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# Morphological Characterization Study of a Hot-Pressed Synthesized Polymer Nanocomposite Electrolyte Membrane (1-X)[70PEO:30AgCl]:X $Ag_2SO_4$

***Arti Verma***

*Department of Physics, Late Bindeshwari Baghel Government College Kumhari, District Durg, Chhattisgarh, India*

*Email: arti.verma577@gmail.com*

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**ABSTRACT**

The present work reports the synthesis and morphological characterization of silver ion conducting polymer nanocomposite electrolyte membranes of composition (1-X)[70PEO:30AgCl]:X$Ag_2SO_4$ prepared by hot press technique. Poly(ethylene oxide) (PEO) was used as host polymer while AgCl and $Ag_2SO_4$ were used as ionic dopants and nanofillers. Structural and morphological investigations were carried out using X-ray diffraction (XRD) and scanning electron microscopy (SEM). XRD results revealed a reduction in crystallinity and enhancement in amorphous nature due to strong polymer-salt interaction. SEM micrographs confirmed homogeneous surface morphology and uniform dispersion of nanofiller particles. The prepared membranes exhibit improved structural stability and are suitable for solid-state electrochemical device applications.



## Introduction

Solid polymer electrolytes (SPEs) have gained considerable importance in recent years due to their potential applications in solid-state batteries, fuel cells, electrochromic devices, supercapacitors, and chemical sensors [1]. Compared with conventional liquid electrolytes, polymer electrolytes offer several advantages such as flexibility, lightweight structure, leak-proof operation, improved thermal stability, and better safety characteristics [2]. These properties make SPEs promising candidates for next-generation energy storage and conversion devices.

Among various host polymers, poly(ethylene oxide) (PEO) has been widely investigated because of its excellent salt solvating capability and ability to form stable complexes with inorganic salts [3]. The ionic transport in PEO-based electrolytes mainly occurs through segmental motion of polymer chains. However, pure PEO exhibits low ionic conductivity at room temperature due to its highly semi-crystalline nature [4]. Therefore, reduction in crystallinity and enhancement of amorphous regions are essential for improving ionic mobility.

Several approaches have been adopted to enhance ionic conductivity in polymer electrolytes, including salt complexation, plasticization, blending, and incorporation of ceramic nanofillers [5]. Silver ion conducting polymer electrolytes are particularly attractive due to their applications in electrochemical cells and sensors [6]. AgCl acts as an effective ionic dopant, while $Ag_2SO_4$ functions as a nanofiller that modifies structural and morphological properties of the polymer matrix.

The hot press technique has emerged as a simple, solvent-free, and environmentally friendly method for preparation of polymer electrolyte membranes [7]. This method provides uniform dispersion of salts, improved mechanical stability, and homogeneous membrane formation.

In the present work, polymer nanocomposite electrolyte membranes of composition (1-X)[70PEO:30AgCl]:X$Ag_2SO_4$ were synthesized using hot press technique and characterized using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The effect of $Ag_2SO_4$ concentration on structural and morphological properties has been systematically investigated.

**Experiment and Synthesis**

High molecular weight PEO, AgCl, and $Ag_2SO_4$ were used for the preparation of polymer nanocomposite electrolyte membranes. Required quantities of precursor materials were weighed accurately and mixed thoroughly in an agate mortar for approximately 30 minutes to achieve homogeneous mixing.

The mixed composition was heated near the melting temperature of PEO (~70°C) until a viscous slurry was formed. The material was then transferred between two stainless steel plates and compressed under a pressure of approximately 1.25 ton/cm² using a hydraulic hot press machine. Free-standing thin electrolyte membranes were obtained after cooling at room temperature.

Table 1: Composition of polymer nanocomposite electrolyte membranes

| Sample | Composition | Description |
|---|---|---|
| S1 | Pure PEO | Host polymer |
| S2 | (1-X)[70PEO:30AgCl]:0.02$Ag_2SO_4$ | Low filler concentration |
| S3 | (1-X)[70PEO:30AgCl]:0.05$Ag_2SO_4$ | Moderate filler concentration |
| S4 | (1-X)[70PEO:30AgCl]:0.10$Ag_2SO_4$ | High filler concentration |
| S5 | (1-X)[70PEO:30AgCl]:0.15$Ag_2SO_4$ | Maximum filler concentration |

**Characterization Techniques**

X-ray diffraction (XRD) analysis was carried out to investigate structural changes and crystallinity of the synthesized membranes. SEM analysis was performed to examine surface morphology and dispersion of nanofiller particles within the polymer matrix.

**Results and Discussion**

The XRD patterns of pure PEO and polymer nanocomposite electrolyte membranes reveal significant structural modifications after incorporation of AgCl and $Ag_2SO_4$. Pure PEO shows sharp diffraction peaks near $2\theta \approx 19°$ and 23°, indicating its semi-crystalline nature. With increasing concentration of $Ag_2SO_4$, the intensity of diffraction peaks decreases and peak broadening occurs.

This behavior indicates suppression of crystalline domains and enhancement of amorphous regions within the polymer matrix.

The reduction in crystallinity is attributed to strong interaction between ether oxygen atoms of PEO and silver ions. Increased amorphous nature facilitates segmental motion of polymer chains and improves ionic mobility.

