Bi-ionic Potential Studies of Artificial Membrane Based on Thermodynamics of Irreversible Processes

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Abstract

Parchment impregnated $PbWO_4$ artificial membrane was prepared by ion-interaction method. SEM reveals the formation of composite material with uniform surface morphology. The membrane was characterized for its thermal stability by TGA. Crystallinity and phosphorylation of the membrane was confirmed by XRD and FT-IR. The values of bi-ionic potential has been measured with various combinations of bi-univalent electrolytes at different concentrations. Theoretical values of BIP have been calculated using the BIP theories developed recently by Toyoshima *et al.* and Tasaka *et al.* based on the principles of non-equilibrium thermodynamics. Comparison of experimental and theoretical BIP values shows that theoretical equations of BIP are applicable to the parchment impregnated membrane. Membrane conductivity in contact with a single electrolyte has been experimentally determined to evaluate the selectivity of the membrane using the predetermined values of intramembrane mobility ratio. The selectivity sequence of the artificial membrane is found to be $Zn^{++}>Ca^{++}>Mg^{++}$. The prepared membrane can be considered as an excellent material for desalination and wastewater treatment applications.

Keywords: *Parchment impregnated $PbWO_4$ artificial membrane, Bi-ionic potential, Electrical conductance, Instrumental techniques.*

1. Introduction

At present, parchment impregnated membrane is most widely used in desalination technology and shows great attention to both academic and industrial areas due to their multi-purpose applications including wastewater treatment, metal ion recovery, electrolysis process and treating industrial and biological effluents. It also plays an important role in food and pharmaceutical processing [1–6]. In this context, various separation membranes such as membranes for microfiltration, nanofiltration, ultrafiltration, reverse osmosis, pervaporation, gas separation and liquid membranes, etc. have been studied, the ion-exchange membrane is one of the most advanced separation membranes among these separation membranes [7]. Because of the development of electrical potential difference between membrane and unequal electrolyte solutions which are in equilibrium, these charged membranes have applications in various processes such as water treatment filtration [8,9], electrokinetic energy conversion [10], fuel cell proton exchange [11,12].

The electrochemical characteristics and electrical conductance of the membrane depends on the nature of membrane forming materials, structure of membrane and concentration of the electrolytic solution in which membrane is operated [13]. The complete physicochemical characterizations are used to analyze the important parameters of membrane like ion-exchange capacity, water content nature, structural and transport properties, porosity, thickness, and thermal stability [14-18].

In the present paper, some theories of bi-ionic potential [19-20] have been applied to evaluate the bi-ionic potential measurements across the membrane and the membrane selectivity of metal ions. Membrane conductance in contact with the various bi-univalent electrolytes has also been determined experimentally to substantiate our findings.

2. Materials and method

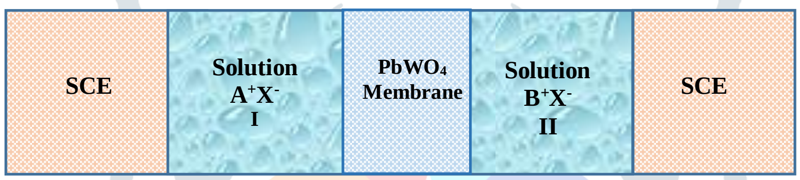
Na_2WO_4 (E. Merck, Mumbai, India), $Pb(NO_3)_2$ (E. Merck, Mumbai, India) and parchment paper (Amol Group of Companies Mumbai, India) were used to prepare the parchment impregnated $PbWO_4$ artificial membrane. All other reagents used were of analytical reagents grade. The bi-univalent chlorides of magnesium, calcium and zinc solutions were used for electrochemical characterization.

2.1. Membrane preparation

Parchment impregnated $PbWO_4$ artificial membrane was prepared by the method of interaction as described by Ansari and coworkers [21-24]. To precipitate these substances in the interstices of parchment paper, a 0.2M solution of Na_2WO_4 was placed inside glass tube, to one end of which was tied the parchment paper previously soaked in deionized water. The tube was suspended for 72 hours in a 0.2M solution of $Pb(NO_3)_2$. The two fresh solutions were interchanged later and kept for another seventy two hours. Thus, parchment paper and inorganic precipitate as a whole act as a artificial membrane. The membrane thus prepared was washed many times with deionized water to wash free electrolytes.

2.2. Measurement of membrane potential

The potential developed by setting up an electrochemical cell of the following type was measured using by a digital multimeter (Rish Multi ®) $4^{3/4}$ digits 18S and saturated calomel electrodes (SCE). The cell potential was taken as a measure of BIP.



Aqueous chloride solutions of Mg, Ca and Zn BDH (AR) grade were used on either sides of the membrane and were rigorously stirred with a pair of electrically operated magnetic stirrers to remove completely or at least to minimize the effect of the film controlled diffusion [25].

2.3. Measurement of membrane conductance

The membrane was sealed between two half cells as described in our earlier communication [26]. The half cells were first filled with electrolyte solutions ($MgCl_2$, $CaCl_2$ and $ZnCl_2$) to equilibrate the membrane (Fig.1). The solutions were replaced by purified mercury without removing the adhering surface liquids. The trapped air, if any, was removed from the membrane-solution interface in order to get reproducible results. Saturated calomel electrodes were used to establish electrical contacts. The membrane conductance was monitored on a direct reading conductivity meter 306 (Systronics) at a frequency of 103 Hz. All measurements were carried at 25±0.1℃. The electrolyte solutions were prepared from AR reagents in deionized water.

