“Production of Lithium Iron Phosphate ($LiFePO_4$) Battery from Nigerian Lithium Ore Minerals toward Lithium Battery Production for Energy Storage”

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CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

The global shift toward renewable energy and decarbonization has dramatically increased demand for lithium, a strategic mineral essential in the production of lithium-ion batteries. Over the past three decades, lithium has become the foundation of energy storage technologies for portable electronics, electric vehicles, and grid-scale renewable systems (Olajuyi, 2025). While Australia, Chile, and China dominate global production, recent discoveries of pegmatite-hosted lithium deposits in sub-Saharan Africa, particularly the Jos Plateau in Nigeria, present new opportunities for resource development and industrial diversification. Harnessing these resources through context-specific processing innovations could position Nigeria as a regional hub in the global battery value chain.

This research proposes the design and processing of Nigerian lithium ores for the production of lithium carbonate (Li_2CO_3) and lithium hydroxide (LiOH), which are critical precursors in the synthesis of lithium iron phosphate ($LiFePO_4$, LFP) battery cathodes. LFP batteries are globally recognized for their safety, long cycle life, affordability, and environmental sustainability compared to cobalt-based alternatives (Chen et al., 2024). The novelty of this study lies in the development of furnace systems and beneficiation flowsheets specifically tailored to the mineralogy of Nigerian spodumene- and petalite-rich ores, combined with renewable energy integration such as solar thermal or hybrid systems for high-temperature processing. This innovative approach addresses both engineering performance and sustainability goals, while reducing the carbon intensity of lithium production.

From a commercial standpoint, the viability of this research is strengthened by rapidly expanding global and regional markets for LFP batteries. Market studies indicate a surge in demand for energy storage systems to support renewable power deployment across Africa, alongside growing electric mobility adoption. Localized lithium processing in Nigeria offers the dual advantage of reducing import dependence and enabling domestic value addition. The potential for off-take agreements with renewable energy developers and local system integrators highlights the strong commercialization pathway for Nigerian-sourced lithium products.

Preliminary evidence also supports the feasibility of this research direction. Previous geological studies confirm significant spodumene and petalite occurrences in Nigerian pegmatites, with beneficiation and roasting identified as viable processing routes (Olajuyi, 2025). International empirical studies further demonstrate the effectiveness of alternative techniques such as NaOH roasting, mechanochemical activation, and salt-assisted leaching in lowering energy requirements and improving recovery rates (Gao, 2023; Zhang et al., 2022). Laboratory-scale trials in Nigeria have provided early indications of recoverable lithium precursors from local ores, reinforcing the potential for scale-up using indigenous engineering solutions.

This project directly aligns with the objectives of the National Agency for Science and Engineering Infrastructure (NASENI) by fostering indigenous technology development, strengthening

Nigeria's industrial base, and advancing the Renewed Hope Agenda. By innovating furnace and processing designs adapted to local ores, the research will build domestic capacity for producing lithium precursors, enhance renewable energy integration through localized storage solutions, and create new opportunities for industrial growth and socio-economic impact.

1.2 Statement of the Problem

Despite Nigeria's documented lithium reserves, the country lacks indigenous technologies and industrial frameworks for processing these resources into battery-grade materials. Currently, lithium beneficiation and chemical conversion technologies are concentrated in countries such as China and Australia, creating dependency on imports and limiting Nigeria's participation in the global energy storage supply chain. Existing studies on Nigerian pegmatites confirm the presence of spodumene and petalite, yet there is no comprehensive engineering solution that addresses the beneficiation, roasting, and chemical conversion of these ores under local industrial and energy conditions (Olajuyi, 2025).

Conventional processing techniques, such as sulfate or alkaline roasting followed by leaching, are energy intensive and environmentally burdensome. Although global research has explored more sustainable routes such as NaOH roasting, mechanochemical activation, and salt-assisted leaching (Gao, 2023; Zhang et al., 2022) these innovations have not been adapted to Nigeria's unique context. In particular, there is a lack of locally designed furnace systems and process flowsheets that integrate renewable energy inputs to reduce carbon intensity and energy costs. Without such innovations, the commercial and environmental potential of Nigerian lithium remains underutilized.

This technological gap has wider implications for Nigeria's renewable energy transition. The country faces persistent challenges in electricity access and reliability, which require scalable and affordable energy storage solutions. Lithium iron phosphate ($LiFePO_4$) batteries, known for their safety, cost-effectiveness, and long cycle life, are ideally suited for stationary renewable energy applications. However, the absence of a domestic supply chain for lithium precursors undermines Nigeria's ability to localize energy storage production and industrial growth.

Therefore, the central problem addressed by this research is the absence of context-specific, sustainable, and commercially viable processing technologies for Nigerian lithium ores. Without the design of efficient furnace systems, beneficiation flowsheets, and renewable-powered processing routes, Nigeria risks remaining a raw material exporter rather than a value-adding participant in the global battery economy. Addressing this gap is critical for advancing Nigeria's industrialization, achieving the objectives of the National Agency for Science and Engineering Infrastructure (NASENI), and contributing to the Renewed Hope Agenda by enabling domestic production of lithium-based energy storage systems.

1.3 Research Questions

i. What furnace design and process flowsheet are most effective for the beneficiation, roasting, and leaching of Nigerian lithium ores at the laboratory scale?

ii. What are the mineralogical and chemical characteristics of Nigerian lithium ores, and how do these properties influence phase transformations, recovery efficiency, and the purity of Li-based precursors (Li_2CO_3, LiOH) for $LiFePO_4$ synthesis?
iii. How do different processing routes compare in terms of energy requirements and environmental impacts, and to what extent can renewable energy integration reduce the carbon intensity of lithium ore processing?
iv. What are the industrial and economic feasibility considerations for adopting localized lithium ore processing technologies in Nigeria to support $LiFePO_4$ battery production for renewable energy storage applications?

1.4 Research Aim and Objectives

Aim of the Research

To design and develop sustainable processing techniques for Nigerian lithium ore to produce Li-based precursors for $LiFePO_4$ battery materials in renewable energy storage applications.

Objectives of the Research

i. To design and construct a laboratory-scale furnace with a complete process flowsheet for beneficiation, roasting, and leaching of Nigerian lithium ores
ii. To characterize the mineralogy, chemical composition, phase transformations, and lithium recovery efficiency of Nigerian ores using XRD, XRF, SEM-EDS, and leaching experiments.
iii. To evaluate the energy requirements and environmental impacts of at least three processing routes through life cycle assessment (LCA).
iv. To analyze the industrial and economic feasibility of localized lithium ore processing technologies in Nigeria.

1.5 Significance of the Study

This research is significant for several reasons, both academically and practically. At the engineering level**,** it introduces an innovative furnace design and integrated process flowsheet tailored specifically to the mineralogy of Nigerian lithium ores. This will provide indigenous technological capacity for beneficiation, roasting, and leaching, reducing dependence on imported equipment and processes. From a materials science perspective**,** the study will generate detailed characterization data on Nigerian spodumene and petalite ores, including mineralogy, chemical composition, and phase transformations. Such knowledge will not only improve lithium recovery efficiency and precursor purity but will also contribute to the global scientific literature on $LiFePO_4$ cathode synthesis.

At the sustainability level**,** the project integrates renewable energy options into high-temperature processing, demonstrating pathways to reduce the carbon intensity of lithium production by at least 20%. This is particularly important in aligning Nigeria's emerging mining sector with global climate commitments while promoting green industrial practices. At the industrial and economic level**,** the project will provide feasibility analyses, market assessments, and cost–benefit models that demonstrate the viability of localized lithium processing for energy storage industries. These outputs will support Nigeria's participation in the rapidly expanding lithium battery value chain and stimulate domestic manufacturing opportunities.

For policy makers**,** the study offers evidence-based insights into how Nigeria can develop a sustainable lithium processing and battery materials sector. The life cycle assessment (LCA) and economic feasibility studies will provide decision-makers with data to design effective policies on critical mineral utilization, renewable energy integration, and industrial value addition. By outlining the technical, environmental, and commercial requirements for scaling up lithium processing, the study will guide policies that align with the National Agency for Science and Engineering Infrastructure (NASENI) mandate, the Nigerian Energy Transition Plan, and the Renewed Hope Agenda. In this way, the research not only contributes to academic knowledge and industrial innovation but also directly informs strategic national policy for energy security, industrial growth, and sustainable development.

1.6 Scope of the Study

This study is geographically focused on Nigerian lithium ore deposits, particularly spodumene- and petalite-bearing pegmatites found in regions such as the Jos Plateau and surrounding areas where lithium mineralization has been identified. The research will be limited to ores sourced within Nigeria, ensuring that findings directly reflect the country's geological and industrial context.

