

INVESTIGATION OF PMMA AND PVdF-HFP WITH NaClO₄POLYMER BLEND ELECTROLYTE SYSTEM– APPLICATION TO AN ELECTROCHEMICAL CELL

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ABSTRACT

A solution-cast process was used to prepare pure poly (methyl methacrylate) (PMMA), and pure poly (vinylidene fluoride-hexafluoropropylene) (PVdF-HFP), their blend, and different weight percentages of sodium per chlorate (NaClO₄) salt added to PMMA: PVdF-HFP polymer blend electrolyte thin films. The polymer electrolyte film was prepared using the solution cast process, which involves combining sodium per chlorate (NaClO₄) ionic salt with poly (methyl methacrylate) (PMMA) and pure poly (vinylidene fluoride-hexafluoropropylene) (PVdF-HFP) blended polymer. These films have been characterized systematically using different experimental techniques to investigate structural, morphological, thermal, and conductivity properties. The degree of crystallinity and porous microstructure of all the thin films were determined by XRD and SEM data and revealed that higher content of the amorphous nature was noticed at higher NaClO₄ salt concentration in PMMA:PVdF-HFP polymer blend. When NaClO₄ salt was added to a PMMA:PVdF-HFP polymer blend, fair complexation/interaction between polymers and salt was observed. In DSC curves of PMMA and PVdF-HFP polymers, a sharp melting temperature (T_m) was seen, which diminished when NaClO₄ salt was added, showing that its amorphous nature had risen to prominence. The ionic conductivity of NaClO₄ salt added PMMA:PVdF-HFP polymer blend electrolyte has been investigated, with an optimal value found to be 1.55382×10^{-4} S/cm at 40% NaClO₄ salt. Electrochemical cells have been fabricated and investigated its characteristics in the configuration of Na/[PMMA:PVdF-HFP: NaClO₄]/(I₂:C).

Keywords: Solution-Cast Process, PMMA, PVdF-HFP, NaClO₄, Ionic Conductivity.

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INTRODUCTION

Solid polymer electrolytes (SPEs), modified solid polymer electrolytes (MSPEs) and gel polymer electrolytes (GPEs) are existing forms of polymer electrolytes. The composite and polymer blend electrolytes are modified polymer electrolytes.¹⁻¹¹ SPEs films may offer low ionic conductivity, gel polymer electrolyte films offer higher conductivity but are limited to use because of their poor compatibility, and Modified polymer electrolyte thin films offer high ionic conductivity with compatible strength for electrochemical devices. Modified polymer electrolytes are made popularly, and act an important role to develop electrochemical devices like batteries, sensors, supercapacitors, etc., because of their long life, and outstanding electrochemical properties, and also provide better safety as compared to liquid electrolytes. Extensive research is being undertaken on PMMA, PVDF-HFP, PEO, and PVC, among other materials, for battery applications and solar cells. By blending two or more polymers, their poor electrical conductivity and low mechanical strength can be improved to make them have high electrical conductivity and mechanical strength.^{12,13} Ionic conductivity has also increased as a consequence

of the addition of several ionic salts, like Na, Mg, Zn, Ca, Li, Na, Cd, and others, to the blending of polymers.¹⁴⁻¹⁸ PMMA is being used as a host polymer due to its greater stability and reduced electrochemical reactivity towards electrodes.^{19,20} The co-polymer PVdF-HFP is employed because it has a good matrix for cross-linking with other polymers and improves ionic conductivity.^{21,22} In the present article, NaClO₄ salt is added to PMMA:PVdF-HFP polymer blend films by solution-cast process and analyzed for various supporting analytical characterizations for structural, morphological, thermal, and optical properties. This polymer electrolyte was used to study the discharge parameters of an electrochemical cell due to its excellent ionic conductivity.

EXPERIMENTAL

Materials Used

PMMA polymer with an average M.W of 1.2×10^5 , PVdF-HFP polymer with an average M.W of 4×10^5 , and other materials, including NaClO₄, were purchased from Sigma Aldrich and used in this research work. THF (Emparta-AR grade), a solvent, was purchased from Merck Millipore in Germany.

