Jones-Dole Coefficients and Conductivity Studies on Electrolyte Solutions of Magnesium Borohydride in Mixtures of 1,2-Dimethoxyethane–Propylene Carbonate and 2-Methoxyethyl Ether–Propylene Carbonate

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Abstract

The study on the competing influences of ion-ion, ion-dipole and dipole-dipole interactions by the species in magnesium borohydride [Mg(BH$_4$)$_2$] organic electrolyte solutions is reported. The organic electrolyte solutions were formulated by dispersing Mg(BH$_4$)$_2$ salt concentrations of 0.01, 0.05, 0.10 and 0.50 M in mixed solvent systems of 1,2-dimethoxyethane (DME)-propylene carbonate (PC) and 2-methoxyethyl ether (DGM)-propylene carbonate (PC). The density, viscosity, conductivity and dielectric constant were determined at different system compositions. The measured viscosity data varied with concentration according to Jones-Dole equation, while the resolved coefficients revealed that DME-PC and DGM-PC mixtures acted as solvates in the Mg(BH$_4$)$_2$ organic electrolyte solutions. The A coefficient values for DME-PC and DGM-PC systems increased progressively with mole fractions over the entire range studied. The B coefficient values for the electrolyte systems were found to exhibit minimum value at 0.822 and 0.095 mole fractions of DME and DGM respectively. The experimental conductivities of the electrolyte solutions measured at temperatures of 298.15, 308.15, 318.15, 328.15 and 338.15K increased with increase in temperature from 0 - 50 % vol. composition range; and thereafter, decreased for both DME-PC and DGM-PC systems. This suggests that the most preferred mix ratio of the studied systems is 50 % vol. of component (DME or DGM) for optimum battery performance.

Keywords: Battery, Ion-dipole interaction, Columbic efficiency, Electrolyte, Jones-Dole Coefficients

1. Introduction

Batteries consist of assembled cells designed solely for the conversion of chemical energy into electrical energy (Hannan et al., 2017). Electrical energy is ubiquitous and its application continues to grow globally. Primarily, electric energy is exploited for heating, lighting and for powering electronics; such as integrated circuit memory retention and biomedical implantable devices. An important battery concept relates to the quantity of load current delivered by the battery and its duration. This is stated as battery capacity (C) and is quoted in Ampere Hours (Ah); for a specific discharge rate to End Point (Output Voltage). The energy density of a cell is quoted in weight (kg) or volume (cm^3) ratio. Another important battery parameter is the coulombic efficiency, defined as the quotient of the charge that leaves the battery in the course of discharge cycle to the charge that passes into the battery in the course of charging cycle. 100 % columbic efficiency is not achievable owing to charge losses from secondary reactions, such as redox reactions in the battery. Better conductivity advances battery reversibility and lessen the voltage differential between the discharge and charge curves. Higher coulombic efficiency means that the level of irreversible drawback reactions in the cell is reduced. The storage of electrical energy necessitates its conversion to another form of energy.

In battery systems, the electrolyte serves as the storage medium and when the battery undergoes discharge, redox reactions generate electrical energy that is harvested as electric current at a particular voltage. The borohydrides have substantial content of gravimetric hydrogen. The absorption and desorption temperatures of hydrogen are relatively low for magnesium borohydride $Mg(BH_4)_2$, spurring immense curiosity and interest (Aurbach et al., 2001 a & b). The borohydrides are employed as reducing agents in chemical syntheses, since their reducing property fosters stability against electrolytic reduction. The deficiencies in lithium battery systems have created the pathway for magnesium batteries as alternatives. Magnesium batteries offer astoundingly high volumetric capacity, improved safety besides the relative abundance of Mg metal. To exploit the vast potential offered by Mg batteries, several hurdles in their electrolyte design must be surmounted for this technology to be feasible (Muldoon et al., 2012). The corrosive nature of perchlorates has been credited to the existence of chlorides in their ions. Traditional electrolytes such as $Mg(ClO_4)_2$ were reckoned incompatible with magnesium anode owing to the growth of an ion-impeding layer, the consequence of their electrochemical reduction (Pour et al., 2011). The discovery of halide free electrolytes is crucial for advancing technology towards the achievement of a rechargeable Mg battery. Without doubt, the shortages of viable electrolytes and feasible cathode materials have restricted demonstrations of rechargeable Mg battery systems to the laboratory (Mohtadi & Mizuno, 2014). Reversible Mg plating / stripping continues to delude many electrolytes. Importantly, it is accepted that the solution coordination structures of Mg complexes in these electrolytes are essential for reversible Mg plating / stripping (Muldoon et al., 2012). The stability of metal complexes is aided by coordinating solvent ligands and the stability of $Mg(BH_4)_2$ coordination structures show no exception to this rule. In the complex $Mg(BH_4)_2 \cdot nL$ (L = ligand), ligands are dislodged by another of higher denticity (Bremer et al., 2005). Magnesium borohydride electrolyte was considered for Mg batteries (Mohtadi et al., 2012 a & b). This choice was made on the assertion that the BH_4^- was relatively a strong reducing agent and could withstand the reducing vicinity of the Mg anode. These findings attest to the fact that ionic salts could be adapted to the magnesium metal, if the anion in the salt has sufficient reductive stability. Pioneering work in hydride chemistry recognize that molecular hydrogen complexes participate significantly in metal complex reactivity (Chatt, 1968).

