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Metal Recovery and Sulphur Removal in Laterite Residue Roasting Process

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Abstract. This study investigates the effect of roasting temperature and duration on sulphur removal and metal recovery from laterite residue obtained through atmospheric leaching of limonitic ore. The residue, characterized by high sulphur (7.43 wt.%) and iron (21.38 wt.%) content, was subjected to roasting at temperatures ranging from 500 °C to 800 °C for up to 120 minutes. XRD analysis revealed the presence of fayalite, hematite, enstatite, and jarosite, indicating stable sulphur-bearing and silicate phases. The results showed that complete sulphur removal (100%) was achieved at ≥750 °C within 30 minutes, whereas only partial removal (~45%) occurred at 500 °C. Iron and manganese recovery increased with temperature, reaching 28.46% and 26.50%, respectively, at 750 °C for 90 minutes. The improved recovery was attributed to the thermal decomposition of sulphates and oxidation of metal species to more leachable oxides. Roasting at 750 °C for 90 minutes was identified as the optimal condition, balancing sulphur elimination, metal recovery, and energy efficiency.

Keywords: laterite residue; roasting; sulphur removal; metal recovery

1. Introduction

Nickel is a critical metal widely utilized in stainless steel production, superalloys, electroplating, batteries, and as a catalyst in petrochemical processes. Traditionally derived from sulphide ores, nickel supply is increasingly constrained due to declining sulphide reserves. As a result, laterite ores, despite their lower nickel content, are gaining prominence as alternative feedstock (Prameswara et al., 2022, 2024; Santha Kumar et al., 2022).

Among extraction routes, pyrometallurgy remains an industrially preferred method due to its operational simplicity, high metal recovery, and absence of liquid waste (Chander & Sharma, 1981; Ilyas et al., 2020). However, conventional technologies such as Blast Furnace and Rotary Kiln Electric Furnace (RKEF) demand high energy input, posing challenges in optimizing laterite utilization and resource efficiency (Tang et al., 2022).

Sulphur contamination in laterite residues, often introduced during earlier metallurgical processing stages such as pressure leaching or reductive roasting, presents a significant challenge for subsequent metal recovery operations (Prameswara et al., 2024). The presence of sulphur compounds can interfere with the selective extraction of valuable metals, promote the formation of refractory phases, and contribute to environmental concerns through the release of SO_2 or other hazardous gases during high-temperature treatment (Wu et al., 2024). Furthermore, residual sulphur may adversely affect product purity and operational efficiency in downstream pyrometallurgical processes (Lei et al., 2020). Therefore, controlling sulphur content through effective thermal treatment is essential to improve metal recovery rates and ensure environmentally responsible residue management.

Despite the increasing reliance on laterite ores for nickel production, research addressing the treatment of laterite processing residues remains limited—particularly in relation to optimizing metal recovery and controlling sulphur content through thermal processes. While pyrometallurgical techniques are widely adopted for primary nickel extraction, their application in enhancing the recovery of residual metals from laterite waste streams is still underexplored. Moreover, few studies have systematically investigated the role of roasting conditions in facilitating sulphur removal while simultaneously improving the efficiency of subsequent extraction stages. This gap highlights the need for a targeted pyrometallurgical approach that maximizing metal recovery while minimizes sulphur-related process complications and emissions. The objective of this study is to investigate the effect of roasting treatment on metal recovery and sulphur removal from laterite residue. In particular, the research aims to evaluate the thermochemical behaviour and identify key process parameters that influence the selectivity and efficiency of metal extraction and sulphur volatilization during roasting.

2. Method

2.1. Materials

The laterite residue used in this study was obtained from an atmospheric leaching process of limonite-type laterite ore using 3 mol/L H_2SO_4 at 90 °C for 180 minutes. Following leaching, the solid residue was separated and thoroughly washed with distilled water at a solid-to-liquid (S/L) ratio of 1:10 to remove residual acid and soluble impurities. The washed residue was then dried in a laboratory oven at 105 °C for 1 hour. The elemental composition of the dried laterite residue was determined using X-ray fluorescence (XRF, PANalytical Epsilon 1), while its mineralogical phases were identified through X-ray diffraction (XRD, Rigaku).

