XVI International Mineral Processing
and Recycling Conference
28-30 May 2025, Belgrade, Serbia

DOI:10.5937/IMPRC25681S Original research article

PRELIMINARY INVESTIGATION ON NIGERIAN LITHIUM ORES FOR EFFICIENT LITHIUM OXIDE EXTRACTION

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ABSTRACT – This paper assesses the compositions of the lithium-rich minerals and the quantity of extractable lithium oxide in the minerals. The x-ray diffraction (XRD) shows 17% - 34% of spodumene and 9% to 30% of lepidiolite. Similarly, atomic absorption microscopy (AAS) indicates that the lithium-rich minerals contain 0.820% - 3.051% and 0.040 - 2.395% of lithium oxide (Li_2O). Further results confirm the presence of lithium-rich minerals in Nigeria, and a high quantity of lithium-rich minerals suggests that high lithium oxide can be extracted. This information is useful for miners, mining and mineral processing industries, and investors in lithium for different industrial applications.

Keywords: Lithium-Rich Minerals, Diluents, Mineralogy, Lithium Oxides.

INTRODUCTION

Lithium is one of the world's most sought-after minerals/metals in 2024, and Nigeria is not left out [1]. In the past year, there has been a notable and abrupt escalation in the activities related to the exploration and extraction of Lithium ore, primarily attributed to the surge in demand stemming from technological advancements [2]. This demand has directly contributed to the heightened interest and efforts in the mining sector. Several sources of lithium have been discovered and explored. However, very little is said about the global lithium deposits. For instance, little is known about the quality and mineralogical composition of lithium-rich minerals in Nigeria. Similarly, the African lithium deposits are poorly documented and reported despite massive mineral extraction and processing in Nigeria and several other Sub-Saharan African countries [3, 4, 5]. Considering the potential of lithium for the green energy transition and energy storage technologies, the knowledge of the reserve estimates of lithium deposits in Africa is critical to meeting the global demands for lithium.

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Lithium metal was discovered in 1817 by Johan Arfvedson. It is a silver-white, light metal with an atomic weight of 6.93 and does not occur naturally in metallic form owing to its reactivity with air [6]. Lithium has excellent properties and is considered a battery element. Typically, lithium has low standard reduction potential, thus making it the lightest metal for high density, high-voltage cell applications. The work of Caprara et al. [7], 2023 showed that lithium may exist in oxide form under the earth as a lithium-rich mineral [7]. These minerals include spodumene, lepidolite, petalite, amblygonite, cookite, and zinnwaldite. Similarly, Dessemond et al. [6] reported that in addition to the lithium-rich minerals, quartz, feldspar, and mica interlock between mineral grains and occupy a more significant portion of the lithium-bearing minerals [6].

Lithium is a vital raw material for energy applications. Lithium may exist as lithium oxide in lithium-bearing minerals, and the quality of the ores depends on the percentage of lithium oxide. Li-bearing minerals are classified based on the percentage of lithium oxide. In a typical lithium run-of-mine, the percentage of lithium-rich minerals varies across different locations within the deposits as the percentage of lithium oxide (Li_2O).

While it is expected that a high percentage of lithium-bearing minerals will translate to high lithium oxide in the mineral, the variation in the quality of mineral deposits or veins depends on several factors. This includes the distribution of lithium-mineral grains within the matrix of the associated minerals, the geology of the deposits and the elemental composition of the lithium ores.

This paper assesses the compositions of the lithium-rich minerals and the quantity of extractable lithium oxide in them using a combination of X-ray diffraction (XRD) and atomic absorption microscopy (AAS). It examines the elemental composition of two different lithium-bearing minerals (spodumene and lepidolite) and analyses the geological characteristics of lithium deposits within Nigeria.

EXPERIMENTAL

Sample Preparation

Twenty (20) randomly sampled lithium ore were picked from two major lithium mining sites in Nigeria (Nasarawa State and Ekiti State), ground into fine particles (< 75 µm) and analyzed using x-ray diffraction (XRD) and atomic absorption microscopy (AAS). The study adopts a multidisciplinary approach involving compositional analysis, mineralogical identification, and comparative evaluation.