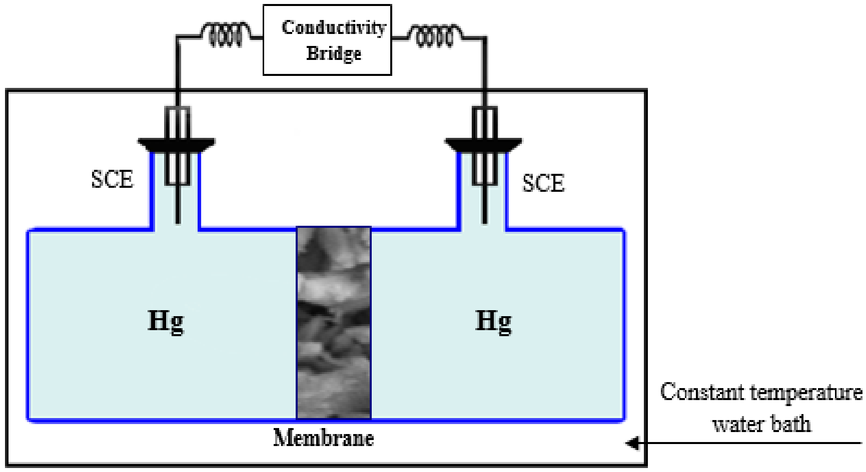


Fig. 1. Measurement of electrical conductance

2.4. Characterization of membrane

In order to judge the performance of the composite membrane, the complete physicochemical characterization has been done and this includes the determination of parameters like membrane water uptake, porosity, thickness, swelling etc. These influence the electrochemical properties of membrane [27].

2.4.1. Water uptake (% total wet weight)

The membrane was soaked in distilled water for 2 hour, blotted quickly with Whatsman filter paper to remove surface moisture and immediately weighted. These were further dried to a constant weight in vacuum over P_2O_5 for 24 hour. The water uptake (total wet weight) was calculated as follows

$$\%\ total\ wet\ weight = \left(\frac{W_w - W_d}{W_w}\right) x100$$

Where

Ww is the weight of the soaked or wet membrane and

Wd the weight of the dry membrane

2.4.2. Porosity

Porosity was determined as the volume of water incorporated in the cavities per unit membrane volume from the water uptake data

$$Porosity = \left(\frac{W_w - W_d}{AL\rho_w}\right)$$

Where A is the area of the membrane (cm^2). L the thickness of the membrane (cm) and ρ_w the density of water (g/cm^3).

2.4.3. Thickness

The thickness of the membrane was measured by taking the average thickness of the membrane by using screw gauze.

2.4.4. Swelling

Swelling was measured as the difference between the average thickness of the membrane equilibrated in 1M NaCl for 24 hour and the dry membrane.

2.4.5. Scanning electron microscopy studies

The characterization, pore structure, micro/macro porosity, homogeneity, thickness, cracks and surface morphology of $PbWO_4$ artificial membrane was analyzed with SEM model phillips 515, USA. Gold Sputter coatings was carried out on the desired membrane sample at pressure 1 Pa.

2.4.6. Thermogravimetric analysis (TGA) studies

The degradation process and thermal stability of the membrane was investigated using thermogravimetric analyzer (Perkin Elmer, Pyris Diamond), under N_2 atmosphere (200 ml/min.) using a heating rate of 10℃ min^{-1} from 25℃ to 1100℃.

2.4.7. X-ray diffraction studies

The XRD image of the $PbWO_4$ membrane was recorded by Mini Flex-II X-ray diffractrometer (Rigaku Corporation) with CuKα.radiation.

2.4.8. Fourier transformed infrared studies

The FT-IR spectrum of parchment impregnated $PbWO_4$ membrane was done by Perkin-Elmer instrument model spectrum BX series Perkin Almer (USA) in the region 400-4400 cm^{-1}. The entrance and exit beams to the sample compartment were sealed with a coated KBr window and there was a hinged cover to seal it from the environment.

2.4.9. Elemental analysis

The elemental analysis of $PbWO_4$ artificial membrane was carried out on the scanning electron microscope (model phillips 515, USA).

3. Results and discussion

The results of thickness, swelling, porosity and water content capacity of parchment impregnated $PbWO_4$ artificial membranes are summarized in Table 1.

Table 1. Water content, Porosity, Thickness, and swelling Properties of impregnated $PbWO_4$ artificial membrane

Water uptake as % Weight of Wet membrane	0.071
Porosity	0.114
Thickness(cm)	0.085
Swelling (%)	0.09

The SEM images of parchment impregnated artificial membrane is shown in Fig. 2(a-d). SEM images appear to be composed of dense and loose aggregation of small particles and formed pores probably with the non-linear channel but no fully interconnected. Particles are irregularly condensed and adopt a heterogeneous structure composed of masses of various size.

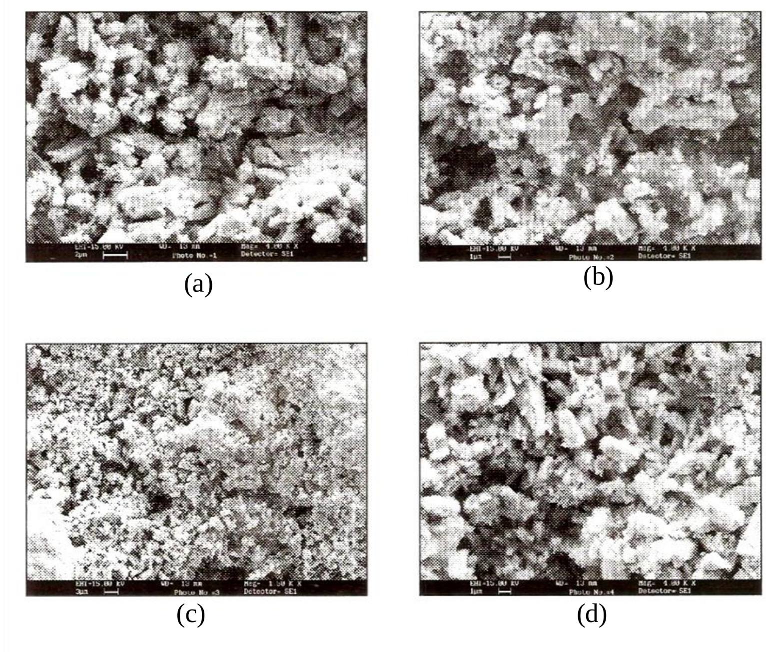


Fig. 2(a-d). Scanning electron micrographs (SEM) of parchment impregnated $PbWO_4$ artificial membrane