2.2. Sulphur removal and metal recovery process

The dried laterite residue was first ground using an agate mortar to obtain a particle size of less than 180 µm. Approximately 2 g of the prepared sample was placed in a ceramic crucible and subjected to roasting in a muffle furnace at target temperatures ranging from 500 °C to 800 °C. The sample was introduced into the furnace once the desired temperature was reached, and the roasting duration was maintained for a predetermined time between 1 and 4 hours. After roasting, the product was cooled to room temperature, weighed to determine the final mass, and subsequently analyzed for sulphur and metal content using XRF. The extent of sulphur removal and metal recovery was calculated based on Equation (1).

$$R(\%) = \frac{m_p C_p}{m_0 C_0} \times 100 \tag{1}$$

where m_p and m_0 represent the mass of roasted product (g) and initial mass (g), C_p and C_0 denote the metal concentration (%).

3. Result and Discussion

3.1. Laterite ore residue characterization

The laterite residue obtained from the leaching process exhibited a complex elemental composition (Table 1), with iron (Fe) as the dominant metallic component at 21.375 wt.%, followed by significant amounts of silicon (Si, 13.95 wt.%), magnesium (Mg, 3.007 wt.%), and manganese (Mn, 1.642 wt.%). Nickel (Ni), although present at a relatively lower concentration (0.599 wt.%), remains a valuable target for secondary recovery due to its economic importance. Minor concentrations of cobalt (Co, 0.031 wt.%) and chromium (Cr, 0.202 wt.%) were also detected, indicating the potential for multi-metal recovery. Notably, the residue contains a high sulphur content of 7.426 wt.%, likely originating from the sulphate species retained during leaching with sulfuric acid. The elevated sulphur level necessitates thermal treatment not only to enhance metal recovery but also to reduce sulphur-associated environmental and processing challenges.

Table 1. Laterite residue composition (wt.%)

Ni	**Fe**	**Co**	**Al**	**Si**	**S**
0.599	21.375	0.031	0.255	13.95	7.426
Mg	**Ti**	**Cr**	**Mn**	**Ca**	
3.007	0.011	0.202	1.642	0.584	

XRD pattern of laterite residue presented in Figure 1. The XRD analysis of the laterite residue reveals a complex mineralogical composition dominated by silicate, oxide, and sulphate phases. The most prominent diffraction peaks correspond to fayalite (Fe_2SiO_4), indicating the presence of iron-bearing silicates as a major crystalline component. Additional peaks identified as hematite (Fe_2O_3) confirm the existence of iron oxides, which are common in thermally altered lateritic materials. The presence of enstatite ($Mg_2Si_2O_6$) suggests magnesium silicates, likely derived from the serpentinization or alteration of olivine-rich parent rocks. The diffraction pattern also displays peaks attributed to quartz (SiO_2) and jarosite [$KFe_3(SO_4)_2(OH)_6$], the latter indicating retention of sulphate-bearing phases likely formed during the prior sulfuric acid leaching. The coexistence of these phases suggests incomplete sulphur removal and the thermal stability of certain sulphate minerals, which may influence subsequent roasting behaviour and metal recovery efficiency.