Analysis of Lithium Ores

Atomic Absorption Microscopy (AAS) Analysis of Lithium Ores

The samples were pulverized, sieved to 75 µm and mixed with Na_2CO_3 (1:1) in a platinum crucible. The mixture was heated in a furnace at 1000 ^{o}C for 1 hour, cooled and dissolved in HCl. The solution was filtered and analyzed using atomic absorption microscopy (AAS) (Thermo-Scientific 3000 Series). The device was calibrated with deionized water, and the filtrate was analyzed using a lithium cathode lamp following standard procedures.

X-ray Diffraction (XRD) Analysis of Lithium Ores

The samples were pulverized using laboratory mortar, sieved to 75 µm, and fed into the X-ray diffraction (XRD) sample holder. X-ray diffractometer Thermo Scientific Model (ARL'XTRA X-Ray and serial number 197492086) was used for the analysis. The samples were inserted into the machine and analyzed at d-spacing appropriate to diffract x-rays. Similarly, the Cu cathode base was used on a reflection-transmission spinner (Theta-Theta setting) and a 2Θ starting position from 0^o and ending at 70^o. The file of d-spacing available in the International Center for Diffraction Data (ICDD) and PDF2 software were used to match the x-ray diffraction peaks.

Data Integration, Analysis and Validation

The compositional and mineralogical data were integrated into the study to evaluate the relative abundance of lithium-bearing minerals (spodumene and lepidolite) and the dilutive effects of non-lithium phases like quartz, muscovite, and albite. Similarly, the variation of lithium oxide in the minerals was examined using the principal component analysis (PCA) and cluster analysis based on the composition and mineralogy of ores. Further analysis of the preliminary findings on the Li_2O content, mineralogy, the presence of dilutive phases, and the quantity of the extractable lithium from the ores were validated with existing literature and industry standards to develop a comprehensive framework for characterizing lithium ores, assess resource utilization and develop extraction strategies.

Testing of Research Hypothesis

The analyzed data were used to test the set research hypothesis (RH) and respond to the research question (RQ).

RQ: Do high lithium-rich minerals in lithium-containing rocks produce higher Li_2O?

RH: High lithium-rich rock minerals are responsible for higher Li_2O in rocks?

RESULTS AND DISCUSSION

Figure 1 compares spodumene and Li_2O fractions, showing that higher spodumene levels often correspond to higher Li_2O fractions. Nevertheless, this correlation does not manifest uniformly across all examined samples. By observing the bars in the graphs, where spodumene levels are highest, Li_2O fractions are also generally elevated. However, there are exceptions where high spodumene does not translate to a correspondingly high Li_2O fraction—of the 10 samples analysed using XRD and AAS, eight samples representing 80%, followed this trend. This indicates that higher spodumene content indicates higher Li_2O content. Also, this implies an 80% chance of higher Li_2O in ores containing lithium when the lithium-rich minerals are high. Hence, XRD analysis can provide relevant information for preliminary economic decisions required in mining and processing when the cost of further analysis is exorbitant or access to analytical devices is limited.

