STRUCTURAL AND ELECTROCHEMICAL INVESTIGATION OF CALCINED LITHIUM TITANATE($Li_4Ti_5O_{12}$)AND COPPER(I) CHLORIDE (CuCl) MIXTURE AS AN ALTERNATIVE ANODE MATERIAL FOR LI-ION BATTERY

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Submitted by

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CANDIDATE'S DECLARATION

I, **NEHA THAKUR (24/MSCPHY/44)** hereby certify that the work which is being presented in thesis entitled "**Structural and electrochemical investigation of calcined mixture of lithium titanate ($Li_4Ti_5O_{12}$) and copper(I) chloride (CuCl) as an alternative anode material for Li-ion battery**" in partial fulfilment of the requirements for the Degree of Master of Science, submitted to the Department of Applied Physics, Delhi Technological University, is an authentic record of my work carried out during the period from April 2025 to May 2025 under the supervision **of Prof. Vinod Singh** and **Dr. Amrish K. Panwar**.

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other university.

Title of Paper: **Structural and electrochemical investigation of calcined mixture of lithium titanate ($Li_4Ti_5O_{12}$) and copper(I) chloride (CuCl) as an alternative anode material for Li-ion battery**.

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Date: 29/05/2026 **Neha Thakur**

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CERTIFICATE BY THE SUPERVISIOR

I, hereby certify that the Project Dissertation titled "**Structural and electrochemical investigation of calcined mixture of lithium titanate ($Li_4Ti_5O_{12}$) and copper(I) chloride (CuCl) as an alternative anode material for Li-ion battery**" which is submitted by **Neha Thakur(24/MSCPHY/44)** Department of Applied Physics, Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Science, is a record of the project work carried out by the students under my supervision. To the best of my knowledge this **work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere**.

Place: Delhi

Date: 29 May, 2026

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ABSTRACT

Spinel lithium titanate ($Li_4Ti_5O_{12}$, LTO) has been considered as a promising alternative to commercialized graphite, which efficiently addresses safety issues such as lithium plating and dendrite formation that are often encountered during high-rate charging. Its zero-strain behaviour and higher operating potential allow excellent cycling stability . However, the intrinsically low electronic conductivity and moderate Li+ diffusion of spinel LTO still limit its high-rate capability and practical application in advanced lithium-ion batteries.

In the present work LTO was synthesized at 800^0C in order to obtain optimized structural and electrochemical profile. To check if halide-assisted copper oxidation and subsequent in-situ formation of CuO can improve the electrochemical performance of spinel, the optimized LTO was mixed with CuCl in Ti:Cu molar ratio of 5:2 and calcined in air. Physical characterization revealed LTO and CuO as two main components in the calcinated mixture of CuCl and LTO. Decrease in crystallite size was seen post calcination of mixture of CuCl and LTO with a minor change in particle size.

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LIST OF SYMBOLS AND ABBREVIATIONS

Li-ion batteries	Lithium-ion batteries
ESS	Energy Storage Systems
LTO	Lithium Titanium Oxide
CuCl	Copper (I) chloride
CL	Mixture of Calcinated Lithium Titanium Oxide and copper(I) chloride
NMP	N-Methyl-2-pyrrolidone
PVDF	Polyvinylidene Fluoride
W-H PLOT	Williamson-Hall Plot
XRD	X-ray Diffraction
SEM	Scanning Electron Microscope
EDS	Energy Dispersive X-ray Spectroscopy

CHAPTER 1

INTRODUCTION: HISTORY

1.1 ENERGY STORAGE DEVICES AND RISE OF LITHIUM-ION BATTERIES

For a long time humans relied almost exclusively on fossil fuels such as coal, oil and natural gas to meet their energy needs. They were convenient, relatively easy to extract and packed with energy. Which is why they powered everything from large industrial factories to everyday vehicles and household appliances. Entire economies were built around these fuels, and for decades they appeared the limitless answer to the world's growing energy needs.

But their drawbacks slowly became too much to ignore. Burning fossil fuels released lots of harmful gases into the atmosphere, causing serious air pollution and contributing heavily to global climate change. What's more, they are finite, they take millions of years to form naturally so once they are used up they cannot be replaced. Growing concerns prompted governments, scientists and industries around the world to seek cleaner and more sustainable alternatives.

The change has led to renewable energy sources such as solar, wind, biomass and geothermal energy [1]. Unlike fossil fuels, they are naturally replenished and give off very little or no harmful emissions during operation. But they had their own major disadvantage, in that they do not generate power in a continuous manner. You have to have the sun shining for solar panels to work , the wind blowing for wind turbines to spin and geothermal or biomass plants might have other operational limits . Renewable energy's intermittent nature and unreliability made it clear that it wasn't enough to simply produce clean energy, but that there should be a reliable way to store it when it was actually needed.

This realization has made energy storage one of the most important research and development areas in modern science and engineering [2]. To meet this challenge, various energy storage systems were explored and developed. These included mechanical systems like pumped hydro storage, where water is pumped uphill and then released to generate electricity when needed. Thermal storage systems save heat or cold for later use. Another option was chemical storage methods, such as producing hydrogen from surplus electricity. But of all these, electrochemical storage, and particularly batteries, have proven to be the most versatile, scalable, and practical means to couple with renewable energy systems.

Before lithium-ion batteries, several earlier battery technologies were developed and used. Lead-acid batteries are some of the first and most commonly used batteries in vehicles and backup power systems. Nickel-Cadmium (Ni-Cd) and Nickel-Iron (Ni-Fe) batteries came next, each with its own strengths and weaknesses. Nickel-Metal Hydride (Ni-MH) batteries increased the amount of energy stored and were often used in hybrid vehicles and consumer electronics. There were also primary lithium batteries , but these were not rechargeable and therefore could not be used long term . Then lithium ion batteries came along, and rapidly superseded all previous technologies. They were lighter, fully rechargeable and could store far more energy in the same amount of space, a property known as energy density. These qualities made them the obvious choice for everything from smartphones and laptops to electric vehicles and large-scale grid storage systems.

All components of lithium-ion battery are important but anode is particularly important[3]. The anode is the electrode that stores lithium ions when the battery is charged, and releases them during discharge. Its characteristics directly affect the battery's lifespan, charging speed, energy output and safety in different conditions. This has made improving the anode one of the most active and important areas of battery research.

1.2 LTO as an Anode Material

Lithium Titanate or LTO ($Li_4Ti_5O_{12}$) is one of the most reliable and unique anode materials that researchers have studied and utilized over the years. What makes LTO unique is a combination of properties that are rarely found in one material. It has a much higher charging rate than conventional anodes, withstands high temperatures without losing structural stability, and can continue to operate reliably even under demanding or harsh operating conditions [4]. Its theoretical storage capacity is 175 mAh/g and in actual experimental situations it consistently delivers between 165 and 170 mAh/g quite close to its theoretical maximum, which in itself is a sign of how efficiently the material works.

Another major advantage of LTO is the stable voltage plateau of ~1.55 V vs. lithium. This is higher than graphite anodes, but means that LTO avoids the formation of lithium dendrites tiny metallic spikes that can grow on conventional anodes over repeated charge cycles and eventually cause short circuits or even fires. This makes LTO inherently much safer than graphite based anodes, a particularly important characteristic for applications where safety cannot be compromised, such as in medical devices, electric vehicles or large scale stationary

energy storage systems . LTO also experiences almost no volume change during lithium insertion and extraction – a property known as “zero-strain” behavior, contributing to its exceptionally long cycle life [5]. LTO anode batteries can be charged and discharged thousands of times with little wear and tear, far longer than most other anode materials .

That said however, LTO is not without its limitations and these have been the subject of considerable research effort . The major disadvantage is the very poor electrical conductivity of the order of 10 -8 to 10 -13 S/cm [6]. In essence, this means that electrons cannot move efficiently through the material. The problem is further compounded by the relatively slow rate of diffusion of lithium ions . This limits how fast the lithium ions can move in and out of the material when it is charging and discharging . Together these two problems make it difficult to fast charge, increase the internal resistance within the electrode, and in the end reduce the usable capacity that can be practically extracted from the battery at high-rate conditions. What this means in the real world is that LTO can do medium charge and discharge rate well but not high power out or in.

To address these limitations, several modification strategies have been explored. The carbon coating, where a thin layer of highly conductive carbon is coated on the surface of LTO particles, is one of the most studied approaches. This forms a conductive network around the material, which promotes the flow of electrons and improves the overall rate performance. Another approach is doping . Doping is the addition of small amounts of foreign elements , either metals or non-metals , into the LTO crystal structure . Doping can change the electronic properties of the material, increase the carrier concentration, and improve the transport of both electrons and ions. Third approach is nanostructuring where the LTO is nanostructured so that the distance for the lithium ions travelling in the material is decreased, thus speeding up the ion diffusion and enhancing the high-rate performance [7].

