



GREEN POLYMER ELECTROLYTES FOR MAGNESIUM – ION BATTERIES

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ARTICLE INFO

Article History:

Received 06th February, 2021

Received in revised form 14th March, 2021

Accepted 23rd April, 2021

Published online 28th May, 2021

Key words:

Green polymer electrolytes, Plasticizers, Impedance studies, Magnesium salts, Magnesium batteries

ABSTRACT

The increasing nature of industrial and domestic applications made the necessity of reasonable energy storage capacity as well as source. This made the development towards electrochemical devices. Biopolymer electrolytes have become the world's attention in facing current issues towards sustainability in energy storage. They aid to reduce the usage of non-renewable petrochemical electrolytes by using the biodegradable product. The current work was on the preparation of blend biopolymer electrolytes with Pectin/Guar Gum as a host. Ethylene carbonate (EC) has been used as plasticizer and Magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) as a doping salt. Electrolytes were prepared by solution casting method using water as a solvent. The electrolytes were prepared with the different compositions of Pectin/GG/ $MgCl_2 \cdot 6H_2O$ /EC. The crystalline/Amorphous nature of the film was studied using XRD analysis by X-ray diffraction technique. The interaction between the host and dopant was affirmed by FT-IR studies using Fourier Transform Infrared Spectroscopy technique. The thermal stability of the film was found by Thermo gravimetric analysis (TGA). The average roughness of the film was determined by 3-D Laser Profilometry. Using AC Impedance analysis, the ionic conductivity of the film was measured and the maximum ionic conductivity value $2.71 \times 10^{-3} S/cm$ was attained for the film containing 40:20:35:5 wt% of Pectin-GG- $MgCl_2 \cdot 6H_2O$ -EC. The maximum transport number of 0.94 was obtained for the film PGMCEC5 (35:15:45:5 M wt% of Pectin/Guar gum/ $MgCl_2 \cdot 6H_2O$ /EC).

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INTRODUCTION

Energy has been used up with the help of fossil fuels. Currently, the depletion in fossil fuels arises and green materials have gained much attention in order to face the energy crises. Many scientists took effort to develop biopolymer electrolyte-based electrochemical devices. So, the environment gets switched towards renewable, clean, healthy, eco-friendly, boundless, and inexhaustible energy sources for living. Energy storage plays a major role in the growing demand for globalization. Electrochemical energy storage is a concept used to store electrical energy under a chemical form. These devices are the most demanding and long-lasting ones which become worldwide attention because of their enormous applications in devices like, sensors and electrochemical cells. Among electrical energy storage devices, a study on batteries has become a vital area in the research. Most of the studies have been done on petrochemical-based polymer electrolytes which have issues towards the environment namely leakage, poor in stability, toxic and non-biodegradable in nature. To reduce the usage of petrochemical-based polymer, biopolymer has been used as hosts because of its low cost, leakage proof, biodegradable, lightweight, etc.

Solid biopolymer electrolytes are formed by the salt dispersed in a polymer matrix [1]. Hassan M.F *et al* reported conductivity of $8.52 \times 10^{-5} S/cm$ for Starch/ $MgSO_4$ system [2]. The K-carrageenan/ $MgCl_2$ system has been prepared by Sangeetha *et al.* and achieved conductivity of $4.76 \times 10^{-3} S/cm$ [3]. The conductivity value of $4.05 \times 10^{-4} S/cm$ has been obtained for cellulose acetate/ $Mg(ClO_4)_2$ system Mahalakshmi *et al.* [4]. The conductivity of $2.18 \times 10^{-3} S/cm$ for the l-carrageenan/ $Mg(ClO_4)_2$ system has been stated by Shanmuga Priya *et al.* [5]. Perumal *et al.* arrived at the conductivity value of $5.66 \times 10^{-4} S/cm$ for the formation of tamarind seed polysaccharide with $Mg(ClO_4)_2$ [6]. Kiruthika *et al.* carried out their work on the pectin/ $MgCl_2$ system and achieved conductivity of $1.14 \times 10^{-3} S/cm$ [7]. Shilpa and Saratha reported conductivity of $7.5 \times 10^{-4} S/cm$ for 50:50 wt % of pectin/ $LiNO_3$ system [8] and they carried out their work on the blend biopolymer electrolyte Pectin- Guar gum- LiTFSI which exhibits conductivity at $1.59 \times 10^{-4} S/cm$ for 0.25 wt % of LiTFSI [9]. The Guar gum (GG)-lithium bis (trifluoromethanesulphonyl) imide (LiTFSI)-glycerol-based electrolyte has been investigated by Abirami *et al.* [10] in which ionic conductivity of $2.041 \times 10^{-3} S/cm$ has been achieved for 60:40 wt% of GG/LiTFSI system. The