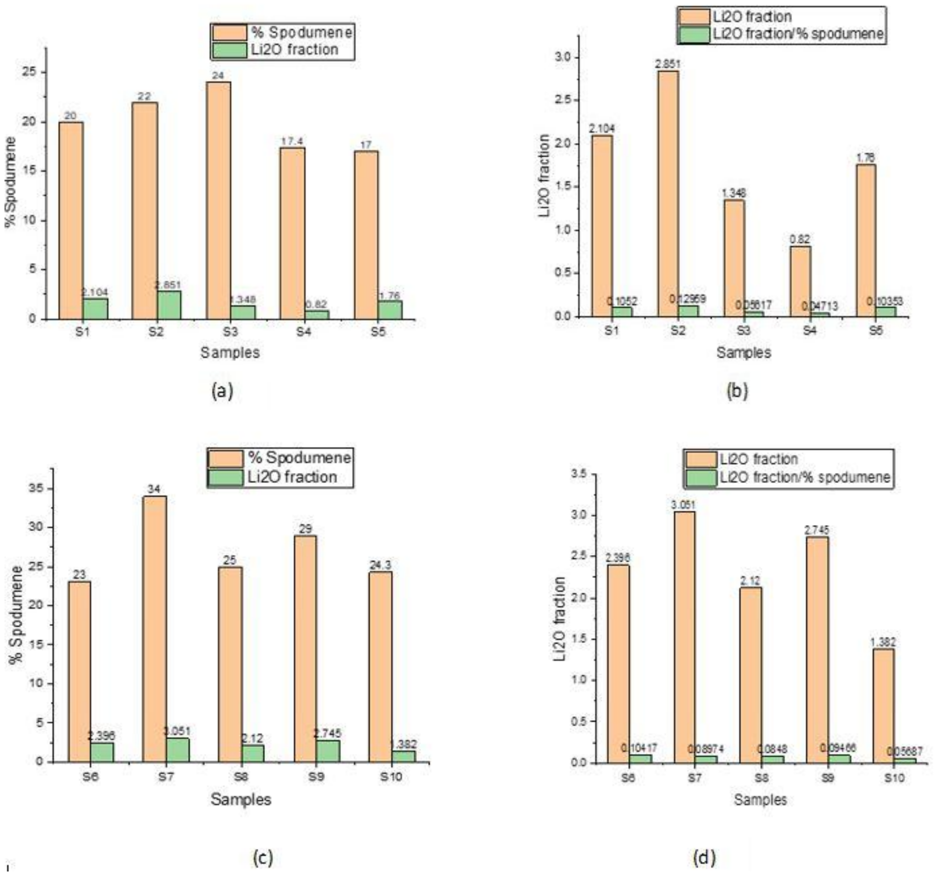
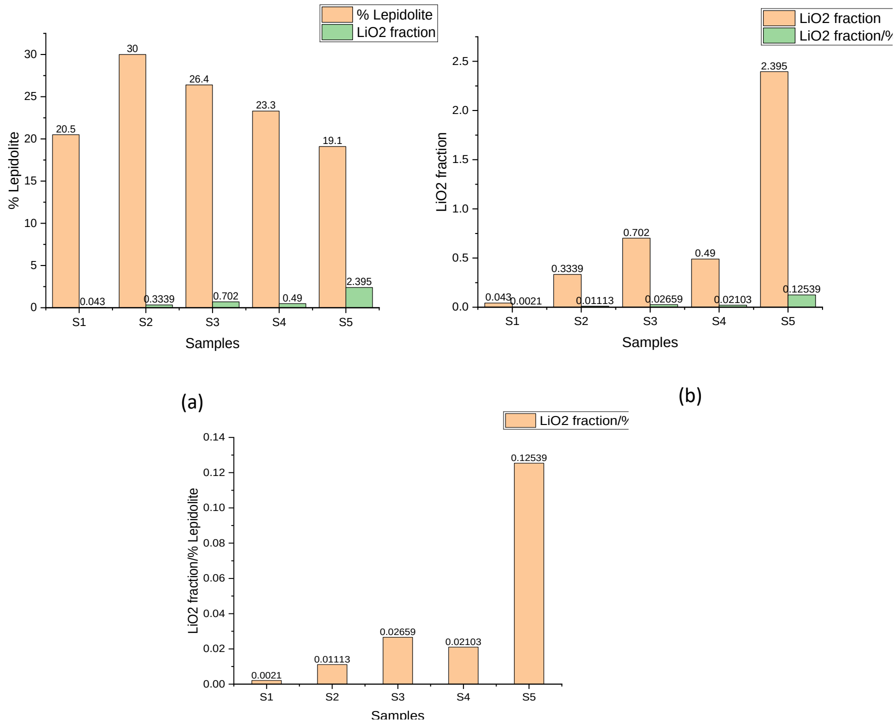


