

# Structural and Ionic Conductivity Studies on Plasticized Pan-Sodium Fluoride Polymer Electrolytes for Electrochemical Cell Applications

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**Abstract:** Ion conducting gel polymer electrolytes based on poly acrylonitrile (PAN) complexed with different weight percent ratios of Sodium Fluoride (NaF) salt were prepared by using solution cast technique. Structural characterization was performed using X-ray diffraction (XRD) technique and Fourier transforms infrared (FTIR) spectroscopy technique. From the X-rd results increase in amorphous phase with the increase of dopant salt concentration was observed. Fourier transform infrared (FTIR) spectroscopic analysis confirmed the complexation of the salt with the polymer matrix. Electrical conductivity was measured that the magnitude of ionic conductivity increased with the increase in the salt concentration as well as temperature. The surface morphology was observed by using Scanning Electron Microscope (SEM), the optical band gap measured from UV-Vis Spectroscopy. The sample containing 30 wt% of NaF exhibited the highest conductivity of  $1.82 \times 10^{-4} \text{ S cm}^{-1}$  at 303K and  $2.96 \times 10^{-3} \text{ S cm}^{-1}$  at 373K. The temperature dependence of ionic conductivity of these films followed Arrhenius relation. Transference number measurements were carried out to investigate the nature of the charge transport species in the polymer electrolyte systems. The transference number data showed that the charge transports in these systems are predominantly due to ions. Using these polymer electrolyte films, electrochemical cells were fabricated and their discharge characteristics were studied. Various cell parameters, such as open circuit voltage, short circuit current, power density and energy density were determined.

**Keywords:** Ionic Conductivity, Gel Polymer Electrolyte, Sodium Fluoride, Plasticizer, Transference numbers.

## 1. INTRODUCTION

Gel polymer electrolytes are rapidly used in such as based batteries, cell technology, notebook computers and smart phone technology. The main reason to use in this way due to safety, no leakage of electrolyte and exhibiting higher energy density [1-2]. Gel polymer electrolytes have become increasingly attractive because their technological application in the development of gel state electrochemical devices. The dissolution of low lattice energy salts into solvating polymer leads to the formation of polymer electrolyte. These can conduct ions and the mobility of these ions can improved by addition of plasticizers [3]. In the present work, we report the study on structural and conductivity studies of plasticized PAN –NaF polymer Gel electrolyte films. Hence present work an attempt has been made to synthesize and characterization of PAN based sodium fluoride salt for battery application. In the present work, the plasticized polymer electrolyte

systems composed of PAN as a host polymer with plasticizers, ethylene carbonate (EC) containing Sodium fluoride (NaF) have been prepared. The conductivity of the samples was measured by ac impedance spectroscopy. The conductivity vs temperature studies were carried out in the temperature range between 303K and 373K. The structure analysis and complexation of PAN based polymer electrolyte films was studied by using the X-Ray diffraction and FTIR technique.

## 2. EXPERIMENTAL PROCEDURE

### 2. 1. Materials & Preparation

Gel polymer electrolyte films (thickness  $134 \mu\text{m}$ ) of Pure Poly acrylonitrile (PAN) and various compositions of complexed films of PAN with Sodium Fluoride (NaF) salt were prepared by solution casting method using Ethylene

carbonate (EC) as a plasticizer and N, N-Dimethyl Formamide as a solvent. PAN and NaF salt were dissolved in N,N-Dimethyl Formamide (DMF) and Ethylene carbonate (EC) stirred at room temperature for 24 hours to get homogeneous solution. The solution was cast onto petridishes and allowed to evaporate slowly at 60°C. Finally the product was dried thoroughly to remove all traces of the solvent. The dried polymer films were peeled off from the petri dishes and stored in a dry vacuum box.