Preparation of the NaClO₄ Salt Added to PMMA: PVdF-HFP Polymer Blend Films

Composite solid polymer blend electrolyte films were made using the solution cast method.^{23,24} PMMA polymer, PVdF-HFP polymer, and various amounts of NaClO₄ were weighed, then transferred to a beaker, and dissolved in THF solvent. The beaker was placed on a magnetic stirrer at room temperature and swirled for 24 hours to attain a homogenous solution. The produced homogenous solution was discharged into a circular glass Petri dish and the solvent was slowly dried to obtain a consistent thin film (thickness ~ 100 µm), and prepared compositions are tabulated in Table-1.

Table-1: Thin Films with PMMA, PVdF-HFP, and NaClO₄ Salted Polymer Electrolyte Composition Ratios

S. No.	Description of the Sample	Polymer PMMA (Wt.%)	Polymer PVdF-HFP (Wt.%)	Salt NaClO ₄ (Wt.%)
a	PM	100	0	0
b	PHP	0	100	0
c	PMPHP200	100	20	0
d	Na	0	0	100
e	PMPHPNA1	100	20	10
f	PMPHPNA2	100	20	20
g	PMPHPNA3	100	20	30
h	PMPHPNA4	100	20	40
i	PMPHPNA5	100	20	50

Characterization Techniques Used in the Present Study

The XRD device SHIMADZU-XRD [Model 6100/7000] was used to examine the crystallinity of all of the thin films. A Hitachi SEM [Model-3700N] was used to analyze the surface morphology of thin films. The DSC-60 SHIMADZU device was used to obtain DSC data in order to determine the melting temperatures of thin films. FTIR Spectroscopy measurements for these blend films to investigate specific interaction/complexation between polymers and salt were recorded with the help of the SHIMADZU [FTIR-8400S] spectrometer. D.C. conductivity studies are obtained from 303K to 373K temperature range using the lab-made conductivity setup with the aid of Keithley-196 System DMM. The characterization of an electrochemical cell with the combination Na / [PMMA; PVDF-HFP: NaClO₄ / (I2:C) was investigated.

RESULTS AND DISCUSSION

X-ray Diffraction Investigation

XRD is a strong tool for expressing crystalline phase transitions and modifications in a polymer-polymer electrolyte. The impact of varied NaClO₄ salt concentrations on the PMPHP200 mix polymer can be seen in Fig.-1. As illustrated in Fig.-1(b), the diffraction angle for pure PVdF-HFP reveals three prominent peaks at 17.60°, 19.52°, and 38.76° degrees, suggesting a semi-crystalline structure, and these reveal α, β, γ phase of the polymer.²⁵ It is observed that the peaks completely disappear in the optimized composition

of the PMPHP200 polymer blend shown in Fig.-1(c). It suggests that the complex formed by PMMA and PVdF-HFP is fair, and the studied range is within the miscibility. The NaClO_4 salt exhibits high-intensity intensity peaks with angles of $2\theta = 16.78^\circ, 23.92^\circ, 25.35^\circ, 27.49^\circ, 31.1^\circ, 36.2^\circ, 40.57^\circ, 49.61^\circ$, and 52.70° which represents crystalline nature of ionic salt.²⁶⁻²⁷ The XRD characteristic peaks in blended PMPHP200 are less intense with the addition of NaClO_4 salt. The XRD diffraction peaks have grown less intense as the NaClO_4 salt concentration in the PMPHP200 polymer blend complex increases, showing a drop in crystallinity and an elevation in amorphosity of the PMPHP200 polymer complex system. For higher concentrations of NaClO_4 , the PMPHP200 blend polymer doesn't have any sharp peaks, which suggests the higher presence of the amorphous phase. Additionally, as shown in Fig.-1(h), the greatest amorphous phase is seen at a content ratio of PMMA:PVdF-HFP: NaClO_4 polymer-salt electrolyte (10:2:4). Further increase in the concentration of NaClO_4 , the intensity of XRD peak is slightly increased representing again possibly reorganizing semi-crystalline nature of the PMPHP200 polymer blend as observed in Fig.-1(i), which maybe the inclusion of higher salt concentration to PMPHP200 polymer blend and lead to an aggregate of Na^+ cation and ClO_4^- anion or phase separation. A further increase in NaClO_4 concentration causes a modest rise in XRD peak intensity and presumably reorganizes the PMPHP200 polymer blend's semi-crystalline character as shown in Fig.-1(i). This incorporation of more salt may cause phase separation or an aggregation of the Na^+ cation and ClO_4^- anion.

