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Optical, Thermal and Electrical studies of PVP based solid Polymer electrolyte For Solid state battery applications

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Abstract: A combination of ion conducting solid polymer electrolytes based on PVP (poly vinyl pyrrolidone) polymer was prepared by solution casting technique. The optical properties were studied by using UV-visible Absorption spectroscopy in the range of 200-800 nm. The absorption band gap is studied it is due to polymer chain complexed with salt. The complexation between (PVP and Mgcl₂6H₂O) has been observed from FTIR. The Glass Transition temperature was measured form DSC.The D.C Conductivity was measured at temperature range 303K to 373K. The ionic conductivity of PVP Polymer electrolyte is about 1.02×10^{-9} S/cm at Room temperature (RT). The highest ionic conductivity is found to be 2.05 $x10^{-5}$ S/cm at 373K, for 85:15 compositions. The ionic conductivity in the conduction process to be the Arrhenius-type thermally activated process. In these polymer electrolytes Charge carries of transference number is calculated by using Wagner's polarization technique. From the Transport Properties it concludes that in this polymer electrolyte the charge carries takes place mostly due to ions. The total ionic and electronic transference number was found to be 0.98 and 0.02in this solid polymer electrolytes. By using these polymer electrolytes a Solid state battery has been fabricated as well as discharge characteristics were studied under the constant load of 100k Ω . With the configuration of Mg⁺/(PVP+Mgcl₂6H₂O)/(I₂+C+electrolyte).Different battery parameters such as open-circuit voltage (OCV), short circuit current (SCC), energy density, and power density has been calculated.

Keywords: FTIR, DSC, Optical properties, Polymer electrolytes, Ionic Conductivity, Transference number, Electrochemical cell, and Discharge Characteristics.

Introduction:

In the past few decades tremendous research has done on Polymer electrolytes due to their characteristics. Ionic conductivity is the key role which takes place in the polymer electrolytes systems. Interest began in this field after the studies of materials based on alkali metal salts complexed with polyethylene oxide (PEO) reported by Wright and co-workers^{1,2}. Solid polymer electrolytes can easily fabricated with desirable sizes and good contact with electrode- electrode interfaces in different electrochemical devices Armand et al^{3 4}. Due to their potential difference in solid polymer electrolytes it is used in many industrial applications such as fuel cell, solid state battery, electro chromic devices, smart windows solar cell etc^{5,6}.

The present paper explains about the solid polymer electrolytes by solvent casting technique. Now a day's Research takes place in the field of rechargeable lithium batteries are directed towards the development of cells with high-energy density (Wh/kg) and high-power density^{7,8}. In these aspects polymers are being doped with inorganic salts for increasing the ionic conductivities for high performance in electro chemical applications⁹. To overcome this problem an attempt has been done in developing of new polymer electrolytes which is having high mechanical, thermal, electrochemical stability and high ionic conductivity^{10,11}. Water

soluble polymer is the main aspect in the industrial point view.Poly (vinyl pyrrolidone) (PVP) is a synthetic biodegradable polymer and it has excellent characteristics like dielectric constant, easy of solubility, stability, resistivity, moderate electrical conductivity and rich in charge transport mechanism and it is low cost which deserves an important role among the conjugated polymers. Another use of PVP is the thermally cross linked and high mechanical strength. The amorphous nature of the PVP gives the low scattering loss which gives the best results in optical applications.

In this present work PVP is chosen because of it characteristics.PVP is the good film forming and it has adhesive behavior on solid polymer electrolytes which gives the good optical properties and mechanical strength. PVP can be easily complexed with many inorganic salts which results the dispersion phase and surface passivation of them.SPEs based on Mg^{2+} ion-conducting salts have attracted considerable attention in the recent years for fabricating all-solid-state electrochemical power sources.

PVP of average molecular weight 360,000 from Sigma Aldrich chemicals. The solid polymer electrolytes were prepared by solvent casting technique. In this paper, solid polymer electrolyte films have been prepared by adding Mgcl₂6H₂O salt in PVP polymer and the films are characterized by several experimental techniques such as FTIR, DSC, Absorption band gap, the electrical properties of solid polymer electrolytes have been studied by DC Conductivity. By using these solid polymer electrolytes, an electro chemical cell has been fabricated and discharge characteristics were studied under the constant load of $100K\Omega$.