## 2. 2. Characterization Methods

The X-Ray spectra were recorded at room temperature in the Bragg angle range 10–80 by using Analytical Xpert PRO (Philips, Netherlands). The FTIR Spectra were recorded with the help of perkin Elmer FTIR Spectrophotometer in range 500 to 4000  $\text{cm}^{-1}$  using KBr pellet method. The DC conductivity of Gel Polymer Electrolyte thick films was measured by using Keithley 6514 electrometer connected to lab made conductivity set up [4-5]. The Scanning Electron Microscope (SEM) is one of the most versatile instruments used for the

analysis of microstructure morphology [6]. The UV-Vis spectrum was recorded by a Hewlett-Packard HP8452A diode array spectrometer. Transference numbers (tion, tele) were evaluated by means of Wagner's polarization technique under a dc bias of 1.5 V [7]. Electrochemical cells were fabricated with configuration Na (anode) / (polymer electrolyte) / (I<sub>2</sub>+C+electrolyte) (cathode) and their discharge characteristics were monitored at a constant load of 100 k $\Omega$ .

## 3. RESULTS AND DISCUSSIONS

### 3. 1. XRD Studies

X-Ray Diffraction pattern of the sample was taken using XPERT-PRO Diffractometer. X-Ray scattering has been a unique and standard characterizing tool to investigate the structural properties of materials. In these studies one basically monitors either the intensity of elastically scattered X-Ray photons as a function of the scattering angle in XRD [8-9]. The XRD characterizations of Pure PAN and NaF with different ratios are given in Fig. 1.

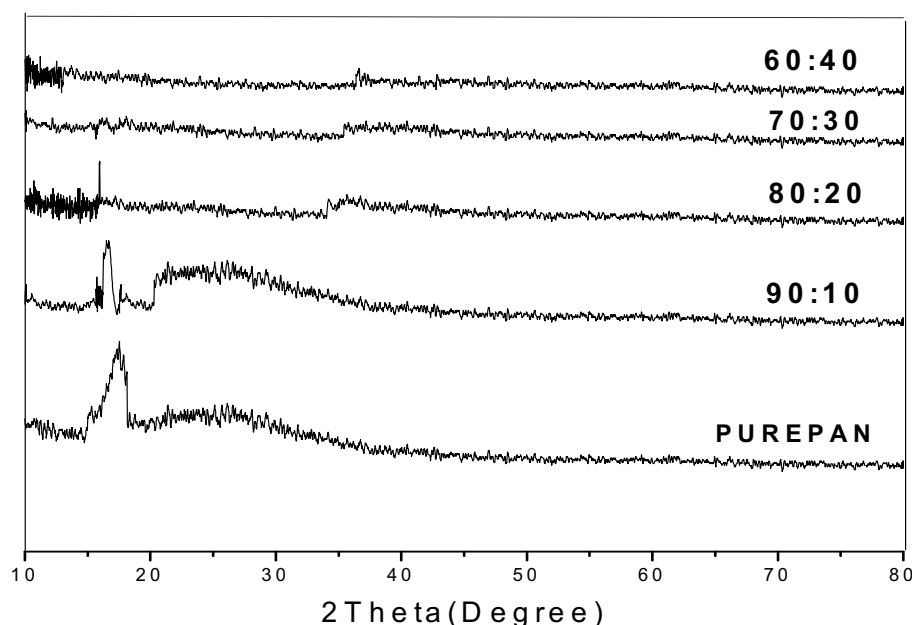


Fig. 1. XRD plots of pure PAN and PAN complexed films for different wt % of NaF.

From the Figure reveals that the XRD pattern of Pure PAN exhibits well defined peaks are observed NaF which are indicates the crystalline nature of salt and also a broad peak is observed for Pure PAN corresponds to crystalline orthorhombic PAN (110) which is centered at around 17.500 and broad peak corresponds to amorphous content lies in between 100-300 which corresponds to amorphous content indicating its semi crystalline nature [10-11]. The characteristic diffraction peaks of PAN-NaF at an angle 17.500 and 16.070 are reduced upon the addition of the plasticizers and NaF salt. The diffractograms in Fig. 1 reveals that the amorphous nature is predominant in the complex which is due to the addition of plasticizers. The diffraction pattern of PAN-NaF shows intense peak at an angles at  $2\theta=17.500$ , 16.680 and 16.070, which reveals the crystalline nature of the ionic salt [12]. The salt content of 30 wt % peak disappeared in the complexes, which indicates the complete dissolution of the salt in the polymer matrix, PAN base NaF salt complex shows the amorphous state. A fully amorphous structure produces a greater polymer flow and ionic diffusivity. Hodge et al [13] established a correlation between the intensity of the peak and degree of crystallinity. From these results the

XRD studies confirmed that there exist a definite complexation between the polymer and salt.

