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A Structural Study of Mixed Ion PVA based Composite Polymer Electrolyte using X-ray Diffraction Studies

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Abstract

A mixed ion composite polymer electrolyte of sodium perchlorate and lithium perchlorate doped with poly-vinyl alcohol (PVA) matrix have been prepared using solvent casting method. In the present paper, the crystallinity and morphological properties of the PVA doped mixed ion polymer composites were studied by X-ray diffraction (XRD) pattern. It was found that the change in structural repositioning and crystallinity of the composites takes place due to the interaction of dopants and also because of complex formation.

Keywords: Polymer electrolyte, Mixed ion composite, PVA, XRD, Sodium perchlorate, Lithium perchlorate and Solvent casting method.

INTRODUCTION

The composite materials are the materials prepared by mixing of two or more components [1]. Composite Polymer electrolytes (CPE) are useful for a variety of electrochemical devices such as batteries, fuel cells, super capacitors, sensors, electrochromic devices and smart windows. Polymer electrolytes are prominently used by the researcher community because of their versatile characteristics in various electrochemical devices [2-3]. In the progression of Composite Polymer Electrolytes, the researchers felt for such a materials which possesses the various characteristics like good conductivity, mechanical strength, salt/polymer interaction with constituents present in the system, temperature operation range, electrode – electrolyte interface etc. The contemporary scientific societies and industries are looking for such composites which are economical, environment friendly. In past two decades, various systems have been comprehensively studied and very commonly studied polymer electrolytes are Li salt complex with various host polymers like PEO, PVA, PAN, PMMA etc. [4-7]. The Mixed Ion CPEs may be one of the alternate promising candidates to substitute Li salt for electrochemical devices. The properties of mixed ion CPE materials depend on the nature, proportion and compatibility of their constituents. In the present paper, we aimed to taking into account the XRD structural study analysis of mixed ion polymer composite of PVA with inorganic salts (sodium perchlorate and lithium perchlorate).

EXPERIMENTAL

Chemicals used:

The host polymer poly-vinyl alcohol with molecular weight of 125,000 g/mol, sodium perchlorate (Sigma Aldrich) and lithium perchlorate (Sigma Aldrich) were used and distilled water was used as a solvent.

Preparation of mixed ion polymer composite films:

The weighing of all chemicals has done with Toledo Metller electronic balance. All the samples of mixed ion composite polymer electrolytes were prepared by solution casting technique. The solid PVA with different concentrations (5 wt%, 10 wt%, 15 wt%, 20 wt%, 25 wt% and 30 wt%) were added with prepared stock solutions of sodium perchlorate and lithium perchlorate to find the mixed ion composites. To obtain a homogeneous mixture, the solution was then stirred continuously using stirrer for 8 hours on a hot plate at room temperature. Finally prepared solutions of mixed ion composites were placed in 80 x 15 mm diameter Borosil Petri dishes for further annealing at room temperature. The Petri dishes are covered with aluminium foil to avoid any outside contaminations.

Characterization:

The prepared mixed ion composite films were cut into 10 mm x 10 mm size for further characterization. To study the crystallinity and its structural morphology, X-ray diffraction (XRD) analysis has been carried out by Rigaku ULTIMA-IV X-ray diffractometer in the range $2\theta = 10°$ to $80°$.

RESULTS AND DISCUSSION

X-ray analysis of pure PVA

Figure-1 represents the XRD pattern for solid powder PVA and figure-2 represents the XRD pattern of water based PVA gel electrolytes. As seen from the figure-1, a broad peak is

noticed for solid PVA at 2θ = 20^0 Pure PVA showed a characteristic broad peak indicating its semi crystalline nature [8] and at 2θ = 19°, the slight decrease in the peak is observed for PVA gel electrolyte suggests the degree of crystallinity of the complex [9-10]. This observed decrease in the peak intensity of XRD pattern results the amorphous nature [11-12].

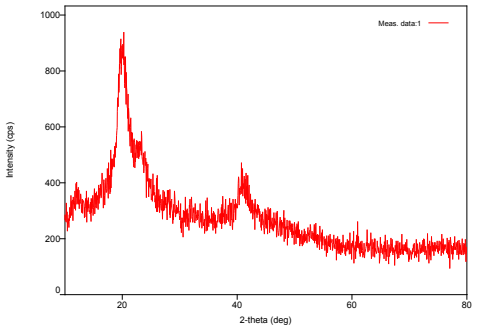

figure-1

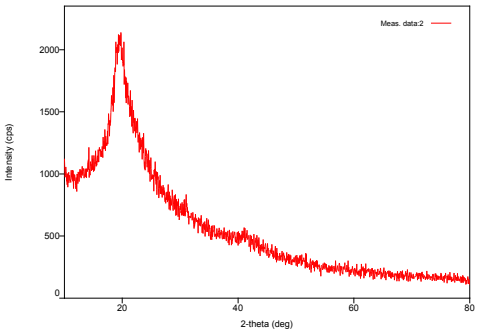

figure-2

X-ray analysis of pure PVA and Sodium Perchlorate Composite

Figure-2, a recorded XRD pattern of pure PVA film was used as a reference for other compositions of different composite systems. To investigate the influence of sodium perchlorate salt on host polymer poly-vinyl alcohol, XRD pattern have been studied (Figure-3). A remarkable decline in the crystallinity of PVA complex due to the addition of $NaClO_4$ salt indicated its amorphous phase which could be authenticated by comparing the XRD patterns of pure PVA film (2θ = 20°) and complexed PVA with $NaClO_4$ (2θ = 24°) [13]. The variation of peaks in XRD patterns of polymer and polymer salt composites clearly proves the coordination between polymer and salt [10].

