STRUCTURAL, ELECTRICAL AND DIELECTRIC PROPERTIES OF LATP-PEO COMPOSITE ELECTROLYTE FOR LI-ION BATTERIES

A DISSERTATION
SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE AWARD OF THE DEGREE
OF

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IN
PHYSICS

Submitted by:

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CANDIDATE'S DECLARATION

We hereby certify that the work which is presented in the Major Project –II entitled in fulfillment of the requirement for the award of the Master of Science in **Physics** and submitted to the Department of **Applied Physics**, Delhi Technological University, Delhi is an authentic record of our own, carried out during a period from August 2023 to May 2024 under the supervision of **Dr. Amrish K. Panwar.**

The matter presented in this report/thesis has not been submitted by us for the award of any other degree of this or any other Institute/University. The work has been accepted in SCI/SCI expanded/SSCI/Scopus indexed journal OR peer-reviewed Scopus indexed conference with the following details:

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CERTIFICATE

I hereby certify that the Project Dissertation titled "Structural, electrical and dielectric properties of LATP-PEO composite electrolyte for Li ion batteries" which is submitted by, Sonik Chauhan (2K22/MSCPHY/40) and Simran (2K22/MSCPHY/38), Department of Applied Physics, Delhi Technological University, Delhi in partial fulfillment 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.

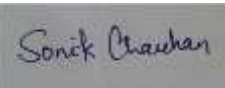Place: Delhi   **Dr. Amrish K. Panwar**

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ABSTRACT

In this study $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$ has been synthesized via solid state reaction route. X ray diffraction has been carried out to confirm the phase formation and XRD peaks are used to calculate the average particle size of the synthesized material. SEM and EDX analysis have been carried out to examine the surface and morphological properties of the material. Further the LATP fillers were incorporated into the polyethylene oxide (PEO) polymer matrix and a thin film has been formed. A.C impedance spectroscopy and dielectric spectroscopy has been carried out at various temperature intervals between 303K and 343K within the frequency range of 25kHz-5MHz. ionic conductivity of the thin film is calculated by Nyquist plot and the activation energy has been calculated by Arrhenius plot. Dielectric studies shows the various phenomenon such as interfacial polarization and space charge polarization.

ACKNOWLEDGEMENTS

Sonik Chauhan

Simran

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CHAPTER 1

INTRODUCTION AND LITERATURE REVIEW

1.1 Introduction

The demand for the renewable energy sources is increasing day by day at a very high rate. The worldwide automotive industry is becoming more electrified in an effort to combat global warming. As a result, the demand of energy storage devices is increasing consequently in recent years the need for high performance rechargeable batteries has grown [1,2]. The development and evolution of the batteries is a reflection of societal improvements in technology. Modern Li ion batteries, solid state batteries and the early electrochemical cells had a significant impact on the industries [3].

1.2 History of Batteries

The first true battery named voltaic pile was discovered by Alessandro Volta in 1800. It had consisted of copper and zinc alternating disc which was separated by the cardboard soaked in saltwater. It showed that electricity could be generated chemically and provided a steady current. Then the lead acid cell as a rechargeable battery was discovered by French physicist Gaston Plante in 1859. In 1866 Georges leclanche developed the leclanche cell which is said to be a precursor to modern dry cell battery. Li ion batteries introduced commercially by 1991 with a variation in technology which allows flexibility and the light weight designs. Batteries are still in

development age and numerous research are going on to increase the energy storage capacity and other useful properties [4].

1.3 COMPONENTS OF BATTERY

There are four main components of batteries:

(1) Anode

(2) Cathode

(3) Electrolyte

(4) Separator

1.3.1 ANODE

Anode is a crucial component in the structure of the rechargeable batteries and due to its structure and characteristics it has significant impact on the overall battery performance [5]. In Li ion batteries some of widely used anodes includes carbon based materials, transition metal oxides and alloy based anode materials [6]. The following reactions occur at anode during charging and discharging in Li ion batteries.

Charging:

$$Li^+ + e^- \longrightarrow Li \tag{1.1}$$

Discharging:

$$Li \longrightarrow Li^+ + e^- \tag{1.2}$$
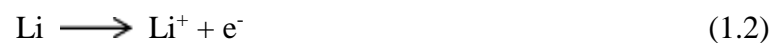
1.3.2 CATHODE

Cathode is also an crucial part for the rechargeable batteries. It plays an important role in developing the efficiency, performance and the overall characteristics of Li ion batteries. The most widely used cathodes in Li ion batteries includes lithium cobalt oxide (LCO), lithium iron phosphate (LFP), lithium manganese oxide (LMO) etc [7,8]. The following reaction occur at cathode during charging and discharging in Li ion batteries.