### 3. 2. FTIR Studies

Fig. 2 shows the FTIR spectra of pure PAN and NaF salt doped PAN complexes of various weight % ratios in the range between 500 and 4000  $\text{cm}^{-1}$ . The complexation of NaF in PAN has also been observed in FTIR spectral studies. The  $\text{Na}^+$  ion may interact either with C=O group of plasticizers (EC and DMF) or with the  $\text{C}\equiv\text{N}$  group of PAN or both [14]. The  $\text{C}\equiv\text{N}$  stretching band in the infrared spectrum is certainly the characteristic feature of nitrile group which appears at  $2244\text{cm}^{-1}$  for the Pure PAN film [15]. It is also observed that the intensity of this peak decreases with increase of salt concentration and the peak is shifted towards the higher wave number side ( $2282$  to  $2313\text{cm}^{-1}$ ).

The C=O group symmetric stretch mode of EC appeared at  $1773\text{cm}^{-1}$ . There is also appeared of new peaks along with changes in existing peaks or disappearance in infrared spectra directly indicates the complexation of PAN and NaF salt. The peaks are shifted from  $1773\text{cm}^{-1}$  to  $1608\text{cm}^{-1}$  the increase in % of NaF in films indicate that there is a weak interaction between the  $\text{Na}^+$  ion and the

