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Jagadeesan Arumugam

ajagadeesanphysics@gmail.com

Nehru Memorial College

Research Article

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Enhancing the Room-Temperature Ionic Conductivity of PVAc–PEMA–TiO₂ Nanocomposite Polymer Electrolytes via Post-Fabrication Shock Wave Treatment

Jagadeesan Arumugam^{*1}

1. PG and Research Department of Physics, Nehru Memorial College, Puthanampatti, Trichy-621007, Tamilnadu, India.

* corresponding author: Jagadeesan Arumugam. Email: ajagadeesanphysics@gmail.com

Abstract:

The escalating demand for wireless electronics in smart technologies underscores the critical need for advanced solid-state energy storage devices. Solid polymer electrolytes (SPEs) offer a promising, leakage-free alternative to liquid variants in lithium battery systems. Blending complementary polymers, such as poly(vinyl acetate) (PVAc) and poly(ethyl methacrylate) (PEMA), alongside incorporating inorganic nano-fillers serves as a highly effective approach to tailoring their electrochemical, thermal, and mechanical characteristics. In this work, novel nanocomposite polymer electrolyte (NCPE) membranes based on a PVAc–PEMA blend complexed with lithium perchlorate (LiClO₄) and varying weight fractions (0 to 10 wt%) of titanium dioxide (TiO₂) nanoparticles were successfully fabricated via a standard solution casting technique. The synthesized membranes were systematically characterized using Fourier Transform Infrared (FTIR) spectroscopy, X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Differential Scanning Calorimetry (DSC). To overcome the ambient ionic transport limitations of dry solid films, the optimized 10 wt% TiO₂ matrix was subjected to a high-energy post-fabrication shock wave treatment, and its electrochemical behavior was thoroughly evaluated via Electrochemical Impedance Spectroscopy (EIS). FTIR structural analysis confirmed successful intermolecular interactions and complexation within the host polymer blend matrix. XRD studies verified the homogenous dispersion of the TiO₂ nano-fillers within a predominantly amorphous polymer network. Prior to shock wave exposure, SEM micrographs of the cast film revealed a highly dense, smooth surface morphology with limited

superficial micro-porosity, which restricted ambient ion pooling and yielded a baseline room-temperature conductivity of $6.74 \times 10^{-8} \text{ S cm}^{-1}$ (sample JCP5). Thermal analysis via DSC demonstrated exceptional thermal profiles, showing that the electrolyte membranes maintain stable operational baselines up to an elevated decomposition threshold of 240°C. Following exposure to the shock wave treatment, the optimized JCP5 electrolyte membrane exhibited a dramatic morphological and electrochemical transformation. Post-shock SEM analysis revealed the development of a highly porous architecture featuring a well-defined, interconnected micro-pore network. This structural reorganization drastically minimized the energy barriers for segmental chain movement and created rapid "ion highways" across the matrix. Consequently, EIS evaluations showed a massive surge in electrochemical performance, with the room-temperature ionic conductivity skyrocketing by nearly four orders of magnitude to an exceptional value of $1.2559 \times 10^{-4} \text{ S cm}^{-1}$ at 303K. By combining the inherent thermal resilience (240°C) of the dry PVAc–PEMA–TiO₂ framework with an innovative shock wave processing technique, this study successfully addresses the long-standing trade-off between mechanical/thermal stability and ambient conductivity. The resulting highly porous, ultra-conducting solid polymer membrane demonstrates outstanding engineering viability for high-performance, solid-state lithium batteries operating under demanding modern conditions.

Keywords: Nanocomposite polymer electrolyte; PVAc–PEMA blend; Shock wave treatment; High conductivity; Interconnected porosity; Thermal stability.

1. Introduction:

The rapid proliferation of wireless electronic devices across diverse sectors, including smart medicine and intelligent building automation, has dramatically accelerated the demand for continuous and high-peak power supplies. While primary harvesting units like photovoltaic cells and biofuels offer green alternatives, robust energy storage systems remain indispensable for ensuring uninterrupted current delivery. Among various energy storage and conversion technologies, rechargeable batteries—operating via reversible electrochemical reactions—play a dominant and vital role. In particular, solid-state lithium batteries have attracted intensive global research interest as next-generation systems, with substantial focus directed toward developing optimized solid electrolyte materials. Among these, solid polymer composite electrolytes (SPEs) stand out due to their processing flexibility, lightweight nature, and desirable interfacial properties[1]. By strategically blending different phases together, it is possible to design an electrolyte matrix that possesses

