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Thermal Decomposition and Kinetic Behaviour of Nitric Acid: Implications for Critical Mineral Processing

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ABSTRACT

Nitric acid (HNO_3)-based leaching processes are emerging and still under development as promising approaches for extracting critical minerals, specifically nickel (Ni) and cobalt (Co), offering potential alternatives to the mature but environmentally intensive high-pressure acid leaching (HPAL) technology. A central challenge in these systems is the potential thermal decomposition of HNO_3 at elevated temperatures, which can lead to reagent loss, reduced leaching efficiency, and the generation of NO_x gases that must be captured and recycled within closed-loop acid circuits. Although HNO_3 decomposition has been studied for several decades, most prior investigations were conducted in non-pressurised, atmospheric-pressure configurations. This study examines the thermal decomposition behaviour of HNO_3 under autoclave conditions using a sealed 1.2 L Parr reactor. Experiments were conducted with 10-40 wt.% HNO_3 at 190-225 °C, following earlier low-temperature work (150-175 °C) that showed negligible decomposition. Determination of residual HNO_3 was carried out by acid-base titration using a standardized NaOH solution (0.1 or 1 M). While Spatially Offset Raman Spectroscopy (SORS) was employed to assess liquid-phase nitrate-nitrite speciation, complementary FTIR spectroscopy was applied to characterise gas-phase NO_x species in the reactor headspace. SORS spectra were dominated by the characteristic nitrate (NO_3^-) and molecular HNO_3 bands. Under these conditions, the aqueous phase retains a high-water content, and decomposition proceeds only to a very limited extent. Consequently, minor species such as HNO_2, or dissolved NO_2 are expected to form only in very trace quantities. The dominant NO_3^- and HNO_3 bands exhibit strong raman intensities that overwhelm and obscure any weak peaks from these trace-level decomposition species, yielding a spectroscopic profile consistent with the absence of significant colour change in the collected samples. Preliminary results indicate small but measurable changes in HNO_3 concentration, driven by a combination of partial decomposition, and the formation of nitrate-derived intermediates. Even at relatively

low concentrations (10-40 wt.%), trace NO_x species were detected, while intermediate and higher concentrations (40-90 wt.%) at 190-225 °C are expected to show notable decomposition. FTIR analysis confirmed weak but detectable NO, NO_2, and minor N_2O_4 spectral features in the headspace phase. The results show that the initial decomposition rate of HNO_3 depends on both acid concentration and temperature, consistent with kinetic calculations, and the experiments quantified both the rate and the overall extent of nitric acid decomposition. The findings provide an understanding of nitric acid decomposition and its relevance to hydrometallurgy and NO_x management in mining.

KEY WORDS

Nitric acid decomposition, Kinetics, NO_x formation, Hydrometallurgy, Nickel and Cobalt Leaching

BIOGRAPHY

Deus Albert Msumange is a PhD candidate in Applied Chemistry at RMIT University, Australia, focusing on industry-collaborative solutions for critical mineral extraction. Before joining RMIT, he worked in both academia and the mining industry, including roles as an Assistant Lecturer in Mineral Processing and Hydrometallurgy at Mbeya University of Science and Technology (MUST) and as an Underground Mining Engineer at Avesoro Resources, Liberia. He holds an MSc in Mining Engineering (Mineral Processing) from Karadeniz Technical University, Türkiye, and a BSc in Geology from Badji Mokhtar Annaba University, Algeria. Deus is interested in sustainable mineral processing, hydrometallurgy, green & industrial chemistry.

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