Information bordering on structure property relationship of solutions continues to advance the studies on viscosities of electrolyte solutions. Ion-ion and ion-solvent interactions are of significant interest especially amongst the complexity of interactions that exist between ions in solutions. Viscosity studies on $Mg(BH_4)_2$ organic electrolyte solutions are useful for understanding interactions at molecular level. Among the main properties of the solvents, low viscosity of DGM and DME and high dielectric constant of PC conduce high conducting electrolytes. It is the interest of this investigation to determine the viscosity and conductivity of $Mg(BH_4)_2$ electrolytes constituted using the mixed solvent systems of DGM-PC and DME- PC at different conditions of experiment; and to apply the outcome of the study in electrolyte design for high capacity energy source.

2. Experimental

2.1. Materials and Methods

The solvents procured for this research are DME (99.5%) and PC (99.5%) products of BDH Chemical Ltd. England, DGM (99.5%) product of BASF Chemical Company England and dry $Mg(BH_4)_2$ (95%) product of Sigma Aldrich. The Binary mixtures of DGM & PC and DME & PC were constituted by mixing the liquid components in varying percentage of 0%, 15%, 25%, 50%, 75%, 85% and 100% (volume percent). The samples were weighed on HR-120 (A&D Japan) electronic balance with a precision of $\pm10^{-4}$ g. The mole fractions range from 0.125 to 1.000 (DME) and 0.095 to 1.000 (DGM). The density of the solutions (prepared in concentrations of 0.01, 0.05, 0.1, 0.5M in solvent) was determined with a single stem capillary pycnometer bottle at temperatures of 298.15, 308.15, 318.15k, 328.15K and 338.15 ± 0.01 K. The kinematic viscosity of the solutions was measured at the desired temperature using a suspended ubbelohde viscometer tube ASTM D 446. The experimental solutions were retained at the required temperatures of 298.15, 308.15, 318.15, 328.15K and 338.15 ± 0.01 K with Koehler electronic thermostat water bath. An electronic timer with precision ± 0.01 s was used to determine the flow time. The conductivity of the solutions was measured with Thermo Scientific Orion conductivity meter (Orion 162A), calibrated with 0.001 M KCl standard (147 µS/cm at 298.15, 308.15, 318.15. 328.15 and 338.15 ± 0.01 K).

2.2. Data Analysis

The kinematic viscosity was determined using the equation (Ibezim-Ezeani et al., 2017 a):

$$V = kt \tag{1}$$

Where *V* is the Kinematic viscosity, *k* is the Viscometer constant and *t* the flow time.
The dynamic viscosity was deduced with the formula:

$$\eta = V\rho \tag{2}$$

Where η is the Dynamic viscosity, *V* is Kinematic viscosity and ρ density of the liquid.

The viscosity of dilute electrolyte solutions (η_r), vary with concentration (*c*) according to the Jones-Dole equation (Jones & Dole, 1929):

$$\eta_r = \frac{\eta}{\eta_o} = 1 + AC^{\frac{1}{2}} + BC \tag{3}$$

Where η and η_o are the viscosities of solution and solvent in turn, while A and B are the Jones - Dole coefficients. The A and B coefficients symbolize ion-ion interactions and ion-solvent interactions respectively. The parameters A and B were obtained by plotting a graph of ($\eta_r - 1$)/($c^{1/2}$) versus $c^{1/2}$ for the Mg(BH$_4$)$_2$ salt solutions in the whole composition range of DME-PC and DGM-PC systems at the temperature of 298.15 ± 0.01 K.