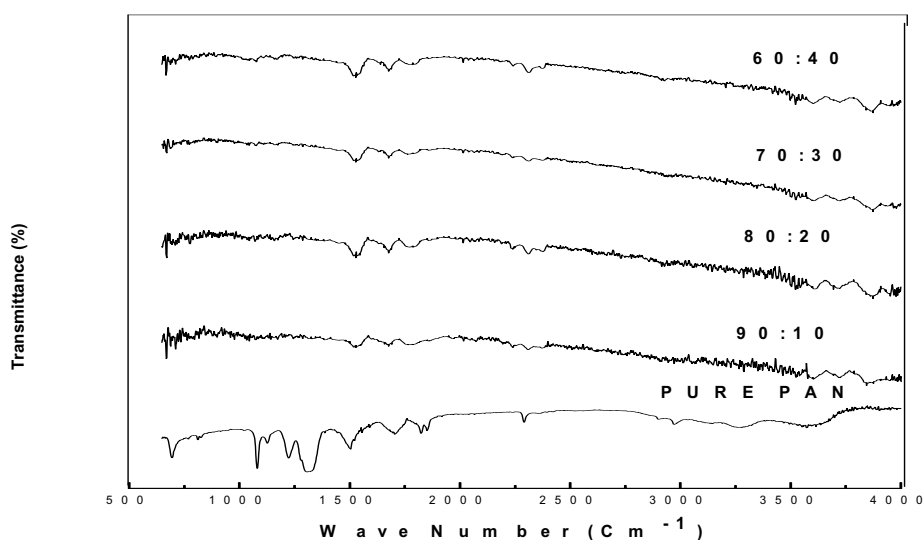


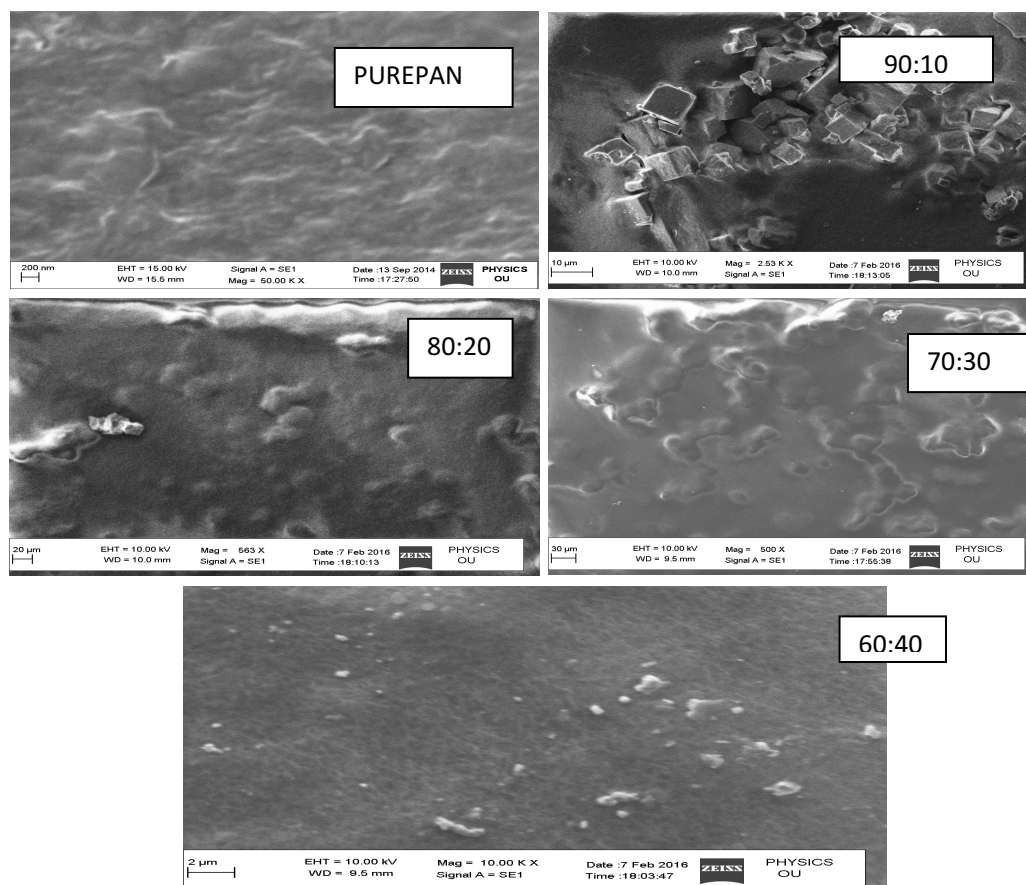
Fig. 2. FTIR Spectra of Pure PAN and PAN-complexed films for different wt % of NaF.

lone pair of the electrons on N of  $C\equiv N$  group of PAN [16].

### 3. 3. SEM Characteristics

The surface morphology of the prepared polymer electrolytes was studied using Scanning Electron Microscopy (FE-SEM, Carl Zeiss, Ultra 55 model). The accelerating voltage was 30 KV with a resolution of 4 nm and magnification 300,000 X. Structural phenomena play an important role in determining the properties of a polymer. Mechanical properties are determined not only by the changes in shape confirmation and by motion of invidual molecules of the polymers, but by the behavior of large and more complex structural formation as well. The interface boundaries of these formations known as super molecular structures, are the sites where

chemical reactions in the polymer are most likely to begin and centers of crack formation and incipient destruction are likely to begin centers of crack formation and incipient destruction are likely to arise. It has been found that extensive occurrences of ordered structures are typical not only of crystalline, but also of amorphous polymers. From the Fig. 3 one can observe the dispersed particles have irregular shape and show agglomeration. These structures are capable for better ion transport through the polymer matrix [17-18]. Fig. 3 presents the SEM images of gel polymer electrolytes with different content of NaF and Pure PAN composite films. These results showed the plasticization effect in the reduction of crystallinity of the host polymer and enhancement in the overall amorphous proportion in the polymer electrolyte. From the result it was understood that incorporation of