Charging:

$$Li_{1-x}CoO_2 + xLi^+ + xe \longrightarrow LiCoO_2 \qquad (1.3)$$

Discharging:

$$LiCoO_2 \rightarrow Li_{1-x}CoO_2 + xLi^+ + xe^- \qquad (1.4)$$

1.3.3 ELECTROLYTE

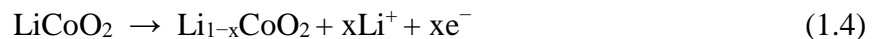Electrolytes serve as a medium for the transport of ions between the anode and cathode during the charging and discharging of rechargeable batteries. The choice of electrolyte directly affects the battery performance including the energy storage capacity, operating voltage, cycle life and other important properties. An electrolyte should exhibits the following properties:

    (1) High ionic conductivity

    (2) Low electronic conductivity

    (3) Thermal stability

    (4) Low viscosity

    (5) Wide operating temperature range

    (6) Chemical compatibility

(7) Non flammable

(8) Cost effectiveness

(9) Low reactivity with electrode material

1.3.4 SEPARATOR

Separator is a physical separation between the anode and the cathode. It also affects the safety and lifespan of batteries, although it does not take part in the electrochemical reaction but allows the ion pass through it [9]. A good separator must have these characteristics:

(1) High ionic conductivity

(2) Good chemical stability

(3) Thermal stability

(4) Uniform thickness and porosity

1.4 LITHIUM ALUMINUM TITANIUM PHOSPHATE ($La_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$)

In this study NASICON type Lithium Aluminum Titanium Phosphate is chosen because of its remarkable properties as a solid ceramic electrolyte. It is a solid state electrolyte which offer advantage over the traditional liquid electrolytes which were used in Li ion batteries. These are the some properties which make LATP useful for the solid state batteries

(1) High ionic conductivity in the range of 10^{-3}-10^{-4} scm^{-1}.

(2) Strong chemical stability.

(3) High oxidation voltage (6V).

(4) Cheap cost of precursors.

(5) Stability in ambient air atmosphere.

However LATP crystallizes in a cubic form called the sodium superionic conductor, or NASICON structure includes MO_6 octahedra and PO_4 tetrahedra. The 3-D Na^+ transporting channel is constructed by the joining of three PO_4 tetrahedra and two MO_6 octahedra linked by the top oxygen atoms. The material converted to Li^+ conductor $LiTi_2(PO_4)_3$ (LTP) after the substitution of M sites and Na atoms by Ti and Li atoms respectively [10-13].
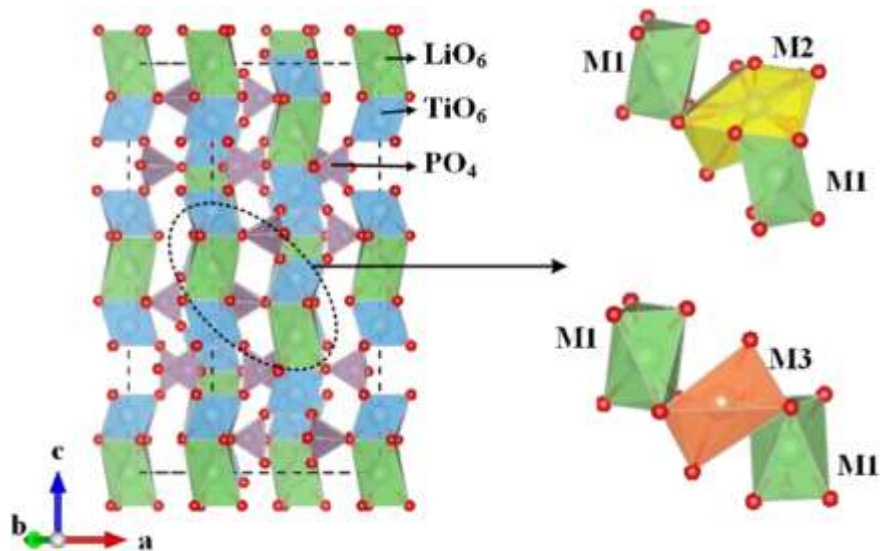

Fig 1.1: Structure of LATP [14].

1.5 POLYETHYLENE OXIDE

Polyethylene Oxide is a polymer electrolyte used widely as a polymer host for inorganic salts to form solid polymer electrolytes for all solid state batteries. It has several drawbacks such as very low ionic conductivity at ambient temperatures, low

mechanical strength, limited electrochemical stability and dendrite growth can still occur under certain conditions [15,16].