The dielectric constant (ε_{mix}) of the binary solvent mixtures (DME-PC and DGM-PC) were calculated with the equation (Rohdewald & Molder, 1973):

$$\varepsilon_{mix} = \frac{[(\%Solvent_1)\varepsilon_1 + (\%Solvent_2)\varepsilon_2]}{100} \tag{4}$$

Where ε_1 and ε_2 are the dielectric constants of pure DME (or DGM) and PC respectively.

3. Results and Discussion

3.1. Jones-Dole Coefficients

The values of Jones-Dole (A and B) coefficients for the DME-PC (Table 1) and DGM-PC (Table 2) electrolyte solutions are positive over the entire constitution range; suggesting strong solvation of Mg^{2+} in both solvent systems, which implies that strong ion-solvent arrangements are prevalent. Observations from Tables 1 and 2 reveal that the values of B coefficients for Mg(BH$_4$)$_2$ electrolyte solutions were at their minimal in 0.822 mole fraction of DME and 0.095 mole fraction of DGM. The discourses of previous researchers did expound this behavior, and noted that the solvent mixture with the minimum value of B would be the most structured (Stokes & Mills, 1965). Therefore, a higher degree of structuring was attained in DGM (15 % vol.) than in DME (85 % vol.).

Results from Table 1, noted that the values of A coefficients increased progressively with increasing composition of DME in the binary mixture, indicating that ion-ion (electrostatic) interactions are reinforced with increasing proportion of DME. Also, from the constitution range of 75 - 100 % vol. DME, ion-ion interactions began to gain preponderance, as the A values compete closely with the B values. This may be credited to the poor solvation of the Mg^{2+} in the binary mixture over this composition range, resulting in very unstable Mg complexes. Further observations revealed that ion-solvent interactions are predominant over ion-ion interactions in the range 0 - 50 % vol. DME. This may be credited to the strong solvation of the Mg^{2+} ions over this composition range in the binary mixture (DME-PC). Furthermore, the values of B coefficient decreased over the constitution range 0 - 85 % vol. DME. This suggests that ion-solvent interactions waned as the DME proportion increased, or it may be reported that DME possesses lesser affinity for electrolyte than for PC molecules. That is, solvent-solvent type interactions prevail over ion-solvent type interactions (DME molecules interacting with PC molecules rather than with cations); signifying that ion solvation is limited by strong electrostatic forces as the dielectric constant of the binary mixture decreases. Again, the relatively sizable dipole moment of DME molecules 1.71D (Smith et al., 1995) may become significantly impaired by dispersion forces. This may undermine the prospect of ion-dipole interactions and concurrently foster attraction between dissimilar molecules (DME and PC molecules).

Results from Table 2, showed that ion-solvent interactions predominate over ion-ion interactions in the constitution range 15 - 85 % vol. DGM. This may be credited to the strong solvation of the Mg^{2+} over this composition range in the binary mixture (DGM-PC). There was gradual increase in A coefficient values as DGM proportion increased in the mixture; but not to the level of surpassing B coefficient values.