synergistic properties tailored for demanding electrochemical applications[2-5]. Poly(ethyl methacrylate) (PEMA) has emerged as an attractive host polymer for ionic conduction, a concept first pioneered by Han et al. and Fahmy et al.[6-7]. Owing to its high transparency, sufficient mechanical strength, excellent elasticity, and robust substrate adhesion, PEMA has been extensively utilized in electrochromic and energy devices, demonstrating superior mechanical integrity compared to poly(methyl methacrylate) (PMMA)[8]. Concurrently, polyvinyl acetate (PVAc) is recognized as an ideal polymer matrix for achieving high room-temperature ionic conductivity when complexed with inorganic salts, primarily due to its excellent mechanical stability, structural flexibility, and ease of film formation[9-10]. Landmark studies by J.Y. Olayemi et al. demonstrated that PVAc forms highly compatible blends with PEMA across a broad composition range, driven by strong intermolecular interactions[11-12]. Notably, a composition featuring 20 wt% PEMA was reported to yield the optimum balance of tensile strength and elongation at break in blended thin films. Maximizing ionic conductivity is a foundational requirement for practical solid polymer electrolytes, prompting researchers to explore diverse structural enhancement methods. Polymer blending represents a highly effective methodology to suppress crystallization and augment ionic transport networks. Typically, polymer matrices are solubilized and cast using volatile solvents to optimize phase distribution[13]. The primary objective of such blending strategies is to yield an electrolyte film with an expanded amorphous volume fraction, which inherently facilitates much faster ion transport than its crystalline counterparts. Furthermore, the dispersion of passive ceramic nanofillers into a polymer matrix has proven highly effective in lowering the glass transition temperature (T_g) and disrupting polymer chain crystallization. The incorporation of these ceramic nanoparticles generates structural defects and induces additional free volume at the filler-polymer chain interface, thereby accelerating ion conduction paths within the amorphous domains[14-18]. In addition to chemical modifications, post-fabrication physical processing techniques have recently gained traction as a powerful tool to alter the microstructural landscapes of polymer frameworks. Specifically, the application of high-energy transient shock waves into solid polymer matrices presents an innovative pathway to mechanically manipulate material morphology. Shock wave propagation induces intense, localized pressure and stress fields capable of forcefully fracturing tightly packed polymer chain arrangements, generating localized defects, and opening up closed microstructures without compromising the core chemical composition of the host matrix. Despite these advancements, a review of the current literature reveals that no data is available regarding composite polymer electrolytes based on the specific PVAc–PEMA–TiO₂ ternary system. To the best of our knowledge, no investigations have explored the combination of a PVAc/PEMA blend matrix reinforced with titanium dioxide (TiO₂) nanoparticles for

lithium-ion conduction, nor has any study evaluated the impact of dynamic shock wave processing on such systems. Motivated by this research gap, the present study details the successful fabrication of a series of PVAc/PEMA–LiClO₄–TiO₂ nanocomposite polymer electrolyte membranes via a controlled solution casting technique. Furthermore, this work investigates the novel application of post-fabrication shock wave treatment on the optimized composite membrane to fundamentally re-engineer its morphology and resolve ambient transport bottlenecks. The resulting structural, thermal, morphological, and electrochemical characteristics are comprehensively evaluated and discussed herein.

2. Experimental details:

Poly(vinyl acetate) (PVAc, Sigma-Aldrich) with an average molecular weight (M_w) $\approx$ 140,000, and high-molecular-weight poly(ethyl methacrylate) (PEMA, Sigma-Aldrich, M_w $\approx$ 515,000), the inorganic dopant salt lithium perchlorate (LiClO₄), and the nano-sized inorganic filler TiO₂ with a particle size of 30 nm (Sigma-Aldrich), and the solvent tetrahydrofuran (THF, Merck Pvt. Ltd.) were used without further purification in this study. The nanocomposite polymer electrolytes were prepared using the solution casting technique.

2.1 Preparation of thin films:

Prior to the preparation of the polymer electrolytes, LiClO₄ was dried at 100°C for 1 hour to remove any trace amounts of water in the material. The incorporation of TiO₂ was expressed as weight ratio and its designation was tabulated in table 1.

Appropriate amounts of PEMA, PVAc, LiClO₄ and nano sized TiO₂ were dissolved in THF. The accuracy of the digital weighing balance was carefully verified beforehand to minimize systematic errors. The solution was then stirred continuously for 24 hours at room temperature and subsequently cast onto a glass Petri dish, where it was allowed to evaporate slowly. Nanocomposite polymer electrolytes were eventually formed in this study, with the resulting membranes having a thickness in the range of 100 to 200 microns.

3. Electrolyte Characterization:

3.1 Fourier Transform Infrared (FTIR) spectroscopy

Infrared spectra exhibited in this work were obtained using FTIR spectrometer (PE IR spectrum ASCII) in the wave number regions between 4000 and 400 cm^{-1} .

3.2 X-ray diffraction (XRD)

The crystallinity or amorphous nature of the blended electrolyte was determined using an X-ray diffractometer (XRD measurement, *.XRDML) with an operating voltage and current of 40 kV and 30 mA, respectively. The X-ray wavelength is 1.5406Å.

