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GAS SENSITIVITY & IONIC CONDUCTIVITY OF PVC BASED MICROPOROUS POLYMER MEMBRANE ELECTROLYTE

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ABSTRACT

Gas sensing properties of a microporous polymer membrane based on polyvinyl chloride (PVC) was studied. These membranes were obtained by a novel polymer dissolution technique in different compositions of (PVC-PVA). Ionic conductivity of polymer membrane electrolytes have been determined by impedance studies in the temperature of 303-393 K by varying the ratio of PVC and PVA. The conductivity increases significantly enhanced about 5.431×10^{-4} S/cm when the weight ratio of PVA was 40 wt%. Finally the effect of different lithium salt concentration was also studied for the micro porous polymer electrolyte of high ionic conductivity system. A maximum ionic conductivity of 1.29×10^{-3} S/cm was obtained for 3M $LiClO_4$ electrolyte solution at ambient temperature. Gas sensing response of the PVC MPEs soaked in 3M molar concentration of Liclo4 was studied for different gases. Highest sensitivity was observed for H_2S gas at an operating temperature of 25 ^{0}c.

KEYWORDS: Microporous polymer electrolytes (MPEs), Gas sensing, Ionic conductivity.

I. INTRODUCTION

Sensors are key elements in this rapidly moving evolution, so the demand for sensors has soared in the last decades. Solid-state sensors that combine integration circuits and micro machining technologies as well as new materials open an avenue that can lead to many families of sensors to meet the new demands in performance, size and cost. Sensor research

and development has flourished during the last decade and a wealth of knowledge has accumulated [1].

Moreover conducting polymer electrolyte have seen intensively interesting to physicist, scientist & engineeriners because of their fundamental physical propertiest & potential applications in various electrochemical devices such as fuel cells, supercapacitors, sensors & display devices. interest in the preparation of polymer electrolytes with high ionic conductivity, good mechanical strength and thermal stability because these polymer electrolytes play a major role in various electrochromic windows, sensors, fuel cells etc.[2]

These plasticized polymer electrolytes could show a room temperature ionic conductivity of the order of 10^{-3} S/cm. However these polymer electrolytes could not completely satisfy the requirements on properties such as good mechanical strength, high ionic conductivity, tensile strength and good abrasion resistance. Microporous polymer membrane electrolytes can be prepared by phase inversion technique[3-5]**.** However, in this process, due to the use of large amounts of expensive, harmful and flammable solvents, contamination of the porous membrane may take place by the residual solvents. Moreover, preparation of MPEs by conventional phase inversion technology is costly and time consuming. For gas sensing characteristics, MPEs based gas sensors are commonly used for environmental monitoring and industrial applications due to their advantages such as small dimensions, low cost and convenient operation [6-7]. Hence in the present investigation we develop a simple, reliable, time saving and industrially feasible process for the fabrication of micro-porous polymer membrane, PVC based micro-porous membrane was prepared by extracting PVA from PVC-PVA blend film by using simple dissolution of PVA in DMSO. Moreover these membranes can be easily detached from the substrate and could be used for Li battery applications or many other applications depending upon the type of dopant to be used. The membrane morphology was investigated by XRD analysis. Gas sensing property of the PVC MPEs and PVC/PEG MPEs are also studied.

II. Experimental descriptions

2.1. Materials and Methods

PVC and PVA (Asldrich USA) were used without further purification for the preparation of micro-porous membranes. Reagent grade anhydrous lithium per chlorate was used after drying in vacuum at 110°C for 24 hours. PVC-PVA was prepared by the solvent casting technique by dissolving appropriate amounts of the corresponding constituents in DMF and DMSO respectively.

The films were dried at 80°C in vacuum oven for 3 hours to remove any further traces of DMF and DMSO. Finally the above prepared cast polymer films were immersed in DMSO for the preferential dissolution of PVA to get uniform micro-porous membranes of PVC which is then soaked in different molar concentration of lithium per chlorate with Propylene carbonate (PC) & Diethyl carbonate(DEC) in 1:1 ratio to obtain micro porous polymer membrane electrolyte.