Some of the most exciting results have been obtained by combining these strategies. For example, when combined with carbon nanotube arrays to create a carbon-composite nanostructure, the LTO anode yielded impressively high capacities over a broad spectrum of charge rates: about 290 mAh/g at 1C, 252 mAh/g at 5C, 229 mAh/g at 10C, and 209 mAh/g at 20C. These numbers are well above the theoretical capacity of LTO, indicating that the composite structure provided additional storage mechanisms besides simple lithium intercalation. Remarkably, after 1000 continuous 10C rate charge-discharge cycles, the anode still maintained about 72% of its initial capacity. Such high cycling rates with this durability is

a great improvement over unmodified LTO and highlights the potential of composite engineering strategies.[8].

Another promising approach was to make a composite of LTO and titanium dioxide (TiO2) and test it in a full cell configuration, which is closer to a real battery configuration. The LTO–TiO2 composite delivered an initial discharge capacity of about 126 mAh/g and retained a substantial part of that capacity after 1000 cycles, indicating good long-term stability. Although the capacity numbers are lower than the carbon nanotube composite, the full-cell testing conditions make the result particularly meaningful, as performance in full cells is generally harder to achieve than in half-cell setups [9]. Taken together these results point to a clear path forward for LTO, whose fundamental strengths in safety, stability, and longevity make it an excellent base material, and research into composites, coatings, and doping continue to steadily address its conductivity and rate limitations.

1.3 How a Rechargeable Lithium-Ion Battery Works?

The operation of a lithium-ion battery is based on a fairly elegant and well-understood electrochemical principle lithium ions shuttling back and forth between two electrodes, the anode and the cathode, through a medium called the electrolyte. This movement of ions, paired with the flow of electrons through an external circuit, is what allows the battery to both store energy and release it on demand.

That is, when energized, the battery is used to power a device , the process starts at the anode. In conventional lithium ion batteries, the anode is typically composed of graphite . The lithium atoms stored in the anode are oxidized to positively charged lithium ions. The lithium ions then travel through the electrolyte, which is a liquid or gel medium allowing ion movement but not electron movement, to the cathode. Electrons can't pass through the electrolyte, so they have to take a different route: they travel through the external circuit, the path that connects the battery to whatever device it's powering. This movement of electrons through the external circuit is the electrical current that powers devices. Reduction occurs at the cathode, which is frequently composed of a material such as lithium cobalt oxide ($LiCoO_2$). They absorb the incoming lithium ions from the electrolyte and the electrons from the external circuit. This completes the circuit. Throughout this process, the electrical balance is maintained naturally, because for each electron that passes through the external circuit, a lithium-ion passes through the electrolyte, preventing any charge buildup in any part of the battery.

All of this is basically the reverse of the charging process. When the battery is connected to an external power source, such as a charger, that power source causes the electrochemical reactions to proceed in the opposite direction. Now you're pushing energy into the battery instead of pulling it out of the battery. But now the cathode is oxidized, releasing the lithium ions and electrons it previously absorbed. The lithium ions reverse their path through the electrolyte to the anode. The electrons are driven through the charging circuit back into the anode as well. The lithium ions and electrons are now again stored in the anode material and ready to be released in the next discharge cycle. At the anode reduction occurs now. Throughout the process charge neutrality is carefully preserved. Once again each electron driven into the anode is precisely matched by one lithium ion arriving through the electrolyte. But a well-designed lithium-ion battery can repeat this cycle of charging and discharging many hundreds or even thousands of times before any significant degradation in performance is noticed. It is this efficiency, compactness and reliability that has made lithium-ion batteries the dominant energy storage technology of the modern era, and why improving their core components, particularly the anode, remains such an important focus of ongoing scientific research [10].

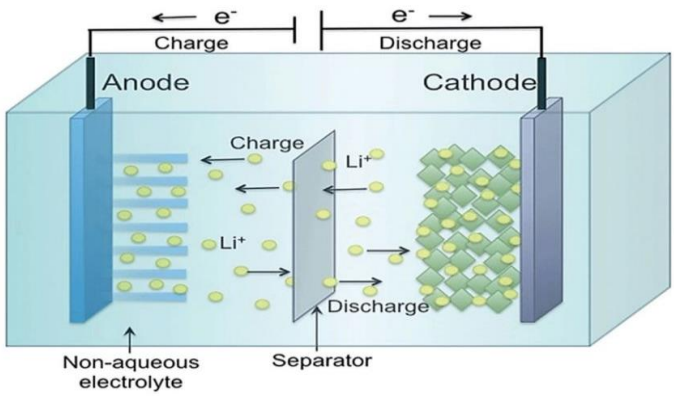


Fig1: Working of rechargeable Li-ion batteries

CHAPTER 2

LITERATURE REVIEW

2.1 Growing Demand for Better Batteries

Technology has developed rapidly in recent years – electric cars, smartphones, laptops and solar power systems are now a big part of everyday life. All of these things require reliable and efficient ways to store energy. Out of all the available options, lithium-ion batteries seem to be the best option so far. They can hold a lot of energy in a small space, last a long time, and charge and discharge efficiently. They've been commercialized for more than three decades, but scientists are still trying to make them better — safer, cheaper, more capable.[11]

A battery's performance is largely dependent on the materials used inside it. They determine the amount of voltage the battery can deliver, how stable that will be, how much energy it can hold, and how long before it degrades.

Traditionally , on the positive electrode side -- the cathode -- a material called lithium cobalt oxide ($LiCoO_2$) was used because it stores a lot of energy . However it is unstable at high temperatures and relies on cobalt, a metal that is expensive and difficult to obtain. Due to these issues, other cathode materials were developed by researchers such as $LiFePO_4$, $LiMn_2O_4$ and nickel rich NMC compounds. They each have their own trade offs between cost , safety , and performance .[12]. Graphite has long been the standard material for the negative electrode side, the anode. It has a layered structure that is good at absorbing and then reversibly releasing lithium ions. But graphite also has serious disadvantages. It has very low operating voltage, so during fast charging or at low temperatures lithium metal deposits on the surface and can create needle-like structures called dendrites [13]. Such dendrites can cause internal shorts within the battery, which can be dangerous. Over time, graphite reacts with the electrolyte to form a layer on its surface that permanently wastes some of the battery's capacity.

These issues have prompted researchers to look for better anode materials that are safer, charge faster and last longer.

2.2 Why Battery Safety Matters?

With the use of lithium-ion batteries in larger and more demanding applications such as electric vehicles and power grids safety has become an extremely important concern. Dendrite growth, electrolyte breakdown and heat-related instability can lead to dangerous failures. Silicon was an attractive alternative anode material as it can host a lot more lithium than graphite but it suffers from large expansion and contraction during charge and discharge which causes it to crack and degrade over time. Other materials such as transition metal oxides have higher energy storage capacity, but usually exhibit unstable voltage behavior and slow reaction speed.

These drawbacks have garnered considerable interest in a material known as lithium titanate or LTO ($Li_4Ti_5O_{12}$). LTO enjoys a good reputation owing to its safety, rigid crystal structure and most importantly the "zero-strain" behavior. This means that, when lithium ions enter or leave the LTO structure during charge and discharge, the material hardly changes its shape or size. This is a rare and valuable property that enables LTO to survive thousands of charge-discharge cycles without breaking down. LTO also operates at a relatively higher voltage than graphite anodes, which effectively prevents lithium plating and makes it much more thermally stable. It has qualities that make it a good candidate for high power applications and situations where extreme conditions are present.

2.3 LTO Features and Performance

LTO has a well-defined crystal structure a cubic spinel structure in which lithium and titanium atoms are located at the specific sites of the structure. When charging, the lithium ions come in and fill up the holes. The whole framework does not get distorted or broken. This is the main reason for the structural stability of LTO and its long cycle life, which can easily go beyond several thousand cycles with little loss of capacity.

LTO does have two major weaknesses, however. First, it has very poor electrical conductivity, meaning that electrons do not easily move through it. Second, it has a lower energy storage capacity of 175 mAh/g compared to graphite, which means that it is less energy dense. These two constraints cause poor performance of LTO, especially when it needs to charge or discharge at high speeds.

The researchers have tried to fix these problems to some extent. Carbon coating of LTO particles facilitates the electron transport through the material. The electronic structure of LTO can be tuned and the transport of electrons and ions can be enhanced by doping, a

process where small amounts of other elements are added [14]. She said, “making the particles smaller makes it easier for the lithium to get through.” LTO can be combined with other conductive materials such as metal oxides, sulfides or halides to form composite structures with better overall performance. Controlled defects introduced on the surface of LTO particles can also be beneficial for accelerating charge transfer. All of these strategies have shown promising results and confirm that LTO, when properly modified, has a strong potential for use in next-generation battery systems.