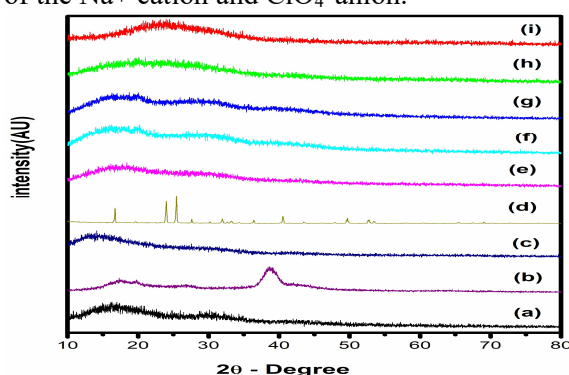


Fig.-1: The XRD of (a) PM, (b) PHP (c) PMPHP200, (d) Na, (e) PMPHPNA1, (f) PMPHPNA2, (g) PMPHPNA3, (h) PMPHPNA4, and (i) PMPHPNA5

Morphology Studies

SEM analysis advances surface morphology studies of polymer electrolyte films. Fig.-2 represents the SEM micrographs of pure polymers and their blends with different composition ratios of salt. Fig.-2(b) revealed the semi-crystalline character of the PVdF-HFP polymer is represented by a rough, granular surface with few accessible microspores.²⁸ As shown in the SEM micrographs, the surface morphology of PMMA has been significantly changed by the addition of PVdF-HFP, with no evidence of phase separation. The surface morphology of PMMA becomes smoother and more porous when PVdF-HFP was added to it. This corroborates the decrease in crystalline nature and the rise in amorphous nature.²⁹ Fig.-2(d)-(h) illustrates the surface morphology of PHP200 polymer electrolytes with different NaClO_4 concentrations. As NaClO_4 concentration in the PMPHP200 polymer blend increases, the structure and pore size reached a sufficient value. Oxygen "O" from PMMA and 'F' in PVdF-HFP may interact with Na^+ ion; it may be available for conduction of ion in this PMPHP200 blend polymer films with NaClO_4 . The observed porous structure is responsible for the flexibility and amorphous nature of this polymer electrolyte. But as the salt concentration attains to a certain level that is (10:02:04) of PMMA: PVdF-HFP: NaClO_4 polymer electrolyte achieves good porous structure. The interlinked porous polymer matrix promotes a path for migration of Na^+ ions which aids for ease the mobility of ions. SEM results reveal an amorphous nature, which is supported by XRD data.

Structural Analysis of the Composed Films by FT-IR Spectra

The functional groups of polymers and their modes of vibrations are studied by Infrared spectroscopy. The addition of salt to the blended polymer leads to bands shifting, broadening, and disappearing of FTIR absorption peaks as a result of the atomic and molecular interaction.³⁰⁻³³

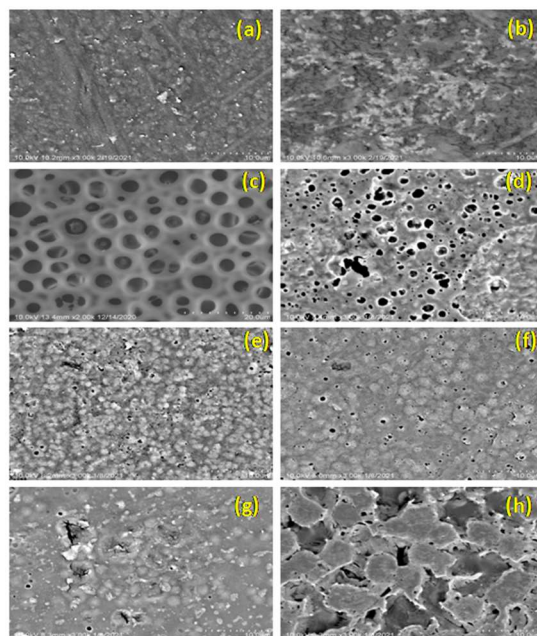


Fig.-2: SEM Images of (a) PM, (b) PHP, (c) PMPHP200, (d) PMPHPNA1, (e) PMPHPNA2, (f) PMPHPNA3, (g) PMPHPNA4, and (h) PMPHPNA5