X-ray diffraction measurement was carried out using PHILLIPS Holland. XRD system PW 171 diffractometer employed with Cu-anode operated at 30kV/15mA. Ionic conductivity of polymer membrane electrolytes have been determined by impedance studies in the temperature of 303-343 K by varying the PVA content in PVC matrix.

III. RESULT & DISCUSSION

3.1. X-ray diffraction analysis

In order to investigate the influence of Lithium salt concentration on the micro-porous polymer membrane, XRD examinations were conducted by soaking the micro-porous polymer membrane in 1M $LiClO_4$, 1.5M $LiClO_4$, 3 M $LiClO_4$ and 3.5M $LiClO_4$ electrolyte solution. The XRD pattern of the PVC based micro porous polymer membrane & microporous polymer membrane soaked in 1.5 M $LiClO_4$ and 3 M $LiClO_4$ electrolyte solution are given in fig.1(a-c). Following observations were made from XRD pattern.

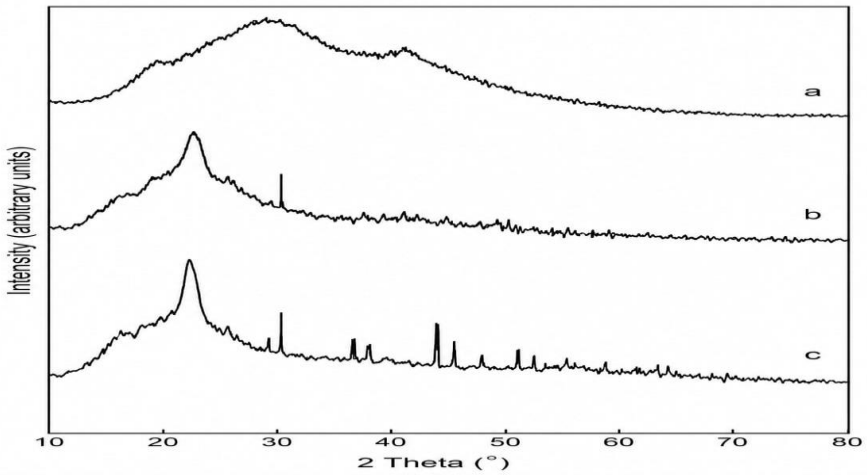

Figure. 1 XRD pattern for (a) micro-porous polymer membrane (b) micro-porous polymer membrane soaked in 1.5 M Liclo4 and (c) micro-porous polymer membrane soaked in 3 M Liclo4 electrolyte solution.

The intensity of the diffraction peaks of PVC based micro-porous polymer membrane increases with increase in salt concentration. This shows a increase in crystalline phase of the micro porous polymer electrolyte. It may be due to the incorporation of LiClO4, which would have disturbed the amorphous phase and increased the crystallanity.

Diffraction peak of PVC based micro-porous polymer membrane is displaced from 2θ =29.1° to 2θ =22.2°, when the polymer membrane is soaked in Li-salt electrolyte solution. It conforms the complex formation between polymer membrane & Li-salt [8].

In addition, further increase in salt concentration (3 M Liclo4) increases the crystalline nature of the polymer electrolyte, which may be due to undissociation of Liclo4 salt, which is evident from fig.(1). The appearance of sharp diffraction peaks confirms the presence of crystalline domains in the polymer electrolyte.

3.2 Conductivity studies

Ionic conductivity values of PVC based micro-porous polymer electrolyte was evaluated from cole-cole impedence. The ionic conductivity of PVC based polymer electrolytes is calculated from σ = l/RbA, where Rb is the bulk resistance of the polymer electrolyte and 'l' and 'A' are the thickness and known area of the polymer electrolyte film respectively. The conductivity data of microporous polymer membrane electrolyte are given in Table 1. It can be seen from the Table 1, that the increase of PVA content in PVC matrix, increases the conductivity due to the higher uptake of electrolyte by PVC in 1 M $LiClO_4$ but decreases the mechanical strength. However the blend ratio of 60 wt. % of PVC and 40 wt% of PVA has exhibited high ionic conductivity of 5.431×10^{-4} (S/cm) and sufficient mechanical strength at room temperature than all other micro-porous membrane electrolytes. Hence above blend ratio is chosen for optimized system.