Experimental Section:

PVP (polyvinylpyrolidone) Sigma Aldrich chemicals based solid polymer electrolytes films doped with pure magnesium chloridehexahydrate were prepared in the different ratios (95:5) (90:10), (85:15) and (80:20) by using solution casting technique as double sterilized water as a solvent. Various concentrations of PVP and Mgcl₂6H₂O are mixed thoroughly, the mixture of these solutions was stirred for 24 hours to obtain a homogeneous solution.later the homogeneous solution was pored into the polypropylene dishes and evaporated slowly at room temperature under vacuum drying process. The polymer electrolyte films were taken off from the dishes and then placed in a desiccator until further test.

The dried polymer films were characterized with FTIR, DSC, and UV-Visible and DC conductivity. The complex formation between PVP and Mgcl₂6H₂O has been studied from the FTIR data. The FTIR spectral peaks were studied by using Perkin Elmer FTIR spectrometer in the range 4000 to 500 cm⁻¹. The effect of the solvent in the solid polymer electrolyte was measured from the UV-visible studies. The effect temperature and salt concentration on the conductivity of PVP was studied with lab made conductivity set up. The D.C conductivities were measured for the films at the temperature range 303-373K. The transport properties of ionic and electronic numbers (t_{ion} and t_{ele}) were calculated by Wagner's polarization technique, at constant voltage 2.5V. The screening current was analyzed as a function of time with Keithley electrometer (Keithley Inc., model 6514). An Electrochemical cell has been fabricated and its discharge characteristics were observed at constant load of 100 K Ω , with the configuration of Mg/(PVP+ Mgcl₂6H₂O)/(I+C+electrolyte).

Measurement Technique:

FTIR analysis:

Fourier transform infrared (FTIR) spectroscopy is promising analyzer which is used to analyze the functional groups and chemical structure involved in the polymeric materials and also identifies vibrational band in the polymeric films. In the present study the FTIR spectra of pure PVP, complexed with Mgcl₂6H₂O with different ratios are shown in Figure.1.



Fig.1 FTIR analysis (a) Pure PVP (b)PVP+Mgcl₂6H₂O (95:5) (c)PVP+Mgcl₂6H₂O (90:10) (d) PVP+Mgcl₂6H₂O (85:15) (e) PVP+Mgcl₂6H₂O (80:20)

An interaction takes place by doping the salt to the host polymer, the complexation occurs between salt and the host polymer matrix. In this view FTIR gives the possible complexation and interaction ions.PVP complexed with Mgcl₂6H₂O various compositions in the wave number range400-4000cm⁻¹. The vibrational band is observed at 600,1100,3000,3500cm⁻¹ are assisted to aliphatic C-H stretching, C-H₂ bending, CH₂ wagging and CN stretching of Pure PVP^{12,13}. The bands at 500-800cm⁻¹ obtained due to C-H stretching, C-H₂ bending of PVP respectively. The band at 2900-3500cm⁻¹ corresponding to salt which is completely mixed with the polymer further. It has been observed from the fig that the vibrational bands of pure PVP at1000, 1300 and1600cm⁻¹ the width of the bands is decreased with an increasing of dopant concentration. There is a difference in the spectral range that has been observed on comparing with the pure PVP. This is due to the stretching and bending of the ions which is completely mixed with the host polymer. In addition C-H bending vibrations takes place at 1100 cm⁻¹ to 3500 cm⁻¹ while the band at 1200 cm⁻¹ indicates that the cations of the salt get coordinated with the oxygen of PVP, this is due to the spectral peaks width changes These shifts in the bands may be attributed to the complex formation between PVP and Mgcl₂6H₂O salt. This influences the local structure of polymer backbones and significantly affects their mobility

DSC Studies:

The differential scanning calorimetry (DSC) curves with various ratios 95:5, 85:15 polymer electrolytes are given in Figure.2



Fig.2 DSC analysis a) Pure PVP b) PVP+Mgcl₂6H₂O(95:5) c) PVP+Mgcl₂6H₂O(85:15)

It is observed that with increasing the salt concentration the melting temperature (Tm) compositions of polymer electrolytes is decreased. From the DSC curves the optimum conduction for low melting point can be determined. The glass transition temperature for different composition is found at 84°C, 85°C. This is due to the plasticization of the polymer electrolyte with the addition of salt. The plasticization effect is relates to the dipole

interactions between the salt and the PVP polymer chains. In other words, lowering of degree of crystallinity of PVP. The lowering of Tg is expected to make the ions move easily in the prepared polymer electrolyte systems. Similar results have been reported by Hiran Kumar et al. for the $PVA:CH_3COONH_4$ polymer electrolyte system¹⁴. In addition, the melting temperature endotherm peak is found to wide with increase of salt concentration. It states that the decrease in the degree of crystallinity and dominant presence of amorphous phase.