The thermal stability of the $PbWO_4$ artificial membrane was analyzed by TGA. The TGA curve measured under flowing N_2 is reported in Fig. 3. TGA of the membrane material showed gradual weight loss of about 3% to 11% from 400℃ to 580℃ which may be due to the removal of external water molecules present at the surface of the membrane material. Further weight loss of 15% to 20% from 800℃ to 1000℃ indicating the start of condensation due to the removal of the lattice water from the material.

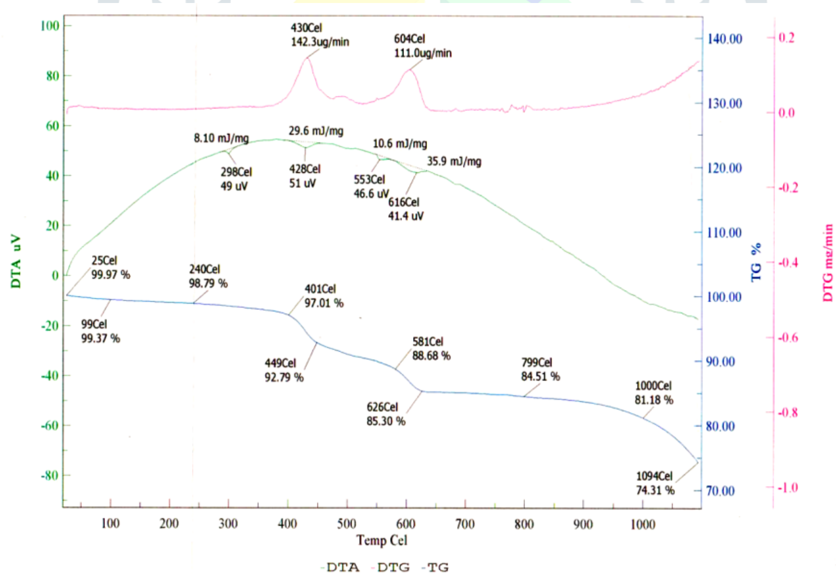
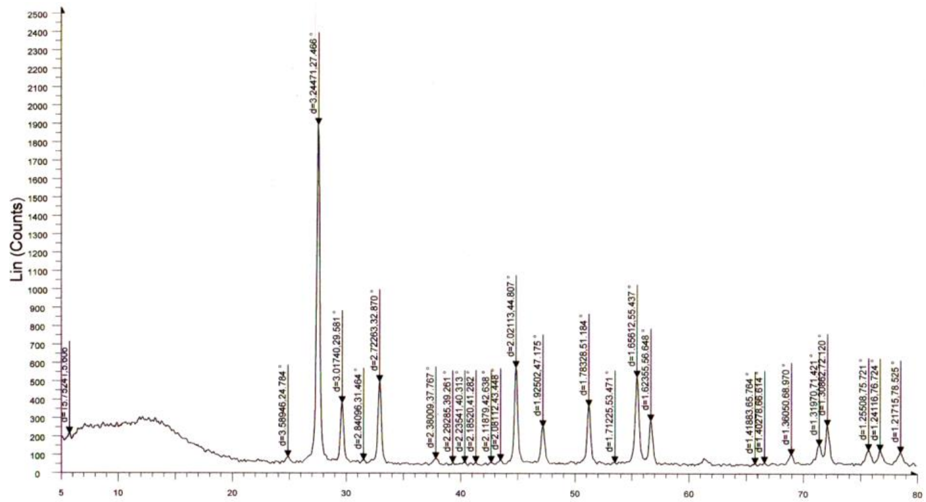


Fig. 3. TGA curve (blue spectra) of the parchment impregnated $PbWO_4$ artificial membrane

Fig. 4. shows X-ray diffraction spectrum of the $PbWO_4$ artificial membrane. The material recorded in powdered sample exhibited some sharp peaks in the spectrum (2θ range) shows semi-crystalline nature of the material.

