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X-RD ANALYSIS & THERMAL PROPERTIES OF PVC MICROPOROUS POLYMER MEMBRANE ELECTROLYTE

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ABSTRACT

A microporous polymer electrolyte membrane based on poly(vinyl chloride) (PVC) and poly(vinyl alcohol) (PVA) was successfully fabricated using the solution-casting & polymer dissolution technique and subsequently impregnated with $LiClO_4$ electrolyte. The structural, thermal, and electrochemical properties of the prepared membranes were systematically investigated using X-ray diffraction (XRD), thermogravimetric analysis (TGA), differential thermal analysis (DTA), XRD analysis revealed a reduction in crystallinity and an increase in the amorphous phase following electrolyte incorporation, facilitating enhanced lithium-ion transport. Thermal characterization demonstrated that the membrane possesses excellent thermal stability with a multistage degradation profile, making it suitable for electrochemical applications.The synergistic combination of PVC and PVA provides desirable mechanical integrity, thermal stability, and electrochemical performance, indicating that the developed microporous polymer electrolyte membrane is a promising candidate for lithium-ion batteries, electrochemical sensors, and other solid-state energy storage devices. Recent studies have similarly highlighted that improving the amorphous phase and polymer–salt interactions enhances the thermal stability in polymer electrolyte systems.

KEYWORDS: *Microporous polymer membrane, XRD, TGA/DTA.*

I. INTRODUCTION

In the recent years, there has been a tremendous interest in the preparation of polymer electrolytes with high ionic conductivity, good mechanical strength and thermal stabilities because these polymer electrolytes play a major role in solid-state batteries, electrochromic windows, sensors, fuel cells etc[1-2]. Poly (vinyl chloride) (PVC) can act as a mechanical stiffener in the electrolyte due to its immiscibility with the plasticizer. A PVC-based polymer electrolyte system plasticized with ethylene carbonate and propylene carbonate has been reported to be applicable to lithium metal and lithium ion secondary batteries[3].

These plasticized polymer electrolytes could show a room temperature ionic conductivity of the order of 10^{-3} S/cm. However these polymer electrolytes could not completely satisfy the requirements on properties such as good mechanical strength, high ionic conductivity, tensile strength and good abrasion resistance [4]. Microporous polymer membrane electrolytes can be prepared by conventional phase inversion technique. However, in this process, due to the use of large amounts of expensive, harmful and flammable solvents, contamination of the porous membrane may take place by the residual solvents. Moreover, preparation of MPEs by conventional phase inversion technology is costly and time consuming [5-6].

Hence in the present investigation we have developed a simple, reliable, time saving and industrially feasible process for the fabrication of microporous polymer membrane. PVC based microporous membrane was prepared by extracting PVA from PVC-PVA blend film by using simple dissolution of PVA in DMSO. Moreover these membranes can be easily detached from the substrate and could be used for Li battery applications or many other applications depending upon the type of dopant to be used. The membrane morphology was investigated by XRD analysis. The polymer PVA is a quick dissolver and it can be easily blended with other polymers, while PVC has been chosen because of its high resistance to oxidation, heat released in burning is considerably low hence PVC membranes are suitable for safety reasons in products close to people's daily lives[7-9].

II. Experimental descriptions

2.1. Materials and Methods

PVA with an average molecular weight (1,15,000) and PVC with an average molecular wt. (2,33,000) (Aldrich USA) were used without further purification for the preparation of microporous membranes. The different compositions of PVC based PVC-PVA polymer membrane were prepared by dissolving PVA in required quantity of DMSO and PVC in DMF, these solutions were stirred well for half an hour until the complete dissolution of PVA

in DMSO and PVC in DMF have been achieved. Both the solutions were added together and was stirred well and heated continuously at 50 °C for half an hour until the mixture becomes a homogeneous viscous solution. The viscous solution was poured on a horizontally leveled glass plate to form the films of required thickness. Finally the films were dried at 80 °C in vacuum oven for 3 hours to remove any further traces of DMSO and DMF. The above prepared cast polymer films were immersed in DMSO for the preferential dissolution of PVA to get uniform microporous membranes of PVC. The thickness of the formed membrane was between the ranges of 195-200 μm. Porosity % and weight uptake of different compositions of PVC based microporous membrane increases with increase in PVA content. The liquid electrolyte used in this study was 1 M $LiClO_4$ in 1:1 (v/v) mixture of propylene carbonate (PC) and diethyl carbonate (DEC). Weight uptake of PVC-PVA blend microporous membranes were measured as a function of soaking time in liquid electrolyte to activate porous polymer membranes for 8 h and calculated as follows [10].

$$\text{Weight uptake (\%)} = (W_1 - W_0)/W_0 \times 100$$

Where, W_1 and W_0 are the weight of the wet and dry polymer membranes, respectively. Porosity of PVC membranes was measured by immersing the membrane into 1-butanol for 2 h and calculated using the following equation [11]:

$$\text{Porosity (\%)} = (W_1 - W_0)/\rho V \times 100$$

Where, W_1 and W_0 are the weight of the wet and dry polymer membranes, respectively. V is the apparent volume of the membrane and ρ is the density of 1-butanol.