3.3 Electrochemical Impedance Spectroscopy (EIS)

The polymer electrolyte films were cut into small disks of 2 cm diameter and sandwiched between two stainless steel electrodes under spring pressure. The impedance of the sample was determined by the complex impedance technique using a BioLogic SP-150 potentiostat over the frequency range from 100 Hz to 1 MHz at temperatures ranging from 303 to 353 K. The resulting impedance plot was used to find the bulk resistance (R_b) value for calculating the ionic conductivity by the equation

$$\sigma = \frac{L}{R_b A}$$

Where σ is the conductivity in Scm^{-1} ,

L- Thickness of the film in cm

R_b - bulk resistance in Ω

A- Surface area of the stainless-steel blocking electrodes in cm^2

The complex permittivity of a system is defined by

$$\epsilon^* = \epsilon' + j\epsilon''$$

Where ϵ' is dielectric constant (the real part of complex permittivity)

ϵ'' is dielectric loss (the imaginary part of the complex permittivity).

Using the measured impedance values, the dielectric constant and dielectric loss can be calculated from the equations.

$$\epsilon' = \frac{Z''}{\omega C_0(Z''^2 + Z'^2)} \quad \epsilon'' = \frac{Z'}{\omega C_0(Z''^2 + Z'^2)}$$

Here Z'' and Z' are the imaginary and real part of the complex permittivity. The ratio of ϵ' and ϵ'' gives loss tangent ($\tan \delta = \epsilon'' / \epsilon'$).

3.4 Scanning Electron Microscope

The SEM images were recorded using a JSM 6610 scanning electron microscope.

3.5 Thermal Analysis

Thermal behavior of the composite polymer membrane was studied using a differential scanning calorimeter (Perkin Elmer Pyris). Each sample was scanned from -20° to 300°C at a heating rate of $10^\circ\text{C}/\text{min}$ under N_2 atmosphere. The crystallinity of the membrane was calculated using the following equation, derived from the DSC curves [19].

$$X_c (\%) = (\Delta H_m / \Delta H_m^\Phi) \times 100,$$

Where ΔH_m^Φ is the crystalline melting heat of pure polymer, and ΔH_m is the heat of fusion of PVAc-PEMA-TiO₂ based membranes. It can be calculated from the integral area of the baseline and each melting curve.

3.6 Shock Wave Treatment Setup and Procedure:

The shock wave treatment presented in this study was carried out utilizing an in-house developed shock tube assembly capable of generating controlled, high-energy transient shock waves. The structural configuration of the shock tube consists of a high-grade polycarbonate tube featuring an inner diameter of 20 mm and an outer diameter of 30 mm. The apparatus is structurally divided into two primary zones: a driver section with a length of approximately 400 mm, and a driven section with a length of approximately 500 mm. Both the driver and driven compartments are securely coupled using a heavy-duty threaded cup arrangement system integrated with an ergonomic handle. This specialized configuration facilitates the rapid and efficient loading of the sacrificial diaphragm between the two high-pressure sections. The photograph of the device used is shown in the Figure 1.

For the post-fabrication shock wave processing, the synthesized composite polymer electrolyte membranes—initially cast within petri dishes—were fixed rigidly against a solid backing wall aligned parallel to the longitudinal axis of the shock tube. The samples were positioned at an optimized distance of

approximately 1 cm from the open exit orifice of the driven section. To generate the shock waves, an art paper diaphragm (90 GSM) was clamped tightly between the driver and driven sections. A high-pressure gas pump connected to the driver section was then engaged to continuously increase the internal pressure until the structural threshold of the paper diaphragm was reached, resulting in a sudden burst. This rapid bursting mechanism creates a sharp, supersonic shock wave that propagates down the driven section toward the sample. This shock wave generation sequence was repeated sequentially by manually replacing the ruptured diaphragm before each pulse. The optimized nanocomposite membranes were subjected to a cumulative exposure of 10 discrete shock impacts. Following the completion of the shock wave processing treatment, the structurally transformed thin films were carefully peeled off from the petri dishes and stored under controlled conditions for subsequent structural, morphological, and electrochemical characterizations.



Fig 1. The photograph of the shock wave tube

4. Results and Discussion:

4.1 AC impedance analysis of NCPE

Ionic Conductivity and Electrochemical Impedance Spectroscopy (EIS) Analysis: The electrochemical transport properties of the prepared solid polymer blend electrolytes were investigated using Electrochemical Impedance Spectroscopy (EIS). The complex impedance technique allows the decoupling of bulk electrolyte resistance from interfacial electrode-electrolyte charging dynamics. The ionic conductivity (σ) of the polymer membranes was evaluated using the standard relationship: $\sigma = l/R_b \cdot A$, where 'l' represents the average thickness of the composite membrane, A is the contact surface area of the blocking stainless steel electrodes, and R_b is the bulk resistance extracted from the high-frequency intercept on the real axis (Z') of the Nyquist plots.