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biodegradable and renewable nature of the pectin made the researchers concentrate on the usage of pectin as the polymer electrolyte. It is non-toxic and water-soluble. It is made up of more hydroxyl groups and it has an oxygen atom that aims to form a coordination bond with cations [11-13]. The Guar-gum (GG) based electrolyte improves conducting nature which possesses low cost, easy fabrication, high efficiency, biodegradable, etc compared to liquid electrolytes [14]. Magnesium is the eighth-most abundant element and can be commercially extracted from mineral deposits or seawater. Magnesium atom releases two electrons during the battery discharge phase which provides it a potential to deliver almost twice the electrical energy. During the battery charging phase, magnesium does not grow dendrites on the metal surface which makes the magnesium batteries easier to handle and much safer [15]. Some of the magnesium salts which are used in solid polymer electrolyte are magnesium acetate $Mg(CH_3COO)_2$ [16], magnesium nitrate $Mg(NO_3)_2$ [17], magnesium triflate $Mg(Tf)_2$ [18], magnesium chloride $MgCl_2$ [19], magnesium sulphate heptahydrate $MgSO_4 \cdot 7H_2O$ [20], magnesium perchlorate $Mg(ClO_4)_2$ [21]. The current study deals with the study of an eco-friendly biopolymer electrolytes pectin and guar gum doped with different concentrations of magnesium chloride and plasticized with ethylene carbonate. The reason for choosing magnesium chloride as a doping salt is that if the concentration of the chloride ions increases, the rate of electrochemical deposition of the host polymer also increases [22].

Experimental Materials used

Host polymer - Biopolymers Pectin and Guar Gum (GG) as a blend

Doping salt - Magnesium chloride hexahydrate

Plasticizer - Ethylene carbonate (EC), and Solvent - Distilled water

Preparation of biopolymer films

Pectin/Guar gum/Magnesium chloride/Ethylene carbonate complex films were prepared using solution casting technique. The blend polymer pectin/guar gum and various compositions of $MgCl_2 \cdot 6H_2O$ (45:25:25, 40:20:35, 35:15:45, 30:10:55 molecular weight percentage of Pectin: Guar gum: $MgCl_2 \cdot 6H_2O$) were dissolved in 40 ml distilled water. Ethylene carbonate (5 wt %) was added to each composition. To get the homogeneous solution, the mixture was agitated for about 24 hours using a magnetic stirrer at ambient temperature. Then the mixture was poured onto Petri dishes, permitted to evaporate the solvent in a hot air-oven at 40 °C for the next 24 hours. Table 1 gives the code and composition of Pectin/Guar gum/ $MgCl_2 \cdot 6H_2O$ /EC blend electrolytes.

Table 1 Code and composition of Pectin/Guar gum/ $MgCl_2 \cdot 6H_2O$ /EC blend electrolytes

Code	Composition
PGMCEC7	45 M wt% pectin: 25 M wt% guar gum :25 M wt% $MgCl_2 \cdot 6H_2O$:5 M wt% EC
PGMCEC6	40 M wt% pectin: 20 M wt% guar gum: 35 M wt% $MgCl_2 \cdot 6H_2O$: 5 M wt% EC
PGMCEC5	35 M wt% pectin: 15 M wt% guar gum: 45 M wt% $MgCl_2 \cdot 6H_2O$: 5 M wt% EC
PGMCEC4	30 M wt% pectin: 10 M wt% guar gum: 55 M wt% $MgCl_2 \cdot 6H_2O$: 5 M wt% EC