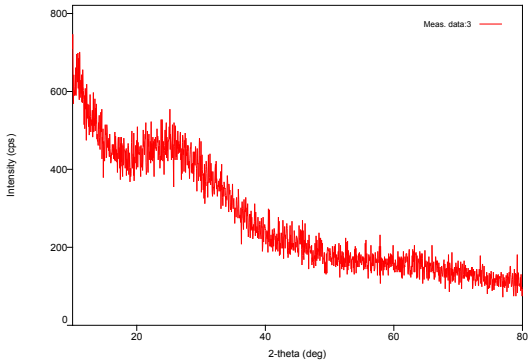

Figure-3

X-ray analysis of pure PVA and Lithium Perchlorate Composite

X-ray diffraction pattern of mixed ion composite of PVA and lithium perchlorate salt is shown in figure-4. It is observed that for pure PVA the peak was at 2θ = 20°, as shown in figure-2, and the diffractogram shown by figure-4 shows some shifting of peaks which provides the information towards low degree crystalline state [10]. The peak found at 2θ = 22° indicate the complete dissolution of salts in host polymer as no peaks corresponding to $LiClO_4$ is found. The obtained XRD pattern shows the crystallinity of the composite form by host polymer PVA and lithium perchlorate salt.

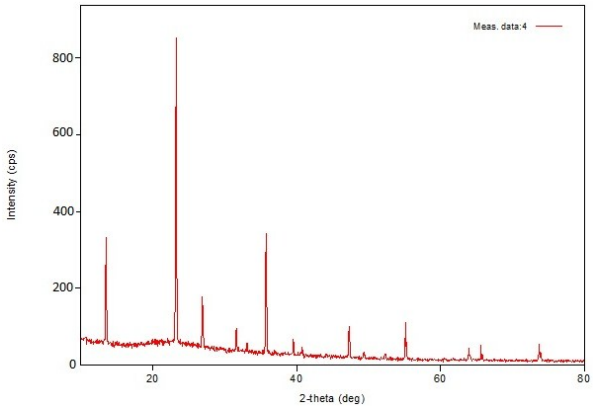

Figure-4

X-ray analysis of pure PVA with Sodium Perchlorate and Lithium Perchlorate Composite

On comparison of XRD pattern of pure PVA (figure-2) with XRD pattern of mixed ion composite of PVA with $NaClO_4$ and $LiClO_4$ (figure-5), it is found that the maximum peak at 2θ = 24° and be a sign of the complete dissolution of salts in host polymer [14] and established the definite complex coordination between polymer PVA and sodium and lithium salts. The intensity peaks shifted from 2θ = 20° to 2θ = 24° and shift in peak values recommend a decrease in the degree of crystallinity of the complex [9]. The observed change may be because of interference of PVA crystalline nature as suggested by Hodge et al as they reported addition of dopant increases the amorphous nature of composite [10]. It was reported that the amorphous polymers having flexible back-

bone supports in greater ionic diffusivity and high ionic conductivity [11-12].

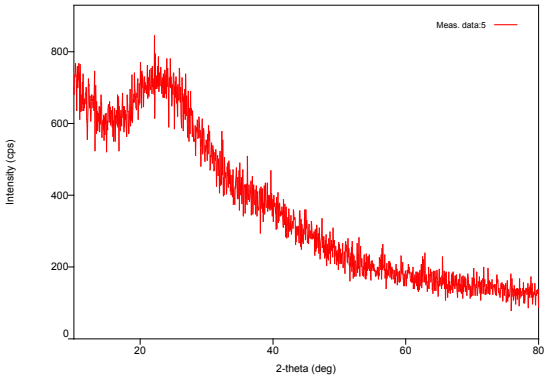

Figure-5

CONCLUSIONS

The X-ray diffraction pattern analysis of pure Poly-vinyl alcohol, Poly-vinyl alcohol doped with sodium perchlorate, Poly-vinyl alcohol doped with lithium perchlorate and a mixed ion composite of Poly-vinyl alcohol with mixed salts sodium perchlorate and lithium perchlorate brought out the foremost structural characteristics and morphological changes by means of doping of the salts in host polymer. X-ray diffractogram recommends the increase in amorphousness with doping of mixed salts sodium perchlorate and lithium perchlorate in the polymer matrix.

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