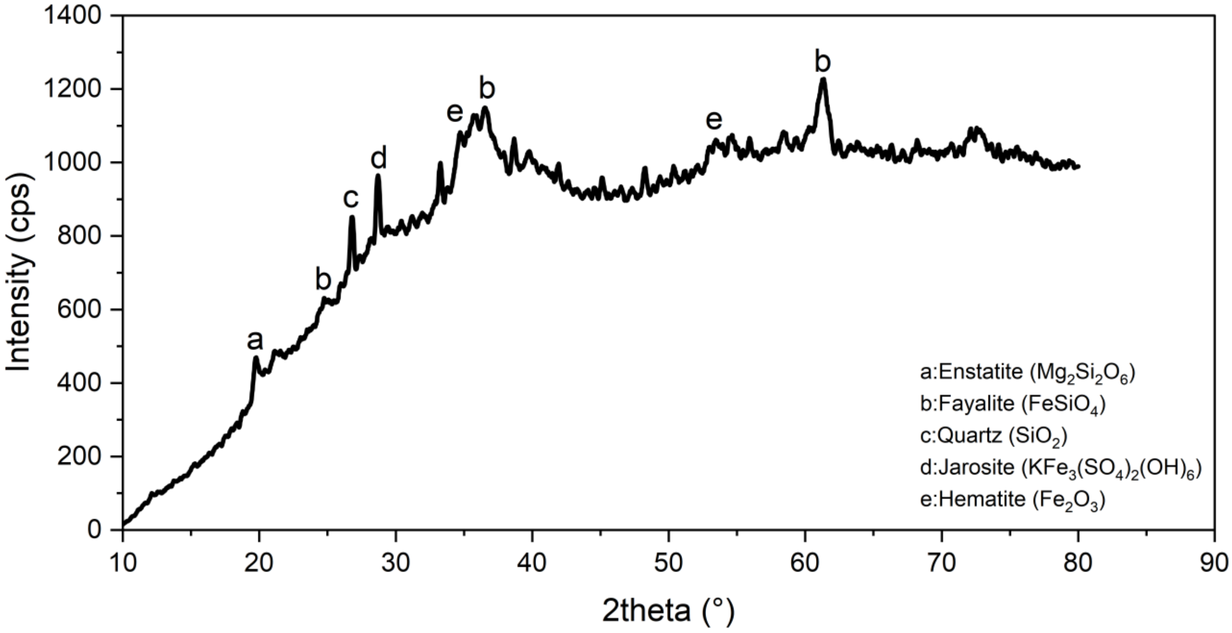

Figure 1. XRD pattern of laterite ore residue

3.2. Effect of temperature and time to sulphur removal and metal recovery

Figure 2 presents the sulphur removal efficiency from the roasting process. The effect of roasting temperature and duration on sulphur removal from laterite residue was systematically evaluated. At 500 °C, sulphur removal efficiency increased gradually with time, reaching 45.05% after 120 minutes. In contrast, complete sulphur removal (100%) was achieved within the first 30 minutes at both 750 °C and 800 °C, indicating that higher temperatures significantly enhance sulphur volatilization. The sharp contrast in removal efficiency between 500 °C and ≥750 °C suggests the presence of thermally stable sulphur-bearing phases that require elevated temperatures to decompose or volatilize effectively.