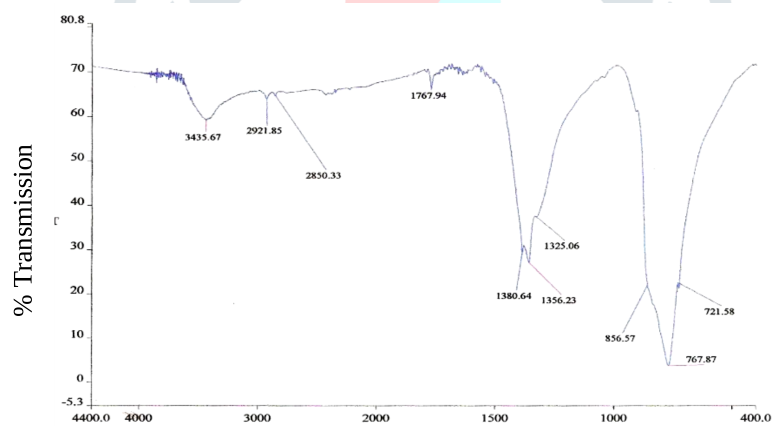


2θ Scale

Fig. 4. XRD pattern of parchment impregnated $PbWO_4$ artificial membrane

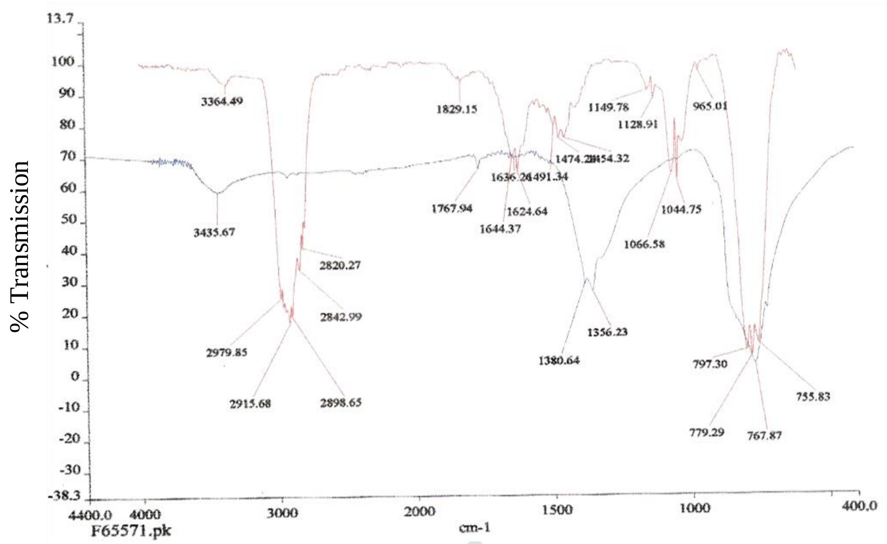
The FT-IR spectra for $PbWO_4$ artificial membrane has been displayed in Fig. 5(a) by the blue spectra. The sample spectra has also been compared with Fluka library to find out possible structure of same membrane as displayed in Fig. 5(b) by red spectra.

On comparison it has been found that it shows the similarities with following compounds with percentage probability given in Table 2. Peak table with % transmission has also been given in Table 3.



Cm^{-1}

Fig. 5(a). FT-IR spectra of parchment impregnated $PbWO_4$ artificial membrane



Cm^{-1}

Fig. 5(b). FT-IR spectra of parchment impregnated $PbWO_4$ artificial membrane compared with Fluka Library

Table 2. Percentage structural probability of the parchment impregnated $PbWO_4$ artificial membrane

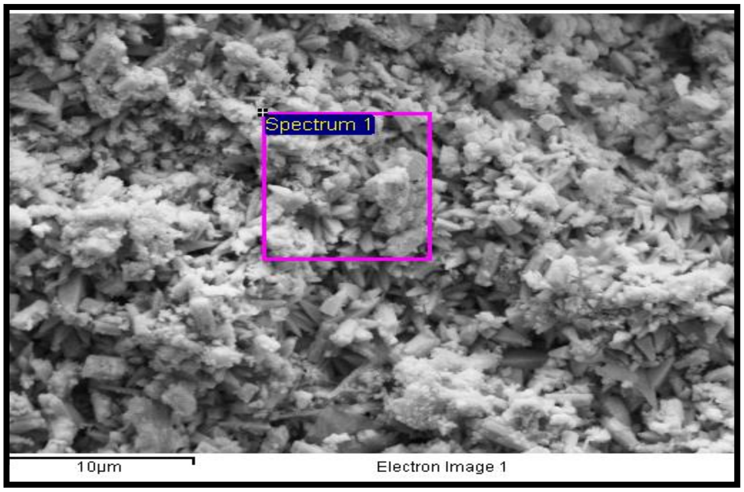
% Probability	Compound
0.447	Methylamine Anhydrous
0.422	1-bromonaphthalene
0.403	Chloral anhydrous
0.382	Carbon tetrachloride

Table 3. Peak table with % transmission of parchment impregnated $PbWO_4$ artificial membrane

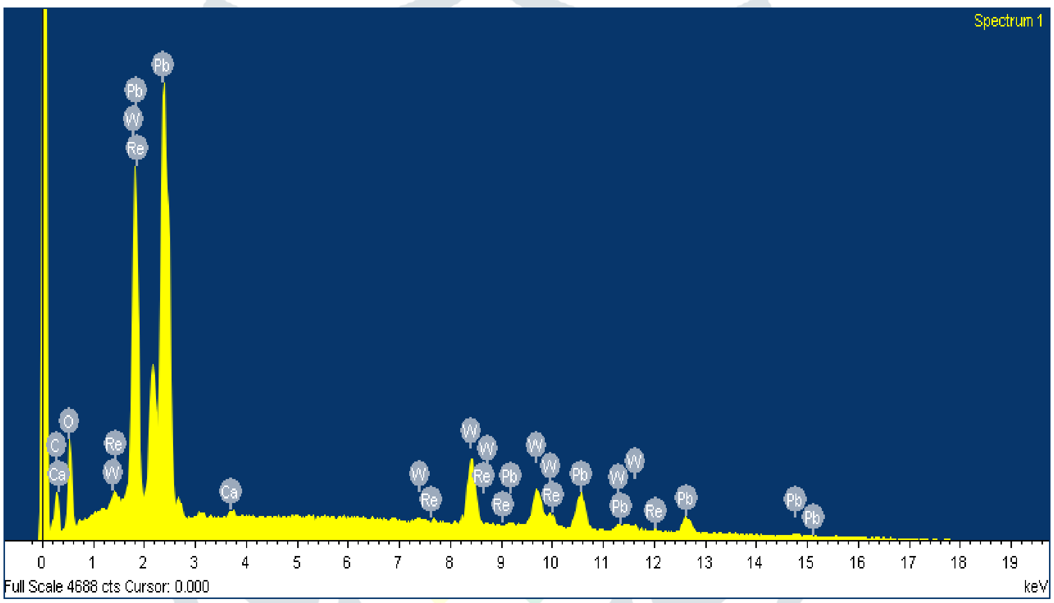
Peak No.	Wave number (cm^{-1})	% Transmission
1	3435.67	59.17
2	17637.94	66.07
3	1356.23	27.03
4	767.37	3.79