Optical absorption studies:

Optical analysis is used for identify the optical band gap of the materials in the transmitting radiation. In an energy level a photon is absorbed in its orbit. When an electron is jumps from lower energy level to higher energy level. Transitions takes place in a band gap energy as it rise in the absorption process called as absorption edge, where the optical band gap energies be determined.

Absorption coefficient (α) was calculated by using the following equitation

$$I=I_{o}\exp\left(-\alpha x\right) \tag{1}$$

Hence $\alpha = 2.303/x$, $\log (I/I_o) = (2.303/x) A$

Insulators and semiconductors are classified in to two types (1) Direct band gap (2) Indirect band gap. If the top of the valence band and bottom of the conduction band are same i.e. zero crystal momentum then direct band gap takes place, where as in indirect band gap If the top of the valence band and bottom of the conduction band are not same i.e. zero crystal momentum. In the indirect band gap transitions takes place from Valence band to conduction band which is accumulated with phonon of magnitude of crystal momentum Davis and shalliday¹⁵. The Direct, Indirect band gaps and Absorption edge values are shown in the Table.1.

Table.1 optical band gap values of solid polymer electrolytes

Polymer electrolyte	Optical band gap		Absorption edge
	Direct(eV)	Indirect (eV)	
Pure PVP	5.2	5.1	4.93
PVP+ Mgcl ₂ 6H ₂ O (95:5)	4.99	4.87	4.84
PVP+ Mgcl ₂ 6H ₂ O (90:10)	4.96	4.86	4.80
PVP+ Mgcl ₂ 6H ₂ O (85:15)	4.45	4.85	4.73
PVP+ Mgcl ₂ 6H ₂ O (80:20)	4.89	4.83	4.65

Table 2. Conductivity values of (PVP+Mgcl₂6H₂O) polymer Electrolyte system

Films	Conductivity at <i>RT</i> (Scm ⁻¹)	Conductivity at 373 K (Scm ⁻¹)	
Pure PVP	1.02×10^{-9}	1.13×10^{-7}	
PVP+ Mgcl ₂ 6H ₂ O	$7.04 \mathrm{x} 10^{-8}$	2.86×10^{-6}	
(95:5)			
PVP+ Mgcl ₂ 6H ₂ O	3.59x10 ⁻⁷	6.52×10^{-6}	
(90:10)			
PVP+ Mgcl ₂ 6H ₂ O	1.37×10^{-7}	$1.57 imes 10^{-5}$	
(85:15)			
PVP+ Mgcl ₂ 6H ₂ O	2.62×10^{-6}	2.07×10^{-5}	
(80:20)			

To calculate band gap energy values graphs were plotted between (α) , $(\alpha hv)^2$, $(\alpha hv)^{1/2}$ as a function of *(hv)*. The linear portion of the (α) versus *(hv)* curves to zero absorption value. Absorption values for 5%,10%,15wt %,20Wt% of Mgcl₂6H₂O doped PVP films lies at 4.84,4.80,4.73 and 4.65 eV, which is observed from Figure.3.



Fig3. (α) vs (*hv*) (Photon energy) plots of (a) Pure PVP (b) PVP+Mgcl₂6H₂O (95:5) (c) PVP+Mgcl₂6H₂O (90:10) (d) PVP+Mgcl₂6H₂O (85:15) (e) PVP+Mgcl₂6H₂O (80:20)

In the energy level the photon is incident by its energy direct band gap exists and the absorption coefficient is given by

 $ahv = C (hv - E_g)^{1/2}$ (2)

Where E_g is the energy band gap, C is Constant which is dependent on the specimen structure, α is the absorption coefficient is the frequency of light and h is a planks constant.