2.4 The Electrode-Electrolyte Interface

Another important factor that affects how a battery works is what happens at the boundary between the electrode and the liquid electrolyte inside the battery. The region along this boundary , called the electrode-electrolyte interface , has a large impact on the ease of ion transfer , the battery stability over time , and the overall long term performance .

For graphite anodes, the electrolyte breaks down at the electrode surface and forms a layer called solid electrolyte interphase, or SEI. A good SEI layer is a protective coating it prevents further unwanted reaction, but also allows lithium ions to pass through. But the SEI on graphite can crack, rebuild or degrade over repeated cycling which causes problems in the long run. In this regard, LTO is quite different.” LTO also works at a higher voltage than the voltage at which the usual electrolytes start to decompose . So it never develops a " typical " SEI layer . This is actually an advantage for safety but it also means that LTO is not always compatible with certain electrolyte additives which are specifically designed to improve SEI formation.

A similar but different layer forms on the cathode side, the cathode electrolyte interphase, or CEI, as a result of oxidation reactions at higher voltages. This layer influences the total resistance and long-time performance of the battery. Because of all this, the selection of the right electrode material, electrolyte and interface engineering strategy must all be done together they cannot be considered in isolation if the aim is to build an optimally performing battery [15].

2.5 LTO Combined with Other Materials

Recent studies have demonstrated that the performance of LTO, especially the rapid charge/discharge capability, can be significantly enhanced when it is combined with other conductive or electrochemically active materials. Carbon based composites for example

LTO with graphene, carbon nanotubes or graphene oxide improve the conductivity of the electrode and ease the charge transport. LTO composites with metal oxides such as TiO2 or Fe2O3 work well by increasing the number of active sites for lithium reactions and by speeding up the transport of lithium ions through the material.

One of the promising and relatively new routes is the hybridization of LTO with metal halides. Halides are compounds that contain chlorine or bromine bonded to metals . When paired with LTO, they can change the surface chemistry, tune the electron distribution within the material, and produce structural defects, which can actually help lithium ions to move more quickly. Particularly, the combination of LTO with copper halides, especially a compound such as copper bromide (CuBr), has been investigated, and the resulting composites can achieve higher capacities compared to conventional commercial LTO [16]. Such improvements are due to the changes in the conductivity and microstructure caused by the interaction between the halide and the oxide.

2.6 Copper(I) Chloride (CuCl) as a Functional Material

Copper(I) chloride (CuCl) is a semiconductor material with a relatively simple crystal structure . Here each copper ion is surrounded by four chlorine ions in a specific geometric arrangement. This leads to stable bonding of CuCl and interesting electrical properties. CuCl has not been used in conventional lithium-ion batteries, but it has become increasingly interesting for new types of battery systems such as chloride-ion batteries due to its low price, easy production, and good electrochemical performance.

CuCl can be prepared by straightforward chemical reactions. For example, Cu metal can react with hydrochloric acid, or a copper chloride precursor can be reduced. Although a structural change can occur at elevated temperatures, controlled heat treatment can be used to promote this transformation to improve the conductivity or to create useful composite structures when CuCl is blended with other materials such as LTO.

Mixing LTO with CuCl is a fairly recent area of research that makes sense when you think about what each material brings to the table. LTO provides structural stability and long cycle life, CuCl provides potential electrochemical activity, improved surface chemistry and better conductivity [17]. When the two are mixed and heated together in a process called calcination, a number of good things can happen: copper species may be partially incorporated into the LTO structure, conductive surface layers may form, structural defects

may be created that help lithium ions move faster, and overall charge transfer within the material may improve greatly.

Early studies on LTO:CuCl composites have reported higher storage capacity, better performance at fast charge rates and improved cycling stability compared to unmodified LTO. These results demonstrate that the use of halide compounds such as CuCl as a modification agent is a practical, cost effective and promising strategy to make LTO based anodes even more suitable for real world battery applications.

CHAPTER 3

EXPERIMENTAL

3.1 MATERIALS AND REAGENTS

Lithium Carbonate (Li_2CO_3) with purity 98.5%, Titanium dioxide (TiO_2) with purity 99%, Copper(I) Chloride (CuCl) with purity 98.0%, were purchased from Sigma-Aldrich.

3.2 SYNTHESIS

3.2.1 SOLID-STATE SYNTHESIS

It is a method of preparing materials in which reactants are in the solid state and they undergo chemical reactions at elevated temperatures forming a new product. Such high temperature processes lead to diffusion reactions forming new phases. Materials here produced have unique structural properties, high purity, fine particle size. Several factors affect the structure of the product such as morphological properties of reagents, their reactivity, surface area etc. Products thus formed can be polycrystalline, single crystal etc. Which have several applications in the field of electronics. This process involves a series of step beginning with the selection of appropriate reagents i.e. reagents with high reactivity, less inertness, well defined compositions and fine powders. These reagents are then weighed to be further mixed in agate mortar or ball mill depending on the quantity. Further if required the resulting product is also palletized in some of the cases. After all this is done, final procedure includes calcinating the resulting product in a crucible, tube or boat where all of these can vary in properties like reactivity, strength, cost etc. Environment plays a crucial role in this process, as presence of air or noble gases do influence the morphology of the product [19].

3.2.2 LTO SYNTHESIS

For preparation of LTO, calculated amounts of lithium carbonate (Li_2CO_3), (Sigma Aldrich, ~99%) and titanium dioxide (TiO_2), anatase/rutile), (Sigma Aldrich, ~99%) were accurately weighed based on the molar ratio required for the formation of the $Li_4Ti_5O_{12}$ spinel phase. The precursors were transferred to an agate mortar and ground thoroughly using ball mill at 206 rpm for 8 hours to ensure uniform dispersion and intimate contact between the lithium and titanium sources. This mechanical mixing serves to enhance solid–solid interfacial contact, which is essential for promoting diffusion-controlled reactions during high-temperature treatment. The ground mixture was then placed in an alumina crucible and subjected to calcination at 800°C in a furnace. The calcination temperature was selected to facilitate the decomposition of lithium carbonate and enable the solid-state reaction between Li_2O (formed in situ) and TiO_2, ultimately yielding the spinel $Li_4Ti_5O_{12}$ phase. After the process is done with the heat treatment, the sample was allowed to cool naturally at room temperature in the furnace itself. The calcined product was lightly reground to break soft agglomerates and stored in airtight containers to prevent moisture uptake prior to further characterization and electrode fabrication.

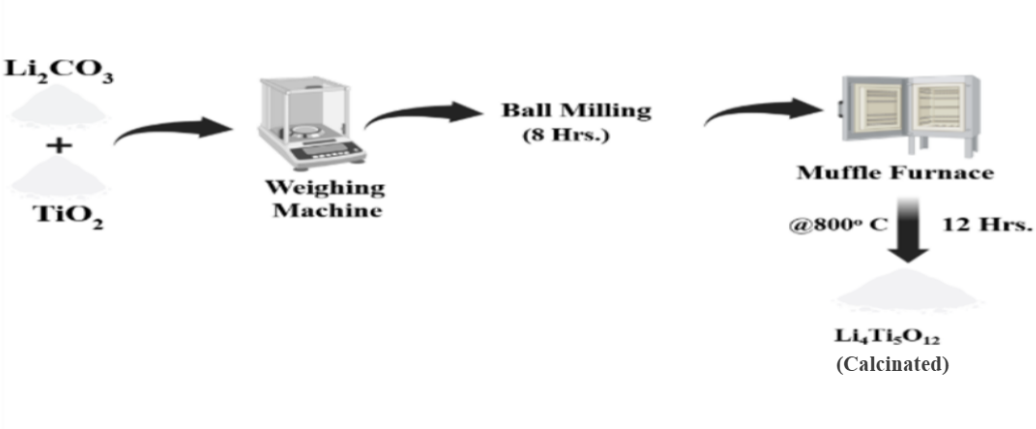


Fig.2 Synthesis of LTO ($Li_4Ti_5O_{12}$)

3.2.3 SYNTHESIS OF LTO AND CuCl MIXTURE

To begin with, 0.918g of LTO and 0.396g of CuCl were precisely mixed together to form a mixture containing Ti and Cu in the molar ratio 5:2. This mixture was then thoroughly grounded in agate mortar for 45 mins. In order to synthesize a uniform mixture. After that step, the resulting mixture was placed in a crucible for calcination in a muffle furnace for 3h at 550°C. This calcinated mixture was then extracted from the furnace after cooling it to the room temperature. Post this the calcined mixture was again grounded in agate mortar for 10-15 min. And then stored in an air tight container for further use. This stored sample was then named CL.