The above studies shows that the parchment impregnated $PbWO_4$ artificial membrane has the presence of IR active bands. This study will be helpful to assign the exact structure of the $PbWO_4$ membrane and then in turn to have its applications in the various field of practical interest.

The result of ultimate analysis of parchment impregnated $PbWO_4$ artificial membrane particles in this study are given in Table 4. The composition of fibers in parchment impregnated $PbWO_4$ artificial membrane has been determined to be approximately Figs. 6 (a-b).



(a)



(b)

Fig. 6 (a-b). Elemental analysis of parchment impregnated $PbWO_4$ artificial membrane

Table 4. Elemental analysis of parchment impregnated $PbWO_4$ artificial membrane particle

Element	% Wt.	Atomic W%
Carbon	7.81	33.07
Oxygen	14.66	46.57
Calcium	0.38	0.68
Tungsten	24.16	6.48
Rhenium	7.25	1.98
Lead	45.74	11.22

Membrane is used to separate two different electrolytic solution at various concentration, the steady potential developed is called bi-ionic potential (BIP) [28] which is used to measure the membrane selectivity of ions.

Wylie and Kannan [29] developed the following equation for the bi-ionic potential:

$$E_{BIP} = \left(RT/FZ\right) \ln a_i^{'} \bar{U}_i \;/ a_j^{'} \bar{U}_j \tag{1}$$

where R,T, F and Z have their usual significance, $a_i^{'}/a_j^{'}$ is the activity ratio of solutions and $\bar{U}_i \;/\bar{U}_j$ is the intramembrane mobility ratio of ions. Wyllie expressed the intramembrane mobility ratio is given by the following equation.

$$\bar{U}_i \;/\bar{U}_j \;= \bar{t}_i \;/\bar{t}_j \;= \bar{m}_i \bar{\lambda}_i \;/\bar{m}_j \bar{\lambda}_j \tag{2}$$

where $\bar{t}_i / \bar{t}_j$ = Transference ratio of artificial membrane

$\bar{m}_i \;/ \;\bar{m}_j$ = Steady state equilibrium concentration and

$\bar{\lambda}_i \;/ \bar{\lambda}_j$ = Ratio of electrical conductivities of membrane

We know that $\bar{m}_i \;/ \bar{m}_j \;= \bar{K}_{ji}$. K_{ji} is the selectivity. This on substitution into eqn. (2) gives:

$$\bar{U}_i \;/\bar{U}_j \;= K_{ji} \left(\bar{\lambda}_i \;/\bar{\lambda}_j\right) \tag{3}$$

The values of bi-ionic potential across $PbWO_4$ membrane with various bi-univalent electrolyte combinations at different concentrations are given in Table 5 and plotted against logC as shown in Fig 7. Equation (1) was used to evaluate intramembrane mobility ratio $\bar{U}_i \;/\bar{U}_j$, given in Table 6.

Table 5. Experimentally values of bi-ionic potential (mV) across parchment impregnated $PbWO_4$ membrane in contact with various bi-univalent electrolytes at 25±0.1℃

Concentrations (mol/L)	Electrolyte Pairs		
	$ZnCl_2$-$CaCl_2$	**$ZnCl_2$-$MgCl_2$**	**$CaCl_2$-$MgCl_2$**
0.1/0.1	1.36	8.19	5.24
0.05/0.05	2.08	8.58	6.36
0.02/0.02	3.19	11.83	9.90
0.01/0.01	5.83	15.17	15.27
0.005/0.005	7.25	18.80	16.59
0.001/0.001	9.39	21.28	17.79

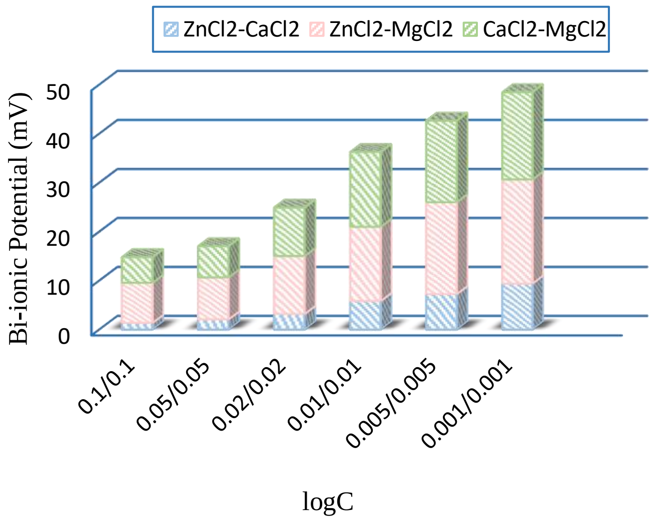


Fig.7. Plots of bi-ionic potential against logC for pairs across $PbWO_4$ membrane