However, the incorporation of inorganic fillers to the PEO matrix can overcome these drawbacks and the composite electrolyte can served as a useful electrolyte in the development of all solid state batteries [17,18].

1.6 OBJECTIVE

The objective of the present work is to enhance the properties like ionic conductivity, mechanical stability of PEO polymer electrolyte by the incorporation of LATP ceramic fillers and various characterization such as electrical and dielectric properties of the formed solid state electrolyte.

CHAPTER 2

SYNTHESIS AND CHARACTERIZATION TECHNIQUES

In this chapter we will discuss about the synthesis and characterization techniques which helps us in this work. There are many characterization techniques used to characterize the samples used accordingly to find out the desired properties of materials are explored. Selected techniques are listed in table 1.1.

Table 2.1: Characterization techniques and properties related to them.

Characterization technique	Related properties
Thermogravimetric Analysis (TGA)	Thermal stability, Kinetics of thermal decomposition
X-ray Diffraction (XRD)	Phase formation, Particle size, Strain etc.
Scanning Electron Microscopy (SEM)	Surface structure
Transmission Electron Microscopy (TEM)	Morphology, Defects in structure.
Atomic Force Microscopy	Topography, Magnetic properties, Molecular interactions.

2.1 SYNTHESIS

There are various synthesis routes such as solid state reaction route, sol gel route, hydrothermal route etc which can be used to synthesized the materials according to their desired properties.

In this study we used solid state reaction route to synthesize our material.

2.1.1 MATERIAL SYNTHESIS

Synthesis of LATP has been carried out via solid state reaction route. Stoichiometric amount of precursors listed in table 3.1 were weighed and hand grinding has been done for 10 hrs in pestle mortar. After this the mixture is transferred into crucible and then heated in furnace at 1000° C for 8 hrs. After heating the sample is again grinded and made ready for the further characterizations.

Table 2.2: List of precursors

Precursor	Assay/purity
Li_2CO_3	99%
Al_2O_3	99%
TiO_2	98%
$NH_4H_2PO_4$	99%

2.1.2 SYNTHESIS OF THIN FILM

Synthesis of thin film has been carried out by the solution casting methodology. Stoichiometric amount of LATP is mixed mixed with dimethylformamide and stirred on magnetic stirrer for 6 hrs and then PEO is added to this solution and stirring is continued for another 8 hrs to get a homogeneous mixture. After this the solution is transferred to a petri dish and dried in vacuum oven for 12 hrs at 60° C and the film is obtained.

2.2 CHARACTERIZATION TECHNIQUES

2.2.1 X-RAY DIFFRACTION

X-Ray diffraction is a useful technique used widely to characterize the samples. it tells us about the phase formation, crystal structure, strain, particle size and several other useful properties of a sample. X-ray beams are incident on the sample and the X-ray diffraction peaks are produced by the constructive interference of the x-ray beam diffracted at specific angles from each set of planes in a material.

In order to observe the peaks in X-ray diffractogram the Bragg's law must be satisfied i.e

$$2d \sin\theta = n\lambda \qquad (2.1)$$

Where, d, θ, n, λ are interplanar spacing, angle between X-ray and atomic plane, order of diffracton and wavelength of X-ray used respectively.
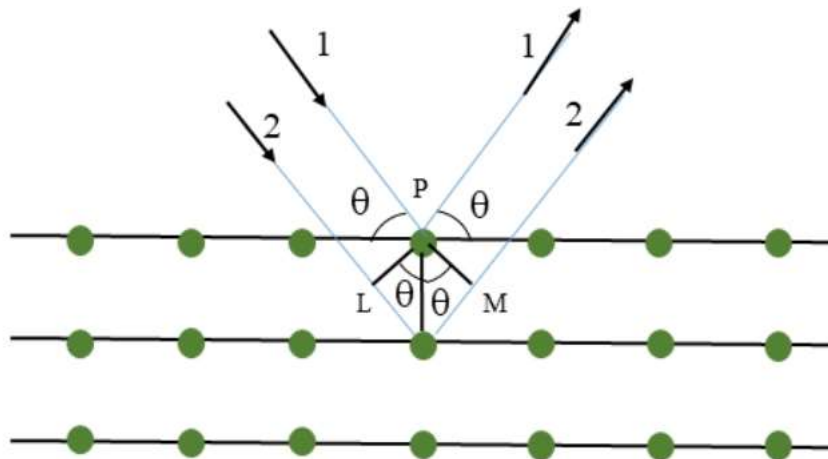

Fig 2.1: Schematic illustration of Bragg's law.

2.2.2 SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy is a powerful tool used to determine the surface and morphological properties of a sample. In this technique a highly focused beam of electrons is made to fall on the sample. There are different interactions occurred as the electron penetrates to the surface of the sample.