The B coefficient values also increased over the constitution range 15 - 50 % vol. DGM in the binary mixture; which suggests that ion-solvent interactions are strengthened as the DGM concentration increased, or it may be stated that DGM has more affinity for the electrolyte than PC molecules; that is, ion-solvent interactions predominate. The values of B coefficient reach maximum at 50 % vol. DGM and above this threshold, ion solvation is not so appreciably impaired as with the DME binary mixture; probably due to stronger coordination with the tridentate O_2 of DGM in the solvated Mg^{2+} complex. Comparison of the values of the B coefficient for both systems studied (Tables 1 and 2); reveal that the values are greater in DGM-PC binary mixture than in DME-PC binary mixture. This further corroborates the fact that solvation of the Mg^{2+} complex is more favorable in DGM-PC system. Again, the relatively sizable dipole moment of DGM molecules 1.91D (Kotera et al., 1962) might not be significantly impaired by dispersion forces when a single DGM molecule orients freely about the Mg^{2+}. Thus, the strength and stability of the ion-dipole attraction is preserved. This stability of the dissolved Mg^{2+} complex in DGM is a consequence of the superior potency of the ion-dipole attraction relative to dipolar and dispersion forces between dissimilar molecules (DGM-PC).
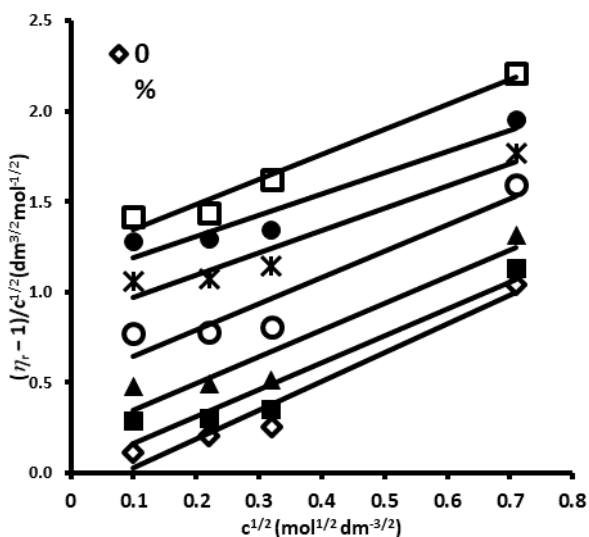

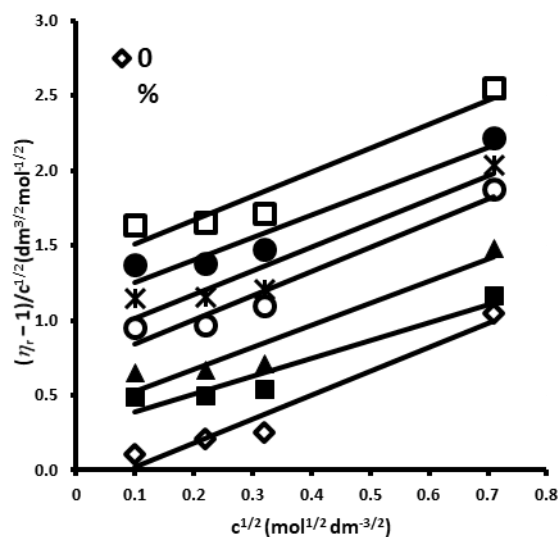**Figure 1.** Plots of Jones-Dole parameter values at different mole fractions of DME

Figure 2. Plots of Jones-Dole parameter values at different mole fractions of DGM

Table 1: Jones-Dole A and B Coefficients at Different Compositions of DME-PC

DME (%)	X_1	Linear Equation	A($dm^{3/2}mol^{-1/2}$)	B ($dm^{3/2}mol^{-1/2}$)
0	0.000	y = 1.596x - 0.135	-0.135	1.596
15	0.125	y = 1.486x + 0.016	0.016	1.486
25	0.214	y = 1.477x + 0.201	0.201	1.477
50	0.448	y = 1.456x + 0.496	0.496	1.456
75	0.710	y = 1.239x + 0.843	0.843	1.239
85	0.822	y = 1.182x + 1.069	1.069	1.182
100	1.000	y = 1.382x + 1.206	1.206	1.382

Table 2: Jones-Dole A and B Coefficients at Different Compositions of DGM-PC

DGM (%)	X_1	Linear Equation	A($dm^{3/2}mol^{-1/2}$)	B ($dm^{3/2}mol^{-1/2}$)
0	0.000	y = 1.596x - 0.135	-0.135	1.596
15	0.095	y = 1.199x + 0.266	0.266	1.199
25	0.167	y = 1.466x + 0.383	0.383	1.466
50	0.374	y = 1.616x + 0.680	0.680	1.616
75	0.643	y = 1.582x + 0.852	0.852	1.582
85	0.772	y = 1.492x + 1.106	1.106	1.492
100	1.000	y = 1.608x + 1.345	1.345	1.608