SEM micrographs provide important information regarding surface morphology of the synthesized electrolyte membranes. Pure PEO exhibits large crystalline flakes and rough surface morphology. After incorporation of AgCl and $Ag_2SO_4$, the surface becomes smoother and more homogeneous.

Uniform distribution of nanofiller particles within the polymer matrix indicates good compatibility between the polymer and salts. At higher filler concentrations, the membranes exhibit granular surface texture with reduced phase separation. Such homogeneous morphology enhances ionic transport and mechanical stability of the electrolyte membranes.

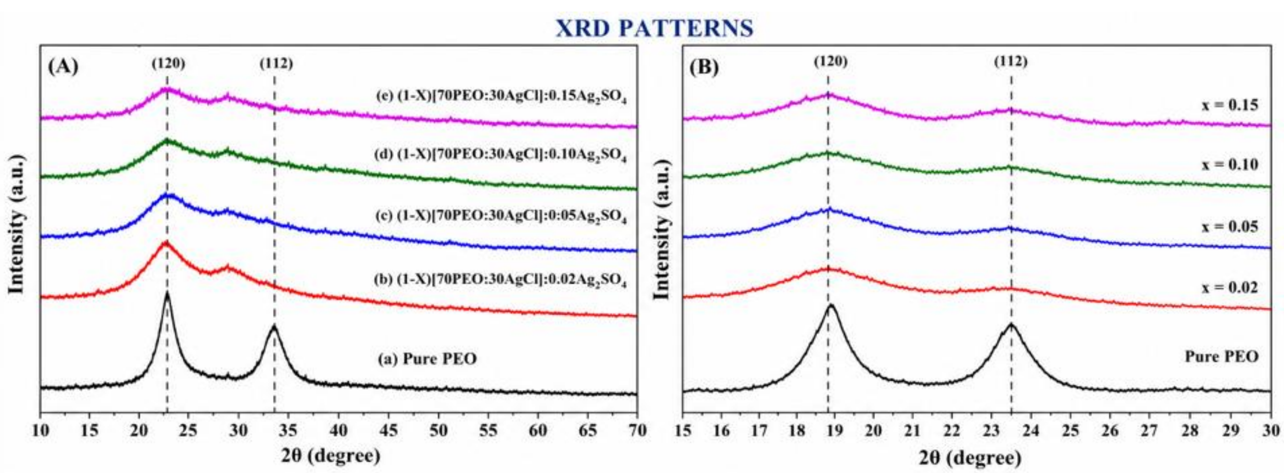

**Figure 1.** (A) Wide angle XRD patterns of pure PEO and (1–X)[70PEO:30AgCl]:X$Ag_2SO_4$ (X = 0.02, 0.05, 0.10 and 0.15) nanocomposite electrolyte membranes. (B) Magnified view of the 2θ range 15°–30° showing (120) and (112) diffraction peaks.

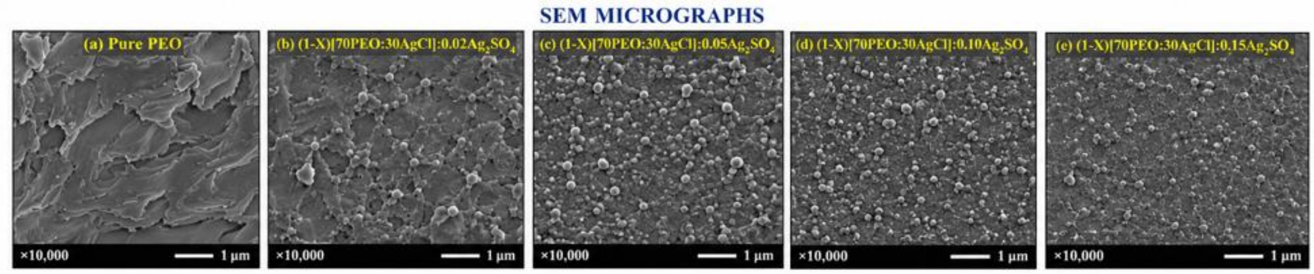


**Figure 2.** SEM micrographs (magnification ×10,000; scale bar = 1 μm) of (a) pure PEO and (b–e) (1−X)[70PEO:30AgCl]:$XAg_2SO_4$ (X = 0.02, 0.05, 0.10 and 0.15) nanocomposite electrolyte membranes.

Table 2: Summary of XRD and SEM observations

| Technique | Observation | Inference |
|---|---|---|
| XRD | Peak broadening and reduced intensity | Reduction in crystallinity |
| XRD | Enhanced amorphous phase | Improved ionic mobility |
| SEM | Smooth homogeneous surface | Uniform salt dispersion |
| SEM | Granular morphology | Enhanced polymer-salt interaction |

**Conclusion**

Hot press synthesized polymer nanocomposite electrolyte membranes of composition (1-X)[70PEO:30AgCl]:$XAg_2SO_4$ were successfully prepared and characterized. XRD studies confirmed reduction in crystallinity and enhancement in amorphous nature with increasing $Ag_2SO_4$ concentration. SEM analysis revealed homogeneous morphology and uniform nanofiller dispersion. These features make the prepared membranes suitable for solid-state electrochemical applications.

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