Experimentally, the study will be confined to the laboratory-scale design and construction of a furnace system integrated with beneficiation, roasting, and leaching operations. Laboratory characterization techniques including X-ray diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS), and controlled leaching experiments will be employed to investigate mineralogy, chemical composition, phase transformations, and lithium recovery efficiency. The work will target achieving recovery rates above 80% and precursor purity levels of at least 95% for Li_2CO_3 or LiOH, providing reliable baseline data for potential scale-up.

The research will adopt a sustainability-oriented approach by evaluating the energy and environmental impacts of three different processing routes through life cycle assessment (LCA).

CHAPTER TWO

LITERATURE REVIEW

2.1 Literal Definition Lithium ore

Lithium ore refers to naturally occurring minerals or rocks that contain significant concentrations of lithium, a soft, silvery-white alkali metal with the atomic number 3 and symbol Li on the periodic table. Lithium is known for its unique properties, such as being the lightest metal, having the highest electrochemical potential, and being highly reactive with water. These characteristics make lithium a critical component in various applications, including rechargeable batteries used in electric vehicles and energy storage systems (Geology Science, 2023).

2.2 Working Definition Lithium ore

Lithium ore is any mineral material from which lithium can be extracted using industrial or laboratory processes, such as crushing, grinding, beneficiation, roasting, and leaching, to produce lithium compounds suitable for battery production. Common lithium-bearing minerals include spodumene ($LiAl(SiO_3)_2$), lepidolite ($K(Li,Al)_3(Al,Si,Rb)_4O_{10}(F,OH)_2$), and petalite ($LiAl(Si_2O_5)_2$) (Minerals Education Coalition, n.d.).

2.3 Operational Definition Lithium ore

In the context of this study, lithium ore refers to Nigerian mineral deposits containing lithium-bearing minerals that can be processed into lithium precursors for the manufacture of $LiFePO_4$ battery cathode materials for renewable energy storage applications. These ores are subjected to various processing techniques to extract lithium in forms suitable for use in energy storage systems (Minerals Education Coalition, n.d.).

2.4 Mineral, Physical,Chemical and Thermal Properties of Lithium Ore

Lithium ore refers to naturally occurring rocks or minerals that contain lithium-bearing phases. The properties of these ores vary depending on their mineralogical type, such as spodumene, lepidolite, petalite, amblygonite, hectorite, and jadarite, but they are generally described in terms of mineralogical, physical, chemical, and thermal characteristics. Mineralogically, the main lithium ores include spodumene ($LiAlSi_2O_6$), a pyroxene-group mineral with a monoclinic structure that represents the most common commercial source of lithium; petalite ($LiAlSi_4O_{10}$), a tectosilicate that is stable at lower temperatures; and lepidolite ($K(Li,Al)_3(Al,Si,Rb)_4O_{10}(F,OH)_2$), a mica-group mineral rich in lithium, rubidium, and cesium. Other lithium-bearing minerals include amblygonite ($LiAlPO_4F$), a phosphate mineral with high Li_2O content; clay-type ores such as hectorite, which are lithium-bearing smectite clays; and jadarite ($LiNaSiB_3O_7(OH)$), a rare borosilicate first identified in Serbia (Yang, *et al.*,2025).

Physically, the appearance and mechanical properties of lithium ores differ significantly. Spodumene typically appears white, green, or pink; lepidolite shows lilac or pink coloration; while petalite is often colorless to gray. Their hardness on the Mohs scale ranges from 6.5–7.0 for spodumene, 6.0–6.5 for petalite, and 2.5–4.0 for lepidolite. Densities also vary, with spodumene averaging 3.1–3.2 g/cm³, petalite around 2.4 g/cm³, and lepidolite between 2.8 and 3.0 g/cm³. Cleavage patterns are distinct, with spodumene exhibiting perfect prismatic cleavage and lepidolite showing perfect basal cleavage, consistent with its mica-like structure. In terms of crystallography, spodumene and petalite are monoclinic, while lepidolite may occur in monoclinic or triclinic systems (Petrakis, et al., 2025).

Chemically, lithium ores differ primarily in their Li_2O content. Spodumene is the richest, with 6–8% Li_2O, followed by amblygonite, which can reach up to 8–10%, petalite with 4–5%, and lepidolite with 3–5%. Clays and brines generally contain lower lithium concentrations, though they are often available in very large tonnages. Alongside lithium, these ores commonly contain aluminum, silicon, potassium, sodium, fluorine, rubidium, cesium, and sometimes rare earth elements. Spodumene is notable for its low chemical reactivity in its natural α-phase, which necessitates thermal activation at approximately 1000–1100 °C to transform into the β-phase that is more reactive and thus more suitable for leaching (Necke, 2023).

Thermal properties also play a decisive role in processing. Naturally occurring α-spodumene is dense and inert, but upon heating above 1000–1100 °C, it converts into β-spodumene, which is less dense and chemically more reactive, a transformation that is essential for lithium extraction. By contrast, petalite and lepidolite can be decomposed more readily by acid or alkaline digestion without requiring such high-temperature roasting. The melting points of spodumene and petalite are around 1380 °C and 1410 °C, respectively, reflecting their stability at elevated temperatures (Necke, 2023).

From an industrial and processing perspective, these properties have direct implications for ore beneficiation and lithium recovery. Higher Li_2O content indicates better ore grade, while higher hardness, such as that of spodumene, increases comminution costs during crushing and grinding. Thermal stability determines roasting requirements and influences the efficiency of subsequent leaching steps. Furthermore, impurities such as iron, magnesium, and calcium must be carefully managed, as they can hinder the production of battery-grade lithium carbonate or hydroxide.

2.5 History of lithium ore

Lithium was first discovered in 1817 by the Swedish chemist Johan August Arfvedson while analyzing the mineral petalite ($LiAl(Si_2O_5)_2$). Arfvedson identified a new element in the mineral, which he named **l**ithium, derived from the Greek word *lithos*, meaning "stone," because it was discovered in a mineral rather than in plant material, unlike other alkali metals such as sodium and potassium.

During the 19th century, lithium compounds were initially used in ceramics, glass production, and as a flux in metal smelting. The first commercial extraction of lithium occurred in the mid-1800s from spodumene, a lithium-rich mineral found in Sweden and later in the United States, particularly in North Carolina. In the early 20th century, lithium carbonate began to be used for medicinal purposes, notably in the treatment of gout and psychiatric conditions.

The demand for lithium increased significantly in the latter half of the 20th century with the development of lithium-ion batteries, which became critical for portable electronics and, later, electric vehicles. Countries with significant lithium ore deposits, such as Australia, Chile, Argentina, and China, became major producers. More recently, exploration in Africa, including Nigeria, has focused on identifying and developing lithium-bearing minerals to support the growing demand for renewable energy storage solutions.

Today, lithium ore is not only valued for industrial and pharmaceutical applications but also as a strategic mineral crucial for global energy transition, particularly in the production of lithium-ion batteries for renewable energy storage systems.

2.6 Experiment I Method on Laboratory-Scale Protocol for the Design and Construction of a Furnace and Process Flowsheet for Nigerian Lithium Ores

The processing of Nigerian lithium ores, particularly spodumene- and lepidolite-bearing pegmatites, requires a systematic flowsheet that integrates beneficiation, roasting, and leaching stages. This is necessary because the natural α-spodumene polymorph is highly refractory to direct leaching and therefore demands thermal or chemical activation prior to hydrometallurgical treatment (Li et al., 2023; ACS Sustainable Chemistry & Engineering, 2022). Nigerian pegmatites have been shown to contain varying grades of spodumene, petalite, and lepidolite, often associated with quartz, feldspar, and mica gangue minerals (Olade, 2024). Understanding this mineralogy is essential, since it informs the choice of beneficiation strategy and roasting pathway.

Beneficiation of Nigerian lithium ores is aimed at concentrating lithium-bearing minerals while reducing gangue to lower the energy and reagent demands of subsequent steps. Crushing and controlled grinding are first applied to liberate spodumene or lepidolite from quartz and feldspar, followed by dense medium separation for coarse fractions and froth flotation for finer fractions (Opoku, 2025; Tadesse, 2019). Liberation analysis by XRD or SEM is typically conducted to determine the optimal particle size for flotation. Froth flotation, in particular, is considered the most effective method for upgrading lithium concentrates when paired with reagent optimisation, while desliming enhances flotation selectivity and reduces downstream acid consumption (Li et al., 2023).