4.1.1 Temperature Dependence and VTF Dynamics:

The variation of ionic conductivity as a function of inverse temperature ($1000/T$) for the host PVAc/PEMA–LiClO₄ matrix integrated with varying weight fractions of passive TiO₂ nanoparticles (0 to 10 wt%, samples JCP0 to JCP5) is presented in Table 1. As observed from the experimental data, the ionic conductivity of all composite configurations scales directly with increasing temperature. This progressive evolution highlights thermally activated behavior, wherein higher thermal energy supplies the necessary kinetic drive to expand the local free volume. This expansion accelerates the segmental motion of the coordinating host chains (PVAc and PEMA), thereby lowering the activation barrier for Li⁺ ions hopping between neighboring coordinating functional groups (carbonyl and ester sites). The non-linear, slightly curved alignment observed in the absolute temperature plots signifies that ion conduction within these dry polymer blends adheres strictly to the Vogel-Tammann-Fulcher (VTF) model rather than standard Arrhenius behavior. The VTF behavior mathematically dictates that ion transport is strongly coupled with the viscoelastic relaxation mechanisms and structural rearrangement modes occurring within the amorphous phases of the polymer matrices. Prior to any dynamic mechanical processing, the un-shocked, optimized 10 wt% TiO₂ formulation (sample JCP5) registered a near-ambient (303 K) conductivity of $6.74 \times 10^{-8} \text{ S cm}^{-1}$, which progressively optimized to $6.26 \times 10^{-5} \text{ S cm}^{-1}$ at an elevated temperature baseline of 353 K. The conductivity values are listed in Table 1.

4.1.2 Enhanced Conductivity via Post-Fabrication Shock Wave Treatment:

To address the transport limitations imposed by the highly dense packing of the solvent-cast films under ambient conditions, the optimized JCP5 composite membrane was exposed to high-energy post-fabrication shock wave pulses. Following this mechanical shock wave exposure, a remarkable electrochemical breakthrough was achieved. The room-temperature (303 K) ionic conductivity of the shock-treated JCP5 membrane surged exponentially to an exceptional value of $1.2559 \times 10^{-4} \text{ S cm}^{-1}$. This represents a near four-orders-of-magnitude leap over its un-shocked ambient baseline ($6.74 \times 10^{-8} \text{ S cm}^{-1}$). The physical and structural mechanism driving this massive enhancement is directly supported by the morphological transitions captured via SEM (Section 4.5). The intense, high-velocity pressure waves break apart the highly dense polymer chain arrangements native to the as-cast THF framework. By forcing a major structural reorganization, the shock wave treatment generates an extensive network of highly porous, interconnected microstructures across the membrane. This structural transformation significantly improves the electrolyte's performance in two key ways: it drastically expands the localized free volume within the amorphous blend

1	jcp0	0	3.00092×10^{-8}	2.45648×10^{-7}	5.48186×10^{-7}	3.37982×10^{-6}	5.54301×10^{-6}	6.48738×10^{-5}
2	jcp1	2	1.7139×10^{-8}	0.773766×10^{-7}	2.49559×10^{-7}	0.398724×10^{-6}	0.476073×10^{-6}	0.13916×10^{-5}
3	jcp2	4	4.23156×10^{-8}	0.852118×10^{-7}	3.73653×10^{-7}	0.552771×10^{-6}	3.14752×10^{-6}	1.48126×10^{-5}
4	jcp3	6	5.32399×10^{-8}	1.11034×10^{-7}	3.9861×10^{-7}	1.19501×10^{-6}	5.9578×10^{-6}	2.68443×10^{-5}
5	jcp4	8	5.89633×10^{-8}	1.27807×10^{-7}	4.18566×10^{-7}	1.20550×10^{-6}	5.97813×10^{-6}	3.52079×10^{-5}
6	jcp5	10	6.74247×10^{-8}	1.47023×10^{-7}	7.29147×10^{-7}	5.35246×10^{-6}	14.5663×10^{-6}	6.25569×10^{-5}

Figure 3 shows the temperature dependent conductivity graph. The figure tells that the conductivity varies systematically with inverse temperature, displaying the characteristic non-linear curvature associated with the VTF framework [20-22].

4.2 Comparative Discussion and Structural Viability

To rigorously evaluate the impact of post-fabrication shock wave processing, the ambient electrochemical performance of the optimized PVAc/PEMA–LiClO₄–TiO₂ nanocomposite polymer electrolyte (sample JCP5) was benchmarked against relevant polymer electrolyte systems documented in recent literature[23-24]. Prior to shock wave treatment, the as-cast JCP5 membrane exhibited an ambient (303K) ionic conductivity of $6.74 \times 10^{-8} \text{ S cm}^{-1}$, which is constrained by the dense, low-porosity matrix generated via standard solution casting. However, following the high-energy shock wave exposure, the room-temperature ionic conductivity skyrocketed to $1.2559 \times 10^{-4} \text{ S cm}^{-1}$. This represents an exceptional leap of nearly four orders of magnitude. A concise comparison positioning the performance of this shock-treated system alongside alternative dry and gel polymer architectures is provided in Table 2.

Table 2: Comparison of ambient ionic conductivity and parameters of related polymer electrolytes.