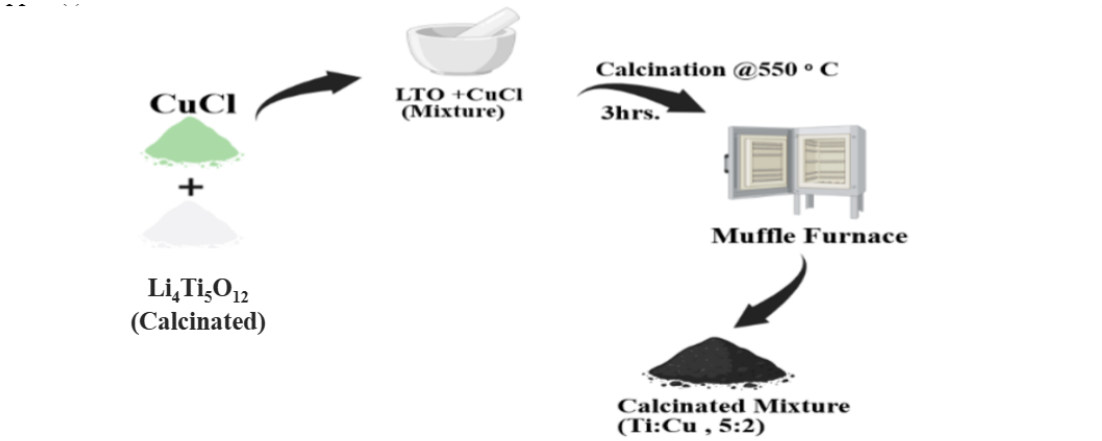


Fig3: Synthesis of LTO and CuCl mixture

3.2.4 PREPARATION OF ELECTRODES

Firstly, a mixture containing optimized LTO, PVDF (polyvinylidene fluoride) and AB (acetylene black) was made carefully, in the mass ratio of optimized LTO, PVDF [18] and Acetylene black to be 8:1:1. Following which various drops of NMP (N-methyl pyrrolidone) were added to this resulting mixture while stirring so as to form a slurry. On reaching a certain thickness the slurry is then carefully spread on a copper foil surface leading to formation of synthesized LTO's electrode. Following which the prepared electrode was dried in oven at 80°C overnight in order to remove any volatile component and generate well - dried electrode. The electrodes prepared from optimized LTO, CuCl and LTO mixture were obtained in this manner and then cut into coin cells for further work.

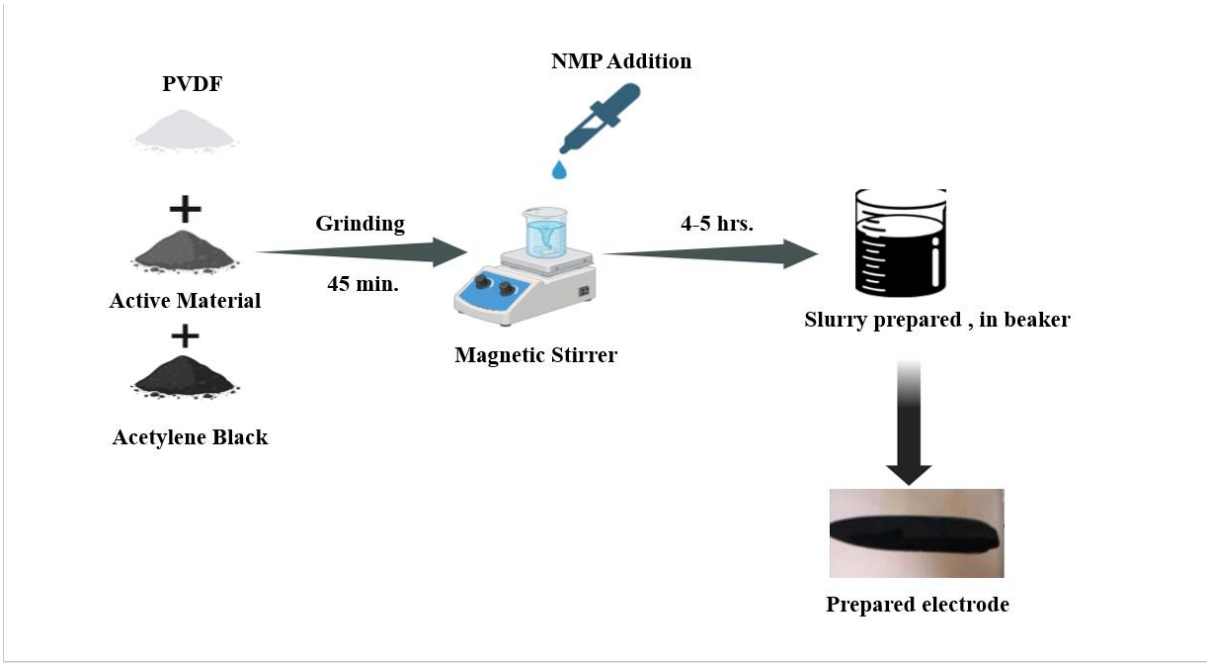


Fig.4 Step-wise synthesis for fabrication of electrodes

Table1: Composition of Slurry for Electrode formation:

Component	Weight (%)	Mass (g)	
Active material	80	0.16	
Activated Carbon	10	0.02	
Polyvinylidene Fluoride (PVDF)	10	0.02	
Solvent	-		NMP (N-Methyl-2-pyrrolidone)

3.3 CHARACTERIZATION

The structural and phase composition of all samples are analyzed using the Rigaku-600 X-Ray diffractometer. The elemental and surface morphological characteristics of different samples are studied using SEM and EDX (Scanning Electron Microscopy and Energy dispersive X-ray spectroscopy.

3.3.1 X-RAY DIFFRACTION (XRD)

X-ray diffraction is an undestructive method that is used for investigating the structure of material at the atomic or molecular scale. Materials with high periodicity i.e. materials that are completely or partially crystalline works as best materials to carry out the XRD. It involves falling of x-ray beams on the surface of the sample, these X ray beams are electromagnetic radiation with wavelength of the order of a few nanometers. The wavelength of the x ray beam is similar to the spacing between the atoms of the sample thus the angle of diffraction is affected by the spacing between atoms of the sample. Contrary to that the use of larger wavelength will show no effect on the diffraction angle. These x rays are scattered or bounced back in a direction different to their incident direction at some different angle namely theta. Theta is known as the angle of diffraction. Some of the beams undergo destructive interference whereas beams with similar wavelength undergo constructive interference resulting in formation of beam with greater amplitude. By convention the angle between incoming and outgoing beam directions is called 2θ. For the simplest sample, constructive interference is observed when Bragg's law is completely satisfied i.e.

$$n\lambda = 2d \sin\theta$$

Where, n= integer (1,2, ...), λ is the wavelength of the incident X-ray beam. There are several measurements made such as single crystal measurements, powde r diffraction etc.

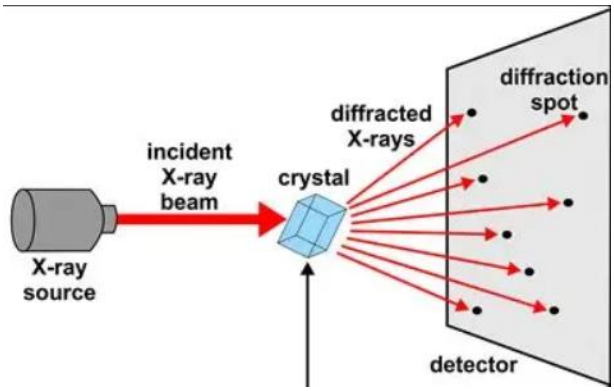


Fig5. Schematic diagram of X-Ray Diffraction

3.3.2 SCANNING ELECTRON MICROSCOPY (SEM)

Scanning electron microscopy (SEM) is a powerful technique for the study of surface of a material. The electron beam is focused on the specimen in SEM with accelerating voltages up to 30 kV (Inkson, 2016). As the electron beam interacts with the specimen, signals are emitted from the specimen and collected by detectors. The recorded signals are combined to create an image. When an electron beam hits a sample, the atomic nucleus or the outer shell electrons cause "elastic scattering" with the same energy. Incident electrons that elastically scatter at an angle larger than 90 degrees are called backscattered electrons (BSEs). BSE signals contain information of the composition and the topography of the sample. When the incident electrons and the electrons.