From the graph $(\alpha hv)^2$ versus hv direct band gap values are obtained. Energy gap values are 4.99, 4.96, 4.45 and 4.89 eV as shown in Figure.4



Fig.4: (a) vs $(ahv)^2$ (Photon energy) plots (a) Pure PVP (b) PVP+Mgcl₂6H₂O (95:5) (c) PVP+Mgcl₂6H₂O (90:10) (d) PVP+Mgcl₂6H₂O (85:15) (e) PVP+Mgcl₂6H₂O (80:20)

where indirect band gap values are obtained by plotting $(\alpha hv)^{1/2}$ versus *hv* as shown in figure5 indirect band gap values are obtained from the graph are 4.87,4.86,4.85 and 4.83 respectively.

In an energy level phonon is required for transitions, where the absorption coefficient is fallowing by

$$\alpha hv = \mathbf{A} \left(hv \cdot E_g \cdot E_p \right)^2 + \mathbf{B} \left(hv \cdot E_g \cdot E_p \right)^2 \tag{3}$$

from the obtained values it is clear that in polymer chains incorporation of small amount of dopant takes place in a host lattice thus the activation energy is decreased such that the ionic conductivity is increased, which is observed from figure.5. The band gap, Direct and indirect band gaps values shifted to lower energies on doping with $Mgcl_26H_2O$ salt.



Fig.5 (a) vs $(\alpha hv)^{1/2}$ (Photon energy) plots (a) Pure PVP (b) PVP+Mgcl₂6H₂O (95:5) (c) PVP+Mgcl₂6H₂O (90:10) (d) PVP+Mgcl₂6H₂O (85:15) (e) PVP+Mgcl₂6H₂O (80:20)



Fig.6 Composition studies plots of (a) Pure PVP (b) PVP+Mgcl₂6H₂O (95:5) (c) PVP+Mgcl₂6H₂O (90:10) (d) PVP+Mgcl₂6H₂O (85:15) (e) PVP+Mgcl₂6H₂O (80:20)

DC conductivity:

The composition studies clear that the raise in conductivity takes place with a blend of Pure PVP and Mgcl₂6H₂O having different ratios as increasing in temp which shown in Figure.6.

The conductivity of pure PVP is found to be 1.02×10^{-9} Scm⁻¹ at RT and its value increases sharply to 1.13×10^{-7} Scm⁻¹ at 373K and the other salt percentage ratios are found to be 2.86×10^{-6} , 6.52×10^{-6} , 1.57×10^{-5} and 2.05×10^{-6} S/cm with 5%,10% 15% and 20% with Mgcl₂6H₂O respectively. From the values it is clear that there is increasing in conductivity as by adding the percentage of salt, and the order of conductivity raise to 2 for Pure PVP. Whereas the conductivity rises of order of 3 for other blends. It is observed that as increase in the temperature the conductivity raises. The conductivity was calculated by using

$$\sigma_{\rm dc} = L/RA. \tag{4}$$

The Arrhenius plots explain the conductivity phenomena, with increasing the salt concentration the conductivity increases, further by up on adding the salt percentage the conductivity is decrease. MacCullam et.al¹⁶ explains that initially the conductivity increases as the charge carriers increases. The conductivity is

decreased at higher concentration due to saggregations of ions, ion triplets and increase in the ion pairs which decrease the mobility and the charge carriers. Form the plot it is observed clearly that there is increase in conductivity for 85:15 as the salt percentage increases the ionic conductivity is decreased for 80:20 which is shown in Figure.7.



Fig.7 Dc conductivity plots (a) Pure PVP (b) PVP+Mgcl₂6H₂O (95:5) (c) PVP+Mgcl₂6H₂O (90:10) (d) PVP+Mgcl₂6H₂O (85:15) (e) PVP+Mgcl₂6H₂O (80:20)

The observed conductivity values, activation energy and transport properties for various compositions of solid polymer electrolytes are as shown inTable.2 and Table.2a.