Fig 2.2 shows a highly focused beam of electron is made to fall on the sample which results in the ejection of secondary electron, backscattered electrons and the characteristic X-rays. Secondary electrons determines the surface properties and produced as a result of inelastic collision between the electron beam and the specimen electron. Their energy is less than 50 eV. Whereas, the chemical composition is determined by the backscattered electrons that are scattered back from the specimen and their energy is greater than 50 eV.
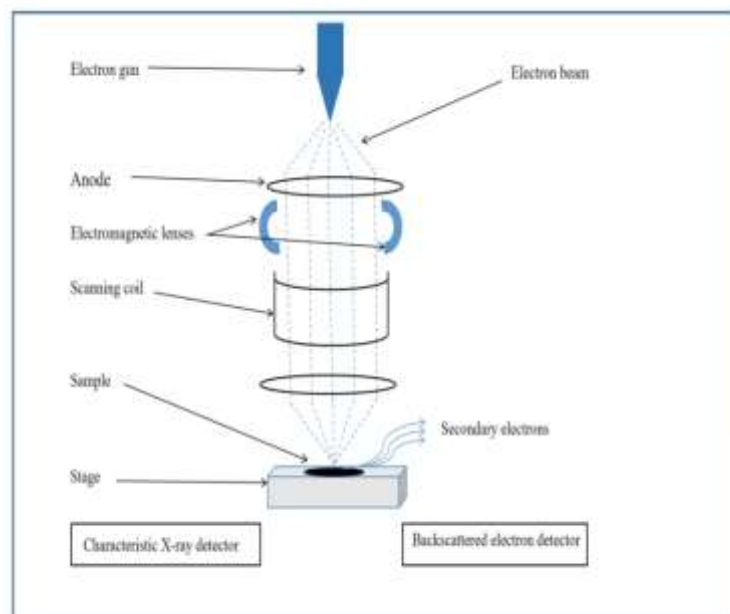

Fig 2.2: Schematic diagram of a SEM apparatus.

CHAPTER 3

RESULT AND DISCUSSION

In this chapter, we will discuss about the xrd analysis of LATP and its structural and morphological properties. After this we will discuss about the AC impedance spectroscopy of the composite electrolyte which reveals its ionic conductivity and the activation energy. Further we will discuss about the dielectric properties of the composite electrolyte.

3.1 XRD ANALYSIS

XRD analysis has been carried out and the observed XRD pattern is shown in fig 3.1. The pattern matches with the pattern (PDF- #035-0754) indicated R-3c space group and the rhombohedral structure. A small peak corresponds to $2\theta = 22^{\circ}$ is also observed which depicts the secondary phase of $AlPO_4$. This is due to the Li ion loss at higher temperature which results in stoichiometric imbalance and Al^{4+} ion of octahedral site restructuralised itself to Al^{3+} ion in stable $AlPO_4$ crystal structure.

The average particle size is also calculated using Debye Scherrer formula which given by:
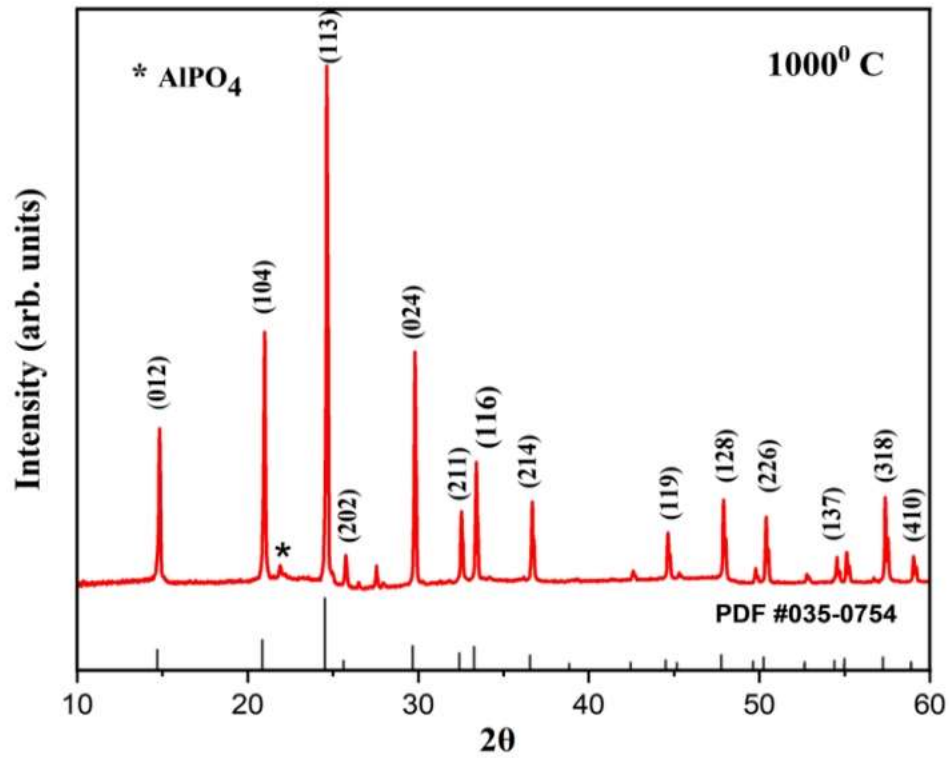
$$D = \frac{k\lambda}{\beta \cos\theta} \tag{3.1}$$

