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Tailoring the electrochemical performance of the polymer electrolyte using Na₂H₂₀B₄O₁₇ for magnesium sulfur battery applications

M.A. Attallah ^{*}^(D), E. Sheha

Physics Department, Faculty of Science, Benha University, 13518, Benha, Egypt

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Keywords: Solid electrolyte Magnesium sulfur battery Na2H20B4O17 Energy storage	Due to the construction's low cost, ecological appeal, friendliness, great theoretical density, and reliability, the Mg-S battery is an encouraging substitute for the pillar lithium battery. Nevertheless, self-discharge, the delayed conversion reaction pathway, and the absence of readily compatible electrolytes continue to restrict its beneficial uses. In this work, polymer electrolyte used 0.7 wt% polyvinyl alcohol (PVA), $(0.3-x)_{wt.\%}$ (Mg(CF ₃ SO ₃) ₂) and $_{xwt.}$ %Na ₂ H ₂₀ B ₄ O ₁₇ (x = 0,0.01,0.02,0.03,0.04,0.05) (PE _x) have been prepared and characterized for usage in Mg-S batteries. Integrating the PVA_(0.3-x)_wt.% (Mg(CF ₃ SO ₃) ₂)_ 0.04Na ₂ H ₂₀ B ₄ O ₁₇ (X ₄) with tetraethylene glycol dimethyl ether (G ₄) demonstrates effective Mg plating and stripping behavior, good anodic stability (versus Mg/Mg ²⁺), and a significant ionic conductivity (8.09 × 10 ⁻⁷ S cm ⁻¹ at 323 K). The Mg-S is assembled using an X ₄ .G ₄ electrolyte and delivers a reversible capacity of 100 mAh g ⁻¹ after 30 cycles.

1. Introduction

Rechargeable next-generation (Mg-S) batteries play a vital role in energy technology storage and conversion as a substitute for lithium batteries [1–5]. This is due to its their production low cheap, ecologically friendly, high energy density (ability to transmit more than an electron every time), low susceptibility for dendrites, which allows the Mg-metal to serve as the battery's anode, safety (limited reactivity to air), Superior theoretical ability (2205 Ah/kg and 3832 Ah/L because of the Mg duality nature) and a plentiful supply of magnesium raw resources [6-15]. In addition, (S) cathode provides nontoxicity, high theoretical capacity (1670 mAh/g), and inexpensive cost (200 \pm ton⁻¹) [16,17]. Despite multiple ongoing research on (Mg-S) batteries, the significant low Mg mobility because of electrode/electrolyte Mg-ion interactions, self-discharge, cathode materials high-performance scarcity, shuttle effect, and the lack of standard Mg electrolytes appropriate to anodes remains a significant barrier to the realize Mg-S battery reliable technology [18-22]. The Mg²⁺ ion's high charge density results in higher migrant barriers; the constructed interfacial layer on the Mg anode, which contains several reduced components (like MgO and Mg $(OH)_2$) of electrolytes unveils higher Mg²⁺ transfer impedance and behave as a passivated layer, causing a high stripping/plating potential [23-26].

In this light, non-nucleophilic electrolytes have been developed

recently using creative ways to be compatible with the electrophilic character of sulfur, such as phenyl complex electrolyte, magnesium-bis (trifluoromethanesulfonyl) imide-based electrolyte [Mg(TFSI)2], magnesium aluminum chloride complex electrolyte [27-32]. Among all types of electrolytes, magnesium electrolytes have achieved significant advances in reversible magnesium deposition in liquid Mg electrolytes [33]. However, flaws and unresolved problems, notably low chemical stability, the cost, and the complex composition of self-discharge, hinder the electrolyte from being commercialized [34–37]. Several studies have been reported on Mg-ion-conducting polymer electrolytes, the electrolyte Mg[Mes₃BPh]₂/THF created by Zhu et al. It exhibits strong electrochemical reversibility for the deposition and dissolution of magnesium and high ionic conductivity. The efficiency of this electrolyte is 80 % and increases to 100 % after 15 cycles later [38]. Chusid et al. investigated a gel-type polymer electrolyte of an Mg organic halo aluminate salt, Mg(AlCl₂-EtBu)₂, PVDF, and tetra glyme, which served as a plasticizer. This gel electrolyte has high ionic conductivity (3.7×10) $^{-3}$ S cm $^{-1}$) and showed reversible cycling for multiple cycles with the Mo_6S_8 cathode [39]. Pandey et al. created a gel polymer electrolyte including magnesium perchlorate (Mg(ClO₄)₂), poly(vinylidene fluoride-co-hexafluoropropylene) (PVdFHFP), magnesium oxide nanoparticles, and organic solvents like ethylene carbonate (EC), propylene carbonate (PC), and tetrahydrofuran (THF). Although this gel polymer had a high ionic conductivity (8 mS cm⁻¹), no cycling data was provided

