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Recovery of Lithium Carbonate from Nigerian Lepidolite Ore via Roasting-Leaching-Precipitation

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Abstract

This study investigated the feasibility of extracting lithium from lepidolite ore sourced from Eggon, Nasarawa State, Nigeria, using a sulfuric acid roasting-water leaching method. The raw ore, a complex aluminosilicate with a low lithium content of 1.12%, underwent a multi-stage process involving calcination, acid roasting, water leaching, and precipitation. Characterization by X-ray diffraction (XRD), X-ray fluorescence (XRF), and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) confirmed successful mineralogical and chemical transformations at each stage. Calcination at 900 °C and subsequent roasting with sulfuric acid at 300 °C effectively activated the ore, converting lithium into a soluble sulfate. Subsequent leaching and purification steps led to the precipitation of lithium carbonate. The final product was a crystalline, lithium-rich powder, although some trace metals persisted. The results confirm the technical viability of this process for producing lithium carbonate, highlighting its potential for industrial application in battery manufacturing.

Keywords: *Lepidolite, roasting, leaching, impurities, characterization, lithium carbonate.*

1.0. Introduction

The increasing global demand for clean energy technologies has intensified interest in lithium, a critical element for the production of lithium-ion batteries, ceramics, glass, lubricants, and other advanced applications. Among these, lithium-ion batteries stand out as the cornerstone of modern energy storage systems, driving the exponential growth of electric vehicles, portable electronics, and grid-scale renewable energy integration (Wang, 2024; Schwich & Friedrich, 2022). Consequently, the development of reliable and sustainable sources of lithium has become a priority for both industrial and academic research, with a strategic focus on diversifying from traditional sources like brines and hard-rock spodumene to new mineral reserves.

Lepidolite, a lithium-bearing mica, represents one of the most important alternative sources of lithium, especially in regions where conventional resources are less accessible. It typically occurs within granitic pegmatites and rare-metal deposits, often associated with minerals such as quartz, feldspar, and amblygonite. Despite its abundance, lepidolite poses significant extraction challenges due to its aluminosilicate framework, where lithium ions are strongly bound within the layered crystal lattice. This structural stability makes conventional methods of direct extraction inefficient and economically unfeasible, necessitating thermal and chemical activation to liberate lithium in a recoverable form (Necke, 2023).

Nigeria, particularly Nasarawa State, is endowed with rich mineral resources, including lithium-bearing ores. Within the Nigerian basement complex, the Eggon Local Government Area has recently emerged as a promising source of lepidolite. However, systematic studies on the beneficiation, extraction, and processing of lithium from this region remain limited. Unlocking this potential not only offers significant economic opportunities for Nigeria but also contributes to diversifying the nation's mineral economy and supporting global efforts toward a sustainable energy transition.

Several approaches have been developed to enhance lithium recovery from lepidolite, with the roasting–leaching–precipitation method being the most widely adopted and technically proven. In this process, calcination at high temperatures alters the mineral structure, removing volatile components and increasing porosity, which is crucial for subsequent chemical reactions (Natasha et al., 2021). The activated ore then undergoes acid roasting with concentrated sulfuric acid, a step that converts the inert lithium aluminosilicate into water-soluble lithium sulfate (Qiu et al., 2022; Su, 2023). This soluble lithium compound can then be efficiently leached into an aqueous solution. Subsequent purification steps, such as the selective precipitation of impurities with a base like sodium

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hydroxide and the final precipitation of the target product with sodium carbonate, yield a high-purity lithium carbonate, a compound directly applicable in industrial and battery-grade production (Wei, 2024; Xu, 2024). The efficiency of this complex multi-stage pathway is typically evaluated through a suite of advanced mineralogical, structural, and compositional analyses using techniques such as X-ray diffraction (XRD), atomic absorption spectroscopy (AAS), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDX).

Beyond technical efficiency, the importance of processing lithium from local sources such as Eggon lepidolite lies in its strategic contribution to national economic resilience. With the projected surge in lithium demand, countries with untapped lithium reserves have the potential to position themselves within the global supply chain of critical energy materials (Gao et al., 2020a). However, to achieve this, comprehensive studies must address not only the lithium recovery rate but also the control of impurities such as silica, alumina, potassium, and transition metals, which can compromise the purity of the final product and its suitability for high-performance battery applications (Suárez, 2024; Gong, 2024). This is a critical challenge in lithium processing, as impurities can significantly impact the performance and longevity of battery cells (Kauppinen et al., 2023; Maslyn et al., 2019). The use of advanced separation technologies like nanofiltration and ion-selective membranes is being explored to address these challenges (Gao et al., 2020b; Huang, 2024; Li et al., 2021).