Figure-3(i to iii) shows the FT-IR patterns of pure PM, pure PHP, PMPHP200, and NaClO_4 salt compositions. The IR spectra of pure PHP have their bands at 3575cm^{-1} due to CH_2 symmetrical stretching, and this band shifted to a lower wave number at 3500cm^{-1} in PMMA: PVdF-HFP: NaClO_4 polymer electrolyte films from Fig.-3(i). The PMMA polymer has bands at 1149cm^{-1} owing to C-C stretching, 854cm^{-1} and 742cm^{-1} owing to C-C and C-O bending, 1724cm^{-1} owing to C-H bending, 1448cm^{-1} , and 1359cm^{-1} owing to C-C stretching³⁰ as shown in Fig.-3. (ii). The band at 1350cm^{-1} is due to the crystalline phase of PVdF-HFP and the band at 750cm^{-1} , and 600cm^{-1} were due to CF_2 bending. It is discovered that with the incorporation of NaClO_4 salt content, the bands in PMPHP200 films were broadened, and various bands were shifted to low wave numbers. Moreover, the intensity of crystalline peaks is reduced by the gradual increase in salt content. Some of the known peaks were found to be disappeared and some new peaks appeared along with shifting of some band positions indicating that the two polymers, PMPHP200 polymer blend, have a significant interaction in presence of NaClO_4 salt and blending was compatible. From Fig.-3(iii), new bands appeared between 640 and 600cm^{-1} are present in all the PMMA: PVdF-HFP: NaClO_4 blend films and ascribed to the stretch of ClO_4^- ions. Based upon earlier report assignments, these bands correspond to the main ClO_4^- species, the first of which is free ClO_4^- ions with a maximum of around 623cm^{-1} and the other being ClO_4^- ions associated with the cation Na^+ at around 635cm^{-1} . The band position around 630cm^{-1} related CH Bending of PVdF-HFP polymer and this band shifted to a lower number in NaClO_4 included blend polymer films. Wiecezorek *et al.*³⁴ and Salmon *et al.*³⁵ ascribed the 623cm^{-1} band position to liberated anions ClO_4^- and 635cm^{-1} envelop to an interaction of an ion pair or anion ClO_4^- with sodium cation. It is observed that the shoulder peak at 635 is obvious in PMMA: PVdF-HFP: NaClO_4 blend films at higher NaClO_4 salt concentration, which indicates the Na^+ and ClO_4^- ion pair formation or phase separation. Interestingly, the ion-pairing band observed at 635cm^{-1} is found to appear with increasing NaClO_4 salt content and this blend appeared at 40 wt.%. This result indicates the formation of cation-anion interaction which led to the aggregation of salt or phase separation.

DSC

DSC technique was used for the investigation of thermal properties i.e., melting temperature, glass transition temperature, and degree of crystallinity of polymeric and glass materials.^{36, 37} The DSC curves of PMMA, PVdF-HFP, PMPHP200 polymer-blend, and different concentrations of NaClO_4 salt doped PMPHP200 polymer blend thin films are shown in Fig.-4. Endothermic DSC curves of PMMA and

PVdF-HFP thin films were showing melting temperatures(T_m) at 162 °C and 145°C respectively. When 20 wt.% of PVdF-HFP polymer was added to PMMA polymer no such melting temperatures(T_m) appeared in the DSC curves of blend films that indicated the phase transition of PVdF-HFP polymer from semi-crystalline nature to amorphous nature, this amorphous nature of thin films offer better ion mobility in electrolytes. Different wt.% (10, 20, 30, 40, 50) of NaClO₄ salt is added to PMPHP200 polymer blend, whose DSC curves have shown in Fig.-4.