3.3.3 ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDX)

This is done by EDS, which measures the energy and intensity of the X-rays emitted from a sample when it is exposed to the electron beam of an electron microscope. When SEM is used the technique is called SEM EDS and when TEM is used it is called TEM EDS. EDS analysis is based on interaction of the high-energy electron beam of the microscope with the atoms of the sample. This interaction has a variety of effects, one of which is the ejection of an inner-shell electron from an atom, leaving an electron vacancy which is quickly filled by a higher-energy, outer-shell electron. The outer shell electron drops to a lower energy level shell and loses energy , this excess energy is emitted in the form of X-rays . The emitted X-ray in this process is called characteristic X-ray, as each element has different energy levels. By detecting and measuring these characteristic X-rays it is possible to find out which elements are present in a sample and in what quantity. The emitted X-rays are detected and measured with an SDD (silicon drift detector). The energies and intensities of the X-rays are recorded for each pixel of measurement. Then the software and a spectral library are used to identify the elements present from the measured characteristic X-ray energies and the amount of the elements from their respective intensities.

CHAPTER – 4

RESULTS AND DISCUSSION

4.1 X-RAY DIFFRACTION (XRD) ANALYSIS

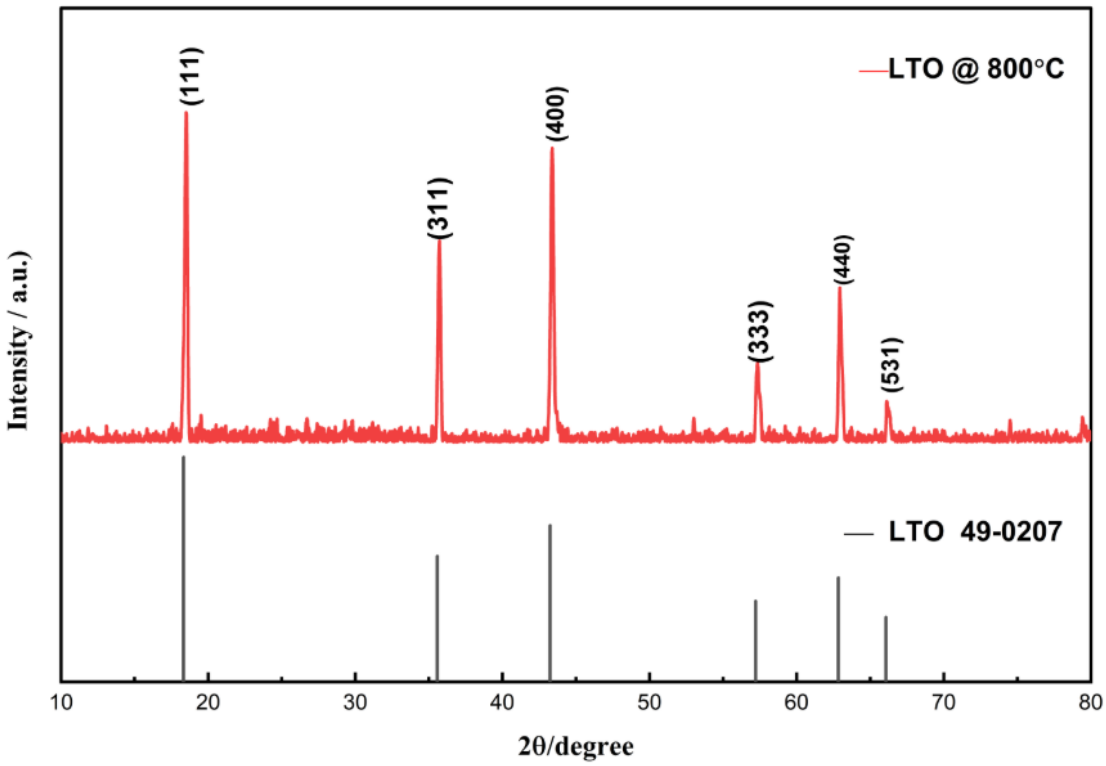


Fig.6 XRD patterns of Synthesized LTO

Fig 6 shows the XRD pattern of synthesized LTO. All sharp diffraction peaks can be indexed based on cubic spinel structure of LTO (JCPDS Card No. 490207) . For synthesized LTO, main diffraction peaks positioned at 2θ equals to 18.3^0, 35.6^0 , 43.2^0,57.2^0 ,62.8^0 and 66.1^0 correspond respectively to crystallographic planes of (111), (311), (400), (333), (440), (531) of LTO (JCPDS Card No. 490207) are clearly displayed indicating formation of LTO with well defined spinal crystal structure at 800^0C [20] . Afterwards the Crystallite size for the

synthesized LTO was calculated using the W-H (Williamson- Hall) plot and it was found to be nearly 35.8 nm.

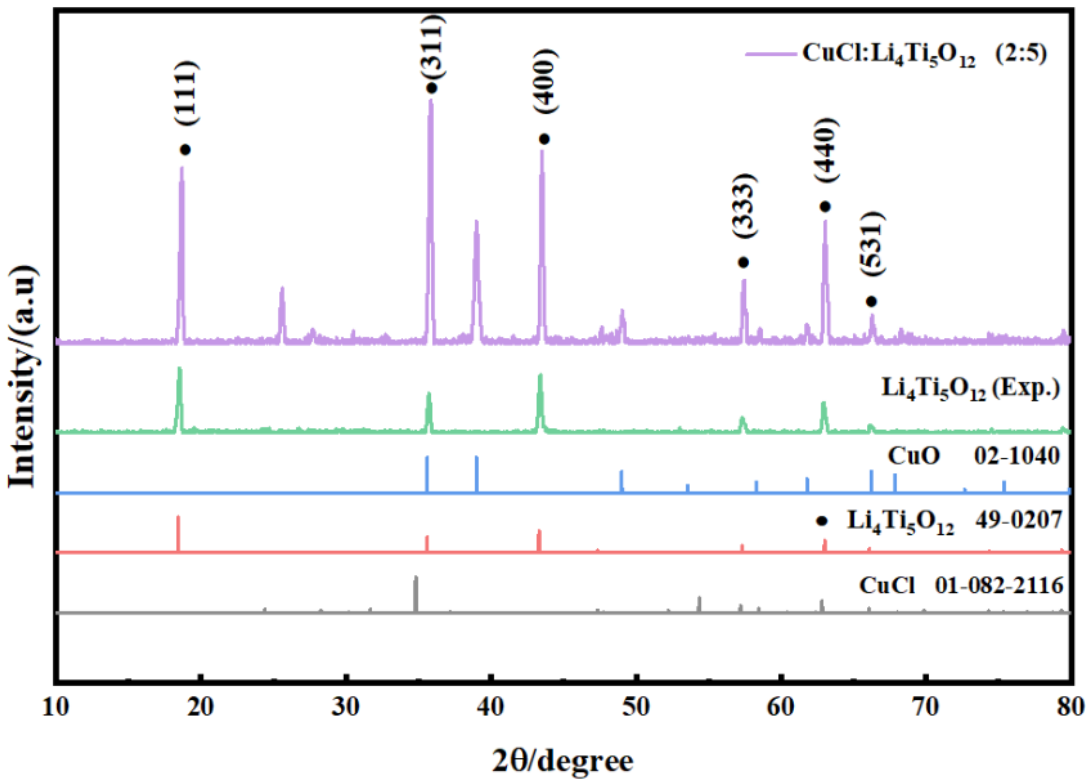


Fig7: XRD pattern of all studied specimen

XRD patterns of the synthesized sample, including the standard XRD patterns of CuO, CuCl and LTO are provided in fig.7. For sample CL , the main diffraction peaks assigned to LTO are clearly shown , in other words , diffraction peaks situated at 2θ equals to 18.3, 35.6 , 43.2 ,57.2 , 62.8 and 66.1 respectively corresponds to crystallographic planes of (1 1 1), (311), (400), (333), (440), (531) of LTO (JCPDS Card No. 490207) are clearly displayed , indicating that LTO with well defined cubic spinal structure is present in the synthesized sample CL which is a mixture of CuCl and LTO with Ti to Cu in the ratio 5:2 . Other than that small characteristic diffraction peaks attributed to CuO (JCPDS , No. 02-1040) was also identified in

sample CL centered at 2θ ~ 35.60, ~39^0, 66.2^0, and 48.6^0, aligned well with the reflection of CuO which indicated the presence of another major substance in the prepared sample. No diffraction peaks assigned to CuCl [21] ,were not found in sample. This might be due to a very small amount of CuCl present in the sample such that it could not be detected. The average full width half maximum (FWHM) values of synthesized LTO and sample CL was found to be 0.24 and 0.25 respectively indicating lower crystallinity in the calcinated sample CL compared to that of LTO displaying that calcination of CuCl and LTO in air can modify the crystallinity of commercial LTO. An extra diffraction peak positioned at 2θ ~ 26^0 shows the formation of TiO_2 as another substance in the sample CL. There could be a possibility that this is due to the structural breakdown of LTO due to addition of CuCl during calcination. When the mixture is heated the Cu^+ ion from CuCl targets (Li^+) lithium ions occupying 8a tetrahedral sites or 16d octahedral sites of LTO matrix. The displaced Li^+ ion combines with the Cl^- ion forming highly stable LiCl compound. Since the peak corresponding to rutile TiO2 can be seen there is a possibility that the LTO crystal structure would have been distorted due to displacement of $Li^{+\cdot}$ Ion by the Cu^+ ion. Removal of Li from the LTO matrix distorts the LTO crystal structure leaving behind the Ti-O arrangement. Amounts of Cu at the 8a tetrahedral sites or 16d octahedral sites of LTO this remaining structure of Ti-O restabilizes as independent binary oxide yielding TiO2 with some amounts of Cu^+ ion at the 8a tetrahedral sites or 16d octahedral. On other hand some amount Cu due to calcination in air produces CuO and thus several peaks of CuO can also be seen here. [22].Additionally the average crystallite size calculated using W-plot for the sample CL was found to be 34.13 nm.

