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Effect of Al₂O₃ on PVP based Polymer electrolyte films doped with Mgcl₂ 6H₂O For Solid State Battery applications

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Abstract : A new Mg^{2+} ion conducting solid polymer electrolytes was prepared with the influence of Nano sized Al₂O₃ particles, PVP (poly vinyl pyrrolidone) has a host polymer with Mgcl₂6H₂O salt by solution casting technique. Several experiments techniques have done to fabricate of a battery. The salvation between (PVP, Mgcl₂6H₂O and Al₂O₃) has been observed from FTIR. The Glass Transition temperature, melting point 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. It was found that the Nano sized Al₂O₃ doped polymer films conductivity is found to be 4.03 $\times 10^{-6}$ S/cm at 373K, for 85:15 compositions. The ionic conductivity in the conduction process to be the Arrhenius-type thermally activated process. From the transference number it is confirmed that the polymer electrolyte with the Nano fillers having charge transport in is mostly due to ions and minority of charge carriers by the electrons and it is calculated by using Wagner's polarization technique. The total ionic and electronic transference number was found to be 0.98 and 0.02in these solid polymer electrolytes.By using these polymer electrolytes an electrochemical cells has been fabricated with the configuration of $Mg^+/(PVP+Mgcl_2O+Al_2O_3)/(I_2+C+electrolyte)$ and discharge characteristics were studied under the constant load of $100k\Omega$ various cell parameters such as open-circuit voltage (OCV), short circuit current (SCC), energy density, and power density has been calculated.

Keywords: FTIR, DSC, Polymer electrolytes, Ionic Conductivity, Transference number, Electrochemical cell, Discharge characteristics.

Introduction:

A primary work in the field of solid polymer electrolyte has been carried by Wright and Co Workers^{1,2} and found that the ionic conductivities is the order of 10⁻⁵ S/cm at 330 K in PEO and NaSCN complexes. Then attention has made and considerable work has done in this field³. A wide range of interest has been made to prepare solid polymer electrolytes over a few decades due to their potential applications as electrolyte materials in high energy density batteries, rechargeable batteries and solid state batteries⁴. Magnesium is used as the excellent material for anode in solid state batteries which is naturally available in earth crust, in addition magnesium based devices is much chipper than lithium devices. Whereas magnesium is used instead of lithium for solid state battery applications⁵. The order of conductivity of pure PVP is around 10⁻⁹ to 10⁻⁷ S/cm at room temperature. The order of ionic conductivity is not suitable for battery applications. The order of ionic conductivity was improved of 10⁻⁷ to 10⁻⁵ S/cm by the addition of Nano particles Al₂O₃ with Mgcl₂6H₂O in solid polymer electrolyte. About 19 years ago Gregory et.al⁶ reported the electro chemical reversible deposition and

dissolution process in (Mg Bph₂Bu₂)₂.Where polyvinyl pyrrolidone (PVP) has major role in displaying dissolubility, stability, high-dielectric constant, compatibility and resistance and PVP films is synthesized lab made at low cost. PVP is soluble in polar solvents as well as ionized water, another advantage of using PVP is that it is thermally stable and cross linked with the composites and having high mechanical strength. Furthermore, the amorphous nature of PVP has low scattering loss, which is more useful for optical application. PVP is chosen because it is having good mechanical, electrical and optical characteristics, one of the properties that it has good film-forming which exhibits good optical properties and mechanical strength. And pyrrolidone group of PVP prefers to complex with many inorganic salts resulting in fine dispersion and Surface passivation of them⁷. Solid polymer electrolytes complexed with inorganic salt and Nano fillers/plasticizers were first

In this present work we report that solid polymer electrolyte films is prepared by adding the Al_2O_3 particles to PVP -Mgcl₂ 6H₂O the purpose of the work to improve the ionic conductivity by comparing with the other polymer electrolytes. The obtained results demonstrate that the electrical conductivity nature is much improved by adding the Al_2O_3 in PVP-Mgcl₂6H₂O.these polymer electrolytes were characterized with several experimental techniques such as FTIR, DSC 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 Ω .

introduced by Weston and Steele in 1982⁸ the addition of the fillers in the solid polymer electrolytes improves

Experimental Section:

the electrical and mechanical strengths of the polymer⁹.

