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A Study of X-Ray Diffraction (XRD) and Surface Morphology Studies of PVC complexes

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ABSTRACT

In this work, the proton conducting polymer electrolytes were prepared by solution cast technique. Poly (vinyl chloride) PVC is used as the polymer host, ammonium triflate ($\text{NH}_4\text{CF}_3\text{SO}_3$) as the doping salt and ethylene carbonate (EC) and butyltrimethyl ammonium bis trifluoromethyl sulfonyl imide ($\text{Bu}_3\text{MeNTf}_2\text{N}$) is used as the plasticizers. Characterization techniques of XRD and FESEM were used to study the properties of the PVC based proton conducting polymer electrolytes. XRD studies shows that amorphous PVC becomes largely amorphous in nature upon addition of $\text{NH}_4\text{CF}_3\text{SO}_3$. Largely amorphous in nature is also obtained upon addition of EC and $\text{Bu}_3\text{MeNTf}_2\text{N}$ to PVC- $\text{NH}_4\text{CF}_3\text{SO}_3$. XRD studies of the salted samples showed that they are largely amorphous in nature. However, XRD studies could not identify for sure the most amorphous sample. Identification of the most amorphous sample is imperative as conduction in polymer electrolytes is known to occur in the amorphous region. The FESEM micrographs gave a qualitative idea of the amorphousness of the salted samples in that A4 had the most grey regions in its micrograph giving a strong indication of it being the most amorphous sample. In case of the EC plasticised system, the micrographs showed that the pore size increased with increasing concentration of EC which in turn caused the amorphous regions (grey area) to decrease. This means that B1, which has the smallest pore size and the most grey area is the most amorphous sample. In the micrographs of $\text{Bu}_3\text{MeNTf}_2\text{N}$ plasticised samples, the sample C3 is observed to be more homogeneous with more white spherulites indicating presence of more trapped ionic liquid. This broadly indicates that C3 is the most amorphous due its homogeneity as compared to the sample C1.

Keywords: Polyvinyl Chloride (PVC); X-ray diffraction; surface morphology; polymer electrolytes; amorphous

INTRODUCTION

X-ray diffraction (XRD) patterns and surface morphology of pure PVC and the polymer electrolytes prepared in this work are presented first. X-ray diffraction studies are carried out to compare the nature of crystallinity and study the occurrence of complexation while changes in surface morphology and structure of the polymer systems can be studied via field emission scanning electron microscopy. In the case of Ammonium Triflate ($\text{NH}_4\text{CF}_3\text{SO}_3$) only the X-ray diffraction pattern will be presented. This is followed by the X-ray diffraction patterns and surface morphology image of the complexes PVC- $\text{NH}_4\text{CF}_3\text{SO}_3$, PVC- $\text{NH}_4\text{CF}_3\text{SO}_3$ -EC and PVC- $\text{NH}_4\text{CF}_3\text{SO}_3$ - $\text{Bu}_3\text{MeNTf}_2\text{N}$ due to Tetrahydrofuran (THF) evaporation. These diffractograms and micrographs are presented in order to study the effects of addition of

plasticizer / ionic liquid on the structure of the PVC- NH₄CF₃SO₃ complexes.

INNOVATION DEVELOPMENT

In the present study, XRD is carried out to investigate the effect of adding another polymer, salt and plasticizer to the crystallinity of the PVC. XRD is also used to examine the occurrence of complexation between polymer host and doping salt. Complexation between a polymer host and a doping salt is usually indicated by the shifts of peaks of the polymer host or salt in the XRD spectra. Sometimes, this is accompanied by the absence or the disappearance of some peaks of the salts and/or polymer (Sekhon et al,1995; Mohamed et al, 1997). Complexation may also be indicated by the presence of new peaks in XRD spectra (Acosta and Morales, 1996, Sekhon et al, 1995). The XRD spectra of the proton conducting polymer electrolytes films studied in this work is recorded using X'pert Pro Analytical Diffractometer for Bragg angles (2θ) varying from 5° to 90°.

In order to understand how the surface morphology of the proton conducting polymer electrolyte film changes with doping of salt and plasticizers, field emission scanning electron microscopy of the polymer electrolyte films were taken. The structure and surface morphology of the polymer electrolyte films is an important property for the polymer electrolyte since it gives us qualitative idea of the degree of crystallinity (X_c) of the polymer films. In the present work, field emission scanning electron microscopy (FESEM) is carried out using FESEM, ZEISS SUPRATM 40VP at Faculty of Applied Sciences, Universiti Teknologi MARA. The polymer electrolyte films were coated with a layer of platinum using Sputter Coater under vacuum chamber with argon gas. The polymer electrolyte films were then attached to FESEM stubs using two-sided adhesive tape. The sample is examined at 5 kV for 500x, 3000x and 10000x magnification in order to investigate the different spherulites size of the samples.

COMMERCIAL POTENTIAL

This product has good performance of PVC based proton conducting polymer electrolytes in electrochemical cells. It suitable for potential application as electrolytes in solid-state electrochemical devices like batteries. Applicability of this polymer electrolytes especially in proton electrochemical cells, primary battery for proton battery and energy storage.

Application of Intellectual Property/Copyright was submitted to Intellectual Property Corporation of Malaysia (MyIPO) with the application number LY2022W00007as shown in Figure 1.



Figure 1

CONCLUSION

XRD results show that the salt and plasticizers have decreased the crystallinity of PVC and salted PVC-based polymer electrolytes. The complexation between the salt, plasticizers and PVC occurs in the amorphous phase as a result of the decrease in the crystalline regions of the complexes. The surface morphology images give a qualitative idea of the degree of crystallinity of the complexes studied in this work. The surface morphology of the polymer electrolytes provides information on the structure of the materials studied which XRD could not.

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