Polymer Electrolyte System	Filler/Plasticizer Type	Max. Conductivity (σ) at Room Temperature (S cm^{-1})	Operating Window / Key Attribute	Reference
Pure PEMA Matrix	None	8.60×10^{-11}	Restricted baseline ion transport	(Anuar et al., 2012)
PEMA - NH_4SO_3 CF_3	Unfilled Dry Salt Matrix	1.02×10^{-5}	Relies on high-concentration salt doping	(Anuar et al., 2012)
PEMA/PVAc Blend	$\text{Mg}(\text{ClO}_4)_2$	2.93×10^{-4}	High baseline salt dissociation	(Bósquez-Cáceres et al., 2021)
PVDF-HFP:PMMA Matrix	LiClO_4 (7.5 wt. %)	2.83×10^{-4}	Formulated as a Liquid Gel framework	(Gohel & Kanchan, 2018)
PVAc/PEMA- LiClO_4 (This Work)	TiO_2 (10 wt% Nanoparticles)Before Shock Wave Treatment	6.74×10^{-8}	Dense, smooth surface; limited ambient paths	Present Study
PVAc/PEMA- TiO_2 (This Work)	Post-Shock Wave Treatment	1.2559×10^{-4}	Dry, highly porous, stable up to 240°C	Present Study

As clearly demonstrated by the comparative data, the shock-treated JCP5 electrolyte successfully overcomes the classic ambient transport bottlenecks characteristic of dry solid polymer matrices. While pure PEMA hosts exhibit extremely low baseline conductivities ($\sim 8.60 \times 10^{-11} \text{ S cm}^{-1}$), the combination of the amorphous-rich PVAc/PEMA blend network and the tailored mechanical shock wave treatment drives our

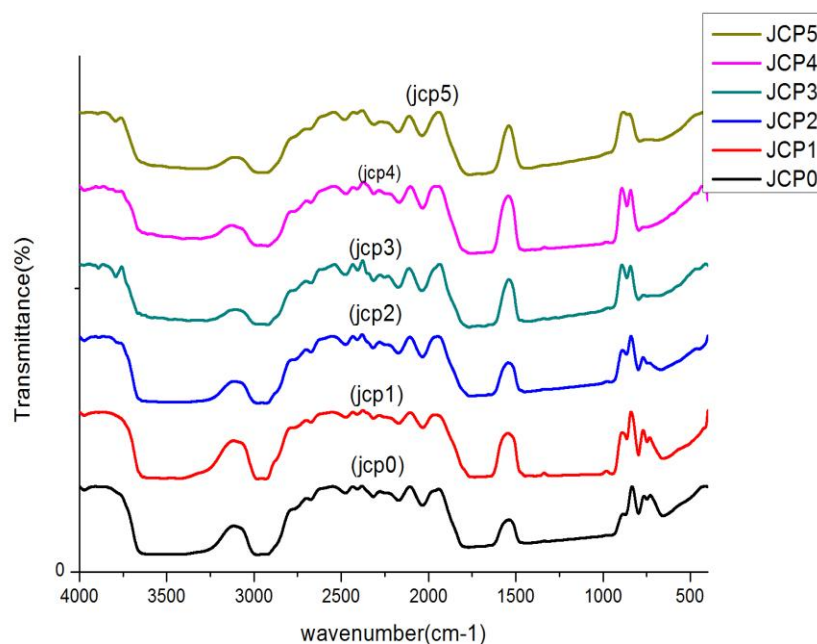
dry electrolyte into the optimal $10^{-4} \text{ S cm}^{-1}$ performance window required for practical solid-state batteries. Remarkably, this conductivity value closely rivals or achieves the same order of magnitude as heavily plasticized or liquid-assisted gel polymer systems, such as the PVDF-HFP:PMMA gel matrix ($2.83 \times 10^{-4} \text{ S cm}^{-1}$). However, liquid-state gel variants inherently suffer from significant drawbacks, including volatile solvent evaporation, poor mechanical rigidity at higher temperatures, and hazardous electrolyte leakage risks under stress[25-26]. In contrast, our shock-treated PVAc/PEMA–TiO₂ electrolyte remains a completely dry thin-film membrane. As proven by the DSC data (Section 4.6), it retains outstanding structural viability and thermal stability up to an elevated thermal degradation threshold of 240°C. The shock wave treatment physically solves the conductivity limitation without requiring chemical additives or volatile plasticizers, ensuring a safe, leak-proof, and high-performance framework perfectly optimized for next-generation solid-state lithium battery configurations operating under highly demanding thermal profiles.

Table 3. Effect of Shockwave treatment on Electrochemical Performance:

Sample Name	Temperature dependent Ionic conductivity values of complexes (S/cm)					
	303K	313K	323K	333K	343K	353K
10wt % of TiO ₂ without shock	6.74247×10^{-8}	1.47023×10^{-7}	7.29147×10^{-7}	5.35246×10^{-6}	14.5663×10^{-6}	6.25569×10^{-5}
10wt% of TiO ₂ with shock	1.2559×10^{-4}	2.9070×10^{-4}	3.17152×10^{-4}	4.85113×10^{-4}	5.321×10^{-4}	5.7124×10^{-4}