The roasting stage is central to converting α-spodumene into the more reactive β-spodumene phase, which is amenable to acid or alkaline leaching. Conventional thermal phase transformation occurs at 1000–1100°C in a tube or muffle furnace, with holding times of one to three hours to ensure complete conversion (MDPI, 2020). Alternative roasting methods, such as sodium carbonate or sodium hydroxide salt roasting at 700–900°C, have been reported to lower energy requirements and improve lithium extraction yields (Gu et al., 2025). Sulphuric acid roasting, performed at 200–400°C, offers another pathway in which lithium sulphates are formed directly and later dissolved in water; however, this method requires careful off-gas scrubbing and corrosion-resistant infrastructure due to the release of hazardous vapours (ACS Sustainable Chemistry & Engineering, 2022).

Laboratory-scale furnaces for these processes are commonly designed as horizontal tube furnaces or muffle furnaces with programmable temperature control and the ability to maintain atmospheres ranging from air to inert gases. Tube furnaces are particularly suited to small-scale roasting studies because they provide uniform temperature profiles and can be equipped with gas inlets for controlled atmospheres (Harper International, 2021). Alumina crucibles or sample boats are preferred because of their resistance to high temperatures and corrosive conditions. When acid roasting is employed, corrosion-resistant vessels and off-gas scrubbing units are indispensable for safe operation (Lab & Furnace/SH Scientific, n.d.).

Following roasting, hydrometallurgical leaching is used to dissolve lithium. For thermally activated β-spodumene, sulphuric acid leaching at 60–90°C with controlled liquid-to-solid ratios is common, producing lithium-rich sulphate solutions (Li et al., 2023). Alkaline leaching after alkali roasting routes often achieves high recoveries with reduced co-dissolution of impurities. Process optimisation involves adjusting leach time, temperature, and reagent concentration to balance lithium recovery with impurity control (Gu et al., 2025). Solid-liquid separation is achieved through vacuum filtration or centrifugation, with careful washing to minimise lithium loss. The pregnant leach solution is purified by pH adjustment to remove aluminium and iron impurities before lithium carbonate or hydroxide is recovered by precipitation (ACS Sustainable Chemistry & Engineering, 2022).

A conceptual laboratory flowsheet therefore begins with representative sampling, comminution, and mineralogical characterisation, followed by beneficiation through dense medium separation and flotation. Concentrates are roasted either thermally or chemically, then leached under optimised hydrometallurgical conditions. Lithium is recovered from the purified solution by carbonate precipitation, while wastes are treated through neutralisation and safe disposal. Each stage requires analytical monitoring, including XRD for phase changes, ICP-OES for lithium concentrations, and SEM for microstructural verification (Opoku, 2025).

Environmental and safety considerations must guide the design of the furnace and leaching systems. High-temperature processes consume substantial energy, while acid roasting generates harmful gases that require scrubbing systems. Wastewater treatment and reagent recycling are necessary to reduce the environmental footprint (MDPI, 2020). Bench-scale experiments are therefore recommended to begin with small (50–200 g) batches in tube furnaces under controlled conditions, gradually scaling to rotary tube furnaces for continuous trials (Harper International, 2021).

In conclusion, a laboratory-scale furnace designed with programmable temperature control, atmosphere flexibility, and corrosion resistance provides an appropriate platform for Nigerian lithium ore processing research. A complete process flowsheet integrating beneficiation, roasting, and leaching is both feasible and necessary to evaluate ore quality and optimise lithium recovery. Nigerian pegmatite deposits present a promising resource for battery material development, and laboratory-scale test work can inform the development of environmentally responsible and economically viable extraction technologies (Olade, 2024; Gu et al., 2025).

2.7 Empirical Studies on the Design and Construction of a Laboratory-Scale Furnace with a Complete Process Flowsheet for Beneficiation, Roasting, and Leaching of Nigerian Lithium Ores

The empirical study of laboratory-scale furnace design and process flowsheets for lithium ore processing is grounded in metallurgical experimentation that integrates beneficiation, thermal treatment, and hydrometallurgical recovery. Previous investigations have shown that the construction of small-scale furnaces enables controlled evaluation of roasting conditions, which are essential for transforming α-spodumene into leachable β-spodumene. In studies of pegmatite-derived spodumene concentrates, tube and muffle furnaces equipped with programmable temperature controls have been used to simulate industrial roasting, providing data on temperature-time transformations, phase stability, and energy consumption (MDPI, 2020). These empirical trials demonstrated that $\alpha \rightarrow \beta$ conversion occurs reliably between 1000 and 1100°C, producing materials amenable to subsequent sulphuric acid leaching, with recoveries above 90% under optimised conditions.

Beneficiation studies carried out on lithium pegmatites in Africa and Asia provide empirical evidence on liberation and upgrading strategies. Opoku (2025), for example, reported that dense medium separation and froth flotation applied to spodumene ores achieved concentrates exceeding 5.5% Li_2O when particle sizes were optimised. Similar findings in other contexts confirm that laboratory-scale flotation tests are critical precursors to furnace-based roasting experiments, as gangue reduction directly lowers reagent consumption in both roasting and leaching stages (Li et al., 2023). Nigerian pegmatites, though under-studied, have shown comparable mineral associations, suggesting that beneficiation results from analogous ores can guide furnace-based flowsheet design (Olade, 2024).

In terms of roasting modifications, empirical research has expanded beyond conventional thermal transformation to include alkali- and salt-assisted roasting. Gu et al. (2025) demonstrated through bench-scale rotary furnace tests that sodium carbonate roasting at 700–900°C reduced energy demand and enhanced lithium extraction efficiencies compared to pure thermal activation. Laboratory data revealed that salt roasting not only promotes phase breakdown but also facilitates the formation of soluble lithium compounds, leading to higher leaching yields. These empirical findings support the integration of salt roasting modules into laboratory furnaces designed for Nigerian ores, where energy and reagent costs are critical variables.

Hydrometallurgical leaching has also been validated through empirical work. Acid leaching of β-spodumene roasted in laboratory furnaces typically achieves lithium recoveries between 85 and 95% under conditions of 3–5 M H_2SO_4, 80–90°C, and 1–2 h reaction times (ACS Sustainable Chemistry & Engineering, 2022). In contrast, water leaching of alkali-roasted concentrates achieves comparable recoveries while reducing impurity dissolution, as demonstrated in multiple laboratory studies. These empirical results underscore the importance of furnace design features such as temperature uniformity, gas control, and corrosion resistance, which influence both roasting success and downstream leachability.

For Nigerian ores, empirical pilot studies remain limited, but initial beneficiation trials indicate that local pegmatites contain spodumene and lepidolite grades suitable for bench-scale processing (Olade, 2024). Laboratory validation of beneficiation, roasting, and leaching using constructed tube furnaces is therefore a necessary step toward developing a country-specific flowsheet. International empirical evidence provides transferable knowledge: furnace modules tested on Australian, Chinese, and Ethiopian ores highlight the key role of small-scale thermal reactors in flowsheet validation and scale-up to pilot operations (Tadesse, 2019).

In summary, empirical studies collectively demonstrate that laboratory-scale furnaces, when integrated into beneficiation-roasting-leaching flowsheets, provide a reliable platform for evaluating lithium ore processing. While most experimental evidence comes from non-Nigerian deposits, the mineralogical similarity of Nigerian pegmatites suggests strong applicability. Laboratory construction of programmable tube or rotary furnaces, supported by beneficiation and hydrometallurgical test work, offers the most practical empirical pathway for developing lithium precursor materials locally.

2.8 Experiment II: Characterization of the Mineralogy, Chemical Composition, Phase Transformations, and Lithium Recovery Efficiency of Nigerian Ores

The characterisation of Nigerian lithium ores is a critical step in developing sustainable beneficiation and extraction strategies, particularly for battery-grade precursors. Nigerian pegmatite-hosted lithium deposits, distributed in states such as Nasarawa, Kogi, and Kwara, are dominated by spodumene, petalite, and lepidolite, with gangue minerals including quartz, feldspar, and micas (Olade, 2024). Proper characterisation is necessary to understand mineral liberation, phase transformation behaviour, and leaching responses, which directly affect lithium recovery efficiency.

Mineralogical identification and quantification are primarily carried out through X-ray diffraction (XRD). XRD enables the detection of crystalline phases, including the refractory α-spodumene and its high-temperature β-polymorph, as well as petalite and lepidolite (MDPI, 2020). In Nigerian ores, XRD studies help establish the mineralogical assemblage and track the transformation of α-spodumene to β-spodumene during roasting between 1000 and 1100°C, a prerequisite for efficient acid leaching (Li et al., 2023). This phase monitoring is essential because incomplete conversion leaves unreactive α-spodumene, lowering lithium recovery.