Characterization

The FTIR spectra of the blend polymer pectin-Guar gum and $MgCl_2$ doped blend polymer samples were done in the frequency range of 4000-400 cm^{-1} using FT-IR (Miracle 10 SHIMADZU) spectrophotometer at ambient temperature. The TG/DTA 6300 Model instrument (EXSTAR) perform thermogravimetric measurements. The sample's roughness can be determined by 3-D Optical Profilometer (Zeta-20). X-ray diffraction patterns of the polymer films were displayed in the range of $2\theta = 10^\circ - 90^\circ$ using XPERT PRO Diffractometer at ambient temperature. The area of the sample was taken as 2 cm × 2cm square dimension and sandwiched between two Aluminium electrodes. Using Ametek PARSTAT MC – 1000, the impedance measurements were done in the frequency range of 1 Hz to 1 MHz at ambient temperature. The transport number measurement was measured using Ametek PARSTAT MC – 1000 in which the DC potential of 0.1 V is applied across the cell in the Al electrode/PGMCEC/Al electrode configuration, and the polarization current is monitored as a function of time.

RESULTS AND DISCUSSIONS

FTIR Analysis

Figure 1 shows the FTIR spectra of Pectin-GG- $MgCl_2$ -EC blend polymer electrolytes. Table 2 gives the peak assignments of pectin/guar gum/ $MgCl_2 \cdot 6H_2O$ /EC polymer electrolytes. In blend pectin-GG, a broad peak around 3394.72 cm^{-1} corresponds to the O-H stretching vibration mode which gets shifted and broadened at 3279.41 cm^{-1} for the other samples of every concentration. This indicates the interaction of the Mg^{2+} ion of the salt with the OH group of blend polymer. A medium peak at 1766.80 cm^{-1} is allotted to the stretching vibrations of the C=O bond of the biopolymer. This peak gets submerged for the biopolymers added with ionic salt.

The O-H bending is observed at 1373.32 cm^{-1} for the blend polymer and gets shifted to 1396.46 cm^{-1} for the samples with the ionic salt. The blend polymer exhibits peak at 1234.44 cm^{-1} gets shifted to 1226.73 cm^{-1} for PGMCEC7 is assigned as C-O-C stretching and 1242.16 cm^{-1} for other compositions [7]. The C-Cl stretching mode of $MgCl_2$ is achieved at 678.94 cm^{-1} for the doped samples [23].

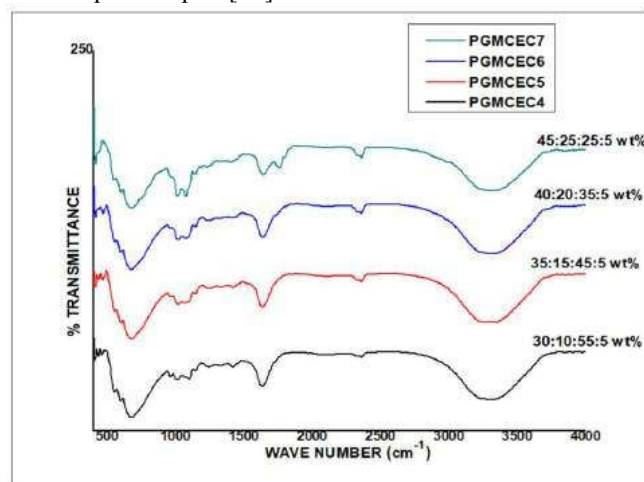


Fig 1 FTIR spectra of Pectin-GG- $MgCl_2$ -EC blend polymer electrolytes

Table 2 Peak assignments of pectin/guar gum/MgCl₂.6H₂O/EC polymer electrolytes

20%Pectin : 20% GG	PGMCEC7	PGMCEC6	PGMCEC5	PGMCEC4	Characteristic groups
	Wave number(cm ⁻¹)				
3394.72	3279.41	3279.41	3279.41	3279.41	OH-stretching
1766.80	-	-	-	-	C=O stretching
1373.32	1396.46	-	-	1327.08	-OH bending
1234.44	1226.73	1242.16	1242.16	1242.16	C-O-C stretching
1149.57	1141.86	1149.57	1149.57	1149.57	C-OH stretching
1041.56	1018.41	1026.13	1018.41	1010.70	CH ₂ twisting
678.94	678.94	671.23	678.94	671.23	C-Cl stretching