Fig 3.1: XRD pattern of LATP calcined at 1000° C for 8hrs

where D = average particle size, k = 0.9, λ = wavelength of x-ray used, β = full width at half maxima (FWHM) and θ = angle corresponds to peak. The average particle size is calculated to be 71 nm.

3.2 SEM AND EDX ANALYSIS

Fig 3.2 shows the SEM images of LATP calcined at 1000° C for 8hrs. Fig (a) shows the agglomerated structure while Fig (b),(c),(d) shows clear picture of LATP particles but there is no clear trend of shapes is observed. This is due to the high calcination temperature.
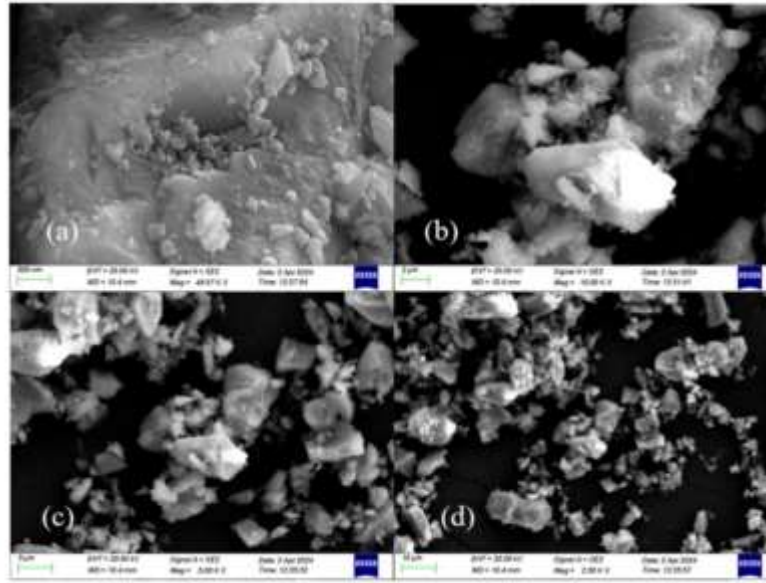
Fig 3.2: SEM images of LATP calcined at 1000° C for 8 hrs

Fig 3.3: (a) shows the elemental distribution is homogeneous whereas a slightly agglomerated phase is observed. Fig 3.3 (b) shows the distribution of P, Fig 3.3 (c) shows the distribution of Ti and Fig 3.3 (d) shows the distribution of Al in the selected region shown in Fig (a).
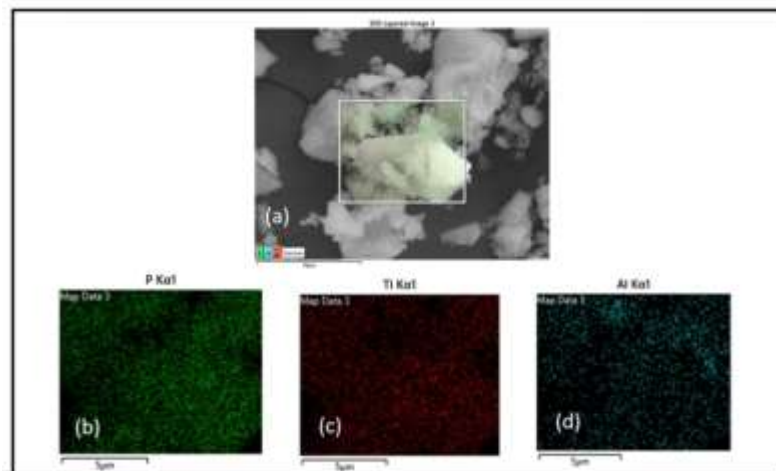