The bulk chemical composition of Nigerian lithium ores is typically assessed using X-ray fluorescence (XRF), which provides major and trace element oxides. Nigerian pegmatites often show Li_2O contents ranging from 1–4 wt.% in raw ores, along with significant levels of SiO_2, Al_2O_3, Na_2O, and K_2O (Olade, 2024). XRF analyses are valuable for establishing baseline chemistry, identifying impurities (such as Fe_2O_3 and TiO_2), and quantifying gangue components that influence roasting and leaching efficiency.

For microstructural and elemental mapping, scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) provides crucial insights. SEM allows the visualisation of grain boundaries, particle morphology, and mineral liberation at different grind sizes, while EDS detects elemental associations within mineral grains. In spodumene-rich Nigerian ores, SEM-EDS can reveal lithium distribution within aluminosilicate lattices and identify accessory phases such as tantalum- or niobium-bearing minerals, which can influence beneficiation performance (Opoku, 2025). These microanalytical studies inform both grinding strategies for mineral liberation and the expected impurity profile during leaching.

Phase transformation studies combine XRD, SEM, and thermal treatment experiments to evaluate roasting efficiency. Literature reports have shown that the α→β transition in spodumene alters crystal structure from monoclinic to tetragonal, enhancing reactivity in subsequent leaching

(MDPI, 2020). Nigerian ores require similar laboratory-scale furnace experiments, supported by mineralogical monitoring, to optimise roasting temperatures and holding times. For lepidolite-rich ores, alkali or salt-assisted roasting may be necessary to achieve favourable phase transformations, as demonstrated in empirical studies elsewhere (Gu et al., 2025).

Finally, lithium recovery efficiency is determined through hydrometallurgical leaching experiments. Sulphuric acid leaching is most commonly employed for β-spodumene, achieving recoveries of 85–95% under optimised conditions (Li et al., 2023). In contrast, alkali-roasted ores may be subjected to water or alkaline leaching, producing lithium recoveries above 90% with lower impurity dissolution (ACS Sustainable Chemistry & Engineering, 2022). For Nigerian ores, systematic leaching tests are required to evaluate reagent concentration, temperature, and liquid-to-solid ratios, with efficiency assessed via ICP-OES or AAS analysis of pregnant leach solutions. Such studies also allow benchmarking of Nigerian ore performance against global counterparts.

In conceptual terms, the integration of XRD, XRF, SEM-EDS, and leaching experiments provides a comprehensive characterisation workflow. XRD reveals mineralogy and phase transitions; XRF establishes bulk chemistry; SEM-EDS details microstructural and elemental associations; and leaching experiments quantify lithium recovery efficiency. Together, these methods generate a detailed understanding of Nigerian lithium ores, guiding beneficiation design, furnace roasting protocols, and hydrometallurgical optimisation. As Nigeria advances toward lithium-based energy storage industries, such multi-method characterisation will be indispensable for validating ore potential and scaling extraction processes sustainably.

2.9 Empirical studies on characterization of mineralogy, chemistry, phase transformation and lithium recovery (XRD, XRF, SEM-EDS, leaching)

Empirical research on lithium-bearing pegmatites consistently demonstrates that a multi-technique approach combining X-ray diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS), and controlled leaching tests is essential to understand ore behaviour and predict laboratory and pilot-scale extraction performance (Li et al., 2023; MDPI, 2020). In these studies the methods are used in complementary roles:XRD identifies crystalline phases and tracks phase transformations, XRF quantifies bulk major/trace chemistry, SEM-EDS documents textural relationships and liberation at the grain scale, and leaching tests quantify practical lithium extraction under controlled reagent/temperature regimes (Opoku, 2025; ACS Sustainable Chem. Eng., 2022).

XRD has been used empirically both as a baseline mineral identification tool and as the primary method to follow the thermal phase transformation of spodumene. Bench studies typically prepare powdered samples (<45 μm) and collect patterns over 2θ ranges that include characteristic spodumene peaks (often 5–70° 2θ using Cu Kα radiation). Researchers report using Rietveld or semi-quantitative peak-area methods to determine relative phase proportions and to detect the α→β conversion after roasting (MDPI, 2020; Li et al., 2023). Empirical datasets show that incomplete α→β conversion detected by residual α-spodumene peaks correlates strongly with poor acid leach extraction, providing an actionable diagnostic for roast optimisation (MDPI, 2020). Nigerian pegmatite case reports that include XRD (Olade, 2024) employ similar protocols to confirm the presence of spodumene, lepidolite and common gangue phases (quartz, K-feldspar, muscovite) and suggest that phase identification is the first filter in choosing thermal vs. chemical pre-treatments.

Bulk chemistry by XRF is a standard empirical step to quantify Li_2O (when laboratories have appropriate standards or use calibrated ICP for Li), major oxides (SiO_2, Al_2O_3, Fe_2O_3, etc.) and deleterious elements (Ti, P, heavy metals). Empirical studies commonly prepare fused glass beads or pressed pellets to minimise matrix effects and run international reference materials for QA/QC (Opoku, 2025). In practice, researchers use XRF results to estimate gangue proportions that control reagent consumption in leaching and to flag high-iron or high-aluminium ores that may require additional purification steps (Li et al., 2023). Nigerian ore studies referenced in the literature use XRF to establish head grades and to track mass-balance changes across beneficiation and roast/leach tests (Olade, 2024).

SEM-EDS is used empirically to resolve grain morphology, intergrowths, surface weathering, and liberation at the micrometre scale. Typical studies mount polished sections or grain mounts, image with backscattered electron (BSE) contrast to reveal compositional contrast, and perform point analyses or area maps to quantify elemental associations (Li, 2023; Opoku, 2025). Empirical outputs from SEM-EDS often reveal important processing constraints for example, spodumene intimately intergrown with albite or mica requires finer grinding for effective liberation, whereas coarser liberation enables efficient dense medium separation (DMS). Several bench studies on hard-rock lithium ores report that SEM-EDS mapping of accessory Ta-Nb phases and iron minerals explains flotation depressants' performance and downstream impurity behaviour in leaching (Tadesse, 2019; Gu et al., 2025).

Leaching experiments in the empirical literature are designed to quantify lithium recovery efficiency as a function of pre-treatment, reagent, temperature and residence time. Bench protocols typically use split concentrates or roasted products in batch reactors with controlled L/S ratios, temperature control (ambient to 95°C), and stirring. For thermally activated β-spodumene, sulphuric acid leaching at elevated temperature (e.g., 1–3 M H_2SO_4, 60–90°C) for 1–4 hours is commonly tested; reported recovery ranges are typically 70–95%, depending on roast quality and particle size (ACS Sustainable Chem. Eng., 2022; MDPI, 2020). Empirical work on alkali or salt roasting followed by water leaching shows comparable or improved recoveries at lower roast temperatures (700–900°C) and often reduces co-dissolution of Al and Fe (Gu et al., 2025). Studies routinely analyse leachates by ICP-OES or ICP-MS to measure Li and impurity concentrations; mass balance calculations then quantify stagewise recoveries and reagent consumptions (Li et al., 2023).

Importantly, empirical studies combine the above techniques in iterative loops:XRD and SEM-EDS verify whether a roast has produced the desired phase transformation and microstructural accessibility, XRF and ICP quantify how much lithium is actually liberated into solution, and mass-balance/energy metrics are used to determine process feasibility. In Nigerian-focused studies, preliminary empirical characterisation (Olade, 2024) confirms spodumene and lepidolite occurrences and indicates that comparable roast-leach strategies used internationally are applicable provided ash chemistry and liberation characteristics are carefully accounted for. Cross-study comparisons highlight recurring empirical findings: (a) complete α→β conversion is necessary but not sufficient; adequate liberation is equally crucial; (b) salt/alkali roasting can reduce energy costs while maintaining high recoveries if followed by proper water or alkaline leaching; and (c) the integration of XRD, XRF, SEM-EDS and targeted leaching experiments yields the most reliable prediction of recovery performance and informs furnace design (MDPI, 2020; Gu et al., 2025; Li et al., 2023).

In summary, the empirical literature shows that a characterisation programme for Nigerian lithium ores must include standardised XRD scans (with Rietveld/quantification), calibrated XRF bulk chemistry, SEM-EDS mapping of liberation and associations, and systematic roast-leach experiments with ICP-based analysis of leachates. Together these methods produce a robust dataset that links mineralogy and microstructure to leach response and recovery efficiency, thereby guiding lab-scale furnace design and the selection of roast-leach pathways for Nigerian pegmatites.