* Corresponding author. E-mail address: Maha.atallah@fsc.bu.edu.eg (M.A. Attallah).

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Fig. 1. (a,b) the SEM images of $PVA_{(0.3-x)wt.\%}$ (Mg(CF₃SO₃)₂)_xNa₂H₂₀B₄O₁₇ (x = 0,0.04 wt%), (c) XRD of (Mg(CF₃SO₃)₂) (d)XRD of Na₂H₂₀B₄O₁₇ and (d) XRD of $PVA_{(0.3-x)wt.\%}$ (Mg(CF₃SO₃)₂)_x Na₂H₂₀B₄O₁₇ (x = 0,0.01,0.02,0.03,0.04,0.05 wt%).

[40]. Shao et al. recently produced a polymer nanocomposite electrolyte comprising Mg(BH₄)₂, PEO, and MgO nanoparticles free of plasticizers and solvents. The Mg-metal cells displayed a highly stable specific capacity for 150 cycles when this polymer electrolyte was combined with the Mo₆S₈ cathode. These cycling trials, however, were carried out at a high temperature of 100 °C. Although the cycling performance is outstanding, the requirement for high temperatures may make it difficult to use this polymer electrolyte in practical applications [2]. Wang et al. have suggested integrated electrolytes using a solvent diglyme with (0.1 mol) Mg[BH₄]₂ and (1.5 mol) Li[BH₄] [36]. After 100 h, this electrolyte exhibits a low overpotential of approximately 0.04 V. Ramasubramonian Deivanayagam et al. describe a PVDF-HFP-based composite polymer electrolyte (CPE) with Mg(ClO₄)₂ salt, Pyr14-TFSI ionic liquid, and TiO₂ ceramic nanoparticle fillers for use in solid-state Mg-metal batteries at room temperature, it was found that the CPE has a good cyclability with a Mg-metal anode [41]. M. Hema prepared PVA/NH₄X (X = Br, Cl, I) electrolyte, in comparison NH₄Cl (1.0×10^{-5} S cm $^{-1})$ and NH4Br (5.7 \times 10 $^{-4}$ S cm $^{-1}),$ PVA doped with NH4I (2.5 \times 10^{-3} S cm⁻¹) has a more excellent conductivity [42]. Duchene et al. successfully created a reliable 3 V all-solid-state sodium-ion battery by combining a sodium metal anode and a NaCrO₂-based cathode with an electrolyte based on closo-borate. It can be demonstrated that

 $Na_2(B_{12}H_{12})_{0.5}(B_{10}H_{10})_{0.5}$ can endure stable and efficient cycling up to a voltage of 3.2 V with a revocable capacity of 85 mA h g^{-1} at a rate of C/20 by enhancing the solid-solid contact between the electrolyte and the cathode [43]. Ould et al. developed a technique for synthesizing a range of sodium borate salts from inexpensive and readily available starting ingredients. The findings show that whereas NaPF₆ exhibits excellent water and air compatibility, the electrolyte salts Na[B(HFIP)₄]. DME and Na[B(pp)₂] offer greater cycling equivalent capacity. This increased chemical stability makes it possible to handle, move, and store salt effectively—all crucial requirements for use in a commercial battery [44]. Wang creates a unique SIC-GPE (PSP-GPE) that combines PVCA and PSTB. The PSP-GPE demonstrates a conductivity of 1×10^{-4} S cm⁻¹ at 30 °C, t_{Na+} (0.88), and an impressive thermal persistence. Also, it is found that the Na₃V₂(PO₄)₃/PSP-GPEs/Na battery has much-improved cycling compared to the cell with a liquid electrolyte based on NaClO₄, particularly at 60 °C [45]. Different fillers, including SiO₂, MgO, Al₂O₃, and TiO₂, can be incorporated into polymers such as PVA and polyvinylidene fluoride (PVDF) to enhance their mechanical and electrical characteristics to be used as a conductive medium for battery electrolyte [46-48]. Amongst other polymers, polyvinyl alcohol (PVA) has garnered a lot of research interest because of its unparalleled qualities, including ease of manufacture, strength, durability, thermal