This study therefore explores the extraction of lithium carbonate from lepidolite ore obtained from Eggon, Nasarawa State, Nigeria. By employing the roasting–leaching–precipitation route and characterizing each transformation stage, the research demonstrates the feasibility of producing high-purity lithium carbonate from Nigerian lepidolite while also identifying challenges associated with impurity removal and process optimization.

2. Methodology

2.1. Description of Study Area

A lepidolite ore sample was obtained from Eggon, a Local Government Area (LGA) in Nasarawa State, Nigeria. It lies within the Nigerian basement complex, a geological formation known for its rich deposits of lithium-bearing minerals. Lepidolite, one of the primary lithium sources in this region, typically occurs in pegmatites and rare-metal granites, with varying compositions and mineral associations. Figure 1 presents the map of Nigeria highlighting Nasarawa State, with a zoomed-in view of the state showing the precise study area in Eggon.

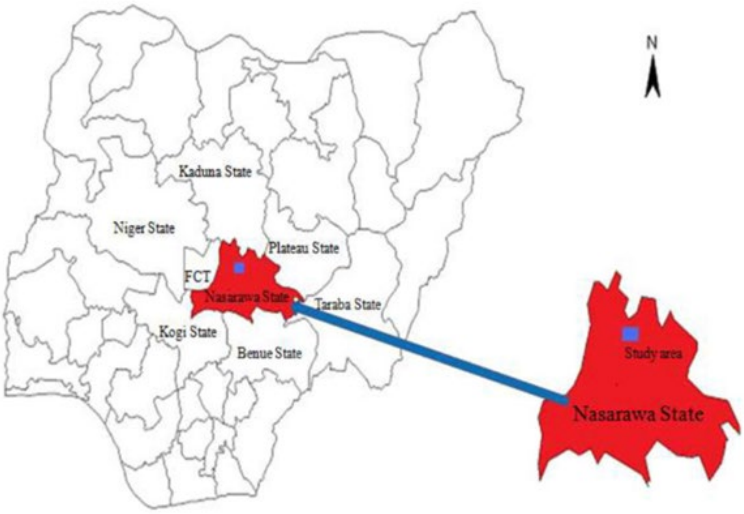

Figure 1: Map Location of Eggon in Nassarawa State Nigeria

2.2 Material Collection and Preparation

In Eggon, Nasarawa State, lepidolite ore was obtained through a combination of mechanical excavation and manual collection. Excavation often extended to depths of 3,000–4,000 feet, where mineralized layers were identified by variations in soil texture and color. Heavy machinery was used to remove bulk material, after which local workers collected the ore in 25–50 kg bags for weighing and valuation. The raw ore was first crushed using a jaw crusher and sieved to 250 µm, a particle size selected to increase surface area and enhance efficiency during calcination, roasting, leaching, and subsequent lithium recovery (Park et al., 2023). Figure 2(a) shows the raw lepidolite ore, a crystalline material with lithium bound within the silicate lattice, which is chemically inert and unsuitable for direct extraction (Natasha et al., 2021).

2.3 Calcination and Roasting

To release lithium from the ore matrix, the material underwent calcination at 900 °C for 2 hours using a muffle furnace (Natasha et al., 2021). This step removed volatiles and enhanced ore porosity, preparing it for the acid roasting stage. The calcined ore was mixed with concentrated sulfuric acid at a solid-to-liquid ratio of 1:1 and roasted at 300 °C for 2 hours. This sulfuric acid roasting method is a common approach for converting lithium-bearing minerals into water-soluble salts, as documented in similar studies (Qiu et al., 2022; Natasha et al., 2021). As shown in Figure 2(b), the roasted ore exhibited a porous, hardened structure resulting from the decomposition of hydroxyl groups, where lithium was successfully converted into soluble lithium sulfate, while silica and alumina remained insoluble.