2.10 Experiment III: Evaluating Energy Requirements and Environmental Impacts of Lithium Ore Processing Routes through LCA

Life cycle assessment (LCA) has become a critical framework for evaluating the environmental sustainability of mineral processing routes, including those for lithium extraction. The methodology follows ISO 14040/44 standards and typically assesses the entire chain from mining, beneficiation, thermal treatment (e.g., roasting), chemical conversion (e.g., leaching), and purification to precursor or carbonate production (ISO, 2006). In the context of Nigerian lithium ores, three main processing routes acid roasting followed by leaching, alkaline/salt roasting with water leaching, and direct leaching of beneficiated concentrates can be comparatively assessed for energy demands and environmental footprints.

Empirical and modelling studies have shown that acid roasting–sulphuric acid leaching routes generally achieve high lithium recoveries (>90%) but are associated with high thermal energy consumption (800–1100°C) and significant acid consumption, both of which increase greenhouse gas (GHG) emissions and environmental burdens (Peters et al., 2017; Nuñez et al., 2022). In contrast, alkali/salt roasting with water leaching reduces the use of strong acids and often operates at lower roasting temperatures (700–900°C). LCA studies report lower acidification and eutrophication impacts but note increased waste generation from sodium or potassium salts, which must be managed to prevent secondary environmental risks (Greim et al., 2020). Direct leaching approaches, particularly those involving mechanical activation or pressure leaching, avoid high-temperature roasting, thereby reducing thermal energy inputs. However, these methods often require higher acid strengths, longer residence times, and more intensive water use, which may shift environmental burdens toward water scarcity and chemical toxicity categories (Yaksic & Tilton, 2009; Nuss & Eckelman, 2014).

Comparative LCA studies also emphasise the role of the electricity source in determining environmental outcomes. For example, processing routes powered by fossil-based grids yield substantially higher CO_2-equivalent emissions than those integrated with renewable energy sources (Peters et al., 2017). Furthermore, waste management strategies such as recycling of sulphuric acid, regeneration of salts, or utilisation of leach residues are shown to significantly alter the impact profile of each route (Nuñez et al., 2022). Recent literature highlights that while acid roasting remains the benchmark for recovery efficiency, alternative routes may provide better long-term sustainability when environmental and energy indicators are fully considered (Greim et al., 2020).

In the Nigerian context, where electricity generation is dominated by fossil fuels and chemical import costs are high, LCA can provide actionable insights into selecting a route that balances lithium recovery efficiency with reduced environmental impact. Conceptually, adopting LCA allows policymakers and researchers to quantify trade-offs across energy consumption, greenhouse gas emissions, acidification potential, water usage, and waste generation. This positions LCA not only as a diagnostic tool but also as a decision-making framework for guiding the design of laboratory-scale experiments and scaling up to industrial processing of Nigerian lithium ores.

2.11 Experiment IV: Industrial and Economic Feasibility of Localized Lithium Ore Processing in Nigeria

The growing global demand for lithium-ion batteries has driven interest in developing localized processing technologies in resource-rich developing countries. In Nigeria, pegmatite-hosted lithium deposits present an opportunity for import substitution and industrial diversification. However, evaluating the industrial and economic feasibility of local processing requires integrating insights from resource availability, infrastructure readiness, cost–benefit trade-offs, and global market dynamics.

Industrial feasibility studies emphasize infrastructure, technology readiness, and integration into existing value chains. Literature on mineral-based industrialization in Africa shows that localized processing often faces challenges such as inadequate energy supply, limited access to high-temperature industrial furnaces, reagent supply chains, and underdeveloped technical expertise (Nkwabi & Moyo, 2021; UNECA, 2020). For lithium, technologies under consideration include beneficiation of spodumene concentrates, thermal conversion (roasting), hydrometallurgical leaching, and precipitation of lithium carbonate or hydroxide. International experience shows that establishing such plants requires reliable electricity and chemical supply, skilled labor, and proximity to mining sites to minimize transport costs (Peters et al., 2017). Studies in Zimbabwe and Namibia highlight that modular or semi-mobile plants can serve as intermediate steps toward full-scale industrial integration (Kamunda et al., 2022).

Economic feasibility is typically assessed through techno-economic analysis (TEA), which evaluates capital expenditure (CAPEX), operating expenditure (OPEX), and potential revenues from lithium carbonate equivalent (LCE) sales (Greim et al., 2020). Literature indicates that roasting and hydrometallurgical plants are energy-intensive, with energy costs accounting for 20–40% of OPEX in hard rock lithium processing (Nuñez et al., 2022). Sensitivity analyses consistently show that profitability depends strongly on global lithium price volatility, energy costs, and recovery efficiency (Nuss & Eckelman, 2014). For Nigeria, additional factors such as port logistics, exchange rate fluctuations, and policy frameworks (tax incentives, mineral beneficiation mandates) significantly influence economic outcomes.

Studies on argue that domestic processing is economically justified when: (i) ore grades are sufficiently high, (ii) there is potential for clustering of industries (chemicals, metallurgy, energy), and (iii) governments provide enabling policies such as subsidies, public–private partnerships, and infrastructure investments (UNECA, 2020; Ayres et al., 2023). Conversely, without such conditions, raw ore export often remains the dominant strategy. However, long-term strategic benefits including job creation, knowledge transfer, and reduced import dependency are increasingly highlighted in literature as essential for justifying localized processing despite short-term economic challenges (Kamunda et al., 2022).

In the Nigerian context, feasibility analysis should therefore consider: (a) ore quality and beneficiation yields; (b) cost of establishing laboratory-to-pilot scale processing facilities; (c) energy availability and costs given fossil-dominated grids; (d) chemical supply chains; and (e) international lithium market trends. Conceptually, the literature supports a staged approach: beginning with pilot beneficiation and roasting facilities, scaling up to hydrometallurgical production, and eventually integrating into regional battery precursor supply chains. Such an approach balances immediate financial risks with long-term industrial and strategic gains.

2.12 Conceptual Framework: Design and Processing of Nigerian Lithium Ore for $LiFePO_4$ Batteries in Renewable Energy Storage

Experiment	Core Concept	Key Elements	Details / Indicators	Instruments / Equipment / Tools	Relevant Standards / References
I	Design & Construction of Lab-Scale Furnace	Furnace design, Heating system, Temp. control, Structural design, Process flow integration, Ventilation, Instrumentation, Safety, Testing	Furnace type (muffle/rotary/tube), batch/continuous, thermal insulation, accurate heating, load-bearing frame, workflow integration, gas exhaust, monitoring, fire protection, optimization	CAD software, refractory materials, thermocouples, PID controllers, steel frame, fume hood, data logger, PPE, test samples	ASTM C27, ISO 9001, IEC 60519, ASTM A36/A992, ISO 14159, OSHA 29 CFR 1910.1450, NFPA 70, ASTM E381
II	Characterisation of Nigerian Lithium Ores	Mineralogy, Chemical composition, Phase transformations, Lithium recovery efficiency, Supporting analysis, Data integration	Crystalline phases, oxides & trace elements, α→β-spodumene, leaching efficiency, particle size & surface area, correlation of properties	XRD, SEM-EDS, Optical microscope, XRF, ICP-MS/OES, AAS, DSC, TGA, HT-XRD, BET analyser, leaching reactors, statistical software	ASTM D2013, ASTM C1365, ASTM E1508, ASTM C114, ASTM D1971, ASTM E1131, ASTM E793,
III	Evaluate Energy & Environmental Impacts (LCA)	LCA framework, Processing routes (spodumene, lepidolite, brine), Energy requirements, Environmental impacts, QC & monitoring, Data & uncertainty	Cradle-to-gate or cradle-to-grave, functional unit, roasting/leaching/evaporation steps, kWh/fuel use, emissions, effluent quality, reproducibility, hotspot identification	LCA software (SimaPro/OpenLCA/GaBi), furnaces, mills, flotation cells, leaching reactors, filtration units, energy meters, gas analysers, XRD/XRF/ICP-MS, pH & conductivity meters, statistical software	ISO 14040/44, ASTM methods, local environmental discharge limits
IV	Industrial & Economic Feasibility Analysis	Industrial feasibility, Economic feasibility, Policy & regulatory assessment, Integration & reporting	Ore reserve assessment, local processing tech, energy/resource requirements, environmental & safety, CAPEX/OPEX, market analysis, ROI, sensitivity/risk, policy compliance, decision support	Geological surveys, XRF/XRD, pilot plants, process simulation software, energy meters, financial models, market reports, MCDA tools	ASTM D420, ISO 9001, ASTM E975, ISO 50001, ISO 14001, ASTM E2338, AACE

CHAPTER THREE

MATERIALS AND METHODS

3.1 Introduction

This chapter described the materials and methods used in this study, these include: the research design, which explained the design and rationale behind its selection, area of the study sample sourcing, instrument and equipment to be used and the experiment to be used to complement the research objectives with justification.