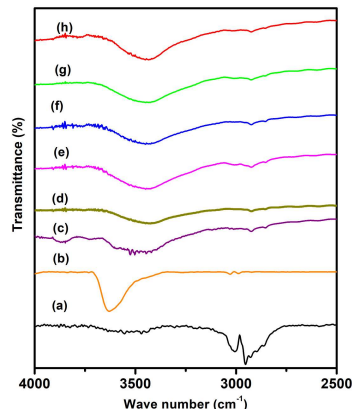


Fig.-3: (i) FT-IR Spectra of (a) PM, (b) PHP, (c) PMPHP200, (d) PMPHPNA1, (e) PMPHPNA2, (f) PMPHPNA3, (g) PMPHPNA4, and (h) PMPHPNA5

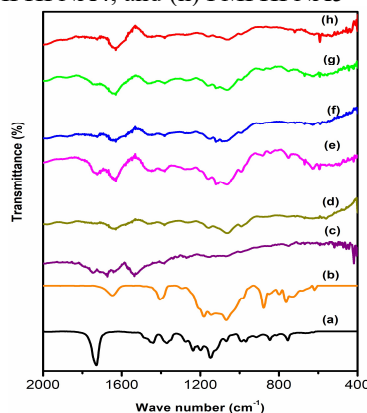


Fig.-3: (ii) FT-IR Spectra of (a) PM (b) PHP, (c) PMPHP200, (d) PMPHPNA1, (e) PMPHPNA2, (f) PMPHPNA3, (g) PMPHPNA4, and (h) PMPHPNA5

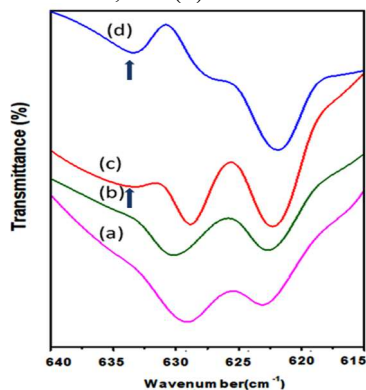


Fig.-3: (iii) FT-IR Spectra of (a) PMPHPNA1, (b) PMPHPNA3, (c) PMPHPNA4, and (d) PMPHPNA5

All the films of NaClO₄ salt added to PMPHP200 polymer blend data have not shown any melting temperature, which indicated that presence of amorphous content in all NaClO₄ salt added to PMMA:PVdF-HFP blend films, and these results also agreed with the XRD, SEM, and IR data. Hence this amorphous nature of NaClO₄ salt incorporated into PMPHP200 polymer blend films may provide higher segmental motion to ions through the polymer chain.³⁸

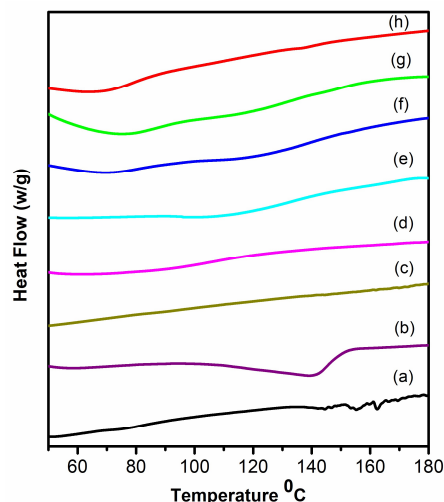


Fig.-4: DSC of (a) PM, (b)PHP, (c) PMPHP200, (d) PMPHPNA1, (e) PMPHPNA2,(f) PMPHPNA3, (g) PMPHPNA4, and (h) PMPHPNA5.

DC Conductivity

The DC conductivity of PMMA and PVdF-HFP complexed with NaClO₄ blends was investigated for the best blend composition with optimum conductivity. The conductivity was calculated using Eqn.-1,

$$\sigma = \frac{l}{AR_b} \quad (1)$$

Where, l represents thickness, A represents the area, and R_b represents bulk resistance.³⁹Figure-5shows the composition dependence conductivity ofNaClO₄ salt added to PMPHP200 polymer blend solid polymer electrolyte. The optimum conductivity is obtained at 40 wt.% NaClO₄ solid electrolyte film. Optimal ionic conductivity is 1.55382×10^{-4} S/cm, this may be due to a higher amorphous phase at this composition, and further increase of NaClO₄ salt to PMPHP200 polymer blend solid polymer electrolyte leads to a decrease in conductivity where ion pair formation or phase separation occurs. The obtained result is well supported by XRD, SEM, FTIR, and DSC studies. The ionic conductivity of NaClO₄ salt added to PMPHP200 polymer blend electrolyte films in the temperature range from 30°C to 100°Care is shown in Fig.-6.