2.4 Water Leaching and Filtration

Following roasting, the ore was subjected to water leaching at 100 °C for 1 hour under continuous stirring to dissolve lithium salts into solution (Gao et al., 2020a). This produced a slurry (Figure 2(c)), in which lithium sulfate dissolved into the aqueous phase while insoluble residues such as silica and alumina remained suspended. Filtration separated the liquid from the solid residue, yielding a brownish filtrate (Figure 2(d)). The coloration indicated the presence of lithium along with impurities such as Fe, K, and trace transition metals, consistent with elemental analyses obtained by AAS and XRF (Gao et al., 2020b).

2.5 Purification and Precipitation of Lithium Carbonate

The leachate was purified by stepwise addition of NaOH to adjust the pH to 9, precipitating impurities such as iron and aluminum hydroxides. This purification is critical for obtaining high-purity lithium products (Gao et al., 2020c). The clear filtrate was then treated with a sodium carbonate solution, leading to the precipitation of lithium carbonate as a fine white solid. Figure 2(e) shows the final lithium carbonate product, which appeared as a crystalline powder. Although purification removed most impurities, traces of silica and alumina persisted, as confirmed by XRD and AAS, which verified that the dominant phase was well-crystallized lithium carbonate (Gong, 2024).

2.6 Characterization and Process Efficiency

Characterization techniques including XRD, AAS, SEM, EDX and XRF confirmed the progressive transformation of raw ore through each stage of processing. As illustrated in Figure 2, the pathway from raw lepidolite to purified lithium carbonate demonstrates the efficiency of the roasting–leaching–precipitation method (Liu, 2024). Lithium recovery rates were determined by comparing lithium content before and after processing, while product purity was validated through compositional analysis. The combined steps consistently produced high-purity lithium carbonate suitable for industrial applications such as lithium-ion batteries, confirming the technical feasibility and efficiency of this extraction method.

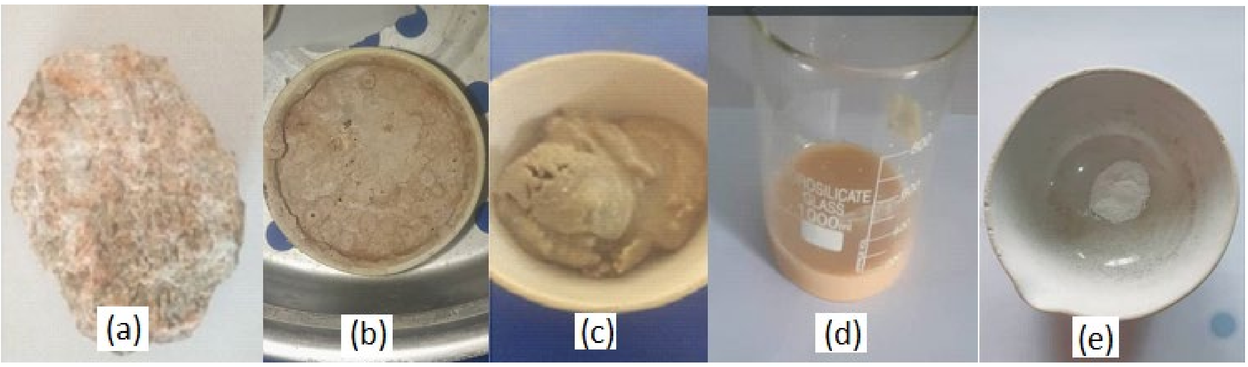

Figure 2. (a) Raw lepidolite sample; (b) Roasted ore after sulfuric acid treatment; (c) Slurry of roasted ore during leaching; (d) Filtrate obtained after water leaching; (e) Final precipitated lithium carbonate product

3.0 Results and Discussion

3.1 Mineralogical Transformations of Lepidolite Ore to Crystalline Lithium Carbonate

The mineralogical composition of the raw lepidolite ore (Figure 3) is dominated by quartz at 25.71% and amblygonite at 51.2%, with smaller amounts of muscovite (10.01%), lepidolite (3.1%), and orthoclase (10.2%). This indicates that lithium-bearing phases such as lepidolite are present but in relatively minor quantities within a gangue-rich matrix. After calcination (Figure 4), significant changes occur in the mineralogical composition. Quartz increases markedly to 53.7%, amblygonite decreases to 30.8%, while lepidolite shows a slight increase from 3.1% to 5.2%, suggesting that calcination enhances the detectability or relative concentration of lithium-bearing phases. Additionally, new phases such as albite (10.6%) and lime (2.04%) appear, reflecting transformations induced by heating. These changes demonstrate that calcination modifies the mineral structure, reduces certain phases like amblygonite, enhances lepidolite presence, and generates new minerals that may improve lithium liberation in subsequent processing steps (Liu, 2024).

