent Lithium-Ion Batteries towards Graphite and Lithium Metal Oxide Recovery Batteries Using Froth Flotation and Magnetic Processes: A Review*. 2024. doi: 10.20944/preprints202410.0952.v1.

[16] M. Al-Harahsheh and S. W. Kingman, ‘Microwave-assisted leaching—a review’, *Hydrometallurgy*, vol. 73, no. 3, pp. 189–203, Jun. 2004, doi: 10.1016/j.hydromet.2003.10.006.

[17] M. Al-Harahsheh, S. Kingman, N. Hankins, C. Somerfield, S. Bradshaw, and W. Louw, ‘The influence of microwaves on the leaching kinetics of chalcopyrite’, *Miner. Eng.*, vol. 18, no. 13, pp. 1259–1268, Nov. 2005, doi: 10.1016/j.mineng.2005.06.006.

[18] T. Wen *et al.*, ‘Effect of microwave-assisted heating on chalcopyrite leaching of kinetics, interface temperature and surface energy’, *Results Phys.*, vol. 7, pp. 2594–2600, Jan. 2017, doi: 10.1016/j.rinp.2017.07.035.

[19] D. R. Baghurst and D. M. P. Mingos, ‘Superheating effects associated with microwave dielectric heating’, *J. Chem. Soc. Chem. Commun.*, vol. 0, no. 9, pp. 674–677, 1992, doi: 10.1039/C39920000674.

[20] Z. Peng and J.-Y. Hwang, ‘Microwave-assisted metallurgy’, *Int. Mater. Rev.*, vol. 60, no. 1, pp. 30–63, Jan. 2015, doi: 10.1179/1743280414Y.0000000042.

[21] Y. Fu, Y. He, Y. Yang, L. Qu, J. Li, and R. Zhou, ‘Microwave reduction enhanced leaching of valuable metals from spent lithium-ion batteries’, *J. Alloys Compd.*, vol. 832, p. 154920, Aug. 2020, doi: 10.1016/j.jallcom.2020.154920.

[22] A. Zhu *et al.*, ‘Microwave-ultra-fast recovery of valuable metals from spent lithium-ion batteries by deep eutectic solvents’, *Waste Manag.*, vol. 156, pp. 139–147, Feb. 2023, doi: 10.1016/j.wasman.2022.11.035.

[23] X. Che, X. Su, R. Chi, and J. Yu, ‘Microwave assisted atmospheric acid leaching of nickel from laterite ore’, *Rare Met.*, vol. 29, no. 3, pp. 327–332, Jun. 2010, doi: 10.1007/s12598-010-0058-7.