Fig. 2. (a, b and c) FTIR spectra, (d) k vs. λ , (e) n vs. λ , (f) $(\alpha hv)^{0.5}$ vs. hv; of PE_x.

stability, resistant to chemicals, and excellent mechanical and electrical qualities [49]. On the other hand, Mg(CF₃SO₃)₂ was used due to its affordable price, non-nucleophilic nature, excessive ionic conductivity, outstanding thermal and oxidative stability, graspability at room temperature, abundance in nature, high solubility in a variety of solvents, commercial accessibility, high purity, and ultralow humidity [50,51]. Because of its solubility, chemical fit with electrodes, initial materials affordable price, great thermal stabilities, low toxicity, strong B-O bonds that endorse chemical stability, adjustable electronic features, and considerable density sodium borate Na₂H₂₀B₄O₁₇ has been selected as the anion from among all of these fillers [52]. Not only tetraethylene glycol dimethyl ether play a crucial role in engineering stable electrolyte interfaces. since alter the chemical composition of the solid electrolyte interface (SEI) layer on the surface of the Mg anode, enabling low overpotential of Mg 2+ extraction/insertion, improving the Mg 2+ transference, and enhancing electrochemical performance of the electrolyte, but also it no decomposition, excellent chemical and thermal stability, Its high boiling point, non-hazardous, environmentally friendly, and abundantly available aqueous [53]. The most significant advantage of using this polymer in electrolyte industries than liquid electrolytes is its low flammability, easy processability, stronger resistance to vibration, stress, and mechanical deformation, and better electrode/electrolyte interfacial contact as well as compatibility than inorganic solid electrolytes [54]. In this study, a solid polymer electrolyte relies on polyvinyl alcohol (PVA), (Mg(CF₃SO₃)₂), and sodium borate Na₂H₂₀B₄O₁₇ has been fabricated to segregate Mg anode surface from the electrolyte and enhance all of the electrochemical

performance, life cycle, safety, the durability of Mg-S battery. Electrochemical impedance spectroscopy (EIS), overpotentials of Mg deposition/dissolution, cyclic voltammetry (CV), and linear sweep voltammetry (LSV) were used to examine the produced electrolytes. Full Mg-S cells have been constructed, and electrochemical performances have been assessed to study the practical application of the produced electrolytes and cathodes. The improved batteries provide the highest starting capacity of 100 mAh g⁻¹. Additionally, energy-dispersive X-ray spectroscopy (EDS), scanning electron microscopy (SEM), and X-ray diffraction (XRD) are used better to understand the reversible magnesium-storage process at various cycle phases.

2. Material and methods

2.1. Electrolyte preparation

PVA, Mg(CF₃SO₃)₂, Sodium borate (Na₂H₂₀B₄O₁₇), and C₁₀H₂₂O₅ (G₄) have been brought from Alfa Aser 98 %. 0.7 gm of PVA was dissolved in 25 ml deionized water and stirred for 48 h at 70 °C then 0.3 gm $(Mg(CF_3SO_3)_2)$ of and xwt.%Na2H20B4O17 (x 0,0.01,0.02,0.03,0.04,0.05) (PVA_(0.3-x)wt.%(Mg(CF3SO3)2)) $_{\%}$ Na₂H₂₀B₄O₁₇) (PE_x) was added. Finally, 20 µl from G₄ have been added to $PVA_{-(0.3-x)wt.\%}$ (Mg(CF₃SO₃)₂)_x Na₂H₂₀B₄O₁₇ with x = 0.04 (X₄). Then, the resulting solution was cast on glass sheets using a mini coater (MC-20, Hohsen) and dried in the open air at 353 k. The resulting film was then cut to a 19.4 mm disc diameter and dried at 333 K in a vacuum oven.