4.3 FTIR spectroscopy Analysis:

The vibrational behavior of the prepared polymer electrolytes was studied using FTIR spectroscopy. Figure 4 shows the FTIR spectra of PVAc, PEMA, and salt with inclusion of TiO_2 nano powder. The peaks appearing at 2936 and 1440 cm^{-1} are ascribed to $-\text{CH}_3$ asymmetric and symmetric bending of PVAc respectively. The characteristic peaks appearing at 1762, 949 and 1305 cm^{-1} are ascribed to $\text{C}=\text{O}$ stretching, $\text{C}-\text{O}$ stretching, and $\text{C}-\text{O}-\text{C}$ stretching vibrations of PVAc, respectively. The vibrational peak at 657 cm^{-1} was assumed to be linked with CH_3 ($\text{C}-\text{O}$) group. The broad band at around 2980 cm^{-1} is attributed to the $\text{C}-\text{H}$ stretching in methyl and methylene groups for PEMA. The strong $\text{C}=\text{O}$ stretching is seen at 1737 cm^{-1} and $\text{C}-\text{H}$ bending for $-\text{CH}_3$ and $-\text{CH}_2$ groups appeared at 1447 cm^{-1} . The broad absorption bands around 1305–949 cm^{-1} are ascribed to the $\text{C}-\text{C}(=\text{O})-\text{O}$ and $\text{O}-\text{C}-\text{ester}$ groups. The methyl rocking vibrations can be seen at 955



cm^{-1} [11-12].

Fig: 4

4.4 X-ray measurement:

Figures 5(a) and (b) show the XRD patterns of pure PVAc, pure PEMA, LiClO_4 , TiO_2 and samples with 0, 2, 4, 6, 8 and 10wt% of TiO_2 to polymer matrices. The XRD patterns obtained for the electrolyte films PVAc-PEMA- LiClO_4 - TiO_2 exhibit crystalline peaks at 2θ values 24, 25, 37, 47, 53, etc. indicating the presence of undissolved TiO_2 in the polymer matrix.

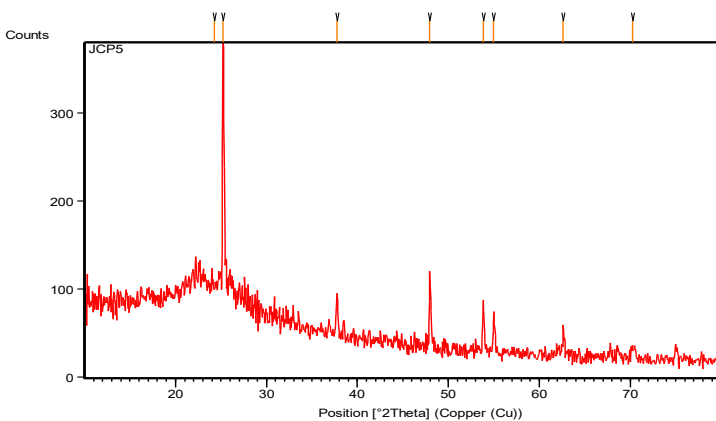


Fig: 5 (a)

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
24.3(2)	21(3)	5.1(4)	3.66534	9.97
25.249(4)	212(14)	0.15(1)	3.52446	100.00
37.73(2)	34(7)	0.20(5)	2.38206	15.98
47.973(8)	79(10)	0.13(2)	1.89485	37.21
53.83(1)	51(8)	0.14(3)	1.70180	24.23
54.99(1)	39(7)	0.17(4)	1.66846	18.58
62.62(2)	23(6)	0.2(1)	1.48227	10.76
70.28(5)	9(3)	0.4(2)	1.33829	4.29

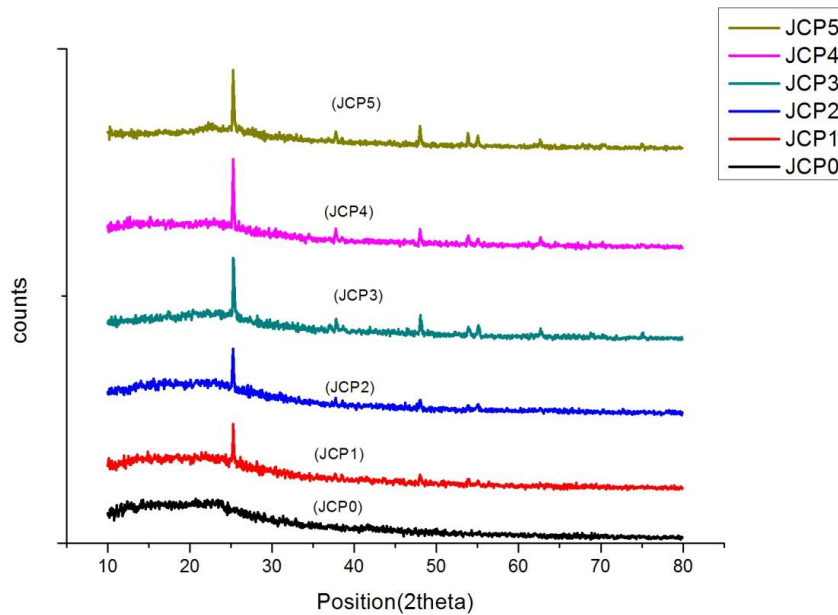


Fig 5(b)