Sample preparation:

PVP (polyvinylpyrolidone) Sigma Aldrich chemicals based solid polymer electrolytes films doped with pure magnesium chloridehexahydrate and Al_2O_3 Nano particles were dispersed 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. The mixture of these solutions was stirred for 24 hours to obtain a homogeneous solution.later the homogeneous solution was placed 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.

Measurement Technique:

FTIR analysis:

The FTIR spectra were recorded with the help of Perkin Elmer FTIR spectrometer in the wavelength range 4000 to 500 cm⁻¹. The complex formation and elimination of hydroxyl groups between PVP, Mgcl₂6H₂O and Al₂O₃ has been studied.

Fourier transform infrared (FTIR) spectroscopy is a versatile instrument used for the identification of functional groups, chemical structure and elimination of hydroxyl groups on the surface of the polymer films. In the present study the FTIR spectra of pure PVP, Mgcl₂6H₂O and Al₂O₃ with different ratios are shown in fig.1

However the transmittance occurs at 1000, 1100, 13000, 3000, 3500cm^{-1} are assisted to aliphatic corresponding to C=O stretching, C=N bending and C=C rocking respectively of Pure PVP^{10, 11}. A broad peak is observed at 1100 cm⁻¹ it is formed due to the vibration oscillation of hydroxyl group (δ O-H) while the similar peak is not observed for pure PVP it may due to the absence of stretching bands. The spectra of PVP, Mgcl₂6H₂O and Al₂O₃ films show variation in peaks at different wavelength. The increasing in the intensity at higher wave length is obtained due to the strong interaction between polymer matrix which is assigned to Mg-O-Al₂ -Mg stretching and dopant, and it is found at 1600 cm⁻¹. Hence it is confirmed that stronger molecular interaction exists between polymers and dopant attributed to the charge of p- π conjugation associated with the amide group of PVP arising dopant. The peak intensity changes at 3100-3900cm⁻¹ is due to the dissolution of nano particles with salt and the polymer. It has been observed from the fig that the vibrational bands of pure PVP at1100, 1300 and1600cm⁻¹ the width of the bands is decreased with an increasing of dopant concentration as a result a strong columbic interaction takes place between PVP and Mg ions. In addition C-H bending vibrations takes place at 1300 cm⁻¹ to 3900 cm⁻¹ while the band at 1600 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 nano particles polymer and salt¹². This influences the local structure of polymer backbones and significantly affects their mobility.



Fig.1 FTIR analysis (a) Pure PVP (b) PVP+Mgcl₂6H₂O+Al₂O₃ (95:5) (c) PVP+Mgcl₂6H₂O+Al₂O₃ (90:10) (d) PVP+Mgcl₂6H₂O+Al₂O₃ (85:15) (e) PVP+Mgcl₂6H₂O+Al₂O₃ (80:20)



Fig.2 DSC analysis (a) Pure PVP (b) PVP+Mgcl₂6H₂O+Al₂O₃ (95:5) (c) PVP+Mgcl₂6H₂O+Al₂O₃ (90:10) (d) PVP+Mgcl₂6H₂O+Al₂O₃ (85:15) (e) PVP+Mgcl₂6H₂O+Al₂O₃ (80:20)

DSC studies:

The Glass transition temperature was analyzed from the TA Instruments DSC Q20 V24.11 Build 124. From the DSC curves the melting point and glass transition temperature can be determined. DSC curves with various ratios of pure 95:5, 90:10, 85:15, and 80:20 polymer electrolytes are given in fig.2

From the figure it is observed that the melting point for pure PVP is found at 100° c. it indicates that the amorphocity nature of the films. By doping the nano particles and the salt concentration to the polymer the melting temperature (Tm) 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 73° c, 70° c, 82° c and 69° c. Due to the influence of Al₂O₃ nano particles on the polymer films dipole interactions takes place between the salt and the PVP polymer chains, at the melting point lowering of degree of crystallinity forms in all the polymer electrolyte films. This results that at the melting point the crystalline nature of the salt and the polymer converts amorphous such that the conductivity nature in polymer film is increasing at the melting point¹³.Similar results have been reported by Hiran Kumar et al. for the PVA:CH₃COONH₄ 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.