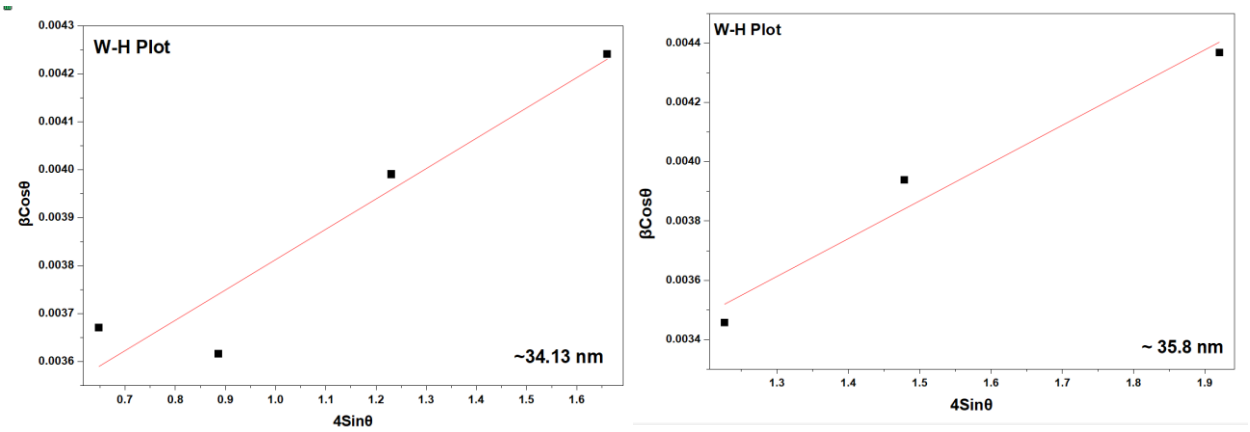


Fig8: W-H plot of CL(a) and synthesized LTO (b)

4.2 SEM

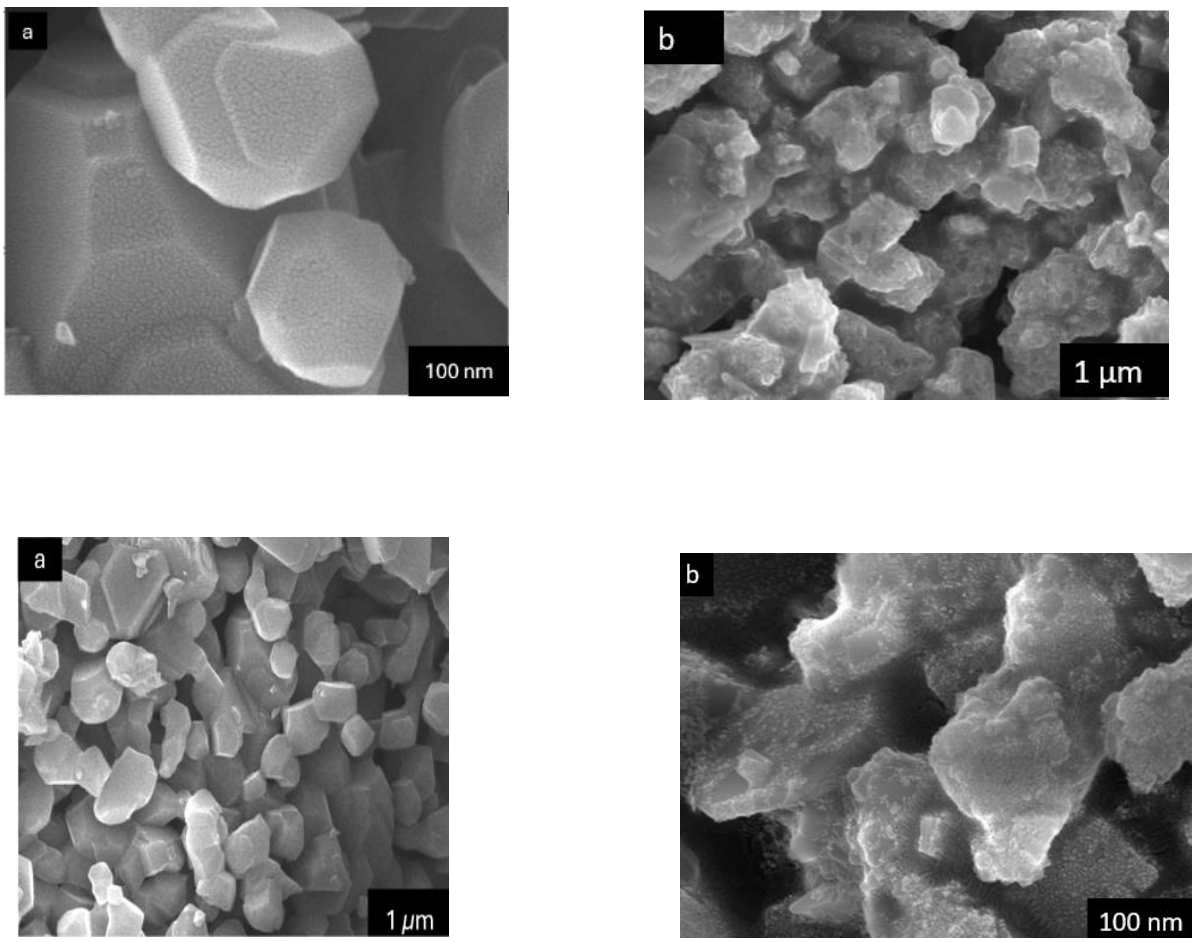


Fig9. SEM images of the materials LTO (shown as a) and CL (shown as b) with scales 1 μm and 100 nm.

The SEM images reveal that the synthesized LTO particles have a well-defined, polyhedral (multi-faceted) morphology essentially, they look like tiny cut gemstones with flat, angular faces. This shape is characteristic of a crystalline spinel structure, which confirms that the material has formed properly during synthesis.

At lower magnification (1 μm), it is evident that the particles are distributed fairly uniformly, with sizes ranging approximately from 110 nm to around 200 nm. The particles seem to be compacted with moderate agglomeration i.e. some of the particles have clustered together. This is typical for oxide powders prepared by solid state or sol-gel methods.

On higher magnification (100 nm) the surface of individual particles appears to have a fine granular texture with a network of shallow micro-cracks or grain boundaries across the faces. This surface roughness is actually beneficial as it increases the effective surface area available for lithium-ion interaction, which can have a positive effect on the electrochemical performance of the material.

Overall, the SEM images indicate that the LTO was successfully synthesized with good crystallinity and a morphology suitable for use as an anode material in lithium-ion batteries, where well-defined particle shape and reasonable surface area are important for stable and efficient charge-discharge behavior.

SEM Analysis of LTO-CuCl Composite (Calcined at 550°C)

The surface morphology of the prepared LTO–CuCl composite was examined by SEM and compared with that of bare LTO. For the pure LTO, as discussed previously, well-defined polyhedral particles with clear facets and an average size of approximately 200–500 nm were observed, which is consistent with the typical spinel LTO crystalline structure.

Importantly, the absence of the large CuCl particle morphology in the composite sample strongly indicates that CuCl was completely converted into other copper-containing phases

during the calcination process, with the copper species now intimately distributed across the LTO surface. Additionally, visible pores and open channels between agglomerated particles were observed, forming a spatially interconnected porous network a feature that is particularly beneficial for electrolyte infiltration and efficient lithium-ion transport during battery operation.

Overall, the SEM results confirm that calcination at 550°C successfully transforms the LTO–CuCl mixture into a composite material with a modified surface architecture, where copper-derived nanoparticles are incorporated onto or into the LTO matrix, potentially enhancing its electrochemical rate performance and cycling stability.