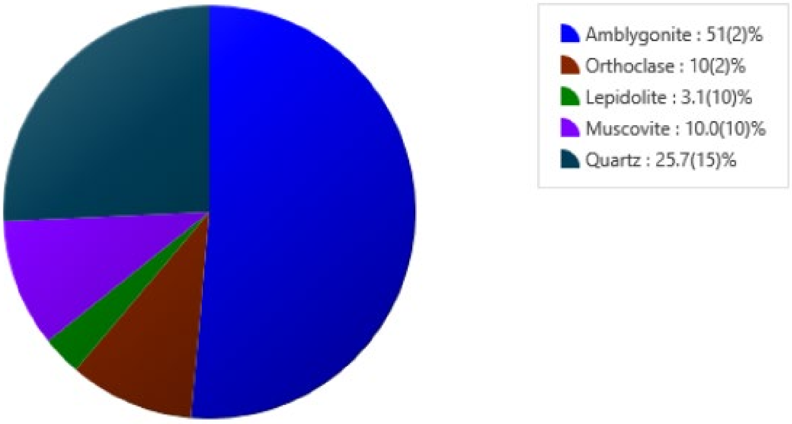

Figure 3: Mineralogical Composition of the Raw Lepidolite Ore from XRD

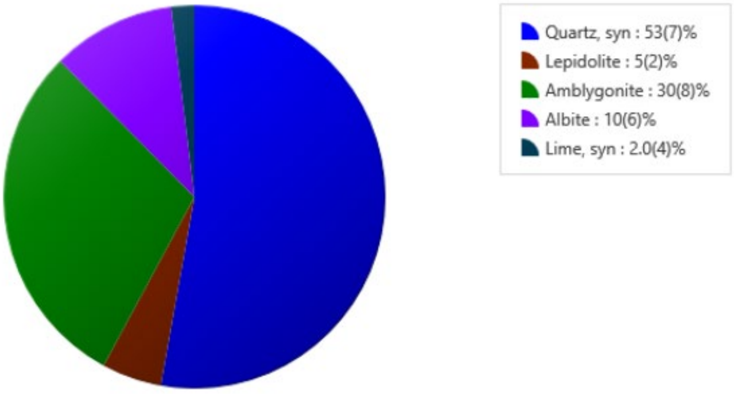

Figure 4: Mineralogical Composition of the Calcined Lepidolite Ore from XRD

The XRD analysis demonstrates the progressive transformation of lepidolite ore through calcination and precipitation stages. In the raw ore (Figure 5: XRD pattern of the raw lepidolite ore), sharp peaks of lepidolite are observed alongside quartz, feldspars, muscovite, and amblygonite, confirming its complex mineralogy and the structural binding of lithium within the mica lattice, which complicates direct extraction (Mubarok et al., 2021). This observation is consistent with the XRF analysis presented in Table 1, which shows that the raw ore is dominated by silicon (30.40%), aluminum (10.70%), and potassium (4.96%), while lithium is present only in small amounts (1.12%). The relatively low lithium concentration relative to the major gangue elements further highlights the challenge of direct recovery and underscores the importance of thermal activation to liberate lithium from the mica structure (Setoudeh et al., 2019). Following calcination (Figure 6: XRD pattern of the calcined lepidolite), the lepidolite peaks decrease in intensity, broaden, or shift, reflecting structural dehydroxylation and collapse of the layered framework. This structural modification highlights the role of calcination in activating the ore and enhancing lithium availability for leaching (Zhu et al., 2023). In contrast, the XRD pattern of the precipitated lithium carbonate (see Figure 7) shows sharp, well-defined peaks with minimal background noise, characteristic of a crystalline and high-purity product (Lei, 2024). The absence of detectable impurity phases confirms selective precipitation. Overall, the results validate calcination as an effective activation step and demonstrate successful recovery of high-purity lithium carbonate, a material essential for the production of battery-grade lithium compounds used in energy storage technologies.