Wt% composition studies:

The variation of DC conductivity (σ) as a function of different wt% of Mgcl₂6H₂O doped with nano particles as shown in fig3. The conductivity of pure PVP is found to be 1.11×10^{-9} S/cm at RT, from the composition studies it is observed that the ionic conductivity of all samples is increases with the addition of salt concentration (wt%). It is observed that at higher concentration the conductivity become slower when comparable with the other samples this may be due to the saggregations of ions in polymer electrolyte. The motion of ions in solid polymer electrolyte is like liquid like flow mechanism by which the moment of ions through the polymer matrix is assisted by a large multiples of polymer segmental motion^{15,16}.



Fig.3 Wt% analysis (a) Pure PVP (b) PVP+Mgcl₂6H₂O+Al₂O₃ (95:5) (c) PVP+Mgcl₂6H₂O+Al₂O₃ (90:10) (d) PVP+Mgcl₂6H₂O+Al₂O₃ (85:15) (e) PVP+Mgcl₂6H₂O+Al₂O₃ (80:20)

DC conductivity studies:

The DC conductivities of the polymer films were screened at the temperature range 303-373K. The variation of temperature with conductivity of polymer electrolyte was studied with lab made conductivity set up.



Fig.4 Conductivity analysis (a) Pure PVP (b) PVP+Mgcl₂6H₂O+Al₂O₃ (95:5) (c) PVP+Mgcl₂6H₂O+Al₂O₃ (90:10) (d) PVP+Mgcl₂6H₂O+Al₂O₃ (85:15) (e) PVP+Mgcl₂6H₂O+Al₂O₃ (80:20)

The conductivity of Pure PVP by adding the Al_2O_3 nano particles in Mgcl₂6H₂O was found to be increasing in temp and shown in fig 4 .The data reveals that the conductivity of pure PVP is 1.11×10^{-9} Scm⁻¹ at RT and its value increases sharply to 1.23×10^{-7} Scm⁻¹ at 373K and the other salt percentage ratios are found to be 2.77×10^{-8} to 2.96×10^{-7} , 6.09×10^{-8} to 8.05×10^{-6} , 3.08×10^{-8} to 1.33×10^{-5} and 1.85×10^{-7} to 1.46×10^{-5} S/cm with 5%,10% 15% and 20% with Al_2O_3 doped in Mgcl₂6H₂O respectively. From the values it is observed that the increase in 2 order in case of Pure PVA where as in case of other blends it is to be 2,3 orders. And it reveals that their increase in conductivity by adding the salt concentration. From the figure It is clear that as increase in the temperature the conductivity is increases. The conductivity was calculated by using

$$\sigma_{\rm dc} = i x l / V x A. \tag{1}$$

Where i is the current, l is the thickness of the film is the applied voltage and A is the area of the cross section of the film.

The observed conductivity values and transport properties for various compositions of solid polymer electrolytes are as shown in Table.1

Films	Conductivity at RT	Conductivity at 373K	Transference numbers	
	(S/cm)	(S/cm)	t _{ion}	t _{ele}
Pure PVP	1.11x10 ⁻⁹	1.23×10^{-7}		
PVP+Mgcl ₂	2.77x10 ⁻⁸	2.96x10 ⁻⁷	0.97	0.02
6H ₂ O(95:05)				
PVP+Mgcl ₂	6.09x10 ⁻⁸	8.05x10 ⁻⁶	0.96	0.03
6H ₂ O(90:10)				
PVP+Mgcl ₂	3.08x10 ⁻⁸	1.33x10 ⁻⁵	0.98	0.01
6H ₂ O(85:15)				
PVP+Mgcl ₂	1.85×10^{-7}	1.46x10 ⁻⁵	0.94	0.05
6H ₂ O(80:20)				