Table 6. Values of the Intramembrane permeability ratios of various bi-univalent electrolyte to pairs across $PbWO_4$ membrane

Concentrations (mol/L)	**Electrolyte Pairs**		
	$\bar{u}_{Zn^{++}} / \bar{u}_{Ca^{++}}$ **$ZnCl_2$-$CaCl_2$**	$\bar{u}_{Zn^{++}} / \bar{u}_{Mg^{++}}$ **$ZnCl_2$-$MgCl_2$**	$\bar{u}_{Ca^{++}} / \bar{u}_{Mg^{++}}$ **$CaCl_2$-$MgCl_2$**
0.1/0.1	2.13	2.36	2.23
0.05/0.05	2.07	2.45	2.27
0.02/0.02	2.18	2.35	2.45
0.01/0.01	2.30	2.89	2.79
0.005/0.005	2.45	2.98	2.90
0.001/0.001	2.43	3.28	2.98

Toyoshima and Nozaki derived the following expression for bi-ionic potential.

$$\Delta E_{BIP} = \left[2 \ln K_A / K_B + \ln \left(JV_A + 1 / JV_B + 1\right)\right] \quad \left(F / RT\right) \tag{4}$$

Where K_A / K_B = Selectivity constant of membrane

V_A / V_B = Membrane parameter and

J= Ionic flux

The values of theoretical E_{BIP} can be calculated using equation (4). For the evaluation of these parameters, equation (4) can be written as

$$(2J + 1) \ln \left(g_A + 2J / g_B + 2J\right) - \ln \left(JV_A + 1 / JV_B + 1\right) - \ln \left(g_A / g_B\right) = 0 \tag{5}$$

$\because \quad N = A, B$

then

$$V_N = 1 + V_N^0 / V_P^0 \tag{6}$$

and

$$g_N = 1 + \left[1 + \left(2K_N C / \overline{X}\right)^2\right]^{1/2} \quad (7)$$

V_N and $\overline{X}$ are the value of the parameters occurring in equations (6) and (7). Toyoshima and Nozaki gave the following equation (8) for membrane potential (Em) arising across a membrane when it is used to separate two solutions of an electrolyte at different concentrations C'_N and C''_N.

$$\left(\frac{F}{RT}\right) Em = -\ln\gamma - \left(1 - \frac{2}{V_N}\right) x \ln \frac{\sqrt{1+\left(2C''_N K_N/\overline{X}\right)^2} + \left(1-2/V_N\right)}{\sqrt{1+\left(2C'_N K_N/\overline{X}\right)^2} + \left(1-2/V_N\right)} + \ln \frac{\sqrt{1+\left(2C'_N K_N/\overline{X}\right)^2} + 1}{\sqrt{1+\left(2C'_N K_N/\overline{X}\right)^2} + 1} \quad (8)$$

Where

$\gamma = C''_N / C'_N$

then

$$(F/RT)E_m = \left(1 - 2/V_N\right) \ln \gamma - 2\left(1 - 1/V_N\right) x \left(1/V_N\right)\left(1 - 1/\gamma\right)\left(\overline{X}/K_N\right)\left(1/C''_N\right) + ... \quad (9)$$

The apparent transference number t_ for co-ion can be calculated by the following equation.

$$-\left(F/RT\right) E_m = \left(1 - 2t\_\right) \ln \gamma \quad (10)$$

The values of observed membrane potential across parchment impregnated $PbWO_4$ artificial membrane in contact with various concentrations of different bi-univalent electrolytes are given in Table 7 and plotted against $\log C_2$ as shown in Fig. 8.

Table 7. Experimentally obeserved values of membrane potential (mV) across parchment impregnated $PbWO_4$ membrane in contact with various bi-univalent electrolytes at 25±0.1℃

Concentration (mol/L)	Electrolytes		
	$MgCl_2$	$CaCl_2$	$ZnCl_2$
1.0/0.1	15.5	14.6	13.4
0.5/0.05	20.9	18.8	17.6
0.1/0.01	29.5	26.4	24.7
0.05/0.005	40.9	37.7	35.4
0.01/0.001	49.5	49.3	45.6
0.005/0.0005	59.9	55.6	52.9

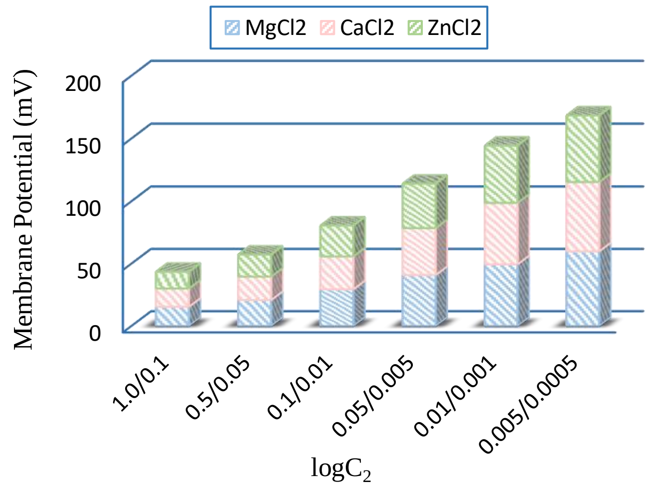


Fig. 8. Plots of observed membrane potential against $\log C_2$ using for various bi-univalent electrolytes with $PbWO_4$ membrane

The values of apparent transference number t_ are calculated from observed membrane potential (Table 7) using equation (10) for parchment impregnated $PbWO_4$ membrane are given in Table 8 and plotted against $1/C_2$ as shown in Fig. 9.