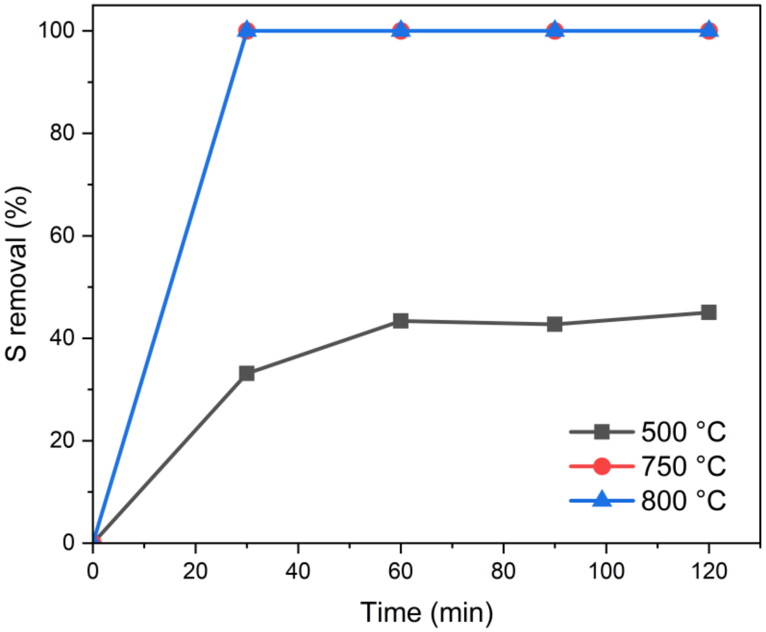

Figure 2. Sulphur removal efficiency at 500-800 °C for 0-180 min

The thermal decomposition behaviour of sulphur-bearing phases in the laterite residue plays a critical role in the efficiency of sulphur removal during roasting. At lower temperatures (~500 °C), the primary sulphur-containing phase, jarosite [$KFe_3(SO_4)_2(OH)_6$], undergoes partial decomposition (Equation (2)), releasing sulphur primarily in the form of SO_3 gas (Zulhan et al., 2022). However, the thermal stability of jarosite and related sulphates at this temperature limits the extent of sulphur volatilization, as reflected in the incomplete sulphur removal observed experimentally. In contrast, at higher temperatures (≥750 °C), complete decomposition of residual metal sulphates—such as those of Fe, Mg, and Ni—occurs, leading to full conversion of sulphur species into gaseous SO_3 (Equation (2)) (Lei et al., 2020). This process is thermodynamically favourable at elevated temperatures and results in near-instantaneous sulphur elimination.

Additionally, SO_3 may further dissociate into SO_2 and O_2 in the gas phase at these higher temperatures, especially under reducing or low-moisture conditions.

$$KFe_3(SO_4)_2(OH)_6 \rightarrow Fe_2O_3 + K_2SO_4 + H_2O + SO_3 \uparrow \tag{2}$$

$$MSO_4 \rightarrow MO + SO_3 \uparrow (M = Fe, Mg, Ni, Co) \tag{3}$$

$$SO_3 \rightarrow SO_2 + \frac{1}{2}O_2 \tag{4}$$

The recovery behaviour of iron (Fe) and manganese (Mn) from laterite residue during roasting shows a clear dependence on both temperature and time (Figure 3). At 500 °C, Fe recovery remains relatively stable across the roasting durations, ranging from 20.68% to 21.42%, indicating limited reactivity or transformation of iron-bearing phases at this lower temperature. In contrast, elevated temperatures significantly enhance Fe recovery, with values increasing to 25.41% and 28.46% at 750 °C, and reaching a maximum of 29.42% at 800 °C after 120 minutes. This improvement suggests enhanced phase transformation or increased solubility of iron species formed at higher thermal input. A similar trend is observed for Mn recovery, though the results are more variable. At 500 °C, Mn recovery remains relatively low and stable, but increases noticeably with temperature and time, peaking at 26.50% and 25.65% at 750 °C and 800 °C, respectively. These observations suggest that higher roasting temperatures facilitate the decomposition of complex Mn- and Fe-bearing minerals, thereby promoting the formation of more extractable oxides. The enhanced recovery at elevated temperatures also correlates with effective sulphur removal, supporting the hypothesis that sulphur elimination improves the accessibility and reactivity of target metals during subsequent processing.