Table 1. Ionic conductivity values of PVC microporous polymer membrane soaked in 1 M concentration of $LiClO_4$ electrolyte Solution at room temperature

S. No	PVC	PVA	Conductivity, S cm^{-1}	Film strength
1	100	0	0.129×10^{-4}	Excellent
2	90	10	0.334×10^{-4}	Excellent
3	80	20	0.876×10^{-4}	Good
4	70	30	2.638×10^{-4}	Good
5	60	40	5.431×10^{-4}	Good
6	50	50	5.412×10^{-4}	Poor

The effect of different lithium salt concentrations in the optimized system of PVC-PVA microporous polymer electrolyte was studied similar to the above conducted experiment, the ionic conductivity of polymer membrane soaked in 1 M, 1.5 M, 3 M, 3.5 M of $LiClO_4$ electrolyte solution at various temperatures ranging from 303 to 343 K is given in Table 2. It can be seen from the table, the conductivity increases with increase in temperature for the all concentration [9]. Hence the polymer electrolyte obeys the Arrhenius relation. It is due to the amorphous nature of the present polymer electrolyte which facilitates the fast Li ion motion in the polymer network and also it provides a higher free volume on increasing the temperature[10].

Table2. Ionic conductivity values of PVC microporous membrane soaked in different concentration of $LiClO_4$ electrolyte solution at various temperatures

(Ionic conductivity $\times 10^{-3}$ (S/cm)					
Concentration	**303 K**	**313 K**	**323 K**	**333 K**	**343 K**
1 M $LiClO_4$	0.54	0.57	0.58	0.65	0.73
1.5 M $LiClO_4$	0.62	1.01	1.98	2.78	3.29
3 M $LiClO_4$	1.29	1.75	2.34	2.96	3.43
3.5 M $LiClO_4$	1.03	1.67	2.26	2.59	3.27

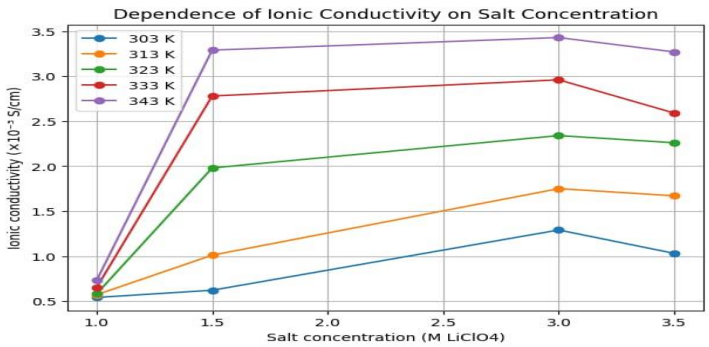

Figure 2. Dependence of ionic conductivity on salt concentration for PVC microporous polymer electrolyte at various temperatures.

The ionic conductivity vs. salt concentration (mole fraction) for (60-40) wt. % system of (PVC-PVA) at various temperatures of 303 K–343 K is shown in Figure 2. The ionic conductivity for the PVC microporous polymer electrolyte increases with the increase in Li salt concentration up to 3 M $LiClO_4$. The microporous polymer electrolyte consists of 3 M $LiClO_4$ electrolyte solution has high ionic conductivity of 1.29×10^{-3} S/cm at room temperature. Beyond this concentration (3 M $LiClO_4$) ionic conductivity decreases. This is due to the formation of ion pairs and the ion triplets, which causes constraints in the polymer

segmental motion and also retards the ionic mobility due to the increase in crystalline nature of microporous polymer electrolyte[11-12].

3.3. Microporous Polymer Electrolytes (MPEs) as a Gas Sensor:

Gas sensing study of the PVC MPEs with 1M, 1.5M, 3M and 3.5M of $LiClO_4$ concentration which has been synthesized by the method as discussed above, was carried out at R. L. College, Parola, Jalgaon. For PVC MPEs 1M, 1.5M and 3.5M of $LiClO_4$ are not found to be effective for gas sensing while better results are obtained for 3M $LiClO_4$ and hence only these results are reported in figures 3.