Table 8. Transference number t_ of co-ions derived from observed membrane potential at various concentration for $PbWO_4$ membrane

Concentration (mol/L)	Electrolytes		
	$MgCl_2$	$CaCl_2$	$ZnCl_2$
1.0/0.1	0.76	0.65	0.59
0.5/0.05	0.70	0.59	0.50
0.1/0.01	0.65	0.49	0.48
0.05/0.005	0.60	0.45	0.39
0.01/0.001	0.51	0.33	0.30
0.005/0.0005	0.42	0.29	0.24

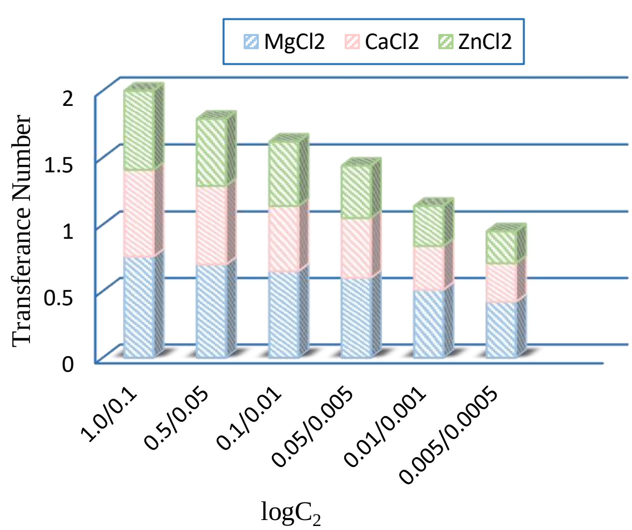


Fig. 9. Pots of 1/t- against $1/C_2$ for $PbWO_4$ membrane using bi-univalent electrolyte solutions

Adding equations (9) and (10), to get the following equation (11)

$$1/t\_ = V_N + (V_N - 1)\left[\frac{\gamma - 1}{\gamma \ln \gamma}\right](\overline{X}/K_N)(1/C''_N) + ... \quad (11)$$

In equation (11) the values of V_N and $(\overline{X}/K_N)$ can be determined from the ordinate intercept and initial slope of a plot for 1/t_ against $1/C_2$ (Fig. 9). The values of V_N and $(\overline{X}/K_N)$ thus derived for the artificial membrane and using bi-univalent electrolytes are given in Table 9.

Table 9. Values of the thermodynamically effective fixed charge density (eq/1) and V_N of parchment impregnated $PbWO_4$ membrane

Electrolyte	Parameters	
	V_N	$(\overline{X}/K_N)$
$MgCl_2$	1.782	4.7
$CaCl_2$	1.995	3.9
$ZnCl_2$	2.649	4.9

These values of V_N and $(\overline{X}/K_N)$ were used to calculate J and g_N using equations (5) and (6). The values of membrane parameters V_N, g_N, J and $(\overline{X}/K_N)$ are used to calculate the theoretical bi-ionic potential in equation (4). These theoretical values of BIP thus obtained are plotted against logC and are shown by broken lines in Figs. (10-12). For comparison the observed values of BIP are also plotted and are shown by solid lines in the same graph. The comparison demonstrates that the theoretical predictions are quite satisfactorily borne by our experimental observed results, thereby confirming the theories of Toyoshima and Nozaki under investigation.

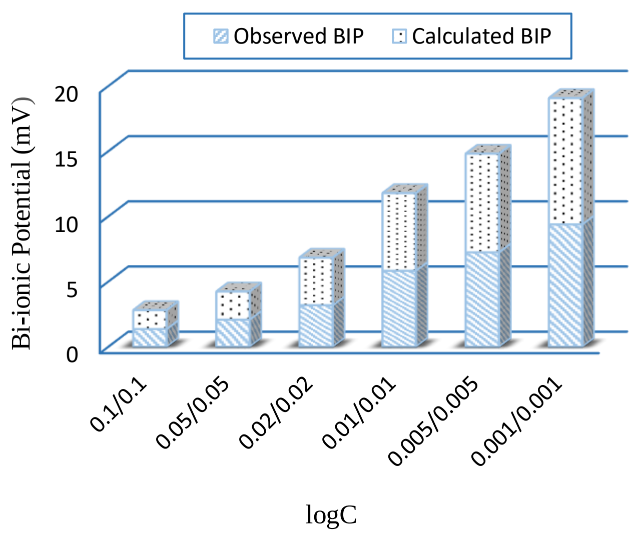


Fig. 10. Plots of bi-ionic potential against logC for $ZnCl_2$-$CaCl_2$ pair across $PbWO_4$ membrane

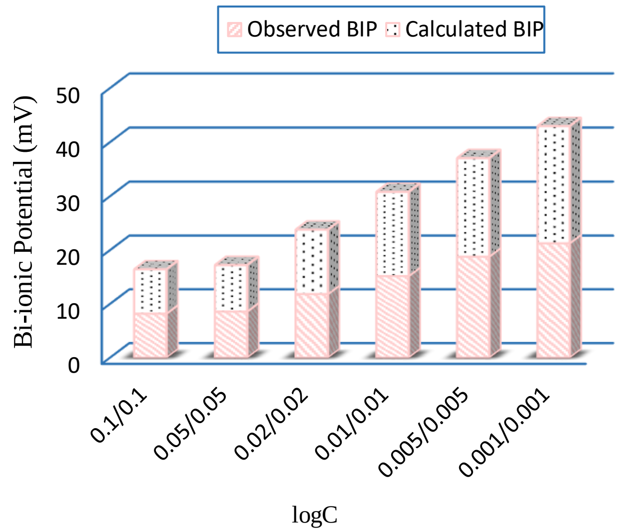


Fig. 11. Plots of bi-ionic potential against logC for $ZnCl_2$-$MgCl_2$ pair across $PbWO_4$ membrane