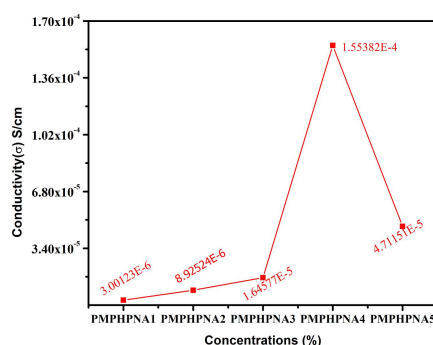


Fig.-5: Composition Dependence Conductivity of NaClO₄Salt Added to PMMA:PVdF-HFP Polymer Blend Films

As the temperature rises, the ionic conductivity rises due to the polymer complex becomes more amorphous. As the temperature of the polymer rises, the ion transport phenomenon is enhanced via ion transfer from one site to another with a modest amount of activation energy. The dissociated Na⁺ ion is hopping between the two blended polymer chains. Na⁺ ion moves from one site to another, where it is linked with an "O" of PMMA, and "F" of PVdF-HFP, in the PMPHP200 blended polymer films. As the temperature increases the blended polymers attain a more amorphous nature, hence the ions easily migrate in the amorphous nature of polymer electrolyte.

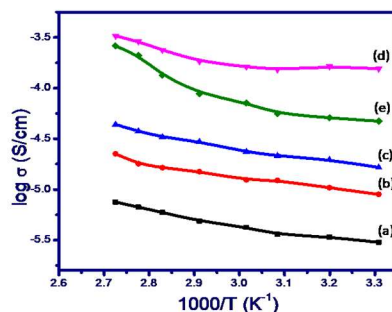


Fig.-6: Temperature Dependent DC Conductivity of (a) Na100, (b) Na200, (c) Na300, (d) Na400 and (e) Na500

Electrochemical Cell

The structure of a fabricated electrochemical is Na / [PMMA: PVdF-HFP: NaClO₄] / (I₂+C). Figure-7 illustrates the discharging properties of the electrochemical cell at room temperature with a constant load of 1 kΩ. The Na/[PMMA: PVdF-HFP: NaClO₄]/(I₂+C) electrochemical cell has an open-circuit voltage of 2.55 V and a short-circuit (I_{sc}) current of 11.2 mA. The open-circuited (V_{oc}) voltage of Na/[PMMA:PVdF-HFP: NaClO₄](100:20:40)/(I₂:C) is dropped to 2.4 V. But when it is coupled to a load of 1KΩ and its stability was observed for 80 hours. After 80 hours, the voltage drops sharply, which could be attributed to polarization and a thin salt film forming at the electrode-electrolyte interference. Additionally, efforts are being made to enhance the cell characteristics and capacity.

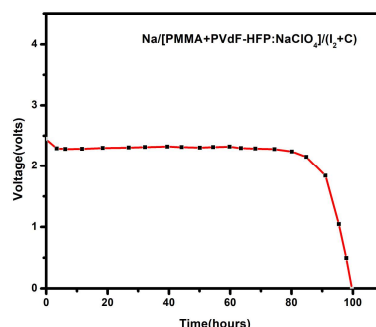


Fig.-7: Discharge Characteristics of an Electrochemical Cell of Na/[PMMA:PVdF-HFP: NaClO₄](100:20:40)/(I₂:C)

CONCLUSION

The uniform thin films of PMMA, PVdF-HFP, and PMMA: PVdF-HFP blend, and NaClO₄ salt doped PMMA:PVdF-HFP polymer blend electrolytes were prepared by the solution-cast process. The structural, morphological, thermal, and optical properties were examined. The observations suggested a reduction in the degree of crystallinity with raising NaClO₄ concentration in PMMA:PVdF-HFP blended polymer. The temperature variation ionic conductivity of all prepared thin films was examined and among all the films higher ionic conductivity was obtained at 40 wt.% of NaClO₄ salt present PMMA: PVdF-HFP polymer blend film. An electrochemical cell of Na/ [PMMA:PVdF-HFP: NaClO₄] / (I₂+C) was made from the analysis of all the findings, and its features were also investigated.

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