XRD Analysis

Figure 2 shows the XRD spectra of pectin-guar gum blend polymer complexed with various concentrations of MgCl₂.6H₂O and plasticizer ethylene carbonate. For the pure pectin-guar gum, the crystalline region is located at an angle 2θ = 10, 20, 44.6° [24]. For the doped samples, the peak was observed between 20° and 35°. The relative type of peak was observed in the Premalatha *et al* [25]. In this study, an increase in the concentration of ionic salt (MgCl₂.6H₂O), decreases the intensity of a broad peak. This might be feasible only if there is complete interaction of salt with the polymer matrix [26]. As a result, conductivity and amorphous nature get increased in polymer films. The polymer electrolyte of PGMCEC6 exhibits maximum amorphous nature.

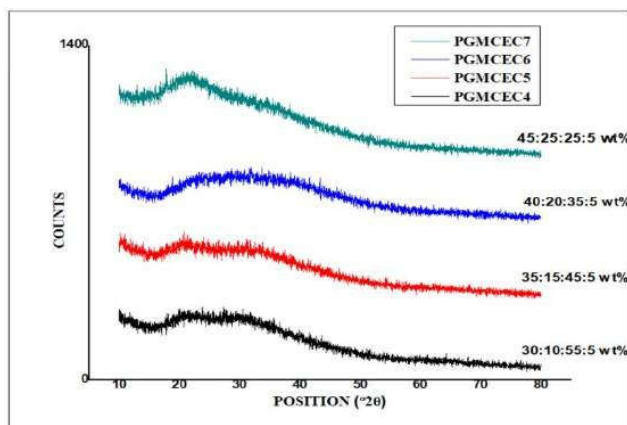


Fig 2 XRD spectra of Pectin-GG-MgCl₂-EC blend polymer electrolytes

TGA Analysis

Figure 3 shows a thermogravimetric curve of different compositions of pectin-guar gum-MgCl₂-EC blend biopolymer electrolytes. Table 3 gives the percentage weight loss of pectin-GG-MgCl₂.6H₂O-EC polymer electrolytes. For Pectin-Guar gum, the first weight loss of 18% at 200°C is due to the breakdown of trapped moisture content. The major weight loss of 65% at 250°C was observed on a further increase in temperature.

For PGMCEC7 blend polymer electrolytes, the loss of weight with 27% at 117°C takes place in the first zone. The reason is due to the vaporization of residual moisture in the sample. Beyond this zone, a major loss of 81.3% at 321°C was observed. The depletion can be assigned to the breakage of C-O-C bonds in mannose and galactose backbones, results in CO₂ expulsion from the guar gum backbone [27]. Similar weight loss was observed in the second zone for PGMCEC6,

PGMCEC5, and PGMCEC4 blend polymer electrolytes such as 70, 84, 81.5 % at 293°C, 305°C, 299.5°C respectively due to the breakage of C-O-C bonds. TGA results revealed that on adding ionic salt and plasticizer have significantly affected the thermal stability of polymer film [28].

