



ISSN: 2319-5967

ISO 9001:2008 Certified

International Journal of Engineering Science and Innovative Technology (IJESIT)

Volume 3, Issue 4, July 2014

Studies on electrical properties of PVA: NiBr₂ complexed polymer electrolyte films for battery applications

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Abstract: Nickel ion conducting solid polymer electrolytes based on polyvinyl alcohol (PVA) complexed with different weight percent ratios of nickel bromide (NiBr₂) salt were prepared by using solution cast technique. Structural characterization was performed using X-ray diffraction (XRD) technique and Fourier transform 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. Differential scanning calorimetry (DSC) was used to determine the glass transition and melting temperatures of pure PVA and PVA: NiBr₂ complexed polymer films. Electrical conductivity was measured using ac impedance analyzer in the frequency and temperature ranges 1 Hz - 1 MHz and 303 - 373 K respectively. It was observed that the magnitude of ionic conductivity increased with the increase in the salt concentration as well as temperature. 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 transport 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: Electrochemical cell, Ionic conductivity, Polymer electrolyte, Transference numbers.

I. INTRODUCTION

Solid polymer electrolytes (SPEs) have been extensively studied in the last two decades because of their potential applications in many technological areas, such as solid state batteries, electro-chemical sensors and electrochromic devices [1]-[3]. The pre-requisites for a polymer electrolyte for solid state battery applications are high ionic conductivity at ambient temperature, good mechanical strength, appreciable transference number, good thermal and electro chemical stabilities and better compatibility with electrodes. Pioneering work by Wright and co-workers [4] and Armand et al [5] has inspired a large number of experimental investigations on the properties of solid polymer electrolytes. The ionic conductivity of SPEs is due to the motion of dissolved ionic species (cations and anions) in a polymeric matrix and, in most cases, occurs in the amorphous phase. In recent years, considerable efforts have been devoted to improve the ionic conductivity and mechanical properties of SPEs.

PVA is a potential material having a high dielectric strength, good charge storage capacity and dopant dependent electrical properties. It has carbon chain back bone with hydroxyl groups attached to methane carbons. These O–H groups can be a source of hydrogen bonding and therefore, assist the formation of polymer complexes [6]. It has excellent mechanical properties and shows both ionic and electronic conduction [7]. When polymers are doped with inorganic salts, they show appreciable change in their structural as well as electrical properties. Polymer electrolytes containing divalent cations are suitable for electrochemical applications. Recently PVA doped with divalent metal salts like CuCl₂, MnCl₂ and MgBr₂ etc. In the present work NiBr₂ is doped into PVA polymer host matrix to enhance its electrical properties. Different characterization techniques (XRD, FTIR, DSC, AC impedance spectroscopy, etc) have been carried out to study the structural and electrical properties of PVA: NiBr₂



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complexed polymer electrolyte films. Using these polymer electrolytes, electrochemical cells were fabricated and their discharge characteristics were evaluated at a constant load of 100k Ω .

II. EXPERIMENTAL

Films (thickness 150 μm) of pure PVA and various compositions of complexed films of PVA with NiBr_2 salt were prepared by solution casting technique using double distilled water as a solvent. PVA and NiBr_2 salt were dissolved in double distilled water and stirred at room temperature for 10–12 h to get a homogeneous mixture. The solution was cast onto polypropylene dishes and allowed to evaporate slowly at room temperature. The final product was dried thoroughly to remove all traces of the solvent. The dried composite polymer films were peeled off from the polypropylene dishes and stored in a dry vacuum box. In the present study, X-ray diffraction spectra were recorded at room temperature in the Bragg angle range 10–70 $^\circ$ by a SEIFERT X-ray diffractometer. CuK_α radiation was used along with Zr filter as monochromatic radiation. Fourier Infrared spectra were recorded using EO-SXB IR spectrometer with a resolution of 4 cm^{-1} in the wave number range 400–4000 cm^{-1} . Differential scanning calorimetry (DSC) was used to determine the glass transition and melting temperatures of pure and NiBr_2 complexed PVA films. The impedance measurements were carried by a computer controlled phase sensitive multimeter (PSM 1700) in the frequency range 1Hz–1MHz and temperature range 303–373 K. The temperature was measured by copper–constantan thermocouple. Transference numbers (t_{ion} , t_{ele}) were evaluated by means of Wagner's polarization technique under a dc bias of 1.5 V. Electrochemical cells were fabricated with configuration Ni (anode) / (polymer electrolyte) / ($\text{I}_2+\text{C}+\text{electrolyte}$) (cathode) and their discharge characteristics were monitored at a constant load of 100 k Ω .