3.2 Research Design

The philosophical orientation of this study is grounded in a pragmatic paradigm, which integrates positivist, interpretivist, and transformative elements to address both scientific and societal questions around lithium ore processing.

At the positivist level**,** the work assumes that the properties of Nigerian lithium ores can be objectively measured and analyzed using established scientific methods. Experimental design, standardised instruments (XRD, SEM-EDS, DSC, ICP-MS), and compliance with ASTM/ISO standards reflect a belief in empirical observation, measurement, and reproducibility as the pathway to valid knowledge. This aligns with the natural sciences' tradition of seeking universal, testable truths through controlled experiments. At the interpretivist level, the research recognizes that processing routes are not only technical phenomena but also embedded in socio-economic and environmental contexts. The use of life cycle assessment (LCA) and techno-economic feasibility studies reflects an interpretive effort to understand how different stakeholders (industry, policymakers, communities) might perceive and evaluate energy consumption, environmental impacts, and market feasibility. Here, the philosophy acknowledges that meaning is shaped by context, interpretation, and policy frameworks.

At the transformative and pragmatic level**,** the study adopts a problem-solving stance: it is not research for knowledge's sake alone but directed at developing sustainable and localized processing technologies for Nigeria. This trend reflects pragmatism, where the value of knowledge is judged by its capacity to generate solutions, reduce environmental harm, create industrial value, and support renewable energy transitions. The integration of safety standards (OSHA, NFPA), environmental regulations (ISO 14040/44), and economic assessment frameworks reflects a sustainability-oriented pragmatism**,** ensuring that scientific findings translate into actionable outcomes.

In summary, the philosophical trend of this work can be described as scientific realism under a pragmatic paradigm: it combines positivist rigor for material characterization, interpretivist awareness for socio-environmental assessment, and a transformative orientation towards sustainable energy development.

3.3 Method of Material Sourcing

The materials required for this research will be sourced systematically in line with the four core experimental components: **(I) Design and Construction of a Laboratory-Scale Furnace, (II) Characterisation of Nigerian Lithium Ores, (III) Evaluation of Energy and Environmental Impacts through Life Cycle Assessment, and (IV) Industrial and Economic Feasibility Analysis**.

Sourcing of Ore Samples

Lithium-bearing ore samples will be collected from selected Nigerian pegmatite deposits located in states such as Nasarawa, Kogi, and Plateau, which have been reported to host spodumene- and lepidolite-rich pegmatites. Sampling will follow ASTM D2013 protocols to ensure representativeness and homogeneity. Samples will be obtained in collaboration with local mining operators and the Nigerian Geological Survey Agency (NGSA). The ores will be transported in sealed, contamination-free containers to the laboratory, where they will be crushed, milled, and homogenised before testing.

Procurement of Furnace Construction Materials

Refractory bricks (high-alumina, insulating firebricks) and structural steel (ASTM A36/A992) will be sourced from certified local and international suppliers. Temperature control devices, including thermocouples, PID controllers, and programmable data loggers, will be procured from laboratory equipment distributors. Ventilation systems (fume hoods, exhaust fans) and fire safety installations will be obtained in compliance with NFPA 70 and OSHA 29 CFR 1910.1450. Procurement will prioritise locally available materials where feasible, to enhance cost-effectiveness and support local industries.

Acquisition of Characterisation Instruments

Specialised characterisation instruments such as X-ray diffraction (XRD), scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS), optical microscopy, and X-ray fluorescence (XRF) will be accessed through research collaborations with established Nigerian universities and research centres. Advanced chemical analysis facilities (ICP-MS, AAS, DSC, TGA, HT-XRD, BET analyser) will be sourced through partnerships with regional laboratories and, where necessary, outsourced to internationally accredited facilities. Access agreements will be formalised to ensure timely and cost-efficient utilisation.

Energy and Environmental Monitoring Tools

Energy meters, gas analysers, and effluent monitoring devices (pH and conductivity meters) will be procured from laboratory equipment suppliers. LCA software packages (SimaPro, OpenLCA, GaBi) will be sourced under institutional academic licenses, while statistical software (SPSS, R, MATLAB) will be acquired under existing research agreements. Data from pilot-scale experiments will be integrated into LCA modelling in compliance with ISO 14040/44 standards.

Economic and Industrial Data Sources

Industrial and economic feasibility analysis will draw on multiple sources of data. Geological reserve information will be obtained from the NGSA and published academic reports. Market data (lithium carbonate/hydroxide prices, demand forecasts, CAPEX/OPEX benchmarks) will be sourced from international mining and energy reports, trade publications, and financial databases. Process simulation software (Aspen Plus, COMSOL) and multi-criteria decision analysis (MCDA) tools will be acquired under institutional research licenses.

3.4 Materials to be Used

In this research, the materials, equipment, and standards required are organized in line with the four core experimental components: **(I) Design and Construction of a Laboratory-Scale Furnace, (II) Characterisation of Nigerian Lithium Ores, (III) Evaluation of Energy and Environmental Impacts through Life Cycle Assessment, and (IV) Industrial and Economic Feasibility Analysis**.

Experiment I (Design and Construction of a Laboratory-Scale Furnace), the study will employ refractory materials such as high-alumina bricks and insulating firebricks to ensure durability and heat retention, together with a structural steel frame fabricated in accordance with ASTM A36/A992 standards. Temperature control and heating precision will be enabled by thermocouples, PID controllers, and programmable data loggers, while process visualization and structural integration will be supported through CAD software. A fume hood and gas exhaust ventilation system will be incorporated to manage gaseous emissions, while fire protection and personnel safety will be ensured using personal protective equipment (PPE) and compliance with NFPA 70 and OSHA 29 CFR 1910.1450.

Experiment II (Characterisation of Nigerian Lithium Ores), materials will include representative ore samples collected from Nigerian pegmatite deposits, prepared in accordance with ASTM D2013. Characterisation will utilize a range of instruments: X-ray diffraction (XRD) for crystalline phase identification, scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) for microstructural and elemental analysis, and optical microscopy for petrographic observations. Complementary chemical analysis will be conducted using X-ray fluorescence (XRF), inductively coupled plasma mass spectrometry (ICP-MS/OES), and atomic absorption spectroscopy (AAS), aligned with ASTM C114 and ASTM D1971. Thermal behaviour, including $\alpha \rightarrow \beta$ spodumene transformation, will be investigated using differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and high-temperature XRD (HT-XRD) under ASTM E1131 and ASTM E793 protocols. Surface area and porosity of milled ores will be determined via Brunauer–Emmett–Teller (BET) analysis, while leaching reactors will support lithium recovery testing.

Experiment III (Evaluation of Energy and Environmental Impacts through Life Cycle Assessment), materials include SimaPro, OpenLCA, or GaBi software packages to model life cycle impacts under ISO 14040/44 guidelines. Experimental data will be sourced from furnaces, comminution mills, flotation cells, leaching reactors, and filtration units to capture energy consumption, reagent use, and waste generation. Energy meters, gas analysers, and effluent monitoring devices (pH and conductivity meters) will provide quantitative measures of inputs and outputs, while XRD/XRF/ICP-MS analyses will validate compositional data. Statistical software will be used to handle data uncertainty and ensure reproducibility.

Experiment IV (Industrial and Economic Feasibility Analysis), materials and tools will include geological survey data and reserve assessments for Nigerian lithium deposits, processed through XRD and XRF techniques for ore quality validation. Pilot plant facilities will serve to scale laboratory findings to semi-industrial levels, while process simulation software will model energy and resource balances in compliance with ISO 50001 and ISO 14001. Financial models, capital and operating cost estimates (CAPEX/OPEX), and market reports will provide the economic basis for feasibility studies, supported by ASTM E2338 and AACE guidelines. Multi-criteria decision analysis (MCDA) tools will further assist in integrating technical, economic, and policy parameters into a decision support framework.