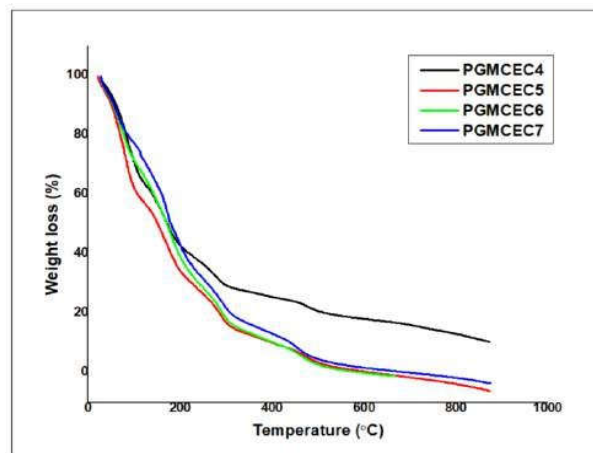


Fig 3 TG curve of Pectin-GG-MgCl₂-EC blend polymer electrolytes

Table 3 Percentage weight loss of Pectin-GG-MgCl₂.6H₂O-EC polymer electrolytes

Code	Composition of Pectin-GG-MgCl ₂ .6H ₂ O-EC polymer electrolytes (M wt %)	Total weight loss (%)
Pure Pectin/GG	20%Pectin/ 20%GG	98.9
PGMCEC7	45:25:25:5	80.3
PGMCEC6	40:20:35:5	86.4
PGMCEC5	35:15:45:5	89.1
PGMCEC4	30:10:55:5	88.9

3-D Laser Profilometry Analysis

The morphology of the film surface was studied using 3-D Laser Profilometer. When the average roughness (Ra) increases, the roughness of the surface also increases. Figure 4 shows the Zeta images of Pectin-GG-MgCl₂-EC polymer electrolytes. The Ra values for the various concentrations of the blend biopolymer films are given in Table 4.

Among all four biopolymer thin films of Pectin-GG-MgCl₂-EC, PGMCEC7 and PGMCEC5 exhibit lesser average roughness values of 2.88% and 11.8% respectively. Hence, the above two compositions of biopolymer films were comparatively smoother than the other two compositions i.e., PGMCEC6 and PGMCEC4 exhibit a higher Ra value of 26.3% and 22.7%.

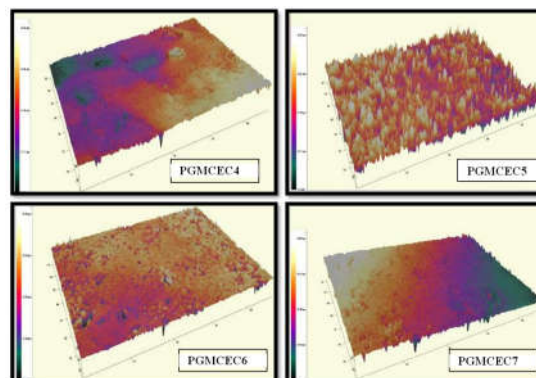


Fig 4 Zeta 3D images of Pectin-GG-MgCl₂-EC blend polymer electrolytes

Table 4 Average roughness values of Pectin-GG-MgCl₂-EC blend polymer electrolytes

Composition of Pectin-GG-MgCl ₂ -EC (M wt%)	Average roughness (Ra) values
45:25:25:5	2.88%
40:20:35:5	26.3%
35:15:45:5	11.8%
30:10:55:5	22.7%

AC Impedance Studies

Impedance spectroscopy is a powerful device for the analysis of the electrical properties of the solid electrolyte material and their interfaces. The ionic conductivity depends on their mobility and the conducting species. This method is used to establish the mechanism of conductivity, observing the polymer chain participation and carrier generation processes. The blend solid polymer film was kept between the two aluminium electrodes. The values of real and imaginary impedance were taken. Nyquist plots for Pectin-Guar Gum doped with different concentrations of MgCl₂ with plasticizer at ambient temperature (303K) were shown in fig 5. A plot of real impedance on the X-axis versus negative imaginary impedance on the Y-axis was drawn. In the graph, two well-defined regions can be seen, a semicircle that is in the high-frequency range and an inclined spike at the low-frequency region. From the complex impedance plot, the (R_b) bulk resistance can be calculated from the point of intersection of the semicircle and the inclined spike.