Fig 3.3 : Elemental mapping of LATP calcined at 1000° C fir 8 h

3.3 A.C. IMPEDANCE SPECTROSCOPY

The ionic conductivity of the prepared PEO-LATP thin film is calculated by the equation given below:

$$\sigma = \frac{L}{R * A}$$

$$(3.2)$$

Where σ = ionic conductivity (S cm^{-1}), L = film thickness (mm), R = bulk resistance of thin film (ohm), A = surface area of electrode in contact (mm^2).
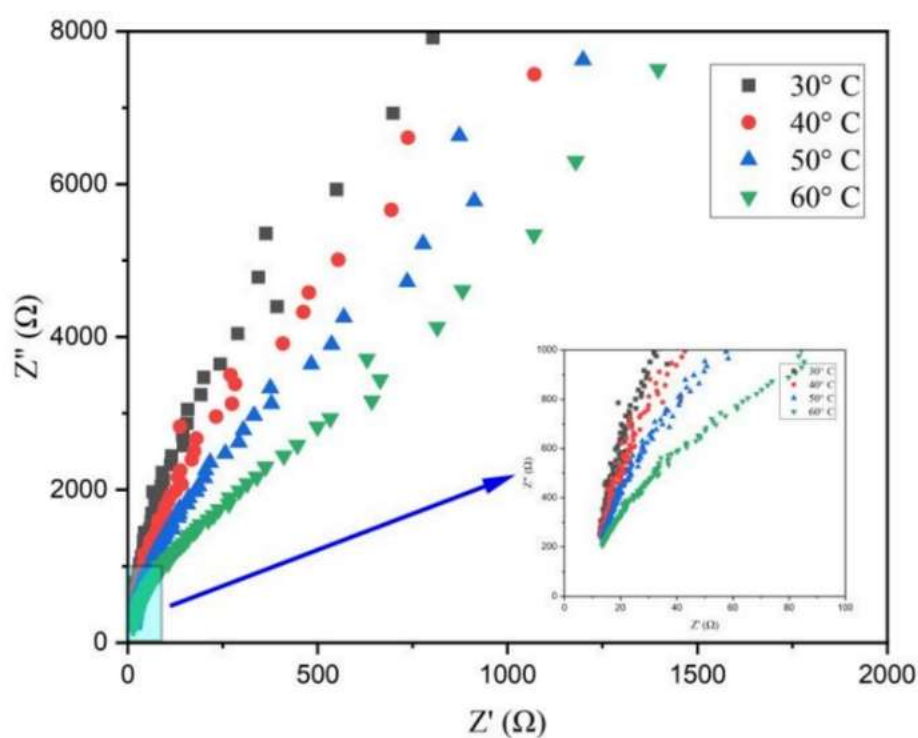

Fig 3.4: Nyquist plot of PEO-LATP thin film.

Fig 3.4 shows the Nyquist plot of PEO-LATP thin film. The values of bulk resistance and ionic conductivity of the thin film at various temperatures has been obtained by this plot and shown in table 4.1.

Table 3.1: Bulk resistance and ionic conductivity of PEO-LATP thin film at various temperature

Temperature (K)	Bulk resistance (Ω)	L/A (cm⁻¹)	Ionic conductivity (S cm⁻¹)
303	54093.78	0.008457	0.156
313	36459.77	0.008457	0.232
323	26608.78	0.008457	0.318
333	12228.11	0.008457	0.691

There is a straight line along with semicircular arcs corresponding to lower and higher frequency regime respectively can be seen in the fig 4.1.[19]
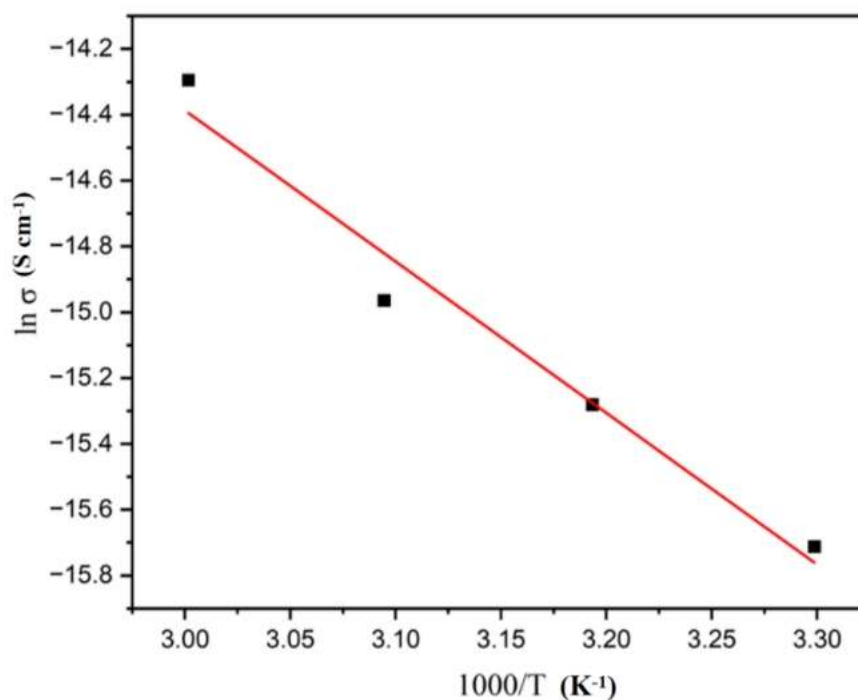