Fig. 3. (a) Nyquist plots of SS//electrolytes//SS at 303k (b) Arrhenius plot, (c) Stripping/plating of Mg//X₄//Mg cells (d) LSV of Mg//X₄//SS cells at 303k (e) Nyquist plots of symmetric Mg|X₄|Mg cells before and after polarization at 303k (f) polarization current curve vs time of symmetric Mg|X₄|Mg cells.

2.2. Cathode preparation

Sulfur(S) and super carbon (C) have been brought from Alfa Aser 99 %. 0.8 gm of Sulfur and 0.2 gm of Super carbon P were hand-ground for 30 min. The mixture was ground by using a ball mill (500 rmp) for 24 h. Then add 0.066 gm of Polyvinylidene fluoride (PVDF) and dissolve in Nmethyl-2-pyrrolidinone (NMP). Finally, the solution was coated on Al foil using a mini coater (MC-20, Hohsen) and dried in an oven at 100 °C for 120 min with a thickness of 50 micros.

2.3. Material characterization

The XRD patterns were obtained using a Rigaku Miniflex 600 diffractometer. The morphology of the samples was examined using a Jeol JMS-700 scanning electron microscope with an integrated energy dispersive X-ray (EDS).

2.4. Electrochemical measurements

CHI604E electrochemical workstation in symmetric stainless steel

(SS) SS|X₄|SS electrolytic cell, the ionic conductivity of the electrolyte samples (σ) have been calculated from 1 MHz to 10 Hz at various temperatures using the equation $\sigma = \frac{t}{R \times A}$, where t is the distance between the two SS discs, R is the resistance, and A is the SS disc's impactful area. The electrolyte electrochemical stability was investigated using a two-electrode coin cell with Mg disc as the reference and counter electrode and stainless steel as the working electrode. The Mg-S coin-type cell (CR2032) was assembled in an argon atmosphere to test the electrochemical activity. The discharge and charge tests were conducted among 0.1 and 2.5 V cut-off voltages (versus Mg²⁺/Mg). The cells were disassembled, and the recovered cathode disks were cleaned by repeatedly washing in acetonitrile and dried at 65 °C for 1 h before XRD, SEM, and EDS investigations.

3. Results and discussion

Fig. 1a and b depicted the SEM images of the PE_x at x = 0 and 0.04 wt %. It can be seen that (Mg(CF₃SO₃)₂) has been distributed in the PVA matrix with pores forms. Introducing Na₂H₂₀B₄O₁₇ in the PVA_(Mg (CF₃SO₃)₂) matrix makes the distribution more regular with the same



Fig. 4. (a) FTIR spectrum, (b)Absorbance,(c) SEM image of X₄_G₄.



Fig. 5. (a) Nyquist plots of SS//X₄_G₄//SS at 303k (b) Arrhenius plot, (c) Stripping/plating of Mg//X₄_G₄//Mg cells (d) LSV of Mg//X₄_G₄//SS cells at 303k (e) Nyquist plots of symmetric Mg| X₄_G₄|Mg cells before and after polarization at 303k (f) polarization current curve vs time of symmetric Mg| X₄_G₄|Mg.

shape. Fig. 1c and d depicted the XRD of (Mg(CF₃SO₃)₂) and Na₂H₂₀B₄O₁₇ salt, respectively. The (Mg(CF₃SO₃)₂) pattern shows a significant match with PDF Card No:2100509 QM:C .Na2H20B4O17 major peaks have been detected corresponding to the PDF Card No:90155567 QM:C. Fig. 1e shows the XRD of PE_x , x = 0,0.01,0.02,0.03,0.04, and 0.05 wt%. It can be observed that there is a principal peak at 2 $\theta = 20^{\circ}$ on every sample [49], symbolizing the semi-crystalline PVA polymer nature. This peak intensity was reduced by raising the $Na_2H_{20}B_4O_{17}$ concentrations, indicating a decrease in crystallinity. This might result from new bonds being formed between the polymer components and the $\mathrm{Na_2H_{20}B_4O_{17}}$ components at their contact. Additionally, a distinct peak at $2 \theta = 12^{\circ}, 14^{\circ}, 18^{\circ}, 21^{\circ}, 25^{\circ}, 30^{\circ}$ have been noticed; this peak may be caused by some crystalline patterns of (Mg(CF₃SO₃)₂). As the Na₂H₂₀B₄O₁₇ wt% increases, this peak intensity completely vanishes, indicating a powerful interaction between the PVA (Mg(CF₃SO₃)₂) matrix and the Na₂H₂₀B₄O₁₇. This indicates that the interaction between PVA and $(Mg(CF_3SO_3)_2)$ is promptness up by the Na₂H₂₀B₄O₁₇.