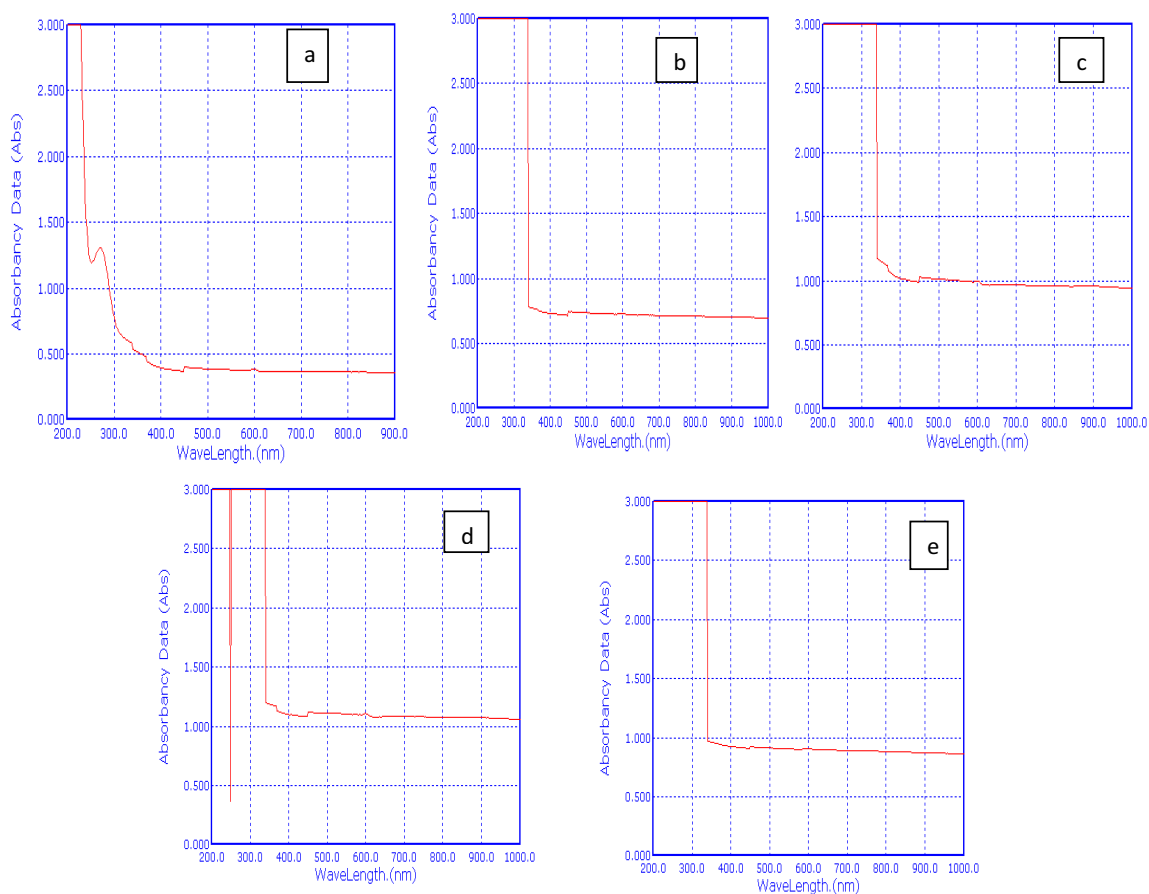


**Fig. 3.** SEM images of (a) Pure PAN (b) PAN: NaF (90:10) (c) PAN: NaF (80:20) (d) PAN: NaF (70:30) and (e) PAN: NaF (60:40).

NAF in the PAN electrolyte gave favorable conduction path for faster migration Na<sup>+</sup> in the electrolyte. These structural modifications result in disordered polymer chain and sustain the degree of amorphousity which improved the ionic conductivity.

### 3. 4. UV-VIS Spectra

The band gap energy was determined based on the numerical derivative of the optical absorption coefficient. The fundamental absorption method refers to band to band transitions by using energy



**Fig. 4(a-e).** UV-VIS Spectroscopic images of PAN: NaF polymer electrolyte films in different wt. % ratios at room temperature.