III. RESULTS AND DISCUSSIONS

A. XRD analysis

The XRD profiles of pure and NiBr_2 doped PVA with different weight percentages of NiBr_2 are shown in Fig. 1.

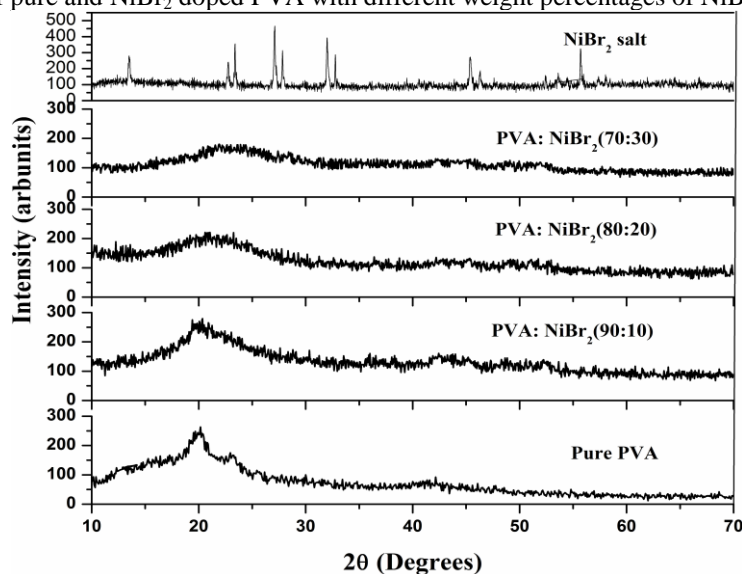


Fig. 1: XRD patterns of pure PVA and NiBr_2 doped PVA polymer electrolytes.

From the figure, well defined peaks were observed in NiBr_2 salt which indicates the crystalline nature of the salt. Pure PVA showed a characteristic broad peak for an orthorhombic lattice centered at 20 $^\circ$ indicating its semi crystalline nature [8]. With the addition of salt, the intensity of this peak decreases suggesting a decrease in the degree of crystallinity of the complex [9]. This could be due to the disruption of the PVA crystalline structure by NiBr_2 salt as suggested by Hodge et al [10]. They reported that the intensity of XRD pattern decreases as the amorphous nature increases with the addition of dopant. This amorphous nature results in greater ionic diffusivity and high ionic conductivity, which can be observed in amorphous polymers having flexible back-bone [11], [12]. No peaks corresponding to NiBr_2 salt were observed in complexed polymer films which indicates the complete dissolution of the salt in the polymer matrix.

B. FTIR analysis

Fourier transform infrared (FTIR) spectroscopy is a versatile tool to identify the chemical structure of polymeric materials and analyze their interaction behavior with dopant salt. It provides information about the interaction between the polymer and the ions. The FTIR spectra for pure PVA, NiBr₂ complexed PVA and pure NiBr₂ salt are shown in Fig. 2.