Fig. 2a, b, and c depicted FTIR transmission spectra of PE_x . At x = 0, it is obvious that some transmission bands exist at wavenumbers 3250, $2950,1550,1230,1180,1030,850 \text{ cm}^{-1}$. These bands are attributed to the OH stretching, the CH anti-symmetric stretching [45], the C-H bending vibration of CH₂, the twisting vibrations, the crystalline sequence peak of PVA, the CO stretching vibrations, and the C-C stretching vibration, respectively [55-57]. Another band appeared at wavenumbers 780 and 640 cm⁻¹ is due to symmetric CF₃, symmetric stretching of SO₃ [58]. With the introduction of Na₂H₂₀B₄O₁₇ in the framework of the PVA_(Mg(CF₃SO₃)₂) matrix; The original band intensity decreases due to the free radical's production, cross-links etc. where the carbon-carbon connection cleavage and the -H removal from the polymer chains backbone were all linked to this drop in these vibrational bands intensity. These data suggest that the structural change between PVA and (Mg(CF₃SO₃)₂) is caused by Na₂H₂₀B₄O₁₇. These structural alterations in the host PVA $(Mg(CF_3SO_3)_2)$ caused by the $\mathrm{Na_2H_{20}B_4O_{17}}$ doping are consistent with the observed changes in the samples' absorption and optical energy gap. Because the extinction coefficient (k) represents the energy lost quantity (as a scattering or reflection) that is induced by material particles, Fig. 2d displays the extinction coefficient (K) of PEx. The extinction coefficients (K) in the UV band (200-250 nm) decreased considerably with increasing wavelength. This is because the UV spectrum region's (high energy) photons are strong enough to cause the electrons to leave the valence band and



Fig. 6. (a) Nyquist plot, (b) Linear fitting of Warburg impedance before and after cycling, (c)CV curve at scan rate 20 mV/s, (d) discharge capacity vs cycle number (e)galvnostatic discharge-charge curves.

 $C_2(\mu F)$

0.2

3.89

Table 1								
EIS parameters of symmetric Mg/Mg cells.								
EIS Parameters	$R_s(\Omega)$	$R_{SEI}(\Omega)$	$R_{ct}(\Omega)$	C ₁ (µF)				
before	18.95	299.6	390.4	0.01				

269.7

500

1.56

48.45

after

enter the conduction band, decreasing energy loss. The extinction coefficients rise with increased wavelength in the visible range. This is because there isn't enough energy in the incident photon for the electrons to go from the valence band to the conduction band. Consequently, a significant amount of energy was lost, producing an increasing extinction coefficient. The refractive index of PEx is shown in Fig. 2e. As the wavelength of the incident light increases, the refractive index reduces, which coincides well with the Cauchy dispersion relation [59]: $n(\lambda) = A + \frac{B}{\lambda^2}$. Further, the light velocity decreases in the PE_x matrix with the increase in the content of the Na₂H₂₀B₄O₁₇; hence, the refractive index increases. The indirect band gap has been determined from the fitting $(\alpha hv)^{0.5}$ vs (hv) linear plots extrapolate. Increasing the concentration of Na₂H₂₀B₄O₁₇ causes the indirect bandgap values to descend. This might be connected to the interaction of the polymer molecules, and $\mathrm{Na_2H_{20}B_4O_{17}}$ forms new bonds that change the structure of the polymer, so new states begin to arise in the region between the valence band and conduction band that is prohibited [60,61].