3.5 Methods to be used

3.5.1 Experiment I Methods on design and construct a laboratory-scale furnace with a complete process flowsheet for beneficiation, roasting, and leaching of Nigerian lithium ores

Core Concept	Key Elements	Details / Indicators	Instruments / Equipment	Relevant Standards
Furnace Design	Type Selection, Capacity, Material	Decide furnace type (muffle, rotary, tube), batch vs. continuous, refractory material, thermal insulation	CAD software, Refractory materials, Thermal insulation	ASTM C27 (Refractories), ISO 9001 (Quality management)
Heating System	Power Source, Temperature Range	Electric, gas, or induction; max operating temperature; uniform heating	Heating elements, Power supply, Thermocouples, PID controller	IEC 60519 (Safety in furnaces)
Temperature Control	Sensors & Feedback, Safety	Accurate measurement & regulation; over-temperature protection	Thermocouples, Temperature controller, Safety cut-off switches	ISO 13709 / IEC 60519-1
Structural Design	Frame, Supports, Housing	Load-bearing frame; furnace chamber; insulation; access ports	Steel frame, Refractory bricks, Hinged doors, Latches	ASTM A36/A992 (Steel structures)

Process Flow Integration	Material Handling, Workflow	Incorporate ore preparation, beneficiation, roasting, and leaching steps	Feed trays, Conveyors, Crucibles, Stirring systems	ISO 14159 (Ergonomics and safety of machinery)
Ventilation & Exhaust	Fume Extraction, Safety	Proper exhaust for gases; maintain lab safety	Fume hood, Exhaust fan, Ducting	OSHA 29 CFR 1910.1450 (Laboratory safety), ISO 14175 (Industrial gases ventilation)
Instrumentation & Monitoring	Measurement & Control	Track temperature, process duration, and energy consumption	Data logger, Thermocouples, Multimeter	IEC 61010 (Safety of lab equipment), ASTM E230 (Thermocouples)
Safety Measures	Fire Protection, PPE	Protect operators and equipment	Fire extinguisher, PPE (gloves, goggles, lab coat), Emergency shutdown	NFPA 70 (Electrical safety), OSHA 1910.157 (Fire protection)
Testing & Optimization	Trial Runs, Parameter Adjustment	Test furnace performance; adjust heating rates, insulation, and loading	Test samples, Data recording sheets, Optimization software	ISO 9001 (Quality management), ASTM E381 (Thermal testing of furnaces)

3.5.2 Experiment II Method Characterisation of the Mineralogy, Chemical Composition, Phase Transformations, and Lithium Recovery Efficiency of Nigerian Ores

Core Concept	Key Elements	Details / Indicators	Instruments / Equipment	Relevant ASTM Standards
Objective	Characterisation of mineralogy, chemical composition, phase transformations, and lithium recovery efficiency	Nigerian ores: spodumene, lepidolite, petalite, amblygonite	Sample crushers, mills, drying oven	ASTM D2013 (coal/ore sample preparation, adapted)

Mineralogy	Crystalline phase ID & microtextures	Mineral phases, crystallinity, ore–gangue association	XRD, SEM-EDS, Optical polarising microscope	ASTM C1365 (XRD for crystalline phases), ASTM E1508 (SEM/EDS)
Chemical Composition	Major, minor, and trace elements	Oxides (SiO_2, Al_2O_3, Fe_2O_3, MgO, etc.), critical metals (Li, Be, Ta, Nb, Rb, Cs)	XRF, ICP-MS, ICP-OES, AAS, EPMA	ASTM C114 (chemical analysis of oxides), ASTM D1971 (ICP for trace elements), ASTM D4326 (XRF analysis of oxides)
Phase Transformations	Thermal stability & roasting transformations	α → β-spodumene conversion, salt roasting intermediates, decomposition	DSC, TGA, HT-XRD, DTA	ASTM E1131 (TGA), ASTM E793 (DSC), ASTM C892 (thermal analysis of clays/minerals)
Lithium Recovery Efficiency	Extractable Li yield & leaching efficiency	% recovery, kinetics, solution chemistry	Leaching reactors, autoclaves, filtration, ICP-MS/ICP-OES (Li analysis), flame photometer	ASTM D2340 (Li in water by flame photometry), ASTM E1915 (chemical analysis of refractory materials – adapted)
Supporting Analysis	Particle size, surface area, morphology	Liberation size, porosity, leaching surface area	Laser diffraction particle size analyser, BET analyser, crushing/grinding mills	ASTM B822 (particle size by laser diffraction), ASTM D3663 (BET surface area)
Data Processing & Integration	Correlate mineralogy, chemistry & recovery	Relationships between ore properties and processing	Statistical software (R, SPSS, Python), MLA	ASTM E2554 (data reporting for

				analytical methods)
Expected Outcomes	Comprehensive ore profile & recovery pathway	Mineralogical/chemical data → optimised beneficiation & precursor production	Technical reports, flowsheet design	Best practice guided by ASTM + ISO 14000 (if integrated with LCA)

3.5.3 Experiment III on Method of Evaluating Energy Requirements & Environmental Impacts of Lithium Ore Processing Routes (with instruments)

Core concept	Key elements	Details / indicators	Instruments / equipment (recommended)
Objective	Evaluate energy requirements & environmental impacts	Compare chosen lithium ore processing routes using LCA (ISO 14040/44)	Project laptop, lab notebook, data storage, version control (Git)
Framework	Life Cycle Assessment (LCA)	Boundary setting (cradle-to-gate / cradle-to-grave), functional unit, sensitivity/uncertainty analysis	LCA software (SimaPro / OpenLCA / GaBi), Excel / Python (pandas, brightway2)
Processing routes — Spodumene (roasting & leaching)	Roasting, grinding, concentration, leaching, purification	Thermal roast temperature/time, leach kinetics, yields	Rotary kiln / tube furnace / muffle furnace; programmable furnace controllers; ball mill / rod mill; cone crusher; vibrating screens; flotation cells; stirred autoclave / leaching reactors; filtration press; centrifuge; hotplate / reflux setups
Processing routes — Lepidolite (salt roasting & leaching)	Salt roasting, water leaching, solvent handling, brine treatment	Salt chemistry, recovery of Li/Rb/Cs, residues handling	Rotary/tabletop furnace; salt handling hoods; leaching reactors; solvent-compatible stirrers; corrosion-resistant tanks (PTFE-lined); decanter centrifuge; vacuum filtration

Processing routes — Brine / evaporation & chemical processing	Brine concentration, precipitation, solvent extraction, evaporation ponds	Evaporation rates, crystallisation, chemical consumption	Solar/forced evaporation ponds (pilot) or laboratory evaporators; evaporative concentrators; solvent extraction columns; pilot-scale evaporators; crystallisers; pumps and piping; conductivity / salinity meters
Energy requirements (measurement)	Thermal energy, electricity, fuel use per process step	kWh, MJ, fuel mass/volume, specific energy per kg Li	Power meters / energy loggers; clamp meters; data loggers; thermal flow meters; calorimeter (for lab calorimetry); gas flow meters; fuel flow counters; smart submeters for pilot-scale equipment
Environmental impacts (measurement & sampling)	GWP (CO_2-eq), water use, emissions (SOx, NOx, PM), effluent toxicity, solid wastes	Emission rates, effluent concentrations, COD/BOD, heavy metals	Stack gas analyser (NDIR CO_2, O_2), FTIR or portable multi-gas analyser, NOx/SOx detectors; particulate samplers / PM monitors; high-volume air sampler; gravimetric filters; water sampling bottles; autosamplers
Chemical & materials analysis	Feed/mineral characterisation, product purity, trace contaminants	Mineral phases, elemental concentrations (Li, Be, Ta, Nb, Rb, Cs), particle size	X-ray diffraction (XRD); X-ray fluorescence (XRF); ICP-MS / ICP-OES for trace & major elements; SEM-EDS; particle size analyser (laser diffraction); loss-on-ignition furnace; moisture analyser; LOI muffle furnace
Effluent & waste characterisation	Toxicity, leachability, pH, heavy metal content	TCLP or other leach tests, pH, conductivity, COD, total suspended solids	pH meter; conductivity meter; turbidity meter; COD / BOD test kits; TOC analyser; leach test apparatus (TCLP setup); centrifuge; filtration units

Quality control & process monitoring	On-line / lab QC for reproducibility, mass balance	Recovery %, yield, reproducibility, measurement uncertainty	Analytical balances (±0.1 mg); volumetric glassware; automated titrator; spectrophotometer; temperature probes; RTD thermocouples; flow meters; PLC / SCADA for pilot plant monitoring; calibration standards
LCA data & uncertainty analysis	Inventory data collection, databases, sensitivity and Monte Carlo	Emission factors, dataset gaps, scenario analysis	Access to LCA databases (Ecoinvent, USLCI, GREET); statistical software / Python (Monte Carlo libraries); spreadsheet modelling; uncertainty propagation tools
Regulatory / standard tests	Compliance & comparability	ISO, ASTM, local environmental discharge limits	Reference method kits; certified reference materials (CRMs); access to accredited lab (if needed)
Expected outcomes	Hotspot identification, energy-intensive steps, mitigation options	GWP per kg Li, water footprint, recommendation matrix	Report generation tools, figure software (Inkscape / Illustrator), presentation slides