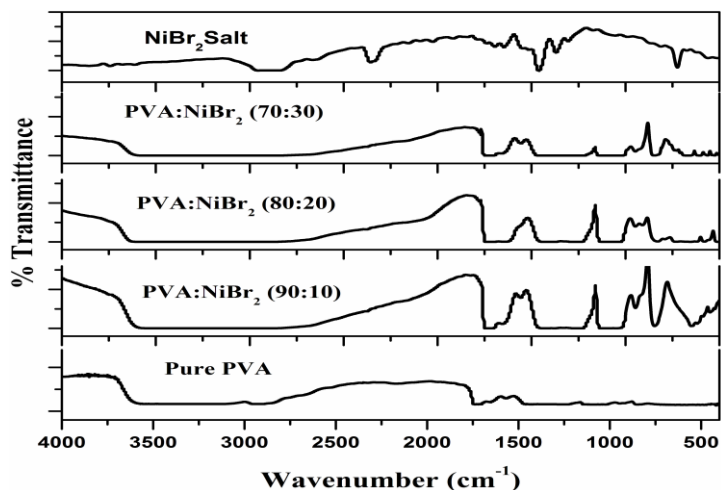
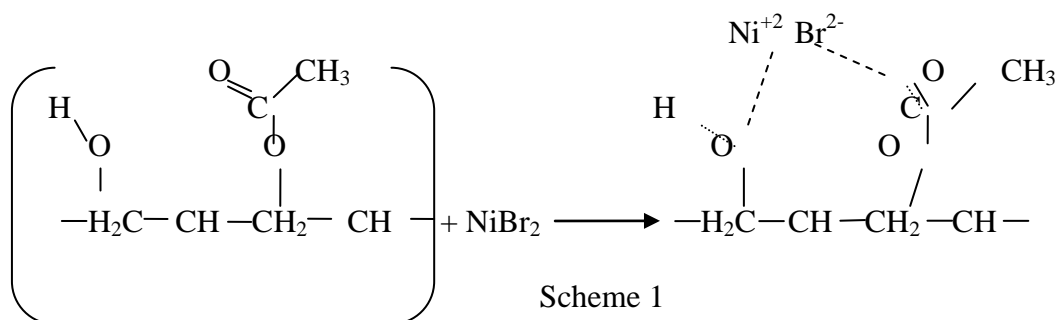


Fig. 2: FTIR patterns of pure PVA and NiBr₂ doped PVA polymer electrolytes.

The predominant O–H vibrational band of alcohols at 3581 cm⁻¹ (3400–3600 cm⁻¹) is shift towards the lower wave numbers and appeared as broad hydroxyl band in all the complexes as compared to pure PVA. It gives a strong indication of specific interactions between the polymer matrix and dopant salt. C–C stretching, C–H stretching of CH₂, C=O in pure PVA exhibited an absorption band at 1149 cm⁻¹, 1479 cm⁻¹ and 1750 cm⁻¹ respectively. These bands were shifted to lower frequencies and some peak intensities decreased with increase in salt concentration. The shift in CH₂ bending vibrations was from 1440 cm⁻¹ to 1389 cm⁻¹ while the band at 1409 cm⁻¹ indicates the chemical interaction of NiBr₂ ions with PVA matrix. The shift in acetyl C–O stretching of PVA was from 865cm⁻¹ to 862 cm⁻¹ with a sharp decrease in the peak intensity while the at 860 cm⁻¹ and 858 cm⁻¹ support the existence of chemical interaction. Scheme 1 represents the interaction of NiBr₂ salt with PVA matrix [13]. The observed changes in the intensities of bands, shifting of vibrational bands and disappearance of some bands indicates the formation of complexes [14].



The shift in frequency is correlated with force constant and bond length. The force constant values can be calculated from the expression [15]

$$\nu = \frac{1}{2\pi c} \sqrt{\frac{k(m_1 + m_2)}{m_1 m_2}} \quad \text{----- (1)}$$

where ν is the wave number (cm^{-1}), c the velocity of light ($3 \times 10^{10} \text{cm sec}^{-1}$), k the force constant (N cm^{-1}) and m_1 are m_2 atomic weights of the two atoms respectively. NiBr_2 interacts with the O–H group of PVA and C=O to form a complex. Due to this interaction, the frequencies corresponding to other vibrations in PVA such as C–H bending of CH_2 and C–O groups will be affected. The force constant values calculated for C–O stretching for pure PVA and PVA doped with different NiBr_2 salt concentrations varied from 3.03 N cm^{-1} to 2.98 N cm^{-1} . The force constant decreases with increase in salt concentration; this indicates that the Ni^{2+} ion interacts with the oxygen atoms in the polymer matrix which confirms the complexation.