**Table 1.** Optical energy band gap for PAN based polymer electrolytes:

Polymer Electrolyte	Planck's constant (h) Joule- seconds	Light velocity (C) m/s	Wavelength (λ) A <sup>0</sup>	Optical energy Band gap in (ev)
Pure PAN	6.626 x 10 <sup>-34</sup> Joule- seconds	3 x 10 <sup>8</sup> m/s	350.12	3.54842
90:10	6.626 x 10 <sup>-34</sup> Joule- seconds	3 x 10 <sup>8</sup> m/s	340.0	3.6540
80:20	6.626 x 10 <sup>-34</sup> Joule- seconds	3 x 10 <sup>8</sup> m/s	612.0	2.0300
70:30	6.626 x 10 <sup>-34</sup> Joule- seconds	3 x 10 <sup>8</sup> m/s	702.0	1.7697
60:40	6.626 x 10 <sup>-34</sup> Joule- seconds	3 x 10 <sup>8</sup> m/s	346.0	3.5906

relation  $E = h\nu$ . Where  $h$  is Planck's constant and  $\nu = c/\lambda$ , where  $c$  is the speed of light in vacuum and  $\lambda$  is the wavelength of the spectrum [19]. The structural effect of salt on the conductivity was also confirmed by UV-Vis spectroscopy. The Fig. 4 shows that the UV-Vis spectra of PAN with different wt. % ratios of NaF salt at room temperature. The UV-Vis spectrum was recorded by a Hewlett-Packard HP8452A diode array spectrometer. The optical absorption particularly studying the shape and shift of absorption edge is very useful technique for understanding the basic mechanism of optically induced transitions in crystalline and non crystalline materials [20-21]. The absorption rate is slightly changed by increasing the salt ratio of solvents. The optical band gap of the polymer electrolytes was determined using UV-Visible spectra. From the spectra of UV-Vis the wavelength values are given in Table 1. It is cleared that the optical energy band gap is minimum for 70:30 samples and cleared that the conductivity is more when compared to remaining samples due to low optical band gap. These results suggest that there may be interaction between PAN and complexed NaF Salt.

### 3. 5. Conductivity Studies

The ionic conductivity versus salt concentration of polymer electrolyte PAN and

NaF system with varying weight percentage of salt in the temperature range 303 K to 373 K is shown in Fig. 5. The conductivity data at room temperature and at 373 K is represented in Table 2. From this we conclude that the conductivity increases as the salt content increases up to 30 wt%. Due to the high amorphousness of the polymer electrolyte films which provides more free mobile protons thus giving rise to the higher conductivity [22]. The enhancement in conductivity is not only due to the increment of mobile charge carriers but also due to Ethylene carbonate (EC), which allows greater dissolution of the electrolyte salt resulting in increased number of charge carriers and hence increase in conductivity. The maximum value of conductivity obtained at room temperature is  $1.82 \times 10^{-4} \text{ S cm}^{-1}$ . This conductivity value is 10 orders greater than that of Pure PAN ( $10^{-14} \text{ S cm}^{-1}$ ) as reported by Pan and Zou [23]. However, when more than 30 wt% of NaF has been added, the conductivity decreases and this might be due to ion pair or aggregates formation [24]. The polymer electrolytes leads to increase the viscosity of polymer electrolyte film due to incorporation of high amount of salt content the proton transportation is reduced and then impedes the mobility of charge carriers result in proton conductivity decreased. The variation of log conductivity as a function of inverse of absolute

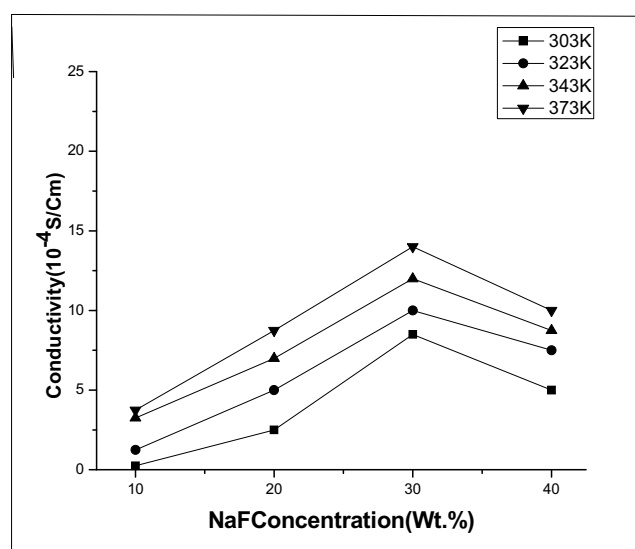


Fig. 5. Ion conductivity ( $\sigma$ ) of polymer electrolyte films as a function of NaF concentration.