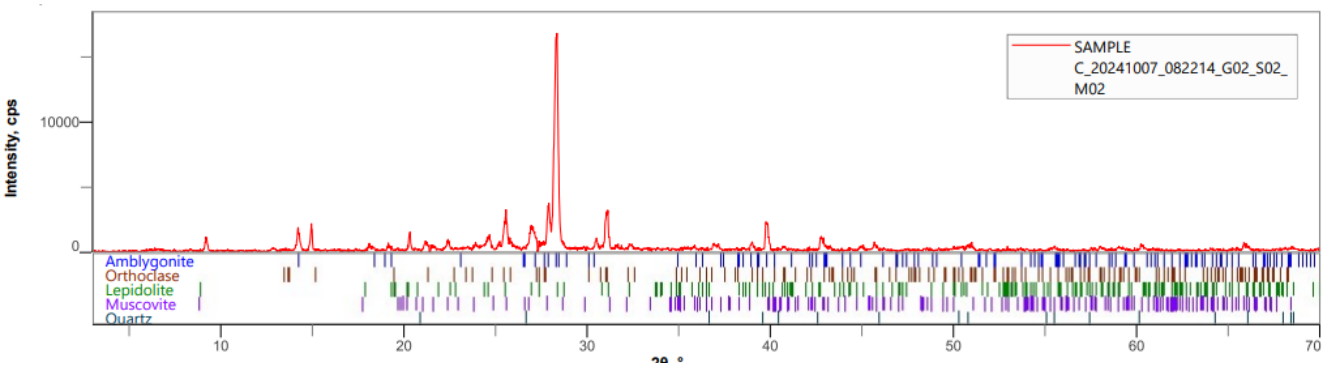

Figure 5: XRD pattern of the raw lepidolite ore

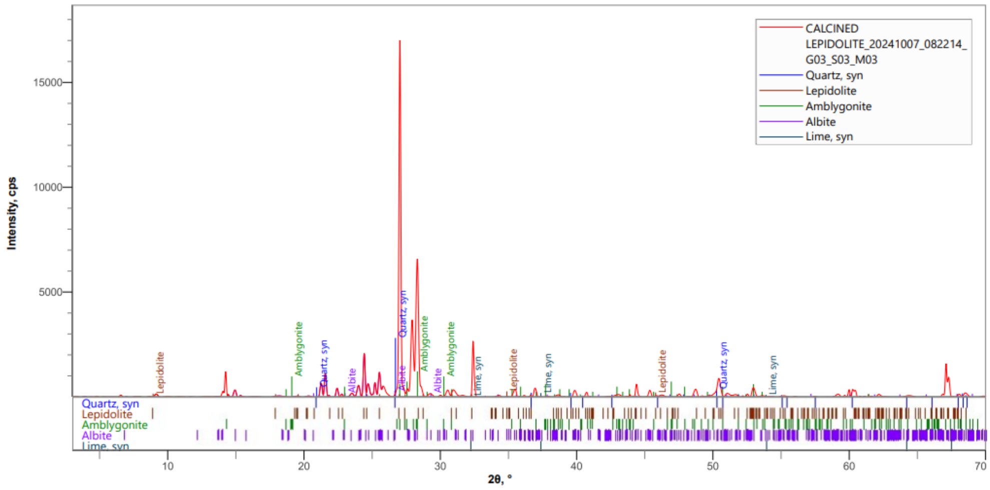

Figure 6: XRD pattern of the Calcined lepidolite Ore

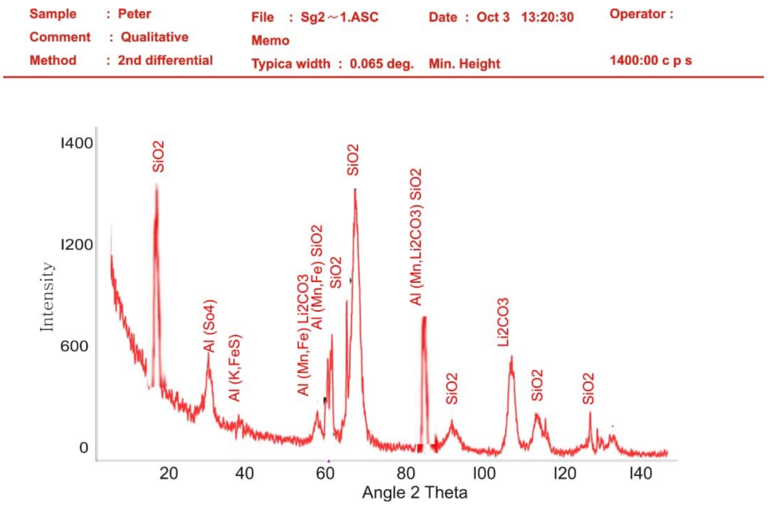

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Figure 7: XRD pattern of the precipitated Li_2CO_3

Table 1: XRF Analysis of Raw Lepidolite Ore

Element	Si	Al	K	Fe	Rb	Mn	Li	Ca	Ti	Cs	O + Others	Total
%	30.40	10.70	4.96	1.78	1.54	0.33	1.12	0.07	0.05	0.04	49.01	100.00

3.2 Impurity Removal and Lithium Enrichment during Precipitation

The results in Table 2 show compositional changes between the leachate and the final lithium carbonate product, reflecting the effectiveness of purification and precipitation. Silica and alumina remain high in both matrices (SiO_2: 48.95% to 47.97%; Al_2O_3: 28.92% to 29.96%), indicating limited removal and likely co-precipitation of silicate/aluminate residues with Li_2CO_3, underscoring a need for improved impurity control. Iron and manganese decreased (Fe_2O_3: 0.14% to 0.10%; MnO: 0.15% to 0.12%), consistent with NaOH treatment and precipitation reducing transition metals. Calcium decreased (CaO: 0.12% to 0.08%), while magnesium increased slightly (MgO: 0.22% to 0.30%), suggesting partial co-precipitation of Mg with