C. DSC analysis

Differential scanning calorimetry is a technique to study the thermal transitions of a polymer. Using this technique the glass transition temperature (T_g) melting temperature (T_m) and the relative percentage of crystallinity ($\% \chi_c$) of the material can be identified. Fig.3 shows the DSC curves of pure PVA and PVA: NiBr_2 complexed films in the temperature range 300–550 K.

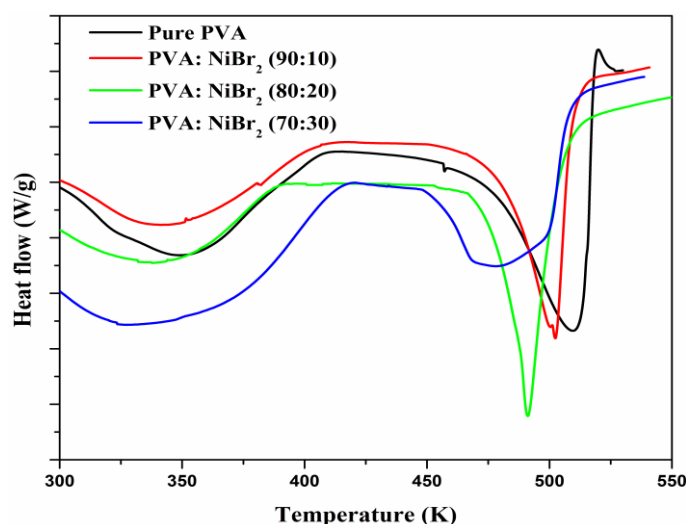


Fig. 3: DSC spectra of pure and NiBr_2 doped PVA polymer electrolytes.

DSC analysis has been done to correlate the glass transition temperature with the amorphousness and ionic conductivity of the polymer electrolytes. From DSC thermo grams an endothermic peak was observed around 320–350 K corresponding to the glass transition temperature (T_g) of the polymer electrolytes. T_g decreased with increase in salt concentration. The low glass transition temperature causes the higher segmental motion of the polymer electrolyte. Hence the ions move easily throughout the polymer chain. This enhances the ion transportation which achieves the ionic conductivity. The observed shift in T_g values of the polymer electrolytes in DSC thermo grams indicates the interaction between the polymer and the salt [16]. Another endothermic peak was observed around 498–520 K corresponding to the melting point of PVA based polymer electrolytes. Melting temperatures and enthalpies were determined from the position of the endothermic peak and from the peak areas respectively. The relative percentage of crystallinity ($\% \chi_c$) was estimated using the equation [17]

$$(\% \chi_c) = \frac{\Delta H_m}{\Delta H_m^0} \times 100$$

Where ΔH_m^0 is the melting enthalpy of pure PVA and ΔH_m is melting enthalpy of related NiBr_2 complexed PVA. The measured values of glass transition temperature, (T_g), melting temperature (T_m) and relative percentage of crystallinity ($\% \chi_c$) values are listed in Table I. From the table it is clear that T_g values and relative crystallinity ($\% \chi_c$) decrease with the increase of salt concentration. The decrease in melting temperature in the melting endotherm is a clear indication of decrease in the degree of crystallinity and dominant presence of amorphous phase. The T_g and χ_c has been found to be low for 30 wt% NiBr_2 doped polymer electrolyte system. The lower values of T_g and χ_c represent the high amorphous nature of the above mentioned polymer electrolyte as confirmed from the XRD results.

D. Impedance analysis

Impedance spectroscopy was employed to establish the conduction mechanism and to investigate the participation of the polymeric chain in the transportation process. Fig. 4 shows the Cole-Cole plots of PVA: NiBr₂ (70:30) polymer electrolyte film at different temperatures. Each plot consists of a high frequency semicircle and a low-frequency spike. The high frequency semicircle is due to the parallel combination of bulk resistance (due to the migration of ions) and bulk capacitance (due to the immobile polymer chains). The presence of the depressed semicircle reveals the non-Debye nature of the sample [18] due to the potential well for each site, through which the ion transport takes place, not being equal. The inclined spike represents formation of double layer capacitance at the electrode-electrolyte interface due to the migration of ions at low frequency. The bulk resistance (R_b) of the polymer electrolytes was determined from the interception of the semicircular arc on the real impedance (Z') axis. From the figure it is observed that the bulk resistance (R_b) decreases with increasing temperature. The ionic conductivity values of the polymer electrolytes were calculate at different temperatures and were found to increase with increase of temperature.