Table 3. Gas sensing response for PVC MPEs sample soaked in 3M$LiClO_4$

Sample	Temp°C	LPG	CO_2	NH_3	Ethanol	H_2	Cl_2	H_2S
PVC MPEs	40	0.06	0.09	0.12	0.02	0.09	0.10	0.20
	35	0.05	0.10	0.14	0.05	0.07	0.11	0.31
	30	0.03	0.11	0.13	0.07	0.06	0.12	0.31
	25	0.02	0.10	0.10	0.08	0.05	0.10	0.36
	20	0.02	0.10	0.09	0.12	0.05	0.09	0.32
	15	0.01	0.06	0.04	0.16	0.02	0.06	0.20
	10	0.01	0.01	0.01	0.19	0.01	0.05	0.17

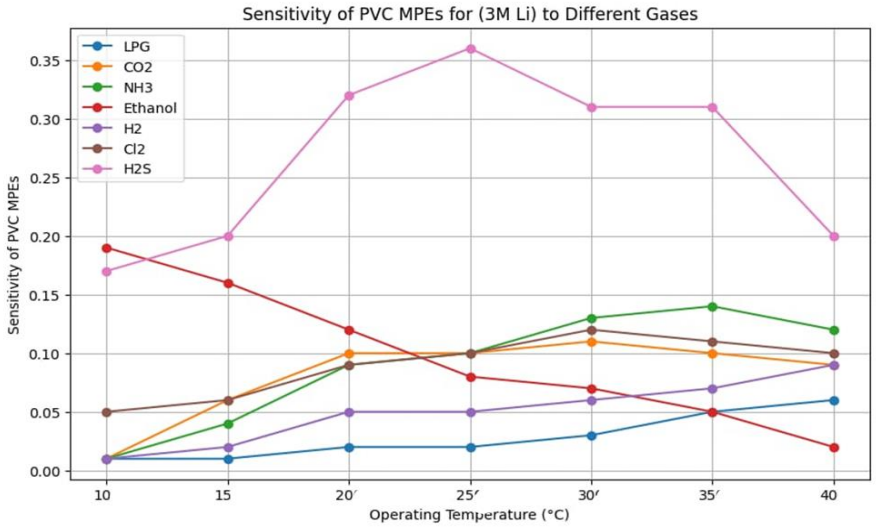

Figure 3. Sensitivity of PVC MPEs for (3M Li) to different gases as a function of operating temperature

From above table 3 and figures 3, it is clear that 3M $LiClO_4$ sample shows good sensitivity for H_2S at all temperatures (10°C to 40°C) and has highest sensitivity (0.36) at an operating temperature about 25°C. MPEs sample show high sensitivity towards H_2S as compared to other gases. This sensitivity increases with increase in temperature from 10°C to 25°C. From

25°C onwards, sensitivity increases slowly with increase in temperature, while at 10°C highest sensitivity is seen for Ethanol gas. In recent years, several studies are going on to improve the performance of MPEs sensors in order to increase the sensitivity to the lower operating temperature. In the present research work an attempt was made towards the use of Li salt for incorporation into PVC membrane to improve the sensitivity for selective sensing of H_2S, NH_3 and Cl_2 gas [13-15].

CONCLUSION

1. It is possible to prepare PVC based microphorus polymer membrane electrolyte by novel polymer resolution technique.
2. The x-ray diffraction peak was observed at $2\theta = 13.2°$ with less intensity indicates that the synthesized microporous PVC membrane is highly amorphous in nature.
3. The maximum ionic conductivity of 5.431×10^{-4} S/cm is observed for (60-40) wt% blend ratio of PVC-PVA at room temperature for 1 M $LiClO_4$ concentration and has sufficient mechanical strength.
4. The effect of Li salt concentration in the polymer matrix on ionic conductivity reveals that the ionic conductivity increases with the increase in Li salt concentration up to 3 M $LiClO_4$. The microporous polymer electrolyte consists of 3 M $LiClO_4$ electrolyte solution has high ionic conductivity of 1.29×10^{-3} S/cm at room temperature.
5. Gas sensing response of the PVC MPEs soaked in 3M molar concentration of Liclo4 was studied for different gases. Highest sensitivity was observed for H_2S gas at an operating temperature of 25 ^{0}c.

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