Li_2CO_3. Potassium was nearly unchanged (K_2O: 10.02% to 10.04%), and sodium decreased slightly (Na_2O: 0.30% to 0.25%), reflecting the challenge of separating alkali salts with similar solubility behavior.

Among trace elements (ppm), cesium increased (230 to 245 ppm), rubidium decreased slightly (560 to 550 ppm), tin decreased markedly (12.8 to 10.2 ppm), niobium decreased (3.60 to 3.30 ppm), and gallium remained unchanged (6.60 ppm). Lithium was enriched in the product (1980 ppm; not detected in leachate), confirming successful precipitation as Li_2CO_3. Loss on ignition (LOI) remained stable (4.33% to 4.30%), indicating similar volatile content (hydroxyls, carbonates). Overall, lithium recovery was effective, with reductions in several impurities, though substantial SiO_2/Al_2O_3 and K_2O persisted.

Table 2a: Bulk composition of leachate and lithium carbonate (oxides and LOI, %)

Element	**Leachate (%)**	**Li_2CO_3 (%)**
SiO_2	48.95	47.97
Al_2O_3	28.92	29.96
Fe_2O_3	0.14	0.10
MnO	0.15	0.12
CaO	0.12	0.08
P_2O_5	0.05	0.02
K_2O	10.02	10.04
TiO_2	0.01	0.02
MgO	0.22	0.30
Na_2O	0.30	0.25
LOI	4.33	4.30

Table 2b: Trace and special elements (ppm)

Element	**Leachate (ppm)**	**Li_2CO_3 (ppm)**
Li	—	1980
Ta	0.40	0.37
Cs	230	245
Sr	1.45	1.55
Sn	12.8	10.2
Nb	3.60	3.30
Rb	560	550
Ga	6.60	6.60