The ionic conductivity values at room temperature were calculate and are given in Table I. From the table it is clear that the ionic conductivity increases with increasing NiBr₂ content from 10 wt% to 30 wt%. Hopping mechanism between coordinating sites, local structural relaxations and segmental motions of the polymer chains are essential to assure high conductivity of the electrolyte [19]-[22].

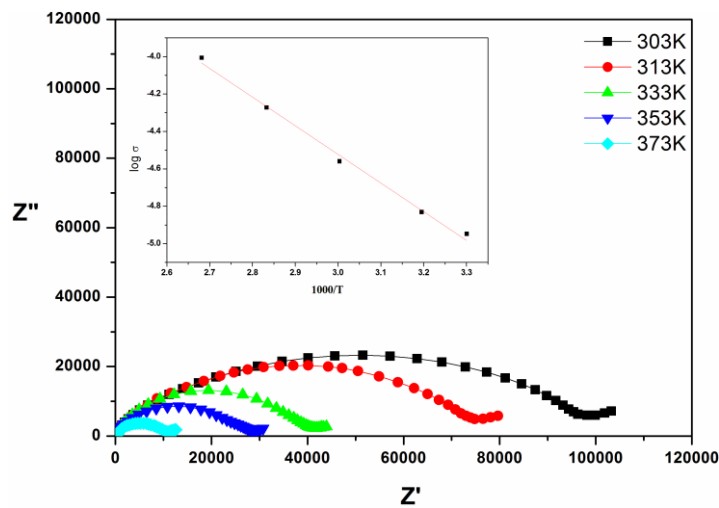


Fig. 4: Cole-Cole plots of PVA: NiBr₂ (70:30) at different temperatures. Variation of conductivity with temperature is inserted inside the figure.

E. Temperature dependent conductivity

The variation of electrical conductivity as a function of inverse temperature for 30 wt% NiBr₂ doped PVA polymer electrolyte film is inserted inside Fig. 4. The linear variation in logσ vs 1000/T plot suggests an Arrhenius type thermally activated process represented by the relation

$$\sigma_{dc} = \sigma_o \exp\left(-\frac{E_a}{kT}\right)$$

Where σ₀ is a pre exponential factor, E_a, the activation energy, k, the Boltzmann constant and T, the absolute temperature. The conductivity increased with increasing temperature for all complexes. Druger et al [23], [24] attributed the change in ionic conductivity with temperature in solid polymer electrolytes to segmental motion, which results in an increase in free volume of the system. Thus, the segmental motion either permits the ions to hop from one site to another or provides a pathway for ions to move. In other words, the segmental movement of the polymer facilitates the translational ionic motion. From this, it is clear that the ionic motion is due to translation motion or hopping facilitated by the dynamic segmental motion of the polymer. As the amorphous region increases, the polymer chain acquires faster internal modes in which bond rotations produce segmental motion to favour inter

and intrachain ion hopping, thus increasing the conductivity. The activation energy is a combination of the energy of defect formation and the energy of migration. The calculated activation energies values of all polymer electrolytes are listed in Table I. Polymer electrolytes having low activation energies are desirable for electrochemical applications [25].