Table 3: Experimental Conductivity (κ) for DME-PC Binary Mixtures at 298.15 K

DME (%)	X_1	0.01M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	0.05M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	0.10M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	0.50M $Mg(BH_4)_2$ κ ($mScm^{-1}$)
0	0.000	0.31	0.86	1.23	4.14
15	0.125	0.49	1.06	1.57	4.64
25	0.214	0.60	1.20	1.77	4.96
50	0.448	0.82	1.54	1.99	5.42
75	0.710	0.73	1.38	1.86	5.11
85	0.822	0.55	1.15	1.64	4.73
100	1.000	0.13	0.63	1.06	4.04

Table 4: Experimental Conductivity (κ) for DME-PC Binary Mixtures at different temperatures

DME (%)	X_1	308.15 K 0.01M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	0.05M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	318.15 K 0.01M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	0.05M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	328.15 K 0.01M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	0.05M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	338.15 K 0.01M $Mg(BH_4)_2$ κ ($mScm^{-1}$)	0.05M $Mg(BH_4)_2$ κ ($mScm^{-1}$)
0	0.000	0.32	0.91	0.35	0.97	0.37	1.00	0.38	1.02
15	0.125	0.51	1.13	0.53	1.15	0.56	1.17	0.56	1.21
25	0.214	0.61	1.28	0.65	1.30	0.66	1.32	0.67	1.35
50	0.448	0.83	1.58	0.87	1.64	0.90	1.68	0.92	1.72
75	0.710	0.77	1.45	0.80	1.47	0.82	1.53	0.84	1.57
85	0.822	0.58	1.20	0.62	1.26	0.62	1.31	0.64	1.35
100	1.000	0.15	0.67	0.19	0.72	0.21	0.75	0.22	0.80

Table 5: Experimental Conductivity (κ) for DGM-PC Binary Mixtures at 298.15 K

DGM (%)	X_1	0.01M $Mg(BH_4)_2$ K (mScm^{-1})	0.05M $Mg(BH_4)_2$ K (mScm^{-1})	0.10M $Mg(BH_4)_2$ K (mScm^{-1})	0.50M $Mg(BH_4)_2$ K (mScm^{-1})
0	0.000	0.39	1.08	1.38	4.77
15	0.095	0.60	1.30	1.73	5.42
25	0.167	0.78	1.47	1.97	5.74
50	0.374	1.10	1.85	2.3	6.03
75	0.643	0.98	1.64	2.13	5.67
85	0.772	0.80	1.47	1.92	5.36
100	1.000	0.27	0.89	1.28	4.54

Table 6: Experimental Conductivity (κ) for DGM-PC Binary Mixtures at different temperatures

DGM (%)	X_1	308.15 K 0.01M $Mg(BH_4)_2$ κ (mScm^{-1})	308.15 K 0.05M $Mg(BH_4)_2$ κ (mScm^{-1})	318.15 K 0.01M $Mg(BH_4)_2$ κ (mScm^{-1})	318.15 K 0.05M $Mg(BH_4)_2$ κ (mScm^{-1})	328.15 K 0.01M $Mg(BH_4)_2$ κ (mScm^{-1})	328.15 K 0.05M $Mg(BH_4)_2$ κ (mScm^{-1})	338.15 K 0.01M $Mg(BH_4)_2$ κ (mScm^{-1})	338.15 K 0.05M $Mg(BH_4)_2$ κ (mScm^{-1})
0	0.000	0.39	1.11	0.43	1.15	0.45	1.21	0.47	1.27
15	0.095	0.62	1.36	0.66	1.41	0.69	1.46	0.72	1.51
25	0.167	0.80	1.52	0.83	1.60	0.86	1.67	0.89	1.71
50	0.374	1.11	1.89	1.13	1.95	1.15	2.00	1.18	2.07
75	0.643	0.99	1.68	1.00	1.72	1.03	1.78	1.07	1.85
85	0.772	0.83	1.50	0.83	1.54	0.85	1.60	0.88	1.68
100	1.000	0.29	0.92	0.30	0.97	0.33	1.02	0.36	1.08

Table 7 Calculated Values of Dielectric Constant at Different Solvent Compositions