4.3 EDX ANALYSIS

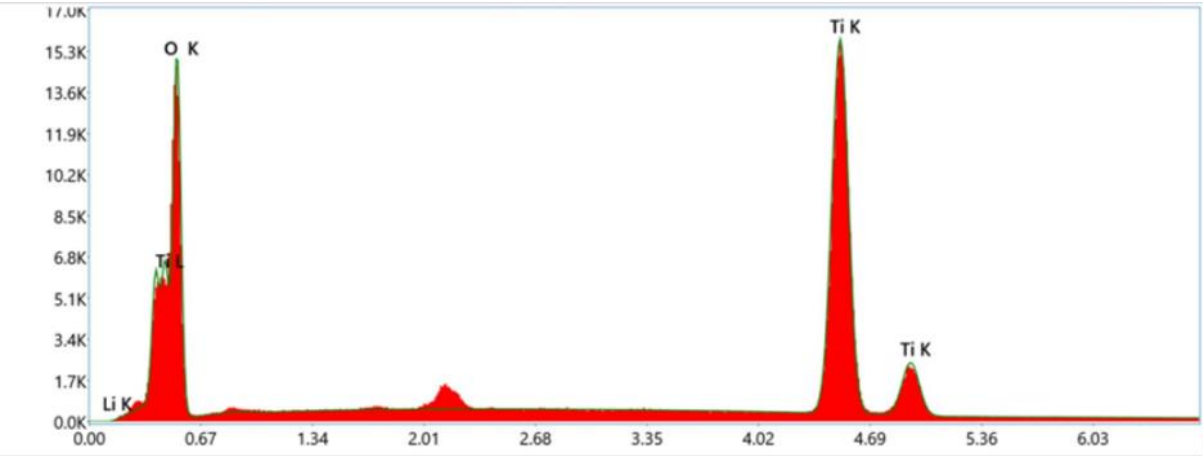


Fig.10 EDS spectrum of optimized LTO @ 800^0C

Table2: Elemental composition of LTO (Li4Ti5O12)

Element	Weight %	Atomic %	Error %
Li K	0.0	0.0	100
O K	31.7	58.1	10.8
Ti K	68.3	41.9	3.8

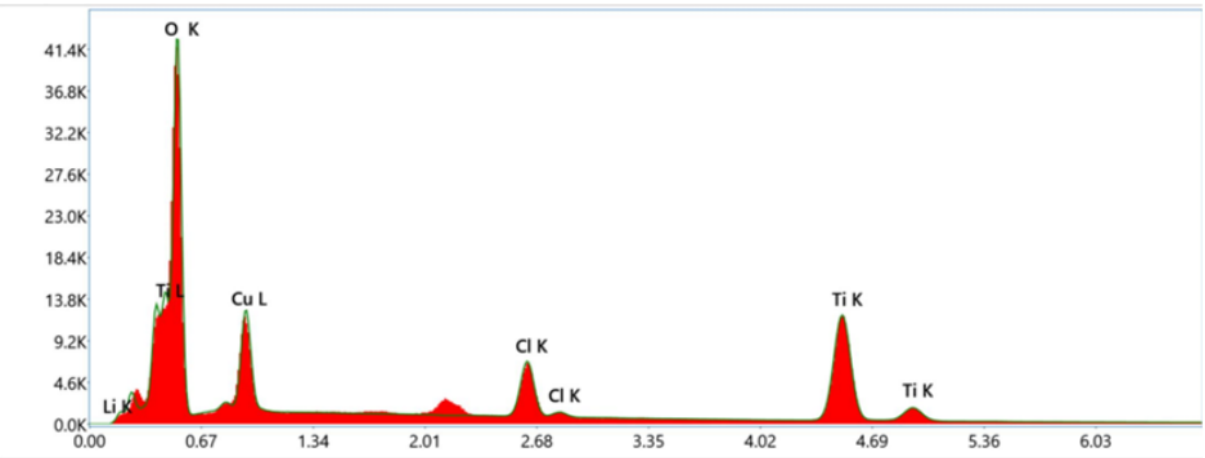


Fig.11 EDS spectrum of CL (LTO+CuCL)

Table 3 : Elemental composition of LTO and CuCl mixture

Element	Weight %	Atomic %	Error %
Li K	0.0	0.1	100
O K	44.6	70.9	10.0
Cl K	5.4	3.9	3.9
Ti K	39.1	20.7	4.0
Cu L	10.8	4.3	8.8

EDS patterns of the synthesized samples i.e. LTO and CL are provided in the fig(a) and fig(b). In case of optimized LTO as shown in fig(a). Three peaks that are assigned respectively to oxygen (O) and titanium(Ti) are clearly shown, in which the atomic contents of O and Ti were approximately 58.1%, 41.9 % . With calculation it is found that the atomic ratio of O to Ti in case of optimized LTO is found to be nearly 2.41 , which is approximately same as that of the atomic ratio of O and Ti in case of commercial LTO (2.4).

On other hand the peaks designated to elements Cu , Cl , O ,Ti are also displayed in the pattern of prepared mixture of LTO and CuCl (Ti:Cu , 5:2) , fig(b). The atomic content of oxygen (O) and titanium (Ti) in the sample CT are found to be 70.9% and 20.7% respectively . Further the atomic ratio was calculated to be around 3.42 which is apparently greater than the atomic ratio of oxygen(O) to that of titanium (Ti) in case of pure LTO (2.4) reflecting the information about possible synthesis of some kind of oxide. Again, the atomic contents of Cu and Cl were found to be 4.3 %, 3.9% which is very low compared to the atomic content of other elements

present in the synthesized sample Cl. Thus it can be assumed that the Cl was consumed during the calcination process of the sample CL. This observation is in agreement with the fact that no diffraction peak belonging to CuCl is seen in the XRD results of the synthesized sample CL(fig). from the above information it can thus be concluded that the three main components found in the sample CL were O followed by Ti and Cu , which can also be seen in the XRD result i.e. LTO and CuO were the main components in the synthesized sample CL.