Films	Activation energies (eV)		Transference numbers		
	Region I	RegionII	t _{ion}	t _{ele}	
Pure PVP	0.948	0.2831			
PVP+ Mgcl ₂ 6H ₂ O	0.7857	0.1492	0.94	0.06	
(95:5)					
PVP+ Mgcl ₂ 6H ₂ O	0.7927	0.1563	0.96	0.04	
(90:10)					
PVP+ Mgcl ₂ 6H ₂ O	0.1257	0.1394	0.98	0.02	
(85:15)					
PVP+ Mgcl ₂ 6H ₂ O	0.1257	0.1394	0.98	0.02	
(80:20)					

Table.2a Activation Energies and Transference Numbers of (PVP+Mgcl₂6H₂O) Electrolyte system

This change may occur when the transition takes place from semi-crystalline phase to amorphous phase¹⁷. A segmental motion occurs when the polymer chain acquires faster internal modes for which bond rotation takes place. Thus hopping mechanism takes place in between the ions and polymer chains and therefore the conductivity becomes high.

From plot it is observed that the highest conductivity was found to be 1.57×10^{-5} S/cm, for 15% in the temperature range 30° - 100° C.

Figure.6 composition studies are observed that as the salt percentage (wt %) is increasing the conductivity of all samples also increasing predominately.

Figure.7 explains the variation of activation energy with $Mgcl_26H_2O$ concentration. It is also observed that (PVP+ $Mgcl_26H_2O$) (85:15) has highest conductivity and lowest activation energy when compared with other samples.

Transference number:

To verify conduction process i.e. transference number measurements plays a major role in explaining the conductivity phenomena of the polymer electrolyte systems. The transference number corresponding to t_{ion} and t_{ele} in the polymer electrolyte system, and it was calculated by Wagner's polarizing technique. In this technique sample is sandwiched between the two electrodes (one is blocking and another is non blocking electrode) and is polarized with 2.5V.The screening current is noted with respect to time and shown in figure8.The initial current appears due to the potential and it is directly proportional to the applied field. Later the current starts decreasing with time because of the migration of ions.

The transference number $(t_{ion} \text{ and } t_{ele})$ are calculate from the polarization current verses time plots using the equitation's

$$t_{ion} = \dot{i}_t - \dot{i}_{ele}/\dot{i}_t$$
 and $t_{ele} = \dot{i}_{ele}/\dot{i}_t$ (5)

Where it is the initial current and iele is the final residual current for all compositions of PVP+ Mgcl₂6H₂O

The initial current is the total current of t_{ion} and t_{ele} , due to the applied voltage the polarization takes place and the ions are blocked at the blocking electrode, there by blocking of total and ionic current forms only the electronic current, from the plot the values of ionic transference number are in the range 0.94-0.98. It results that the charge transport in these polymer films is mainly due to ions, as well as minor of transfer of electrons also takes place

As the transference number of all samples is close to unity, here the charge takes place due to the magnesium ions. While the charge transport (t_{ele}) is very small.

Mobility of the ionic species (Mg^+) is measured using transient ionic current method¹⁸. The mobility of ions in SPE is determined using the equation

 $\mu = d^2/TV$

(6)

Where d is the thickness of the sample, T is the time of flight and V is the applied voltage (2.5V). The calculated mobility is found to be $2.45 \times 10^{-8} \text{m}^2/\text{Vs}$ at (85:15) for PVP+ Mgcl₂6H₂O

Electrolytes	Transference number (t _{ion})	Mobility (μ) (in (m²/Vs)
Pure PVA		$0.96 \times 10^{-8} \text{m}^2/\text{Vs}$
PVP+ Mgcl ₂ 6H ₂ O (95	.5) 0.94	$2.12 \times 10^{-8} \text{m}^2/\text{Vs}$
PVP+ Mgcl ₂ 6H ₂ O (90	:10) 0.96	$2.27 \times 10^{-8} \text{m}^2/\text{Vs}$
PVP+ Mgcl ₂ 6H ₂ O (85	:15) 0.98	$2.45 \times 10^{-8} \text{m}^2/\text{Vs}$
PVP+ Mgcl ₂ 6H ₂ O (80	:20) 0.98	$2.98 \times 10^{-8} \text{m}^2/\text{Vs}$

Discharge characteristics:

Solid state battery has been fabricated at room temperature using polymer electrolyte films with a combination of PVP doped with Mgcl₂6H₂O polymer battery was fabricated with the configuration of Mg(anode)/polymer electrolyte/(I+C+electrolyte)/(cathode). Here the magnesium metal is used as anode while, a mixture of I + C + electrolyte is used as cathode. The electrode materials of anode and cathode were made in the form of a pellet having a thickness1mm. In the anode region due to the magnesium metal the charge carrier's takes place and in cathode the mixture of iodine and carbon powder material enhances its electronic conductivity¹⁹. And their discharge characteristics were studied for a constant load of 100 k Ω .