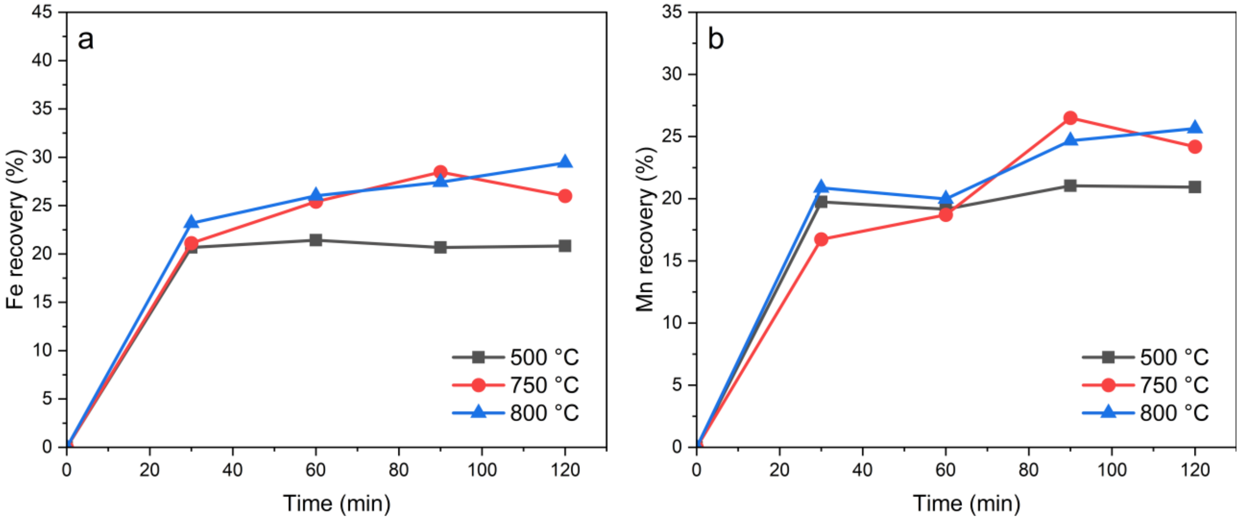

Figure 3. Temperature and time effect on (a) Fe recovery and (b) Mn recovery

The enhanced recovery of Fe and Mn at elevated roasting temperatures can be attributed to a series of thermochemical transformations involving the decomposition of sulphates, oxidation of lower-valent oxides, and breakdown of jarosite-type minerals. At temperatures exceeding 700 °C, metal sulphates such as $FeSO_4$ and $MnSO_4$ undergo thermal decomposition, forming their respective oxides (Fe_2O_3 and Mn_2O_3) and releasing SO_3 gas (Equation (5)) (Lei et al., 2020). These oxides are more chemically stable and exhibit higher solubility under acidic conditions, thereby improving metal extractability. Concurrently, residual Fe^{2+} and Mn^{2+} species present in the form of oxides or sulphates are oxidized to their higher-valent states (Fe^{3+} and Mn^{3+}) in the presence of atmospheric oxygen, forming hematite and bixbyite, which are more favourable for leaching (Equation (6)) (Sissoko et al., 2025). Additionally, the decomposition of jarosite [$KFe_3(SO_4)_2(OH)_6$] and Mn-substituted jarosite structures contributes to sulphur volatilization and releases Fe and Mn into more reactive forms (Equation (7)). These reactions collectively explain the significant improvement in Fe and Mn recovery observed at 750 °C and 800 °C, highlighting the critical role of high-temperature roasting in converting inert or refractory phases into leachable metal oxides.

$$MSO_4 \rightarrow M_2O_3 + SO_3 \uparrow \qquad \text{(M=Fe,Mn)} \tag{5}$$

$$4FeO + O_2 \rightarrow 2Fe_2O_3 \qquad \text{(M=Fe,Mn)} \tag{6}$$

$$KFe_3(SO_4)_2(OH)_6 \rightarrow 3Fe_2O_3 + K_2SO_4 + H_2O + SO_3 \uparrow \tag{7}$$

Therefore, considering the complete sulphur elimination, high metal recovery, and thermodynamic efficiency, 750 °C with a roasting time of 90 minutes can be regarded as the optimum condition. This condition achieves full sulphur removal and high Fe and Mn recovery while offering a more energy-efficient and industrially practical alternative compared to higher temperature regimes. At the identified best condition of 750 °C for 90 minutes, sulphur removal reached 100%, indicating complete decomposition and volatilization of sulphur-bearing phases such as jarosite and metal sulphates. Under the same condition, iron and manganese recoveries were 28.46% and 26.50%, respectively—representing near-maximum values observed in the study. These results confirm that roasting at 750 °C for 90 minutes is highly effective for both sulphur elimination and metal recovery from laterite residue.

4. Conclusion

This study demonstrated that roasting is an effective method for enhancing sulphur removal and metal recovery from laterite residue generated by atmospheric leaching. The residue, which contained significant amounts of iron, manganese, and sulphur, exhibited a complex mineralogical composition including fayalite, hematite, enstatite, and jarosite. Roasting at temperatures below 750 °C resulted in limited sulphur volatilization and moderate metal recovery. In contrast, complete sulphur removal (100%) was achieved at 750 °C and 800 °C within 30 minutes, indicating that higher temperatures facilitate the decomposition of sulphate-bearing phases. Iron and manganese recoveries improved significantly with increasing temperature and time, reaching near-optimal values of 28.46% and 26.50%, respectively, at 750 °C for 90 minutes. These improvements are attributed to the thermal decomposition of metal sulphates and oxidation of metal species into more leachable oxide forms. Considering sulphur elimination efficiency, metal recovery performance, and thermal energy requirements, roasting at 750 °C for 90 minutes is recommended as the optimal condition for treating laterite residues.

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