Table I: Activation energy, glass transition temperature, melting temperature, relative percentage of crystallinity, transference numbers of pure and different compositions of PVA: NiBr₂ polymer electrolytes

Polymer electrolyte	Activation energy E _a (eV)	T _g K	T _m K	% χ _c	Transference number	
					t _{ion}	t _{ele}
Pure PVA	0.45	353	509	---		
PVA:NiBr ₂ (90:10)	0.33	340	501	66	0.96	0.04
PVA:NiBr ₂ (80:20)	0.32	335	492	52	0.967	0.033
PVA:NiBr ₂ (70:30)	0.31	330	485	41	0.972	0.028

F. Transference number

Ionic transference number is one of the most important parameter for the characterization of polymer electrolyte materials. Transference numbers were evaluated using Wagner’s polarizing technique [26]. To determine the ionic contribution to the total conductivity of the polymer electrolyte films, Ni / (PVA+ NiBr₂) / C cell was polarized at 303 K by a constant dc potential of 1.5 V. The polarization current versus time for different polymer electrolytes are shown in Fig.5. The appearance of initial polarization current on application of the potential was proportional to the dc applied field but subsequently it starts decreasing with time. This happens when the migration of ions due to the applied electric field is balanced by diffusion due to the concentration gradient, and hence, the cell gets polarized. The polarization current is then exclusively carried by electrons or holes. The transference numbers (t_{ion}, t_{ele}) were calculated using the equations and the values are listed in table I.

$$t_{ion} = 1 - \frac{I_f}{I_i}$$

$$t_{ele} = 1 - t_{ion}$$

Where I_i is the initial current and I_f, the final current. The ionic transference number (t_{ion}) values are in the range 0.96–0.972. This suggests that the charge transport in these polymer electrolytes are predominately due to ions. The electronic contribution to the total current is negligible in all the polymer electrolyte films. The ionic transference number (t_{ion}) of the present polymer electrolyte films is close to unity and hence these electrolytes are suitable for solid-state electrochemical cells as suggested by the other workers [27]-[29].

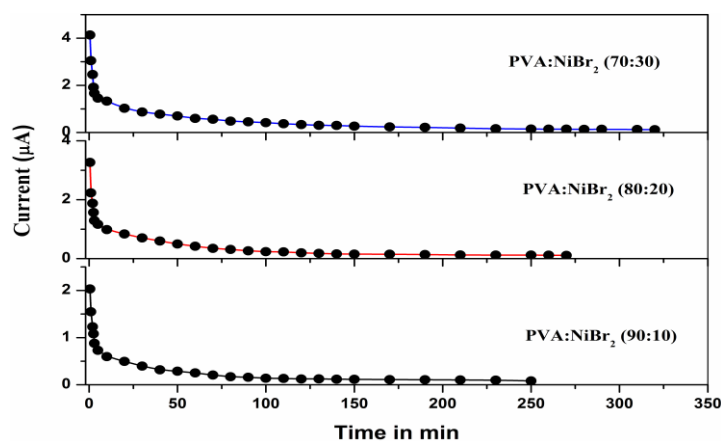


Fig 5: Transference plots of PVA: NiBr₂ polymer electrolyte films for different compositions.

G. Battery discharge characteristics

Solid state electrochemical cells were fabricated using (PVA: NiBr₂) films with in configuration Ni (anode) / polymer electrolyte / (I+C+electrolyte)/(cathode) for various compositions and their discharge characteristics were studied for a constant load of 100 kΩ at room temperature. Nickel metal was used as the anode material and for cathode; a mixture of I + C + electrolyte was made in the form of a pellet after proper mixing of the constituents. The addition of carbon powder to the cathode material enhances its electronic conductivity and hence, cathode can also act as a current collector. The addition of polymer electrolyte to the cathode mixture provides more interfacial contact area between the cathode and electrolyte and hence, reduces the interfacial resistance resulting in better battery performance [30].