Fig.8 Transport characteristics plot (a) PVP+Mgcl₂ $6H_2O$ (95:5) (b) PVP+Mgcl₂ $6H_2O$ (90:10) (c) PVP+Mgcl₂ $6H_2O$ (85:15) (d) PVP+Mgcl₂ $6H_2O$ (80:20)



Fig.9 Discharge characteristics plots (a)PVP+Mgcl₂ $6H_2O$ (95:5) (b) PVP+Mgcl₂ $6H_2O$ (90:10) (c) PVP+Mgcl₂ $6H_2O$ (85:15) (d) PVP+Mgcl₂ $6H_2O$ (80:20)

Figure.9 shows the discharge characteristics of solid state battery with different percentages. At first there is a sharp decrease in the voltage for all the ratios this is may be due to the polarization of current in an electrolyte and formation of a thin layer of magnesium salt between the electrode-electrolyte interfaces²⁰. The cell parameters like open circuit voltage (OCV), short circuit current (SCC), current density, power density etc were calculated for all the batteries and are given in Table4. From the plots of discharge characteristics it clearly shows that, the cell with the composition (PVP+Mgcl₂6H₂O) (85:15) exhibits better performance and stability than other

Cell parameters	PVA+Mgcl ₂ 6H ₂ O			
	(95:5)	(90:10)	(85:15)	(80:20)
Cell weight (g)	1.34	1.32	1.33	1.33
Area of the cell (cm ²)	1.45	1.45	1.45	1.45
Open circuit voltage (O	C V) 1.7	1.87	2.01	1.97
Discharge time (Hrs)	90	97	115	107
Current density (mA/cm	n^2) 0.75	0.8	1.07	1.3
Discharge cell (mA/h)	12.2	12.5	13.5	17.6
Power density (W/Kg)	1.39	1.72	2.35	2.79
Energy density (W h/Kg	() 125.1	166.84	270.2	298.53

Table 4.CellparametersofMg/[Mgcl₂6H₂O]/(I₂+C+electrolyte)

Table 5: cell parameters of polymer electrolyte batteries

Solid state electro chemical cell	Open Circuit voltage (OCV)	(V)Discharge time for plateau region (Hrs)	References
Na/(PVA + NaF)/(I ₂ + C + electrolyte)	2.53	112	28
Mg/(PVA+ Mg(CH ₃ COO)2)/(I ₂ + C+ electrolyte)	1.84	87	16
Mg/(PEO +Mg(NO ₃) ₂)/(I ₂ + C + electrolyte)	1.85	142	3
K/(PVP+PVA+KClO ₃)/(I ₂ + C + electrolyte)	2.00	52	29
$K/(PVP + PVA + KBrO_3)/(I_2 + C + electrolyte)$	2.30	72	30
Mg/(PEG + Mg(CH ₃ COO) ₂)/(I ₂ + C +electrolyte)	1.84	32	31
$Ag/(PEO + AgNO_3)/(I_2 + C + electrolyte)$	0.595	48	32
K/(PVA + CH ₃ COOK)/(I2 + C + electrolyte)	2.0	75	10
Mg/[Mgcl ₂ 6H ₂ O]/(I ₂ +C+electrolyte)	2.1	115	Present

Compositions²¹.cell parameters of the various solid polymer electrolytes are comparable with the present work and it is shown in Table.5.

From these cell parameters demonstrates that the potential applications of these polymer electrolytes in solid state electro chemical cells.

Conclusions

FTIR states that the complexation between the salt and polymer.DSC curves indicates that the melting temperature if found at 84° C.From the conductivity studies the highest conductivity for PVP+ Mgcl₂6H₂O (85:15) was found to be 1.57×10^{-5} S/cm at temperature 373K and the transference of ionic and electronic numbers are found to be in the range 0.96-0.98, this reports that the charge carriers in these polymer electrolyte films are mainly due to ions rather than electrons. By using these polymer electrolyte films a solid state battery has been fabricated and their discharge characteristics were studied.

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