By knowing the bulk resistance, the conductivity of the sample can be determined using the relation

$$\sigma = L/R_b A$$

Where A and L are the area and thickness of the film respectively. The measurement of conductivity was done at room temperature. An impedance plot for the film Pectin: guar gum: magnesium chloride: ethylene carbonate is shown. Table 5 shows the conductivity values of the biopolymer film Pectin and Guar gum with the plasticizer ethylene carbonate. It concludes that the film containing 40:20:35:5 M wt% of Pectin: Guar gum: MgCl₂: Ethylene carbonate exhibited higher conductivity of $2.71 \times 10^{-3} \text{ Scm}^{-1}$. After the addition of salt, the conductivity is found to be decreased. This may be due to the aggregate formation of the salt with the biopolymer which may block the motion of the ions [29].

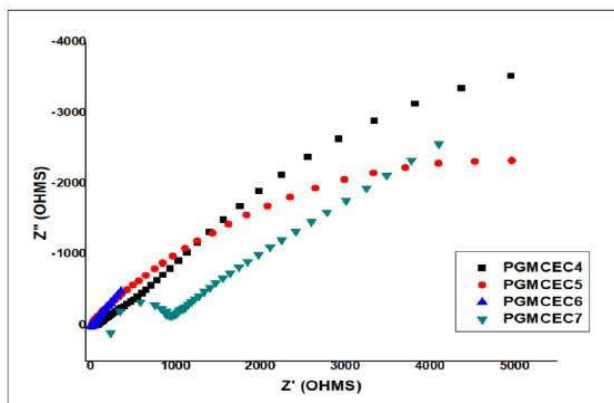


Fig 5 Nyquist plot for Pectin-GG-MgCl₂-EC blend polymer electrolytes

Table 5 Conductivity values of Pectin-Guar gum-MgCl₂-EC blend polymer electrolytes

Code	Conductivity Scm ⁻¹
PGMCEC7	7.87×10^{-3}
PGMCEC6	2.71×10^{-3}
PGMCEC5	3.51×10^{-3}
PGMCEC4	9.225×10^{-4}

Transport Number Measurement

The transport number was measured using chronoamperometry technique and the figure 6 (a-d) shows the transport number curve for Pectin/Guar gum/MgCl₂.6H₂O/EC. The transport number values for Pectin/Guar gum/MgCl₂.6H₂O/EC electrolytes are given in the Table 6.