4.5 SEM Analysis of TiO₂:

Scanning Electron Microscopy (SEM) was performed to scrutinize the surface morphology, phase homogeneity, and structural evolution of the prepared nanocomposite polymer electrolytes before and after post-fabrication shock wave exposure. Surface morphology is highly influential in governing the electrochemical performance of solid state electrolytes, as tortuosity, grain arrangement, and porosity dictate the mechanical path of active ionic species. Figure 6 depicts the comparative SEM micrographs of the optimized PVAc(80 wt%)/PEMA(20 wt%) - LiClO₄(8 wt%) - TiO₂(10 wt%) matrix (sample JCP5) under two distinct processing conditions. As shown in Figure 6(a), the surface of the un-shocked, as-cast film is characterized by a remarkably smooth, continuous, and highly dense morphology. The inorganic titanium dioxide (TiO₂) nanoparticles are uniformly distributed throughout the binary poly(vinyl acetate)/poly(ethyl methacrylate) (PVAc/PEMA) blend matrix, without major micro-phase separation or agglomeration[27]. However, this sample exhibits a prominent absence of superficial micro-porosity or interconnected void networks. This compact and tightly packed structural arrangement is an inherent byproduct of traditional solution casting using volatile tetrahydrofuran (THF) carriers. From an electrochemical perspective, this dense film packing severely hinders ambient ion movement; the lack of porous localized channels restricts the accumulation and transport of free charge carriers, validating the modest room-temperature baseline

conductivity ($6.74 \times 10^{-8} \text{ S cm}^{-1}$) initially observed for the un-shocked matrix. In striking contrast, Figure 6(b) illustrates the morphological transformation of the identical JCP5 sample following exposure to the high-energy transient shock wave treatment. The post-shock micrograph reveals a dramatic and extensive structural reorganization, highlighted by the development of a highly porous network filled with well-defined, interconnected micro-pores. The underlying mechanism driving this transformation is attributed to the intense mechanical stress fields and dynamic pressure gradients propagated during the shock wave impact. The sudden thermal and pressure shock forcefully disrupts the tightly packed, dense polymer chains of the PVAc/PEMA blend, cleaving localized crystalline boundaries and creating vast macroscopic free volumes. This dramatic shift from a dense architecture to a highly porous structure is the primary physical catalyst behind the immense jump in electrochemical performance. The newly formed, interconnected pore channels act as low-resistance, high-speed paths (or "ion highways") across the polymer membrane. Concurrently, the elevated porosity and expansion of local free volume drastically lower the energy barriers inhibiting the segmental motion of the polymer host chains. This facilitates rapid, unhindered migration of dissociated lithium ions (Li^+), providing robust physical evidence that correlates with the near four-orders-of-magnitude surge in room-temperature ionic conductivity to a final value of $1.2559 \times 10^{-4} \text{ S cm}^{-1}$.

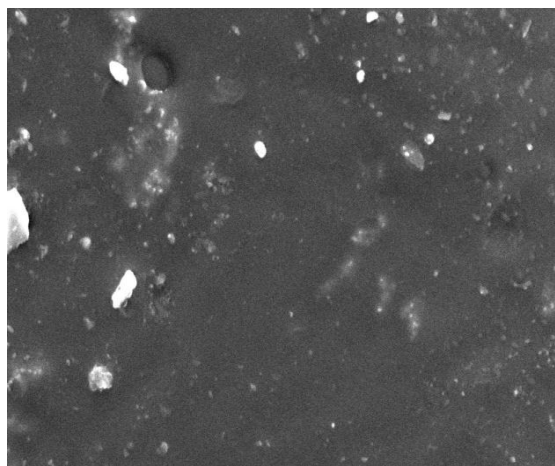


fig 6a

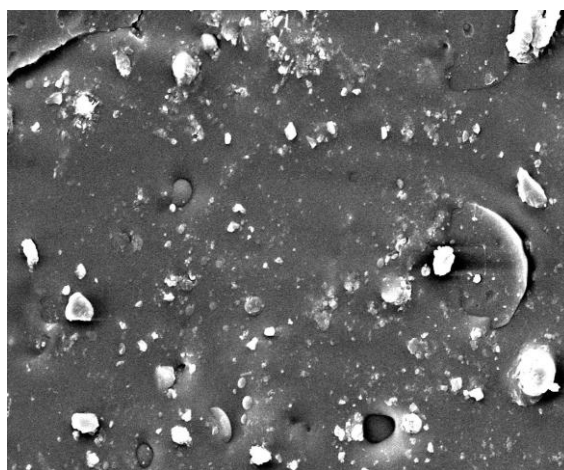


fig 6b

Fig: 6a and b: SEM Images of PVAc-PEMA-LiClO₄-TiO₂ (10 wt%) without and with shock treatment

4.6 DSC Analysis:

The thermal behavior of the prepared films was investigated by DSC, as shown in Figure 7, and the percentage crystallinity was evaluated accordingly. The first thermal anomaly observed at approximately 30 °C corresponds to the glass transition temperature (T_g) of the PVAc phase within the blend network [28]. The second endothermic peak appears at 96°C. The first exothermic is at 280°C. This peak corresponds to the decomposition of the polymer electrolyte along with other active material in contact with it [29]. The decomposition process starts at 240°C. These results show that the composite polymer (PVAc-PEMA- LiClO_4 - TiO_2) can be safely used at below 240°C.