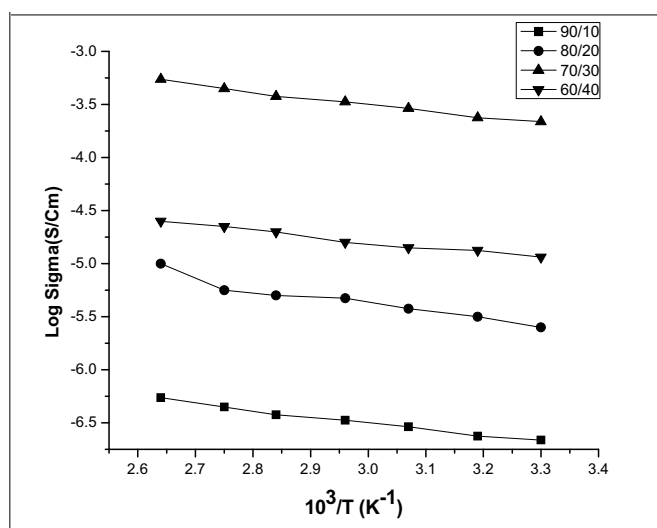


Fig. 6. The variation of log conductivity as a function of inverse of absolute temperature over a temperature range 303-373 K for various concentrations of NaF in PAN.

temperature over a temperature range 303-373 K for various concentrations of NaF in PAN is shown in Fig .6. From the Figure the conductivity can be increased with increasing of temperature. With increasing temperature the no of mobile charge carriers are enhanced resulting ionic conductivity increased [25].and due free volume model [26] and hopping of charge carriers between localized sites [27].

### 3. 6. Transference Number Measurements

Fig. 7. Transference number measurements of PAN: NaF polymer electrolyte films in different weight proportions at room temperature.

The ionic transference numbers of PAN and NaF measured using Wagner’s polarization

method. In this technique, the D.C current was altered as a function of the time on application of a constant dc voltage (1.5V) across the cell: Na/PAN: NaF/Ag cell. After polarization of the cell with 1.5DC, the polarization current versus time plot at room temperature is obtained, which is represent in Fig. 7. The transference numbers were calculated using the equations  $t_{ion}=1-I_f/I_i$ ,  $t_{ele}=I_f/I_i$ . Where  $I_i$  is the initial current and  $I_f$  is the final current. The calculated Transference numbers values are given in Table 2. The current decreases with time show that the total conductivity of the polymer electrolytes is predominantly due to ions. Hence, it proved that NaF salt has provided protons as mobile species in the polymer electrolyte systems. The values of

Table 2. Conductivity, activation energies and transference numbers of PAN: NaF films.

Polymer Electrolyte	Conductivity at		Activation energy(eV)	Transference Number	
	303K (S/Cm)	373K (S/Cm)		$t_{ion}$	$t_{ele}$
90PAN: 10NaF	$5.46 \times 10^{-6}$	$2.85 \times 10^{-5}$	0.47	0.965	0.04
80PAN: 20NaF	$1.62 \times 10^{-5}$	$2.42 \times 10^{-5}$	0.32	0.972	0.014
70PAN: 30NaF	$1.82 \times 10^{-4}$	$2.96 \times 10^{-3}$	0.25	0.989	0.01
60PAN: 40NaF	$2.64 \times 10^{-5}$	$1.32 \times 10^{-5}$	0.28	0.969	0.03

ionic transference numbers are in the range of 0.965-0.989.