For thermal analysis TGA/DTA equipment was used at a scanning rate 10 °C min^{-1} to find thermal stability of microporous polymer membrane. Moreover these membranes can be easily detached from the substrate and could be used for Li battery applications or many other applications depending upon the type of dopant to be used. The membrane morphology & crystallinity of microporous polymer membrane was investigated by XRD analysis.The X-ray diffraction measurement was carried out using PHILLIPS Holland. XRD system PW 171 diffractometer employed with Cu-anode operated at 30kV/15mA.

III. RESULT & DISCUSSION

3.1. X-ray diffraction analysis

In order to investigate the influence of Lithium salt concentration on the micro-porous polymer membrane, XRD examinations were conducted by soaking the micro-porous polymer membrane in 1M $LiClO_4$, 1.5M $LiClO_4$, 3 M $LiClO_4$ and 3.5M $LiClO_4$ electrolyte solution. The XRD pattern of the PVC based micro porous polymer membrane & microporous polymer membrane soaked in 1.5 M $LiClO_4$ and 3 M $LiClO_4$ electrolyte solution are given in fig.1(a-c). Following observations were made from XRD pattern.s

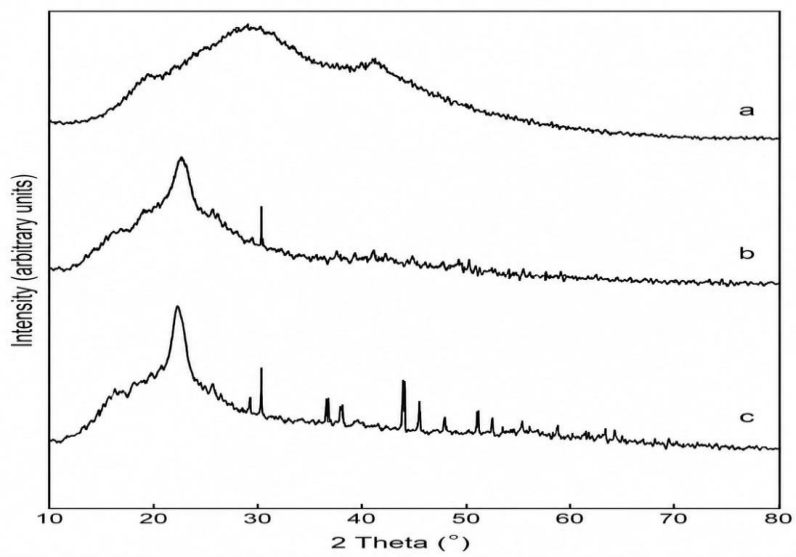

Figure. 1 XRD pattern for (a) micro-porous polymer membrane (b) micro-porous polymer membrane soaked in 1.5 M Liclo4 and (c) micro-porous polymer membrane soaked in 3 M Liclo4 electrolyte solution.

The intensity of the diffraction peaks of PVC based micro-porous polymer membrane increases with increase in salt concentration. This shows a increase in crystalline phase of the micro porous polymer electrolyte. It may be due to the incorporation of LiClO4, which would have disturbed the amorphous phase and increased the crystallanity.

Diffraction peak of PVC based micro-porous polymer membrane is displaced from 2θ =29.1° to 2θ =22.2°, when the polymer membrane is soaked in Li-salt electrolyte solution. It confirms the complex formation between polymer membrane & Li-salt [12].

In addition, further increase in salt concentration (3 M $Liclo_4$) increases the crystalline nature of the polymer electrolyte, which may be due to undissociation of Liclo4 salt, which is evident from fig.(1). The appearance of sharp diffraction peaks confirms the presence of crystalline domains in the polymer electrolyte, Above XRD analysis revealed a reduction in crystallinity and an increase in the amorphous phase following electrolyte incorporation,

facilitating enhanced lithium-ion transport. The changes in diffraction patterns confirmed effective polymer–salt complexation and demonstrated that salt concentration significantly influences the structural characteristics and crystallinity of the membrane [13].

3.2. Thermal analysis:

3.2.1 Thermo Gravimetric Analysis (TGA):

TGA is one of the powerful tools to determine the thermal stability of polymer and its composites. For lithium battery, thermal stability of the porous polymer membrane is an important parameter to guarantee acceptable performances of the membranes in such applications. TGA/DTA analyses of PVC microporous membrane are shown in Figure 2 and Figure 3.

The TGA thermogram (Figure 2) revealed a multistep degradation pattern, indicating the typical thermal decomposition behavior of the PVC/PVA polymer blend. The initial weight loss observed below 100–120 °C is attributed to the evaporation of physically adsorbed moisture and residual solvent retained within the porous membrane structure. This minor weight loss confirms the effective removal of volatile components during membrane preparation.