Table1.	Conductivity a	and Transference	numbers of ()	PVP+Mgcl ₂	6H2O+Al2O3) Electroly	te System
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Jeon et.al¹⁷ reported that the ionic conductivity of the polymer electrolyte is increased with the addition of salt concentration, but further addition of salt to the polymer the conductivity may decreased at a higher concentration, because of saggregations of ions which decreases overall mobility Thus the conductivity reduces MacCullam et.al¹⁸ The increase in conductivity with temperature explained by Arrhenius plots, as the temperature increased the conductivity of the electrolyte films also increased This favors hopping of ions with in between the chains due to the hopping mechanism between coordinating sides, local structure relaxation and segmental motion of the polymer Conductivity in these regions is given by Arrhenius behavior as the ionic conductivity is enhanced almost linearly with increase in temperature Miyamoto and shibayama¹⁹ explained that the ionic conductivity is increased with increase in temperature as a result free volume model where as the temperature increases the polymer electrolyte can explained easily and produce free volumes therefore ions solvated molecules or polymer segments can move in to free volume causing it to increase²⁰ this enhance the ions and the polymer segmental mobility which will in turn with the ionic conductivity

A plot of sigma DC verses 1000/T for pure PVP in the temperature range  $30-100^{\circ}$ C is plotted and the conductivity was found to be  $1.33 \times 10^{-5}$  S/cm

# Activation energy studies:

From the fig.5 it is observed that the activation energies is decreases with increasing the salt concentration this may be due to the mixture of amorphous and crystalline regions. And the conductivity of the films is increases due the amorphousity. The activation energy values for solid polymer electrolytes are given in Table2 .This may be due to fact that the addition of small amount of dopant forms charge complexes in the host lattice²¹.the charge transfer complex increase the electrical conductivity by providing additional charges in the lattice these results in a decrease of activation energy.

Films	Activation energy		Mobility (µ) (m²/Vs)
	Region1	Region 2	
Pure PVP	0.59	0.34	$3.48 \times 10^{-13} \text{m}^2/\text{Vs}$
PVP+Mgcl ₂ 6H ₂ O(95:05)	0.48	0.27	$4.41 \times 10^{-13} \text{m}^2/\text{Vs}$
PVP+Mgcl ₂ 6H ₂ O(90:10)	0.46	0.23	$5.76 \times 10^{-13} \text{m}^2/\text{Vs}$
PVP+Mgcl ₂ 6H ₂ O(85:15)	0.39	0.17	$8.02 \times 10^{-13} \text{m}^2/\text{Vs}$
PVP+Mgcl ₂ 6H ₂ O(80:20)	0.33	0.21	$1.06 \times 10^{-12} \text{m}^2/\text{Vs}$

Table 2. Activation energy and mobility values for (PVP+Mgcl₂6H₂O+Al₂O₃) Electrolyte Systems



Fig.5 Activation energy studies (a) Region I (b) Region II

### **Transference number:**

The ionic and electronic transference numbers ( $t_{ion}$  and  $t_{ele}$ ) were calculated by Wagner's polarization technique, by applying constant voltage 2.5V. The monitored current was calculated as a function of time with Keithley electrometer (Keithley Inc., model 6514).

To verify conduction process a 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 fig.6 The initial current is obtained due to the potential when an electric field is applied. Due to the saturation of ions the current starts decreasing with respect to time.



# Fig.6 Transport analysis (a) PVP+Mgcl₂6H₂O+Al₂O₃ (95:5) (b) PVP+Mgcl₂6H₂O+Al₂O₃ (90:10) (c) PVP+Mgcl₂6H₂O+Al₂O₃ (85:15) (d) PVP+Mgcl₂6H₂O+Al₂O₃ (80:20)

The transference number ( $t_{ion}$  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  $t_{ele} = \dot{i}_{ele}/\dot{i}_t$  (2)

Where  $i_t$  is the initial current and  $i_{ele}$  is the final residual current of Solid polymer electrolyte

The initial current is the total current of  $t_{ion}$  and  $t_{ele}$ , due to the applied voltage of 2.5V the polarization takes place in polymer electrolyte and the ions are blocked at the blocking electrode, from unblocking electrode the ions get drifted due to polarization. Thus blocking of total ionic and electronic current forms only the ionic current, from the plot the values of ionic transference number are in the range 0.94-0.98. It results that the charge transport takes place in these polymer films is mainly due to ions, as well as residual of electrons is also takes place. The charge takes place in solid polymer due to the magnesium ions. The transport properties of all samples are close to unity, while the charge transport ( $t_{ele}$ ) is negligible.