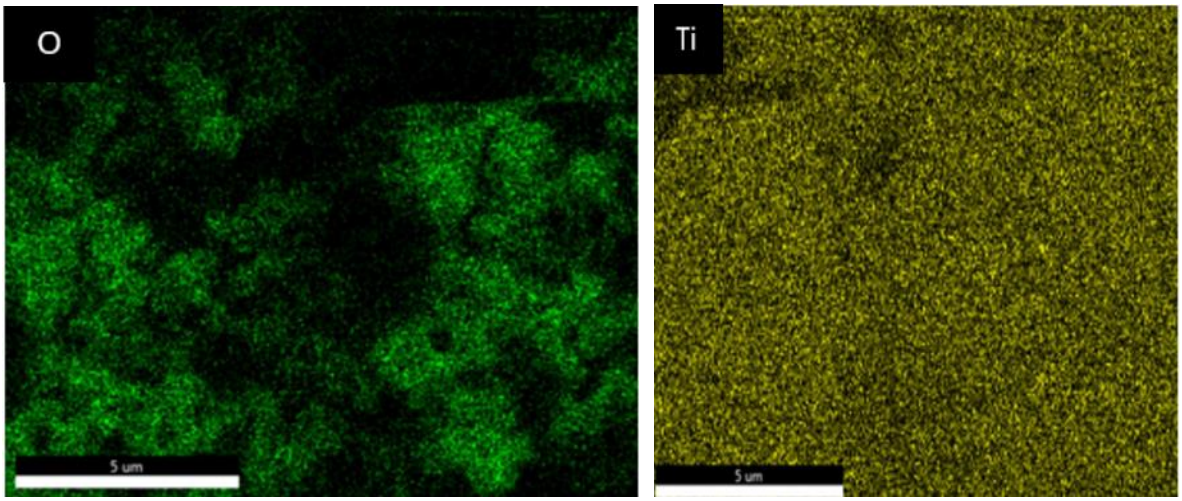


Fig. 12 EDS mappings of Ti, O in Synthesized LTO

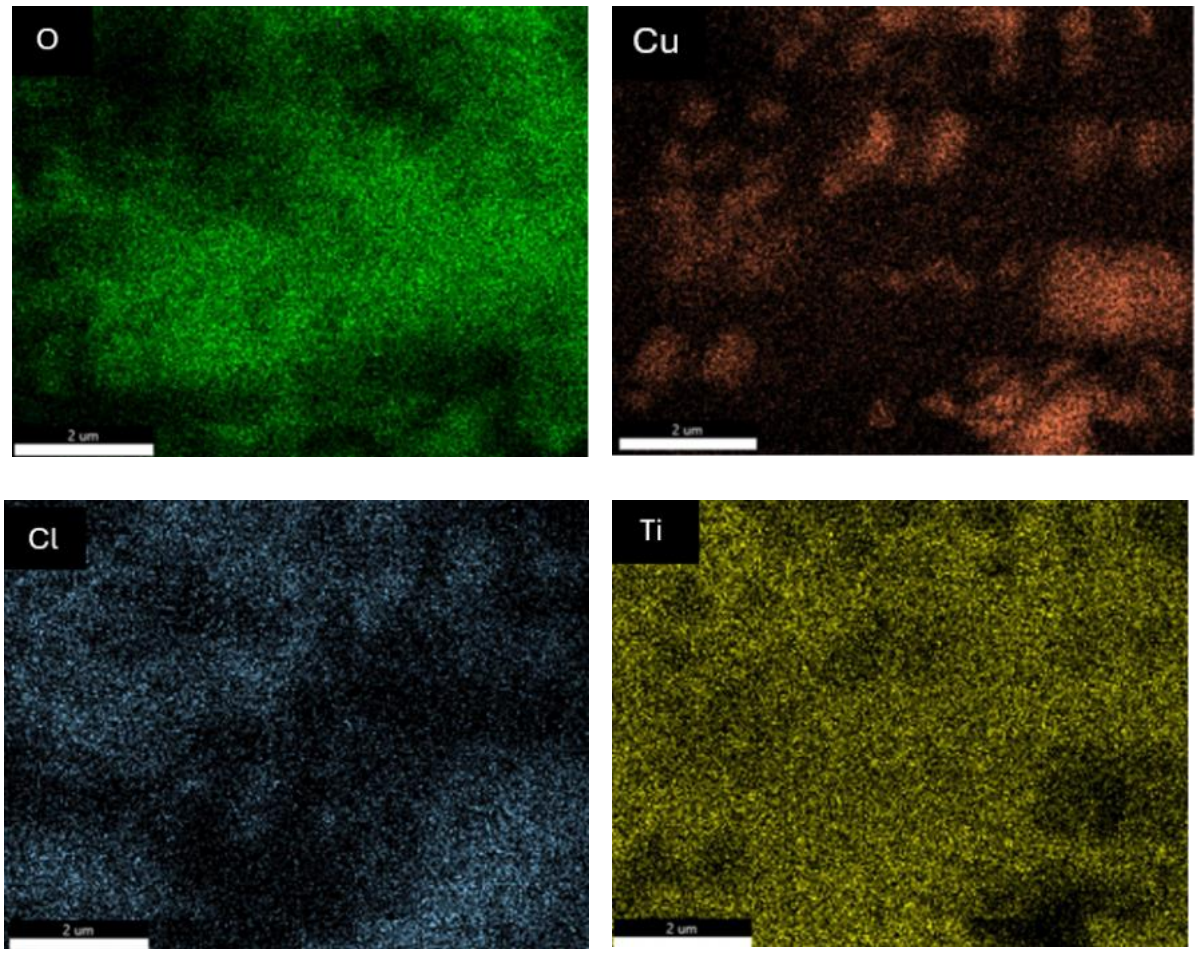


Fig.13 EDS mappings of Ti, O, Cl, Cu in LTO and CL

The element mapping images of the 2 synthesized sample i.e. LTO and CL are displayed in the figure (12) and (13) , in which it can be seen that the green and yellow dot correspond to O and Ti respectively for the case of optimized LTO. The shapes of the dotes quite well resembled the sample CL as shown in fig (12). Displaying the uniform distribution of the elements on the surface of the sample CL and LTO as well. Whereas the element mapping of the element Cl , relatively few dots can be seen indicating the quantity of the substance containing Cl to be very less compared to quantity of the CuO and LTO in case of the sample Cl.

CHAPTER 5

CONCLUSIONS AND FUTURE SCOPE

5.1 CONCLUSION

It is seen that calcining the mixture having LTO and CuCl was a practical way to alter the surface morphologies and particle size of the LTO which is aused commonly as an anode material. With the ease of synthesis of LTO, it is widely used in the industries, thus this work adds on a meaningful contribution to any futuristic development of LTO. In this work LTO has been synthesized initially using precursor's lithium titanium oxide and titanium (IV) oxide via solid state synthesis at 800^0C. Post which the synthesized LTO was then used to prepare a mixture of LTO with CuCl with molar ratio of titanium (Ti) to copper (Cu) equivalent to be 5:2. This specimen was named as CL. Upon further characterization by XRD and EDS it was concluded that the two main components in the prepared specimen CL were LTO and CuO. Then, as shown by the SEM images with a scale of 1μm the synthesized LTO particles had well defined, polyhedral morphology indicating characteristic crystalline spinel structure. With addition of CuCl to LTO in the molar ratio of Ti to Cu (5:2) followed by calcination at 550^0C resulted in considerable change of morphology. The particles no longer were sharp, faceted shape of pure LTO, rather large irregular aggregated were observed. Though the fine granular features visible across particle surface indicated formation of copper-based species most likely Cu_2O or CuO nanoparticles which arrived from the thermal decomposition reaction of CuCl with LTO. Absence of large CuCl particle morphology indicated the conversion of CuCl into other copper containing phases with copper species intimately distributed across the LTO surface.

Overall, calcination of of CuCl and LTO at 550^0C successfully transforms this mixture into a composite material , where Copper- derived nanoparticles were incorporated into/onto the LTO matrix potentially enhancing the electrochemical performance of LTO.

EDS on other also confirmed the presence of two main components in the LTO and CuCl mixture with very low atomic content of Cl indicating possible consumption of Chlorine during the calcination process.

5.2 FUTURE SCOPE

Although the structural profile and surface morphology captured by X-Ray Diffraction (XRD), Scanning electron Microscopy (SEM) reveal about the possible main components in the synthesized sample CL along with changes in the surface morphology of the sample , further research on the electrochemical properties of the synthesized sample are yet to be known to come at a conclusion of implementing this method to enhance the structural and electrochemical properties.

In the previous work, only physical characterization has been done resulting in the change of crystallite size and surface morphologies along with atomic compositions and possible transformation of a component into some other component may be an oxide. Electrochemical tests such as cyclic voltammetry (CV), galvanostatic charge discharge analysis (GCD), electrochemical impedance spectroscopy (EIS) will provide a better insight into the charge-transfer resistance, lithium-ion diffusion kinetics, electrode stability etc. and would help us to know whether the idea of calcinating the mixture containing the CuCl and Calcinated LTO with molar ratio of Ti:Cu to be 5:2 , to enhance the electrochemical properties turned out be favorable or not . Further with advanced structural characterization Techniques such as XPS, TEM, Raman spectroscopy we can further study oxidation state variation, metal ion migration and structural evolution during repeated charge discharge cycle. All of these analysis can provide better insight about the stability of LTO and CuCl mixture as anode for li-ion battery

Future work will involve all the electrochemical analysis such as cyclic voltammetry (CV), galvanostatic charge discharge analysis (GCD) , electrochemical impedance spectroscopy (EIS) for further evaluation on electrochemical properties of the samples synthesized above.

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ANNEXURE-IV

DELHI TECHNOLOGICAL UNIVERSITY

(Formerly Delhi College of Engineering)

Shahbad Daulatpur, Main Bawana Road, Delhi-42

PLAGIARISM VERIFICATION

Title of the Thesis- **Structural and electrochemical investigation of calcined mixture of lithium titanate ($Li_4Ti_5O_{12}$) and copper(I) chloride (CuCl) as an alternative anode material for Li-ion battery.**

Total Pages- **43**

Name of the Scholar - **SATYAM**

Supervisor – **Prof. VINOD SINGH**

Department - **Department Of Applied Physics**

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CERTIFICATE OF FINAL THESIS SUBMISSION

(To be submitted in duplicate)

1. Names: NEHA THAKUR

2. Roll Nos: 24/MSCPHY/44

3. Thesis title: Structural and electrochemical investigation of calcined mixture of lithium titanate ($Li_4Ti_5O_{12}$) and copper(I) chloride (CuCl) as an alternative anode material for Li-ion battery

4. Degree for which the thesis is submitted: Master of Science

5. Faculty: Department of Applied Physics

Thesis Preparation Guide was referred to for preparing the thesis.	YES / NO
Specifications regarding thesis format have been closely followed.	YES / NO
The contents of the thesis have been organized based on the guidelines.	YES / NO
The thesis has been prepared without resorting to plagiarism.	YES / NO
All sources used have been cited appropriately.	YES / NO
The thesis has not been submitted elsewhere for a degree.	YES / NO
All the corrections have been incorporated.	YES / NO
Submitted 2 hard bound copies plus one CD.	YES / NO

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Signatures of Candidates **Signature of Supervisor**