3.3 SEM-EDX Analysis of Lepidolite and Lithium Carbonate

The SEM and EDX results presented in Figures 7 and 8 provide important insights into the structural and compositional transformations of lepidolite during the extraction and precipitation of lithium carbonate. The SEM image of raw lepidolite (Figure 7a) shows a heterogeneous surface morphology with compact, irregularly shaped particles that reflect the crystalline silicate framework typical of lithium-bearing aluminosilicates. In contrast, the SEM image of precipitated Li_2CO_3 (Figure 7b) displays a more homogeneous and porous morphology, with finer and loosely aggregated particles indicating successful breakdown of the silicate matrix during acid roasting and subsequent precipitation (Ma, 2023). This morphological transformation demonstrates that lithium ions were effectively leached from the lepidolite structure and subsequently recrystallized as Li_2CO_3. The EDX spectra further confirm the compositional changes. The raw lepidolite spectrum (Figure 8a) shows significant peaks corresponding to silicon (Si), aluminum (Al), potassium (K), iron (Fe), and oxygen (O), consistent with its aluminosilicate composition. Minor peaks of rubidium (Rb), manganese (Mn), and trace elements are also present, reflecting the multicomponent nature of lepidolite. In contrast, the EDX spectrum of the precipitated product (Figure 8b) reveals prominent peaks associated with lithium carbonate, including oxygen (O) and carbon (C), while the intensity of Si, Al, and K peaks is significantly reduced, confirming that most silicate impurities were left behind after leaching and purification. The appearance of distinct carbonate-related peaks verifies the successful precipitation of Li_2CO_3 (Lei, 2024). Overall, the SEM images illustrate the physical breakdown and recrystallization from a compact crystalline ore to a fine precipitate, while the EDX spectra highlight the compositional shift from a complex silicate matrix to a lithium-enriched carbonate phase. Together, these results confirm the effectiveness of the roasting–leaching–precipitation process in concentrating lithium and producing lithium carbonate, although the persistence of trace impurities suggests that further purification steps may be required to achieve battery-grade quality.

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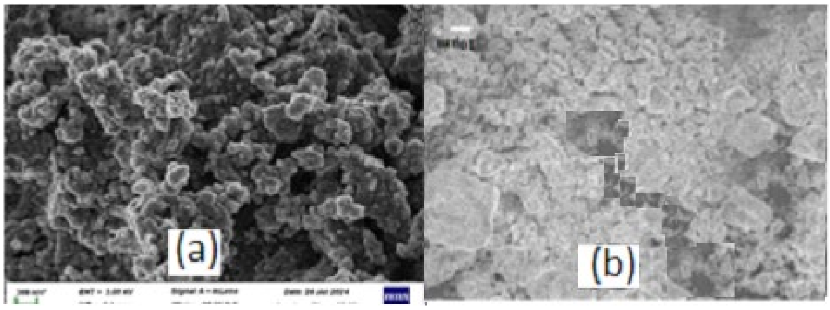

Figure 7: SEM Image of (a) raw lepidolite; (b) precipitated Li_2CO_3

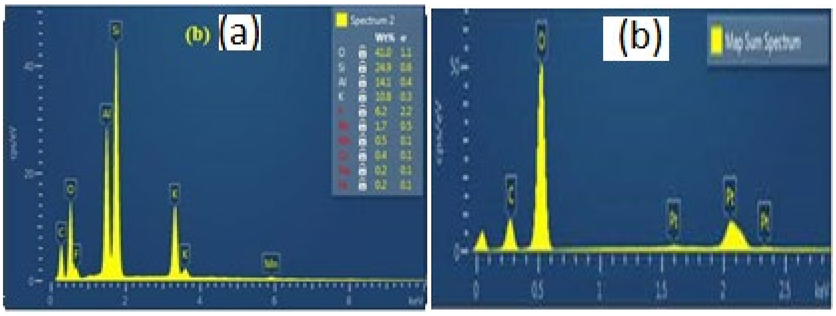

Figure 8: EDX of (a) raw lepidolite; (b) precipitated Li_2CO_3

4.0 Conclusion

The investigation successfully demonstrated the technical feasibility of extracting lithium from lepidolite ore from Eggon, Nigeria, using a sulfuric acid roasting and water leaching process. The multi-stage methodology effectively converted lithium from its inert silicate structure into a soluble sulfate, enabling its subsequent recovery. XRD and SEM-EDX analyses confirmed the progressive mineralogical transformation from a complex aluminosilicate ore to a crystalline lithium carbonate product. While the process successfully produced a lithium-rich material, the persistence of some trace metals persisted highlighted the need for further purification steps to achieve battery-grade quality. Nonetheless, this study validates the method as a viable pathway for lithium carbonate production and provides a strong foundation for optimizing the process to enhance purity for industrial applications.

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