Fig. 3a represents the Nyquist plots of PE_x using SS|PE_x|SS cells; the PE_x displays a spike at low frequencies and an incomplete semicircle at higher frequencies. The semicircle diameter, which symbolizes the bulk resistance (Rb), decreases when the temperature rises. The behavior follows the Arrhenius equation [62,63] $\sigma = \sigma_0 \exp^{\frac{-E_a}{KT}}$, where magnesium ion conduction in polymer electrolytes (PE_x) exhibits a thermally stimulated mechanism. The (PE_x) fitted curves logarithmic conductivity with

temperature were depicted in Fig. 3b. It is evident that conductivity rises as sodium borate concentration does, which could be connected to an increase in free volume, mobility charge carriers and mutation from semi-crystalline to amorphous state as can be seen in XRD [62]. Using the Arrhenius equation least square fitting, the calculated activation energies are 0.19, 0.21,0.22,0.01,0.16 and 0.14 ev, respectively, suggesting that the minimum energy barrier for Mg²⁺ migration in the polymer electrolyte is at x = 0.03. Fig. 3c depicts Mg stripping/plating curves of the symmetric Mg||Mg cells. The cells exhibit cycle stability at ambient temperature with a nominal overpotential 1 V at a current density of 20 μ Acm². This phenomenon is a result of the electrolyte's effective transport capabilities. Fig. 3d depicts LSV scans of the electrolytes run on an asymmetrical cell of the SS X4 Mg cell at room temperature; the X₄ stays stable up to 3V. EIS before and after dc polarization voltage = 0.1 V was recorded in Fig. 5e to determine the initial R0 and steady-state Rs charge-transfer resistances. The polarization current is presented in Fig. 3f. The following Bruce Vincent equation [64,65] $t_{Mg}^{+2}=\frac{I_0(\Delta V-r_0I_0)}{I_0(\Delta V-r_sI_s)}$ was used to calculate the t_{Mg}^{2+} ion transference number where I₀ and I_s denote respectively, the starting current and the steady-state current and r_0 and r_s are the cell resistances before and after the dc polarization. The t_{Mg}^{2+} values was calculated ~ 0.7 .

Fig. 4a depicted the FTIR transmission spectra of $X_4_G_4$. It is obvious that some transmission bands exist at wavenumbers 3250, 2950,1550, 1250,1180,850 cm⁻¹. These bands are attributed to the OH stretching, the CH anti-symmetric stretching [45], the C-H bending vibration of CH₂, the twisting vibrations, the crystalline sequence peak of PVA and the C-C stretching vibration, respectively [55–57]. Another peaks at 780 and 640 cm⁻¹ is are attributed to symmetric CF₃, symmetric stretching of SO₃ [58]. Fig. 4b shows the Absorbance of $X_4_G_4$. It can be seen that the absorbance decreases with increasing the wavelength. Fig. 4c shows the SEM image of $X_4_G_4$. It can be seen that (Mg(CF₃SO₃)₂) has been distributed in the PVA matrix with pores forms. Introducing C₁₀H₂₂O₅



Fig. 7. XRD patterns at various cycle statuses (b) zoom in 2 θ = 37–39° (c) W-H analysis (d) EDS spectra of the sulfur cathodes at different synthetic states (e,f,g) SEM micrographs and element mapping at the fresh state, discharged state and discharged/charged state.

Table 2	
Crystallite size and strain at different cycling states.	

Element	Pristine	discharge	Discharge-charge
Strain($\varepsilon \times 10^{-3}$)	4.9	8.2	0.9
Crystallite size D(nm)	11.14	8.52	181.02