### 3. 7. Discharge Characteristics

The solid state electrochemical cell was fabricated with the configuration of anode Na/PAN: NaF(70:30) /Cathode (I<sub>2</sub>+C+Electrolyte). The thickness of both the electrodes is 1mm. The surface area and thickness of PAN+ NaF Gel polymer electrolyte were 1.34cm<sup>2</sup> and 134 μm respectively. The discharge characteristics of the cell for a constant load of 100KΩ were evaluated at room temperature and were shown in Fig. 8. The initial sharp decrease in the voltage in these cells may be due to polarization and the formation of layer of Sodium salt at the electrode-electrolyte interface. Cell parameters like open circuit voltage (OCV), short circuit current (SCC), current density, power density, energy density and discharge capacity, have been evaluated to the highest conducting gel polymer electrolyte system PAN: NaF (70:30) in said electrochemical cell and the obtaining data are given Table 3. The current density is calculated using SCC value and area of the cell. Power density value is obtained by taking OCV and weight of the cell into consideration. Energy density value is calculated by evaluating the time taken for the plateau region.

From Table 3, it is obvious that the cell with the composition PAN: NaF (70:30) exhibits

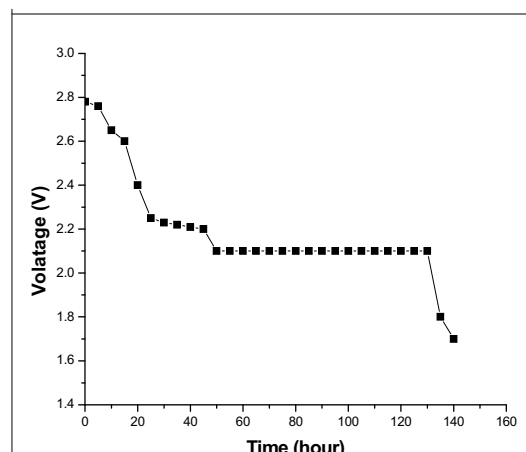


Fig. 8. Discharge characteristics of PAN: NaF (70:30) polymer electrolyte electrochemical cell (load =100KΩ).

better performance and stability than PEO: NaYF<sub>4</sub> and PEO: NaI complexed polymer electrolyte systems [28-30] and almost compatible with the existing reports of PVA:Na Br and PVP:NaI complexed polymer electrolytes [31-33]. Hence, the cell developed in this work offers interesting alternatives for temperature solid state batteries. The cell parameters acquired on this polymer electrolyte system are given in table. It is confirmed that gel state cell parameters are better than the earlier reported sodium based polymer electrolyte cell system [34-36].

Table 3. Cell parameters of PAN: NaF / (I<sub>2</sub>+C+Electrolyte) polymer electrolyte battery

Cell parameters	Na/PAN: NaF (70:30)/ (I <sub>2</sub> +C+Electrolyte).
Open circuit voltage (V)	2.78
Short circuit current (μA)	1.2
Effective area of the cell (cm <sup>2</sup> )	1.30
Weight of the cell (gm)	1.5
Discharge time (h)	118
Current density (μA cm <sup>-2</sup> )	0.982
Power density (W Kg <sup>-1</sup> )	2.942
Energy density (Wh Kg <sup>-1</sup> )	265.4
Discharge capacity (μA h <sup>-1</sup> )	152.4



#### 4. CONCLUSION

The proton conducting gel polymer electrolytes consisting of sodium fluoride salt dissolved in a plasticizing solvent, EC and DMF, immobilized in a host polymer PAN have been synthesized and characterized. The complexation of the salt with the polymer is confirmed by XRD and FTIR studies. Inter planar spacing is also verified theoretically and experimentally. It is clear that with increasing of salt concentration the surface of the films roughness has been decreased and phase segregation is revealed with cylindrical shape. Decrease in the degree of crystallinity and increase in the amorphous nature observed while increase in conductivity with increasing concentration of NaF and temperature. The transference number data indicate that the conduction in these polymer electrolytes is predominantly due to ions rather than electrons. UV-Vis light absorption reveals that the chemical structure of the polymer is identical to that of the polymer formed electrochemically. The surface morphology of the prepared samples is characterized by SEM. The various absorption rates given for different wavelengths and optical energy band gaps were determined. Using PAN: NaF (70:30) gel polymer electrolyte system solid-state battery (Na/PAN: NaF (70:30) +EC+DMF/ (I2+C+Electrolyte)) has been fabricated and their discharge characteristics are studied and these results are found to be comparable with existing results.

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