3.5.4 Experiment IV method on analyzing the industrial and economic feasibility of localized lithium ore processing technologies in Nigeria:

Core Concept	Key Elements	Details / Indicators	Instruments / Tools / Techniques	Standards / References
Industrial Feasibility Analysis	1. Assessment of available lithium ore reserves	Evaluate quantity, quality, and accessibility of lithium ores	Geological surveys, XRF, XRD, ore sampling kits	ASTM D420 (Soil & Ore Sampling), ISO 9001
	2. Evaluation of local processing technologies	Analyze existing or potential processes for beneficiation, roasting, and leaching	Process simulation software (e.g., HSC Chemistry, Aspen Plus), pilot plants	ASTM E975 (Process Evaluation)
	3. Energy and resource requirements	Determine energy consumption, water usage, and auxiliary material needs	Energy meters, flow meters, process modeling	ISO 50001 (Energy Management)
	4. Environmental and safety considerations	Identify emissions, effluents, and occupational hazards	Environmental impact assessment tools, gas analyzers	ISO 14001, OSHA standards
	5. Process scalability	Examine scale-up feasibility from lab to industrial scale	Pilot plant testing, scale-up simulation	ASTM E2338 (Scale-up Studies)
Economic Feasibility Analysis	1. Capital expenditure (CAPEX)	Estimate investment required for plant, equipment, and infrastructure	Cost estimation software, market surveys	AACE International standards
	2. Operational expenditure (OPEX)	Estimate recurring costs: energy, labor, maintenance, consumables	Budgeting software, process flow cost analysis	ISO 55000 (Asset Management)
	3. Market analysis	Evaluate demand, supply, and potential revenue of lithium products	Market reports, pricing analysis	OECD, World Bank mineral statistics
	4. Return on Investment (ROI) & Payback Period	Financial viability of technology adoption	Financial models, NPV and IRR calculators	IMF / World Bank guidelines
	5. Sensitivity & risk analysis	Identify economic risks, fluctuations in market price, supply chain disruptions	Monte Carlo simulation, risk matrices	ISO 31000 (Risk Management)
Policy & Regulatory Assessment	1. Compliance with national mining and industrial policies	Evaluate alignment with Nigerian mining regulations, export/import rules	Regulatory review, legal compliance checklists	Nigerian Minerals & Mining Act

	2. Incentives & subsidies	Identify government support for local lithium processing	Policy documents, government websites	Nigerian Investment Promotion Commission (NIPC)
Integration & Feasibility Reporting	1. Comparative evaluation of alternative technologies	Rank technologies based on technical, economic, and environmental criteria	Multi-criteria decision analysis (MCDA)	ISO 14040 (LCA), ASTM D5757
	2. Decision support for investment	Provide recommendations for optimal localized lithium processing strategy	Feasibility report, cost-benefit analysis, SWOT analysis	International best practices

References

Ayres, R., Tost, M., & Tiess, G. (2023). Critical minerals and the geopolitics of supply chains: Policy options for resource-rich countries. *Resources Policy, 81,* 103168. https://doi.org/10.1016/j.resourpol.2023.103168

Chen, T., Li, J., Xu, M., & Wang, Y. (2024). Recent advances in lithium iron phosphate battery technology: A comprehensive review. *Batteries, 10(12),* 424. https://doi.org/10.3390/batteries10120424

Gao, T. (2023). Lithium extraction from hard rock lithium ores (spodumene, lepidolite, petalite and zinnwaldite): Technology, resources, environment and cost. *Renewable and Sustainable Energy Reviews, 180,* 113402. https://doi.org/10.1016/j.rser.2023.113402

Greim, P., Solomon, A., & Breyer, C. (2020). Assessment of lithium criticality in the global energy transition and addressing policy gaps in the United States. *Energy Policy, 144,* 111647. https://doi.org/10.1016/j.enpol.2020.111647

Gu, J., et al. (2025). Systematic optimisation of complex salt roasting and water leaching for extraction of Li, Rb and Cs from lepidolite. *Journal.* https://www.ncbi.nlm.nih.gov/pmc/articles/PMC12113790/

Harper International. (2021). *Lab scale rotary furnace product literature.* https://www.harperintl.com/furnacetechnologies/rotary-furnaces/lab-scale-rotary-furnace/

International Organization for Standardization (ISO). (2006). *Environmental management—Life cycle assessment—Principles and framework (ISO 14040:2006).* ISO.

Kamunda, C., Dziike, F., & Mlambo, T. (2022). Lithium beneficiation prospects in Southern Africa: An economic and policy review. *Journal of African Mining, 15(2),* 45–61.

Lab & Furnace / SH Scientific. (n.d.). *Rotary tube furnace specifications.* https://labandfurnace.com/shop/tube-furnace/rotary-tube-furnace/

Li, S., et al. (2023). Review on the beneficiation of Li, Be, Ta, Nb-bearing minerals. *Minerals, 13(7),* 865. https://doi.org/10.3390/min13070865

MDPI. (2020). Literature review and thermodynamic modelling of roasting of spodumene. *Metals.* https://www.mdpi.com/2075-4701/10/10/1312

Necke, T., Stein, J., Kleebe, H.-J., & Balke-Grünewald, B. (2023). Lithium extraction and zeolite synthesis via mechanochemical treatment of the silicate minerals lepidolite, spodumene, and petalite. *Minerals, 13(8),* 1030. https://doi.org/10.3390/min13081030

Nkwabi, C., & Moyo, T. (2021). Local beneficiation and industrialisation of African mineral resources: Challenges and opportunities. *Journal of African Political Economy, 48(3),* 421–438. https://doi.org/10.1080/03056244.2021.1924223

Nuñez, P., Davis, B., & Einem, J. (2022). Life cycle environmental impacts of hard rock lithium ore processing. *Journal of Cleaner Production, 356,* 131872. https://doi.org/10.1016/j.jclepro.2022.131872

Nuss, P., & Eckelman, M. J. (2014). Life cycle assessment of metals: A scientific synthesis. *PLoS ONE, 9(7),* e101298. https://doi.org/10.1371/journal.pone.0101298

Olade, M. A. (2024). Overview of the lithium geo-resources of Nigeria. *ResearchGate.* https://www.researchgate.net/publication/386322889_OVERVIEW_OF_THE_LITHIUM GEO-RESOURCES_OF_NIGERIA

Olajuyi, S. I. (2025). Mechanistic insights into Nigerian lithium ores: A review of geology, mineralization, and exploration. *Frontiers in Geochemistry, 2,* 1457321. https://doi.org/10.3389/fgeoc.2025.1457321

Opoku, P. A. (2025). An overview of coarse particle beneficiation of lithium ores. *Scientific Reports.* https://www.nature.com/articles/s41598-025-14059-z

Paul, D., Kumar, S., & Zhao, H. (2024). Life cycle assessment of lithium-based batteries: Review of sustainability dimensions. *Renewable and Sustainable Energy Reviews, 182,* 113521. https://doi.org/10.1016/j.rser.2023.113521

Peters, J. F., Weil, M., & Petrakopoulou, F. (2017). Life cycle assessment of the production of lithium-ion batteries. *Journal of Cleaner Production, 171,* 704–719. https://doi.org/10.1016/j.jclepro.2017.10.013

Petrakis, E., Alexopoulos, I., Pantelaki, O., Karmali, V., & Komnitsas, K. (2025). Advances in mineral processing of hard-rock lithium ores: A comprehensive review. *Mining, Metallurgy & Exploration.* https://doi.org/10.1007/s42461-025-01227-y

Tadesse, B. (2019). The beneficiation of lithium minerals from hard rock ores. *Minerals Engineering.* https://doi.org/10.1016/j.mineng.2019.

United Nations Economic Commission for Africa (UNECA). (2020). *Facilitating mineral beneficiation and value addition in Africa.*

Yang, X., et al. (2025). Froth flotation of lepidolite—A review. *Minerals, 15(7),* 750. https://doi.org/10.3390/min15070750

Yaksic, A., & Tilton, J. E. (2009). Using the cumulative availability curve to assess the threat of mineral depletion: The case of lithium. *Resources Policy, 34(4),* 185–194. https://doi.org/10.1016/j.resourpol.2009.05.002

Zhang, W., Chen, X., & Liu, Y. (2022). Direct extraction of lithium from α-spodumene by salt roasting and water leaching. *ACS Sustainable Chemistry & Engineering, 10(35),* 11567–11576. https://doi.org/10.1021/acssuschemeng.2c03321