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

$$\mu = d^2/TV \tag{3}$$

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  $8.02 \times 10^{-13} \text{m}^2/\text{Vs}$  at (85:15) for PVP+Nano filler+Mgcl₂6H₂O

#### **Discharge characteristics:**

Solid state battery has been fabricated at room temperature using polymer electrolyte 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 the mixture of iodine and carbon powder is act as active cathode material which enhances its electronic conductivity²², their discharge characteristics were studied for a constant load of 100 k $\Omega$ .

An Electrochemical cell has been fabricated and its discharge characteristics were measured at constant load of 100 K $\Omega$ , with the configuration of Mg/(PVP+Al₂O₃+Mgcl₂6H₂O)/(I+C+electrolyte).initially a rapid decrease in voltage is occurs it may be due to the polarization and/or the formation of thin layer of magnesium salt at the electrode/electrolyte interfaces²³.



### Fig.7 Discharge characteristics studies for 85:15 ratio

Fig.7 shows the discharge characteristics of solid state battery hiving highest conductivity for 85:15. 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 Table3. From discharge characteristics it clearly shows that, the cell (PVP+Mgcl₂6H₂O+Al₂O₃) (85:15) is having long durability and exhibits better performance.

Table3. Cell parameters of Mg/[ PVP+Mgcl₂6H₂O+Al₂O₃]/ (I₂+C+electrolyte)

Cell parameters	PVP+Mgcl ₂ 6H ₂ O (85:15)		
Cell weight (g)	1.78		
Area of the cell $(cm^2)$	1.35		
Current density ( $\mu$ A/cm ² )	2.14		
Open circuit voltage (OCV)	1.75		
Discharge time (Hrs)	118		
Power density (W/Kg)	2.85		
Energy density (W h/Kg)	336.3		
Discharge cell ( $\mu$ A/h)	24.5		

Compositions²¹.cell parameters of the various solid polymer electrolytes are comparable with the present work and it is shown in Table.4.

Table 4: Cell parameters of poly	mer electrolyte batteries
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Solid state electro chemical cell	Open Circuit voltage(OCV)	(V)Discharge time for plateau region (h)	Reference
$Na/(PVA + NaF)/(I_2 + C + electrolyte)$	2.53	112	16
$Mg/(PVA+Mg(CH_3COO)_2)/(I_2 + C+electrolyte)$	1.84	87	28
$K/(PVP+PVA+KClO_3)/(I_2 + C + electrolyte)$	2.00	52	29
$K/(PVP + PVA + KBrO_3)/(I_2 + C + electrolyte)$	2.30	72	30
$Mg/(PVP + Mgcl_2 6H_2O)/(I_2 + C + electrolyte)$	1.8	115	31
$Mg/(PEG + Mg(CH_3COO)_2)/(I_2 + C + electrolyte)$	1.84	32	32
$Ag/(PEO + AgNO_3)/(I_2 + C + electrolyte)$	0.595	48	33
$Na/(PVA+CH_3COONa3H_2O)/(I_2 + C + electrolyte)$	1.9	75	34
$Mg/(PVP + Mgcl_2 6H_2O+Al_2O_3)/(I_2+C + electrolyte)$	1.75	118	Present

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  $73^{\circ}c$ ,  $70^{\circ}c$ ,  $82^{\circ}c$  and  $69^{\circ}c$ .From the conductivity studies the highest conductivity for PVP+ Mgcl₂6H₂O+Al₂O₃ (85:15) was found to be  $1.33 \times 10^{-5}$  S/cm at temperature 373K and the transference of ionic and electronic numbers are found to be in the range 0.94-0.98, 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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