Composition (%)	DME X_1	DME Dielectric Constant	DGM X_1	DGM Dielectric Constant
0	0.000	65.00[a]	0.000	65.00[a]
15	0.125	56.33	0.095	56.40
25	0.214	50.55	0.167	50.66
50	0.448	36.10	0.374	36.33
75	0.710	21.65	0.643	21.99
85	0.822	15.87	0.772	16.25
100	1.000	7.20[b]	1.000	7.65[c]

a: Payne & Theodorou, 1972; b: Carvajal et al., 1965; c: Tang & Zhao, 2014

3.2. Conductivity

Conductivity studies on solutions reflect the contributions from different factors namely: the level of ionic concentration, measure of dielectric constant and viscosity behavior of electrolyte systems (Ibezim-Ezeani et al., 2017 b). It is clear from tabulated results that the conductivity of dilute $Mg(BH_4)_2$ electrolyte increased with increase in DME (Tables 3 and 4) and DGM (Tables 5 and 6) for constitution range of 0 - 50 % vol. at the experimental temperatures. This is credited to lower viscosity of the binary mixture which enhanced ion mobility. Above the 50 % vol. composition, a progressive drop in conductivity was recorded, which may be attributed to the effect of decreased dielectric constant of the binary mixture. Conductivity values were higher for DGM-PC mixture in comparison to those for DME-PC mixture; this is likely caused by improved solvation of Mg^{2+} in the mixture, due to stronger coordination with the tridentate O_2 of DGM. It was observed that conductivity values for both DGM-PC and DME-PC mixtures increased with salt concentration (Tables 3 to 6). The composition range of 0 - 50 % vol. for DME and DGM afforded favorable dielectric conditions (Table 7), permitting marginal rise in the degree of ionization with concentration until the composition range of 75 - 100 % vol. for DME and DGM;

where electrostatic forces become appreciable and conductivity values begin to decline. In low salt concentration of electrolytes, Mg^{2+} is synchronized with O_2 of ether and develop large solvation shell distinct from anions, resulting in their relatively lower mobility; while in high concentration electrolytes characterized by small solvation shell, the mobility of Mg^{2+} is higher (Suo et al., 2013).

From Table 7, it was obvious that the dielectric constant of the mixtures decreased with increase in DME and DGM components. Increasing DGM or DME proportion in the binary system reduces the dielectric constant of the mixture and facilitates the creation of ion-pairs in the solution phase. This means that in solvents of low dielectric constants, having diminutive ionizing influence on electrolytes, the electrostatic forces between oppositely charged ions would be substantial and conductance will have diminutive value (Bhattarai et al., 2011). However, solvents of high dielectric constants afford better conducting solutions. With reference to Tables 3 to 6, conductivity increased with temperature by dint of the combination of decreased viscosity of the solution, increased speed of the ions and increased dissociation levels. The dielectric constant is one of several properties that affect ion-pair formation, and decrease in dielectric constant forces conductivity to drop. This effect is more pronounced above the constitution range of 50 % vol. for DME and DGM.

4. Conclusions

The density, viscosity and conductivity relationship between 0 - 100 % vol. of DME-PC (or DGM-PC) and concentrations (0.01, 0.05, 0.10 and 0.50 M) of $Mg(BH_4)_2$ have been investigated at temperatures of 298.15, 308.15, 318.15, 328.15 and 338.15K.

The viscosity data at different mole ratios of DME-PC (or DGM-PC) and concentrations of $Mg(BH_4)_2$ were correlated satisfactorily using Jones-Dole equation. The A coefficient values increased as the % composition of DME (or DGM) increased; while the B coefficient values were more positive than the corresponding values of A coefficient for the studied systems. Hence, the superior strength of ion-dipole interaction over dipole-dipole interaction facilitated stability of solvated $Mg(BH_4)_2$ coordination complexes in solution. Thus, a stable $Mg(BH_4)_2$ coordination structure may be unproblematic to form in the course of Mg stripping, which will benefit the stripping process and enhance the coulombic efficiency. Generally, there was systematic increase in conductivity values of DME (or DGM) with increase in temperature (from 298.15 to 338.15 K) and $Mg(BH_4)_2$ concentrations (0.01 to 0.50 M) in each system. However, the conductivity values of DGM-PC were higher than those of DME-PC in all cases; and increased from 0 - 50 % composition, followed by decreased conductivity values from 75 - 100 % vol. (DME or DGM). The results from this study show that the prime ratio for optimal battery functionality and reversibility is 50 % vol. of DME-PC (or DGM-PC) components.

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