Fig 3.5: Arrhenius curve, variation of conductivity with temperature of PEO=LATP thin film.

The activation energy is calculated from the arrhenius plot shown in fig 3.5. The activation energy of the PEO-LATP thin film is observed as 0.3967 eV.
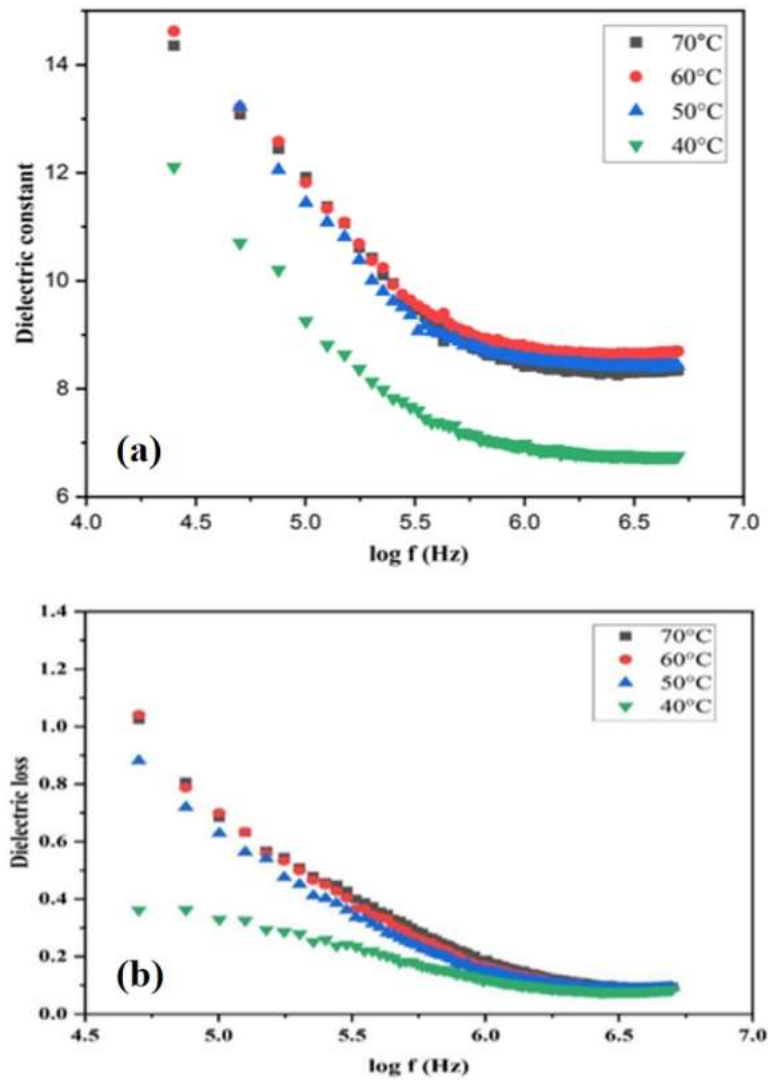
3.4 DIELECTRIC STUDY

Fig 3.6 : (a) Variation of dielectric constant with frequency, (b) Variation of
dielectric loss with frequency

Fig 3.6 shows the change in dielectric constant and dielectric loss at different
temperature 40° C to 70° C in the frequency range 25 kHz-5 MHz.

Fig 3.6(a) shows that the dielectric constant decreases gradually in the lower frequency regime and becomes nearly constant in the higher frequency regime. The observed behaviour of dielectric material can be attributed to various factors such as space, charge, ionic, electronic, dipole polarization. Higher value of dielectric constant in low frequency regime is the result of space charge and dipole polarization. As the frequency increases the oscillations of dipoles lags and dielectric constant decreases gradually. As frequency approaches the higher value, dipoles ceases to flow which results in nearly a constant value of dielectric constant in the higher frequency regime.