Figure 1 Quantity of Lithium-rich Mineral and Lithium oxide in Lithium ores. (a & b) comparison between the percentage of the lithium-rich mineral in the ores and the percentage of lithium oxide present in each mineral, (c & d) lithium oxide-to-lithium mineral ratio for Spodumene ores

Figure 2 shows the correlation measure between the lithium-rich mineral (lepidiolite) and the quantity of lithium oxide present in the lithium ores. Generally, a high lithium-rich mineral (lepidolite) does not translate to high lithium oxide for lepidolite. This outcome is anticipated as there is a wide dispersion of lithium oxide in lepidolite, resulting in low lithium oxide. Similarly, the literature has shown that lepidiolite is a Li-bearing clay mineral with a low Li2O grade. Typical lepidolite run-of-mine contains 1-2% Li2O, while the concentrate contains 2-3.5%. In contrast, the % Li2O in lepidolite is relatively low compared to typical lepidolite run-of-mine, except sample S5, which has a high % Li2O comparable to lepidolite concentrate. While some instances of elevated lepidolite correspond to a relatively high Li2O fraction, there are multiple cases where high lepidolite levels do not translate to a high Li2O fraction. This suggests that while lepidolite confirms the presence of Li2O formation, other factors or limitations might impact the Li2O yield, weakening the direct relationship between the quantity of the minerals in the ores and the extractable lithium oxide.



(c)

Figure 2 Quantity of lithium-rich mineral and lithium oxide in lithium ores (a & b) comparison between the percentage of the lithium-rich mineral in the ores and percentage of lithium oxide present in each mineral, (c) lithium oxide-to-lithium mineral ratio for Lepidolite ores

Figure 3 shows the lithium-rich minerals and other mineral phases in the lithium ores. The non-lithium mineral phases in the ore dilute the overall lithium oxide (Li2O) content. Quartz (SiO_2) dominates non-lithium mineral phases as indicated by its high-intensity peaks (at 2Ө =26.90o). This shows that quartz is the major diluent in lithium-containing rocks, contributing significantly to the overall silica content without increasing the quantity of lithium oxide in the ores. Other diluents in the lithium ores include Albite ($NaAlSi_3O_8$) and Muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$). These contribute Na2O, K2O and alumina (Al2O3) to the composition of the ores. Similarly, Figure 3 shows that quartz, albite and muscovite (three non-lithium-rich minerals) coexist with spodumene, as it was widely reported in the literature, thereby reducing effective lithium concentration.

The fraction occupied by non-lithium-rich mineral phases (66 – 83%), such as quartz, albite, and muscovite, demonstrates the dominance of the mineral phases in contrast to spodumene. This implies that, despite spodumene, the overall lithium content is reduced due to the dominance of these non-lithium-bearing phases.