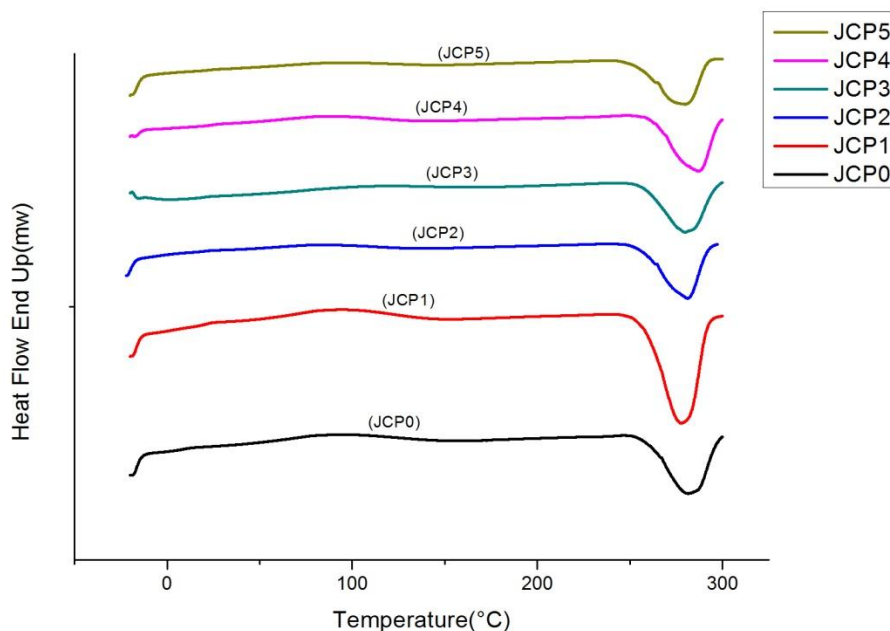


Fig: 7

5. Conclusion:

In this study, novel nanocomposite solid polymer electrolyte (SPE) membranes based on a poly(vinyl acetate)/poly(ethyl methacrylate) (PVAc/PEMA) blend complexed with lithium perchlorate (LiClO_4) and reinforced with titanium dioxide (TiO_2) nanoparticles were successfully fabricated via a scalable solution casting technique. Fourier Transform Infrared (FTIR) spectroscopy verified the successful blending and complexation of the active ionic species with the host polymer functional groups, while X-ray diffraction (XRD) mapping confirmed the homogenous distribution of the TiO_2 ceramic fillers within a

predominantly amorphous matrix. Differential Scanning Calorimetry (DSC) thermograms substantiated the outstanding thermal resilience of the thin-film electrolytes, demonstrating operational stability up to an elevated decomposition threshold of 240°C. Electrochemical Impedance Spectroscopy (EIS) evaluations showed that the ionic conductivity obeys the non-linear Vogel-Tammann-Fulcher (VTF) framework, asserting that ion transport is fundamentally coupled with the segmental motion of the polymer blend host chains. Prior to post-fabrication modification, the optimized 10 wt% TiO₂ composition (sample JCP5) yielded a room-temperature ionic conductivity of $6.74 \times 10^{-8} \text{ S cm}^{-1}$, which was structurally constrained by a highly dense, low-porosity surface morphology native to the solvent casting process. To overcome this transport bottleneck, the optimized JCP5 electrolyte was subjected to high-energy post-fabrication shock wave treatment. This innovative processing technique triggered a profound morphological and electrochemical transformation. Post-shock Scanning Electron Microscopy (SEM) analysis revealed the formation of an extensive network of highly porous, interconnected microstructures. This induced microporosity drastically reduced the mechanical barrier for polymer chain segmental relaxation and established low-tortuosity "ion highways" across the matrix. Consequently, the ambient (303 K) ionic conductivity surged by nearly four orders of magnitude, reaching an exceptional value of $1.2559 \times 10^{-4} \text{ S cm}^{-1}$. By successfully pairing the high thermal stability (240°C) of dry polymer architectures with the excellent room-temperature ionic conductivity of liquid gel frameworks—without requiring hazardous or volatile liquid plasticizers—this shock wave-treated PVAc/PEMA–LiClO₄–TiO₂ ternary composite electrolyte presents outstanding engineering viability for high-performance, leakage-free, solid-state lithium battery applications.

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Data Availability:

Data will be made available on request

Conflicts of interest:

The authors declare that there are no conflicts of interest.

Declaration of competing interest:

The author declares that he has no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author contributions:

Jagadeesan Arumugam: investigation, visualization, Methodology, writing, review and editing.

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