Fig 3.6(b) shows the change in dielectric loss with frequency. Dielectric loss is the measure of power loss or the power dissipated in the material under the influence of electric field. The dielectric loss shows higher value in the lower frequency regime and lower or nearly constant value in the higher frequency regime. The creation of interfacial polarization is responsible for this behaviour. At lower frequency the charge carriers approaches and accumulates at the interface easily which results in significant interfacial polarization and high dielectric loss. Whereas, at higher frequency the charge carriers have limited time for their movement, as a result the interfacial polarization and dielectric loss decreases.

Fig 3.7(a) shows the change of dielectric constant with temperature at the selected frequencies. The dielectric constant increases with increase in temperature. The dielectric constant increases significantly at the lower frequencies but at higher frequency the change in almost negligible. This is because there is no charge accumulation at the interfaces at higher frequency and hence there is no interfacial polarization which results in lower value of dielectric constant [20,21].

Fig 3.7(b) shows the change in dielectric loss with temperature. Dielectric loss increases with increase in temperature. As the temperature increases the mobility of charge carriers increases which results in the higher value of the dielectric loss.
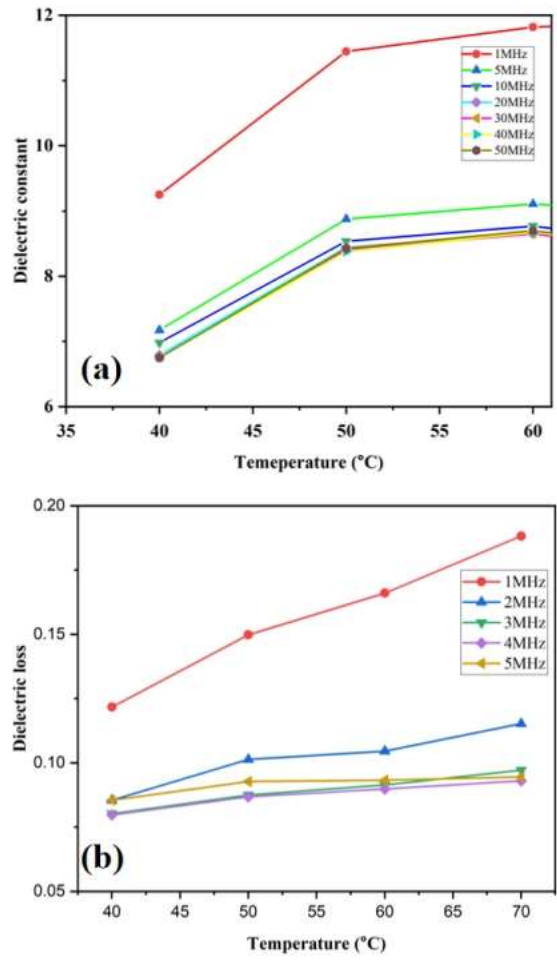
Fig 3.7: (a) Variation of dielectric constant, (b) dielectric loss with temperature at selected frequencies

CHAPTER 4

CONCLUSION AND FUTURE WORK

4.1 CONCLUSION

LATP is successfully synthesized and the XRD pattern matches with the PDF# 035-754 which confirms the R-3c space group and the rhombohedral structure and the average particle size is calculated to be 71 nm. SEM and EDX analysis shows the homogeneous distribution of the elements within the material. After this the LATP ceramic fillers have been successfully incorporated inti the PEO polymer matrix and the impedance and dielectric spectroscopy have been performed. PEO-LATP thin film shows the maximum ionic conductivity of 0.691×10^{-6} Scm^{-1} at 333K and the activation energy is obtained from Arrhenius curve which is calculated to be 0.3967 eV. Dielectric studies shows the variation of dielectric properties with frequency trends can be explained on the basis of phenomenon such as space charge polarization, interfacial polarization.

4.2 FUTURE WORK

In our future work we will try to further increase the ionic conductivity and the electrochemical window of the composite electrolyte and try to test it in a full cell.

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048: "Synthesis and Characterization of Li1.3Al0.3Ti1.7(PO4)3 Based Solid State Electrolyte for Li-ion Battery"

has been accepted for

International Conference on Atomic, Molecular, Material, Nano, and Optical Physics with Applications (ICAMNOP-2023)

Please find the reports beneath.

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REPORT
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Solid state electrolyte (SSEs) development is important for the next generation of electrochemical energy storage. This work may taken further for future research and real demonstration at cell level.

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REPORT
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paper is well written and can be published in present form

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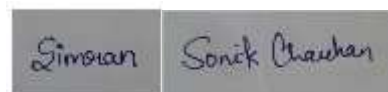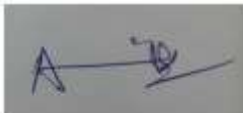|	
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