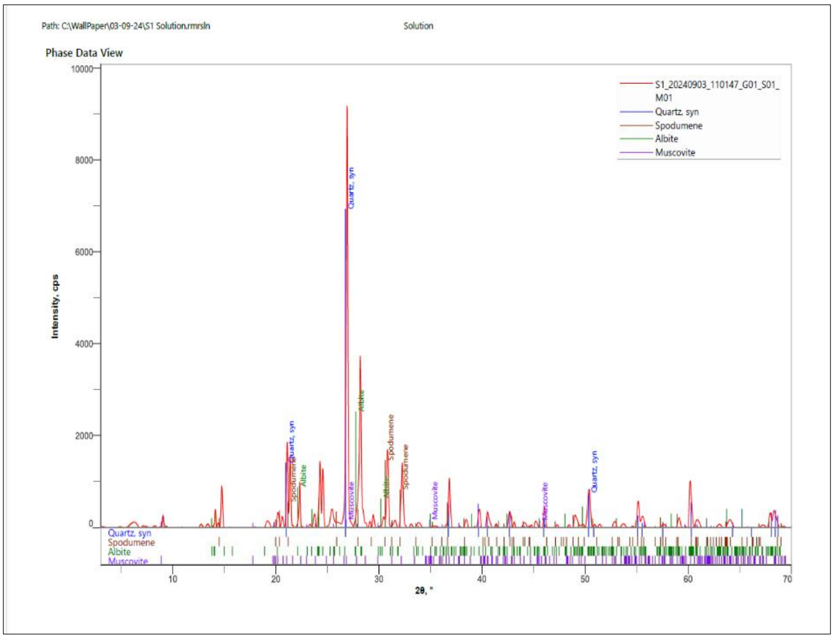


Figure 3 X-ray Diffraction (XRD) for Lithium Ore (Nasarawa State, Nigeria)

While impurities (undesired minerals or gangue) reduce the quality of lithium oxide in the ores, the ores can be concentrated to reduce non-lithium-rich mineral phases and increase the percentage of lithium oxide. However, preliminary analysis of the ores can provide helpful information on the quantity of extractable lithium from the lithium-rich mineral phase (spodumene) prior to the extraction and concentration of the ores.
As observed in Figure 3, Table 1 shows the chemical compositions of the lithium ores. High concentrations of silica (SiO_2), alumina (Al_2O_3) in some samples, and sodium oxide (Na_2O) in lithium ores contribute to the overall composition of the ores. The lithium-rich mineral (spodumene) is associated with a significantly high silica-to-alumina ratio (6.22 – 85.11) due to a relatively low percentage of alumina. Similarly, Na_2O and Fe_2O_3 contribute significantly to the high non-lithium mineral phase in the lithium ores. The presence of non-lithium mineral phases in the ores and their association with lithium oxide will affect the liberation, separation and recovery of design a process flow-sheet for efficient lithium extraction from the ores.

Table 1 Atomic Absorption Spectroscopy (AAS) Analysis of Lithium Ores from Nasarawa State, Nigeria

Samples / **Oxides**	**S1**	**S2**	**S3**	**S4**	**S5**
SiO_2	77.01	79.11	78.13	85.11	81.57
Al_2O_3	12.38	ND	0.14	ND	0.46
SO_3	ND	ND	0.3	ND	0.94
P_2O_5	ND	ND	ND	ND	ND
Na_2O	0.12	13.28	14.98	8.5	10.46
K_2O	2.64	ND	ND	1.2	ND
MgO	0.7	0.5	ND	0.2	ND
CaO	0.07	0.1	ND	ND	ND
TiO_2	0.3	0.4	0.28	0.57	0.52
MnO	0.45	2.75	0.2	0.19	0.37
Fe_2O_3	3.52	ND	3.22	12.51	2.81
Li_2O	2.104	2.851	1.348	0.82	1.76
L.O.I	0.7	0.8	1.2	0.7	0.9