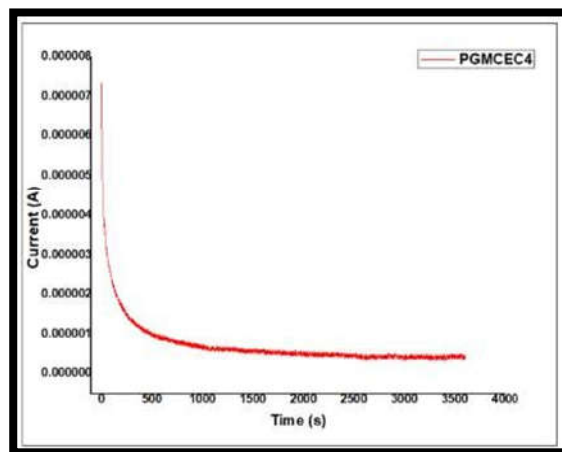


Fig 6 a Transport number curve of PGMCEC4

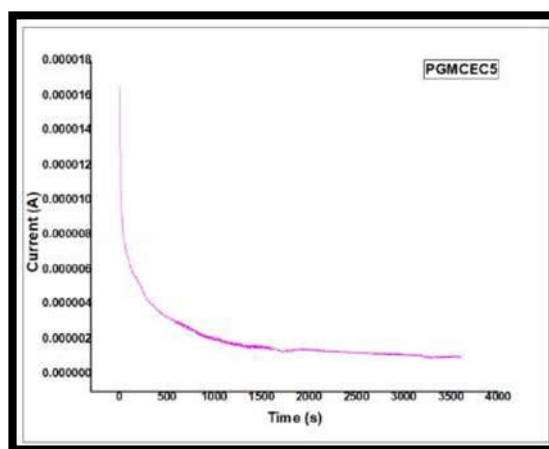


Fig 6 b Transport number curve of PGMCEC5

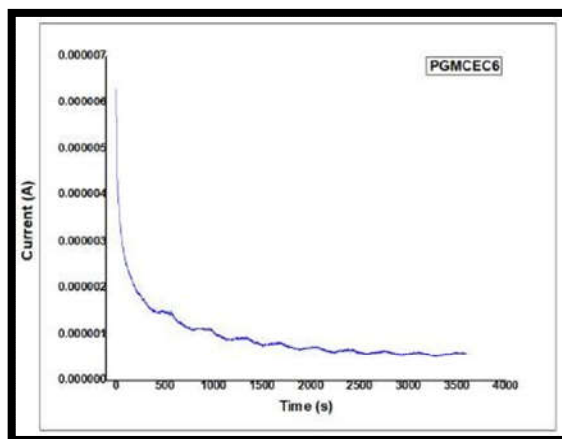


Fig 6 c Transport number curve of PGMCEC6

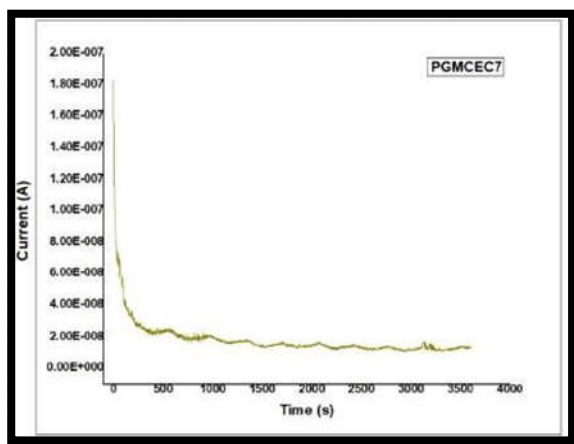


Fig 6 d Transport number curve of PGMCEC7

Fig 6 Transport number curve for Pectin-GG-MgCl₂.6H₂O-EC blend polymer electrolytes

Table 6 Transport number for Pectin/Guar gum/MgCl₂.6H₂O/EC blend polymer electrolytes

Code	Composition	Transport number(t _{ion})
PGMCEC7	45:25:25:5	0.78
PGMCEC6	40:20:35:5	0.65
PGMCEC5	35:15:45:5	0.94
PGMCEC4	30:10:55:5	0.83

From the table 6, it is obvious that the transference number (t_{ion}) for the maximum conductivity polymer electrolyte PGMCEC5 (35:15:45:5 M wt% of Pectin/Guar gum/MgCl₂.6H₂O/EC) was found to be 0.94.

CONCLUSION

A new pectin/guar gum-based blend of biopolymer electrolytes complexed with MgCl₂.6H₂O as a salt and ethylene carbonate (EC) as a plasticizer was prepared via solution casting technique. The interaction between host and dopant was affirmed by FTIR. The prepared blend polymer electrolytes that can withstand up to 320°C were confirmed by Thermogravimetric analysis. XRD shows the amorphous/crystalline nature of the prepared blend electrolytes. From 3D Laser Profilometry, it can be analyzed that PGMCEC7 and PGMCEC5 are comparatively smoother than the other two samples in Pectin/Guar gum/MgCl₂/EC. The maximum ionic conductivity of 2.71×10⁻³ S/cm has been attained for the polymer electrolyte of PGMCEC6. The maximum transport number of 0.94 was obtained for the film PGMCEC5 (35:15:45:5 M wt% of Pectin/Guar gum/MgCl₂.6H₂O/EC). Since the ionic conductivity has been obtained in the range of 10⁻³ S/cm, it can be used in the small gadgets like hearing aid, watches, etc which have ionic conductivity in the same range.

Acknowledgement

The authors wish to acknowledge Gurukataksham Trust for providing financial support and the Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore – 43 for providing the necessary facilities to carry out the present research work.

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How to cite this article:

Nandhini D *et al* (2021) 'Green Polymer Electrolytes for Magnesium – Ion Batteries', *International Journal of Current Advanced Research*, 10(05), pp. 24332-24337. DOI: <http://dx.doi.org/10.24327/ijcar.2021.24337.4826>