The second stage of degradation, occurring in the temperature range of approximately 200–350 °C, is associated with the dehydrochlorination of PVC and the degradation of PVA chains. During this stage, cleavage of the polymer backbone and elimination of HCl from PVC contribute to the major weight loss. The incorporation of $LiClO_4$ promotes polymer–salt interactions, which influence the thermal decomposition pathway while preserving the structural integrity of the membrane.

A final decomposition stage above 350 °C corresponds to the degradation of the remaining polymer backbone and oxidation of the carbonaceous residue, resulting in complete thermal decomposition. [14-15].

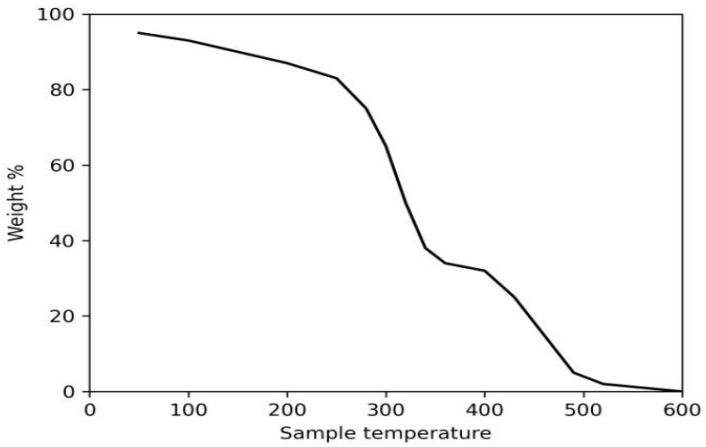

Figure 2. TGA curve for PVC based microporous polymer membrane.

3.2.2 Differential Thermal Analysis (DTA)

The DTA thermogram exhibits characteristic endothermic and exothermic transitions around 250 °C corresponding to moisture evaporation, polymer chain rearrangement, and decomposition reactions. The absence of significant thermal events below the operating temperature indicates that the membrane possesses sufficient thermal stability about 280 °C for electrochemical applications.

Furthermore, the enhanced thermal stability can be attributed to strong intermolecular interactions between PVC, PVA, and $LiClO_4$, which restrict polymer chain mobility and improve the overall stability of the polymer matrix. The porous morphology of the membrane facilitates efficient electrolyte uptake without significantly compromising its thermal resistance. These observations are consistent with recent studies, which have reported that lithium salt incorporation into polymer blend electrolytes enhances polymer–salt interactions, increases the amorphous phase required for ion transport, and maintains adequate thermal stability for solid and gel polymer electrolyte applications[16-17].

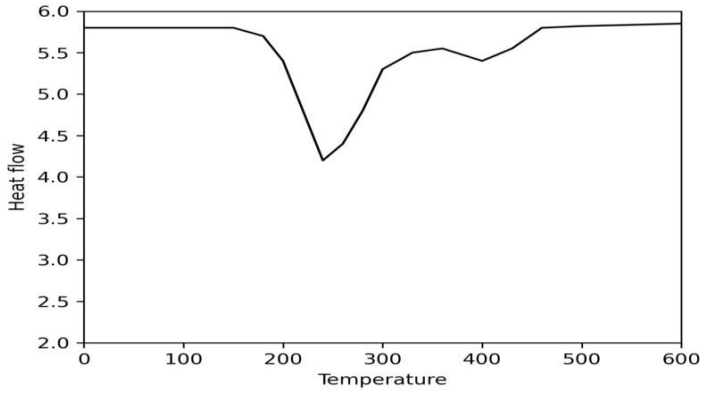

Figure 3. DTA curve for PVC microporous polymer membrane.

CONCLUSION

1. A simple, cost-effective, and environmentally favorable polymer dissolution technique was successfully employed to fabricate a PVC-based microporous polymer membrane without using conventional phase inversion methods.
2. The x-ray diffraction peaks with less intensity indicates that the prepared membrane exhibited a highly amorphous structure, as confirmed by XRD analysis, which is beneficial for enhanced ion transport in polymer electrolyte systems.
3. Upon incorporation of $LiClO_4$, the observed changes in diffraction patterns (peaks displaced from $2\theta = 29.1°$ to $2\theta = 22.2°$) confirmed effective polymer–salt complexation and demonstrated that salt concentration significantly influences the structural characteristics and crystallinity of the membrane.
4. Thermal characterization by TGA/DTA revealed that the developed membrane possesses excellent thermal stability up to approximately 280 °C, making it suitable for electrochemical applications requiring high thermal endurance.
5. The combination of desirable structural and thermal properties indicates that the prepared PVC microporous membrane is a promising candidate for lithium-ion battery separators and polymer electrolyte systems. Furthermore, the proposed fabrication method offers a simple, scalable, and economically viable route for producing high-performance microporous membranes, with potential applications in advanced energy storage devices, electrochemical sensors, and other solid-state electrochemical technologies.

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