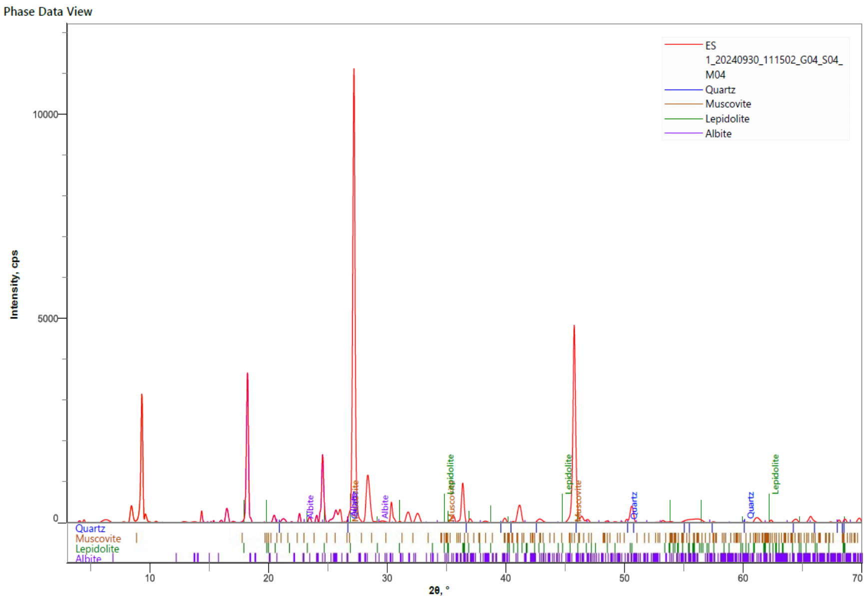


Figure 4 X-ray diffraction (XRD) for Lithium Ore (Ijero-Ekiti, Nigeria)

Figures 3 and 4 show the mineralogy of lithium ores and describe the crystalline structure of the minerals contained in the ores. The figures also present additional information on the structure of the ores at atomic scale. Both Figures identify three non-lithium mineral phases associated with the lithium-rich mineral phases (spodumene and lepidiolite). Most XRD peaks are thin and short, except those representing quartz. The information presented by the XRD results agrees with the chemical compositions of the elements in oxides and is relevant for mineral separation and recovery. As observed in lithium-caesium-tantalum (LCT) pegmatites, an assemblage of K-feldspar, quartz, muscovite and albite dominates the lithium ores. This detail describes the mineralogy of lithium ores and provides other relevant information for lithium extraction and process optimisation for enhanced lithium extraction.

Table 2 AAS Analysis of Lithium Ores (Ejero Ekiti, Nigeria)

Samples / **Oxides**	S1	S2	S3	S4	S5
SiO_2	62.25	56.34	54.02	56.78	71.33
Al_2O_3	26.25	36.15	37.39	35.07	20.74
SO_3	ND	ND	0.2	ND	ND
P_2O_5	ND	ND	0.98	ND	ND
Na_2O	1.59	0.2	0.6	0.09	0.5
K_2O	4.34	1.08	1.06	1.4	1.09
MgO	ND	ND	ND	ND	ND
CaO	ND	ND	ND	ND	ND
TiO_2	0.3	0.4	0.28	0.57	0.52
MnO	0.15	0.28	0.14	0.3	0.41
Fe_2O_3	1.17	0.76	0.69	0.9	1.33
Li_2O	0.043	0.334	0.702	0.49	2.395
L.O.I	4.2	4.7	5.3	4.7	2.2

Tables 1 and 2 show the chemical composition/mineralogy of the lithium ores randomly selected from Ejero Ekiti in Nigeria. The percentage of lithium oxide (Li_2O) ranges from 0.04 to 2.395%. The quantity of Li_2O in most samples is below 1%, except S5. Low Li_2O can be traced to a low silica-to-alumina ratio due to relatively high alumina within the muscovite and albite mineral phases, as shown in Figures 2 and 3. Tables 1 and 2 and Figures 3 and 4 confirm relatively low-grade lithium ores, dominated by three non-lithium mineral phases as diluent of the lithium-bearing deposits.

CONCLUSION

The study confirmed the presence of lithium ores in Nigeria and the quantity of extractable lithium oxides. Three densely dominated non-lithium mineral phases were identified as significant diluents of the lithium-rich minerals. While these mineral phases in the lithium ores lower the percentage of lithium oxide in the ores, the value is comparable to lithium ore deposits in countries where lithium processing is reported on a large scale. The preliminary information on the mineralogy, atomic arrangement of the ores, and crystal structure of the minerals is useful for the extraction and optimisation of lithium extraction in Nigeria.

ACKNOWLEDGEMENT

The authors appreciate the Regional Scholarship Innovation Fund (Rsif) for the PhD scholarship offered to the first author. The authors are also grateful for the matching fund provided by Dr. Afolayan David Oluwasegun of the African University of Science and Technology, Abuja, Nigeria and Professor Ogunmodimu of Pennsylvania State University*, PA, USA.*

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