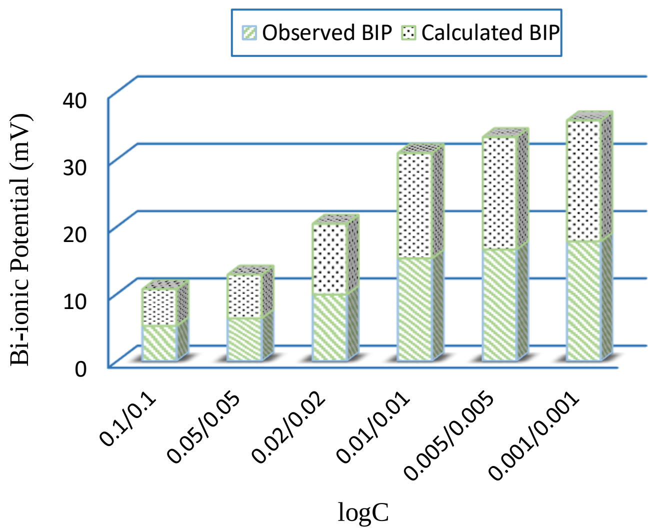


Fig. 12. Plots of bi-ionic potential against logC for $CaCl_2$-$MgCl_2$ pair across $PbWO_4$ membrane

Experimental values of electrical conductance$\times 10^3$ ($S\ m^{-1}$) across parchment impregnated $PbWO_4$ membrane of various bi-univalent electrolytes at 25±0.1℃ as shown in Table 10. The values are relatively more dependent on the concentration of the electrolytes within the membrane as shown in Fig. 13.

Table 10. Experimental values of membrane Electrical Conductance X10^3 (MHOS) across $PbWO_4$ membrane at 25±0.1℃

Concentration (mol/L)	Electrolytes		
	$MgCl_2$	$CaCl_2$	$ZnCl_2$
0.1	10.26	11.28	12.30
0.5	6.49	7.52	8.55
0.05	5.59	6.60	7.67
0.02	4.66	5.68	6.70
0.01	3.95	4.98	5.99

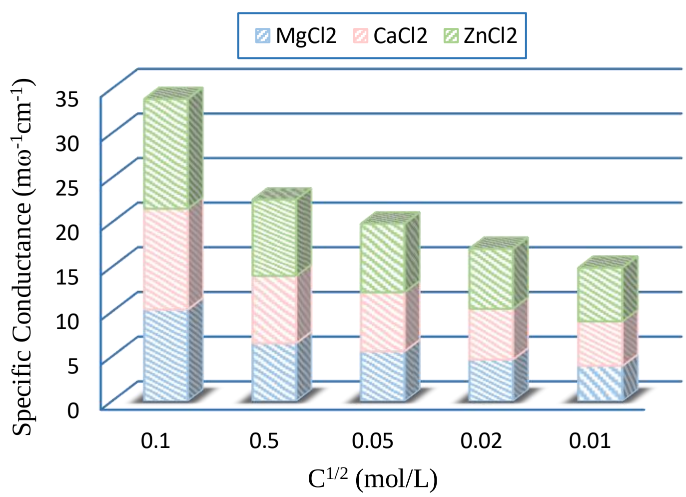


Fig. 13. Plots of specific conductance (mΩ^{-1}cm^{-1}) against square root of concentrations across $PbWO_4$ membrane at 25±0.1℃

This implies that the membrane has a relatively high Donnan uptake of anion and a low selectivity constant value. The values of selectivity K_{ij} evaluated for the $PbWO_4$ membrane from the ratio of their electrical conductivity using the value from Tables 6 and 10 are given in Table 11. The order of selectivity sequence of the membrane for the cations is Zn^{++}>Ca^{++}>Mg^{++}. This order of selectivity on the basis of the Eisenman-Sherry model of membrane selectivity points towards the weak field strength of the charged groups attached to the membrane matrix.

Table 11. Values of the selectivity K_{ji} $\left(K_{ij} = 1/K_{ji}\right)$ evaluated from ratio of electrical conductivities at various electrolyte concentration for $PbWO_4$ artificial membrane

Concentration (Mol/L)	Selectivity		
	K_{CaZn}	K_{MgZn}	K_{MgCa}
1.0/0.1	0.91	0.97	1.15
0.5/0.05	0.99	0.99	1.19
0.1/0.01	0.17	0.19	1.46
0.05/0.005	0.98	1.50	1.69
0.01/0.001	1.57	1.69	1.57
0.005/0.0005	1.67	1.75	1.79

4. Conclusion

The parchment impregnated $PbWO_4$ artificial membrane was successfully synthesized by using ion-interaction method. The values of bi-ionic potential across the membrane using various combinations of bi-univalent electrolytes at different concentrations were measured by applying the theory of Toyoshima and Nozaki based on thermodynamics of irreversible processes. Artificial membrane was used to evaluate the effective fixed charge density values of the membrane electrolyte system. Involving the membrane potential measurements in these equations, various thermodynamic membrane parameters were evaluated. The values of apparent transference number t_ has been important factor during the electrochemical characterization of the membrane.

The conductance order of various electrolytes were found to follow the sequence for the cations $Zn^{++}>Ca^{++}>Mg^{++}$. This order of selectivity $Zn^{++}>Ca^{++}>Mg^{++}$ on the basis of the Eisenman-Sherry model of membrane selectivity points towards the weak field strength of the charged groups attached to the membrane matrix. The prepared membrane can be considered as an excellent material for desalination and wastewater treatment applications.

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