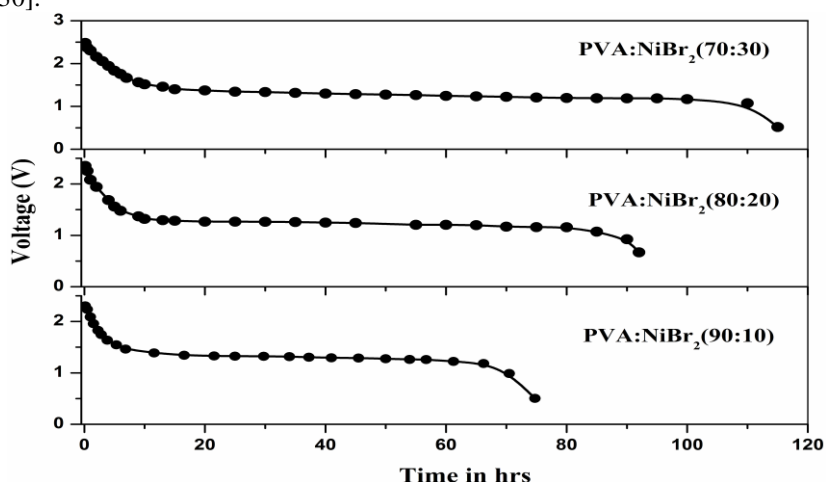


Fig 6: Discharge characteristics of (PVA: NiBr₂) electrochemical cell at a constant load of 100 kΩ.

Fig 6 shows the discharge profiles of the electrochemical cells of various compositions. The initial sharp decrease in the voltage for all the cells may be due to the polarization and / or formation of a thin layer of nickel salt at the electrode-electrolyte interface [31]. The cell parameters like open circuit voltage (OCV), short circuit current (SCC), current density, power density etc were evaluated for all the batteries and are given in Table II. From the table it is clear that, the cell with the composition (PVA: NiBr₂) (70:30) exhibits better performance and stability than other compositions. This data demonstrates the potential application of these polymer electrolytes in solid state battery applications.

Table II. Cell parameters of Ni/(PVA : NiBr₂)/(V₂O₅+C+electrolyte)polymer electrolytes

Cell parameters	(PVA+NiBr ₂) (90:10)	(PVA+NiBr ₂) (80:20)	(PVA+NiBr ₂) (70:30)
Open circuit voltage (V)	2.30	2.35	2.48
Short circuit current (μA)	120	210	998
Area of the cell (cm ²)	1.32	1.32	1.32
Weight of the cell (g)	1.31	1.30	1.32
Discharge time for plateau region (hours)	75	92	115
Current density (μAcm ⁻²)	91.6	161.5	212.1
Power density (W kg ⁻¹)	0.21	0.37	0.54
Energy density (Wh kg ⁻¹)	15.75	34.96	62.10
Discharge capacity(μA h ⁻¹)	1.60	2.28	2.43



ISSN: 2319-5967

ISO 9001:2008 Certified

International Journal of Engineering Science and Innovative Technology (IJESIT)

Volume 3, Issue 4, July 2014

IV. CONCLUSIONS

XRD analysis suggests an increase in the degree of amorphosity with increasing NiBr₂ salt concentrations in the polymer matrix. FTIR studies confirmed the complexation of the polymer with the dopant salt. Thermal analysis indicates the glass transition temperature and crystallinity to be low for PVA: NiBr₂ (70:30) complexed polymer electrolyte. The temperature dependence ionic conductivity follows the Arrhenius relation. The PVA: NiBr₂ (70:30) complexed polymer electrolyte showed the highest conductivity $1.13 \times 10^{-5} \text{ Scm}^{-1}$ and lowest activation energy 0.31 eV. Transference number data shows that the conductivity is mainly due to ions rather than electrons. Electrochemical cells were fabricated using these polymer electrolyte films and their discharge characteristics were studied.

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ISSN: 2319-5967

ISO 9001:2008 Certified

International Journal of Engineering Science and Innovative Technology (IJESIT)

Volume 3, Issue 4, July 2014

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M. Ravi received his Ph.D in Physics in 2012 from Sri Venkateswara University, Tirupati, India. He is 5 years of research experience in the field of polymer electrolyte for battery applications and 2 years of teaching experience in physics for B.Sc./M.Sc. students. He has published 15 research papers in international reputed journals. Presently he is working as postdoctoral fellow at Harbin institute of technology, P.R.China, to work on the project "multi walled carbon nano tubes (MWCNT) dispersed polymer electrolytes for ionic device applications".

V.V.R. Narasimha Rao former professor of physics at Sri Venkateswara University, Tirupati, India after serving for more than 35 years. He was specialized in the area of "thin films". He has published more than 100 research papers in international journals of high repute. Around 20 students were awarded Doctorate degrees under his supervision. He has passion for research and teaching.