(G₄) in the PVA_(Mg(CF₃SO₃)₂)/Na₂H₂₀B₄O₁₇ matrix makes the distribution more regular with the same shape. Fig. 5a represents the Nyquist plots of X4_G4 using SS X4_G4 |SS cells; the X4_G4 displays a spike at low frequencies and an incomplete semicircle at higher frequencies. The X₄ G₄ fitted curves logarithmic conductivity with temperature were depicted in Fig. 5b. It is evident that conductivity rises as temperature increases, which could be connected to an increase in mobility charge carriers. Fig. 5c depicts Mg stripping/plating curves of the symmetric Mg||Mg cells. The cells exhibit cycle stability at ambient temperature with a nominal overpotential 1.25 V. This phenomenon is a result of the electrolyte's effective transport capabilities. Fig. 5d depicts LSV scans of the X₄ G₄ run on an asymmetrical cell of the SS | X₄ G₄ | Mg cell at room temperature; the X4_G4 stays stable up to 2.5V. EIS before and after dc polarization voltage = 0.1 V was recorded in Fig. 5e to determine the initial R0 and steady-state Rs charge-transfer resistances. The polarization current is presented in Fig. 5f. The ion transfer number t_{Mg}^{2+} has been calculated by using Bruce Vincent equation and its value was ~0.9. By adding a G₄ to X₄ it can be seen that all the structure and electrochemical properties have been enhanced. It can be seen that in the FTIR spectrum, not only the main peaks at 3250, 2950, 1250, 1100 cm^{-1} have become broad and also their intensities increase but also the energy band gap decrease from 4ev for X₄ to 3.7ev for X₄_G₄. Electric conductivity has been increased from $4.1\times10^{-8}\,S\,cm^{-1}$ for X_4 to $8.09\times\,10^{-7}\,S\,cm^{-1}$ for $X_4_G_4$ at temperature 323 $^{\circ}$ k, The cells exhibit better cycling at an ambient temperature where the number of cycle increases from 80 for X₄

cycle to 100 for $X_4_G_4$ cycle, the stability of the cell have been improved whereas $X_4_G_4$. stays stable up to 3V than X_4 where stay stable at 2.5 V, the over potential decrease from 2 V for X_4 to 0.5 V for $X_4_G_4$, finally the ion transfer number increase from 0.7 for X_4 to 0.96 for $X_4_G_4$.

Fig. 6a shows the Mg $|X_4_G_4|$ S coin cells' Nyquist plots before and after CV. Using the equivalent circuit provided by the ZView program, the battery impedance parameters were estimated, where Rs, RSEI, and Rct stand for the respective ohm resistance of the bulk cell, interfacial layer resistance, and charge-transfer resistance, Table 1. It can be seen that each ohm resistance and charge-transfer resistance increase from 18.95 Ω , 390.4 Ω before discharge to 48.45 Ω and 500 Ω after discharge, respectively. The interfacial layer resistance decreases from 299.6 Ω before discharge to 269.7 Ω after discharge. The diffusion coefficient was calculated from the equation: $D_{Mg^{2+}} = \frac{R^2 T^2}{2A^2 n^4 F^4 C^3 A_{\omega}^2}$ [66] where the Warburg coefficient (A_w) can be determined by fitting the formula -Z' = $R_s+R_{ct}+A_\omega w^{-0.5}$ in the low frequencies region, Fig. 6b. The D_{Mg} $^{2+}$ values ~4.3 \times 10 $^{-17}$ cm 2 s $^{-1}$ before discharge and increases to ~ 3.8 \times $10^{-15} \text{ cm}^2 \text{ s}^{-1}$ after discharge, a sign of the S cathode's sluggish electrochemical kinetics due to high electrostatic interaction with the storage Mg-ions; Nevertheless, the results show good kinetics in comparison to conventional Mg-storage materials similar organic and inorganic cathodes (Mo₆S₈, TiS₂,VS₂, TiSe₂, VSe₂, and PI@CNT are 10^{-12} , 10^{-22} , 10^{-22} , 10^{-19} , 10^{-19} , and 10^{-12} cm² s⁻¹, respectively) [16]. Fig. 6c, depicts CV curves between 0.3 and 2.5 V at 20 mV/s scan rate of Mg X_4 G₄|S. The cathodic peak for Sulfur was clearly visible at 0.1–0.3 V. Additionally, the discharge products re-oxidation is responsible for the anodic peak at 2.2 V. Fig. 6d depicts the discharge capacity vs cycle number. Fig. 6e shows the discharge/charge curves of Mg| X₄_G₄|S coin cells at the current density of 20 $\mu A~cm^{-2}$. The cell delivers an initial discharge capacity of 100 mAh g^{-1} at a voltage of 0.3 V. This fully proves that the X4_G4 protective layer significantly enhances the interfacial compatibility of Mg metal by reducing the side reaction between the Mg metal and the electrolyte

The Sulfur cathode morphology, compositions, and structural evolution at different electrochemical states were examined using XRD, SEM, and EDS examinations on a Mg $|X_4_G_4|$ S cell discharged and discharged/charged at a fixed capacity. To investigate Mg²⁺ initial magnesiation/demagnesiation effects in sulfur structure, Fig. 7a displays the XRD patterns at various cycle statuses. It can be obvious that there are some peaks at 2 θ = 16.5 °,23 °, 33.5 °,38.2 °,44.7 ° and 65.3 ° related to (022), (220),(331),(422),(062) and (468) sulfur structure lattice planes. During the initial discharge, not only did the peak area of sulfur significantly decrease, but there was also a peak appearance at 2 θ = 46.5 ° degrees, corresponding to the S complex. Also, after the introductory discharge, the Sulfur predominant peak (422) is right-shifted to higher 2 θ values, Fig. 7b. Williamson-Hall equation [67,68] $\beta \cos \theta = \frac{k\lambda}{D}$ +4 $\varepsilon \sin \theta$ was used to calculate particle size (D), microstrain (ε) Where β is the full width at half maximum, θ is the Bragg's angle, k is the factor shape, and λ is wavelength. Fig. 7c illustrates the relationship between β $\cos \theta$ and $4\sin \theta$ at various cycling states. This yields a straight line with an intercept of $\frac{k\lambda}{D}$ and a slope of ε . Table 2 provides the crystallite sizes and strains at various cycling phases. It can be observed that crystallite size increase so much after charge. This could be because of the conversion reaction, in which substantial volumetric growth occurs along with the extraction of Mg $^{2+}$ from the structure of the $(Mg(CF_3SO_3)_2$ skeleton-like disrupting the skeleton and shortening the cycle life. The presence of Mg²⁺ ions in the sulfur structure had an impact on the shear microstrain, and the value is shown to change from 0.0049 to 0.0082 after the initial discharge to 0.0009 after recharge, indicating reversible Sulfur deformation and compression upon Mg²⁺ incorporation and retrieval. Fig. 7d depicted the EDS spectra at different cycling statuses; the EDS results showed that the Mg weight percentage has risen since discharge and recharge, confirming the previously stated Mg ²⁺changeable converting reaction procedure during a discharge/charge cycle. Thanks to employing PVA as an adhesive in the electrode procedure and glass fiber as a separator, characteristic patterns related to F and Si were distinctly created. Fig. 7(e,f and g) presents SEM images and mapping analysis at the fresh, discharged, and discharged/charged states. At the early discharge fracture creation, porosity and Mg indicator buildup were noticed concerning the fresh electrode. Succeeding the electrode's charging, the Mg- S growth unit befitted more evident and this confirmed in XRD.

4. Conclusion

 $(Mg(CF_3SO_3)_2)$ _xNa₂H₂₀B₄O₁₇ PVA_(0.3-x)wt.% (x 0,0.01,0.02,0.03,0.04,0.05) polymer electrolytes have been prepared and characterized. XRD and FTIR spectrum confirmed a powerful interaction between the PVA (Mg(CF_3SO_3)₂) and the Na₂H₂₀B₄O₁₇. Furthermore, adding Na₂H₂₀B₄O₁₇ to PVA (Mg(CF₃SO₃)₂) enhances optical properties, ionic conductivity, ion transfer number, and diffusion coefficient. Adding G₄ to X₄ demonstrates effective Mg plating and stripping behavior, good anodic stability (versus Mg/Mg²⁺), and a significant ionic conductivity (10^{-7} S cm⁻¹ at 323 K). Mg | X₄ G₄|S cell delivers a reversible capacity of 100 mAh g^{-1} after 30 cycles. This fully proves that the X₄_G₄ is a promising gel polymer electrolyte magnesium sulfur battery, where a workable method of stabilizing the S cathode for Mg-ion batteries is suggested. A longer cycle life and a greater reversible capacity (100 mAh g^{-1}) are provided.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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