Paving the Way towards Highly Stable and Practical Electrolytes for Rechargeable Magnesium Batteries

Oscar Tutusaus[a, b] and Rana Mohtadi*[a]

1. Introduction

Over the past two decades, rechargeable batteries have played an instrumental role in driving the evolution of new products and technologies for portable, automotive, and grid-stationary applications. Nowadays, in a state-of-the-art rechargeable Li-ion battery, 240 Wh kg\(^{-1}\) (640 Wh l\(^{-1}\)) can be provided for thousands of cycles.\([1]\) Yet, there is an increasing gap between the energy demand and the amount of energy that current rechargeable battery technologies can offer.

The energy density of a battery is determined by the anode and cathode materials, specifically by their capacity (total number of electrons stored in the active material) and their voltage difference. Therefore, owing to a high volumetric capacity, that is, 3832 mAh cm\(^{-3}\), Mg metal has recently attracted increased attention as a battery anode candidate.\([2]\) Despite the fact that Mg metal is a priori less competitive than lithium metal, on both specific capacity (2205 mAh g\(^{-1}\) vs. 3862 mAh g\(^{-1}\) for lithium) and redox potential levels, the electrochemical processes related to Mg reversible plating/striping have, so far, demonstrated the absence of dendrite formation,\([3]\) alleviating the safety concerns associated with lithium-metal anodes.\([4]\) Moreover, the use of Mg may offer an opportunity for battery cost reductions because of its abundance in the earth’s crust (it is the fifth most abundant element).\([2, 3a]\)

Despite the traits of Mg metal, the development of rechargeable Mg batteries has been faced with several technical challenges. These are manifested by the absence of practical high capacity, high-voltage cathodes, and appropriate electrolytes.\([2]\) As electrolytes are the medium that link the anode to the cathode, they should, at the very minimum, possess a sufficient electrochemical stability window against electroreduction and electrooxidation together with acceptable electrochemical transport. To become practical, other physical and chemical properties are also desirable (Figure 1). Indeed, the ultimate need to simultaneously satisfy a myriad of requirements makes the development of electrolytes a challenging endeavour.

Figure 1. Properties desired for Mg battery electrolytes.

A key challenge of electrolyte development for rechargeable Mg batteries stems from the instability of simple ionic salts and polar aprotic solvents in the proximity of Mg metal. For example, conventional Mg salts such as \([\text{ClO}_4]^{-}\), \([\text{BF}_4]^{-}\), and \([\text{TFSI}]^{-}\) \([\text{TFSI} = \text{bis(trifluoromethanesulfonyl)imide}]\), and polar aprotic solvents such as nitriles and carbonates were found to...
decompose on the metal surface.\textsuperscript{[5]} The resulting interfacial layer was found to be impermeable to Mg\textsuperscript{2+}, preventing battery function.\textsuperscript{[10]} This unfortunate phenomenon has excluded a myriad of salt/solvent combinations from being considered as Mg battery electrolytes.\textsuperscript{[2,3a,5]}

Over the past two decades, a number of systems with wide electrochemical windows and high conductivities have been developed. However, a common shortcoming of all of these electrolytes is their incompatibility with non-noble metals at operating voltages above around 2.5 V vs. Mg, representing a key hurdle towards their practical use in Mg batteries.\textsuperscript{[2,7]} In this Concept, we will summarize recent advances aimed at tackling the existing challenges. We will focus on those developed by our group, involving a new design platform based on salts containing the B–H motif that were shown to greatly alleviate the aforementioned issue.

2. Established Electrolyte Design Strategies

Over 80 years ago, it was discovered that ethereal solutions of Grignard reagents were capable of supporting the plating of highly pure Mg metal.\textsuperscript{[8]} However, the relatively low oxidation stability of most Grignard reagents (about 1.5 V vs. Mg; 2. Established Electrolyte Design Strategies

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<table>
<thead>
<tr>
<th>Electrolyte</th>
<th>Anodic stability on Pt (V vs. Mg)</th>
<th>Corrosivity threshold on 316 stainless steel (V vs. Mg)</th>
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</thead>
<tbody>
<tr>
<td>nBuMgCl</td>
<td>1.3\textsuperscript{(11)} 1.4\textsuperscript{(11)}</td>
<td>N/A</td>
</tr>
<tr>
<td>HMD5MgCl\textsuperscript{(2)}</td>
<td>2.1\textsuperscript{(1a)} (1.0 m in THF)</td>
<td>N/A</td>
</tr>
<tr>
<td>Mg\textsubscript{1} Bu\textsubscript{1} Ph\textsubscript{2}</td>
<td>2.0\textsuperscript{(1a)} 1.6\textsuperscript{(1a)}</td>
<td>N/A</td>
</tr>
<tr>
<td>(4-F-Ph)MgBr</td>
<td>2.4\textsuperscript{(1c)} (1 m in THF)</td>
<td>N/A</td>
</tr>
<tr>
<td>(HMD5)\textsubscript{1} Mg\textsubscript{2} AI\textsubscript{2}Cl\textsubscript{5} \textsuperscript{(4)}</td>
<td>3.3\textsuperscript{(1b)} (0.25 m in THF) 3.5\textsuperscript{(1b)} (0.35 m in diglyme) 2.6\textsuperscript{(1b)} (0.35 m in diglyme) 1.6\textsuperscript{(1b)} (0.25 m in THF) N/A</td>
<td></td>
</tr>
<tr>
<td>MgCl\textsubscript{2}/AI\textsubscript{2}Cl\textsubscript{3}</td>
<td>3.3\textsuperscript{(1b)} (0.4 m in THF)</td>
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</tr>
<tr>
<td>Mg\textsubscript{2} Cl\textsubscript{2} \textsubscript{(THF)}\textsubscript{2}</td>
<td>3.3\textsuperscript{(1c)} (0.2 m in THF)</td>
<td>N/A</td>
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<tr>
<td>MgCl\textsubscript{2}/AI\textsubscript{2}Cl\textsubscript{3}</td>
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<td>1.8\textsuperscript{(1c)}</td>
</tr>
<tr>
<td>MgCl\textsubscript{2}/2AI\textsubscript{2}EtCl\textsubscript{2}</td>
<td>2.3\textsuperscript{(1h)} (0.67 m in THF) 3.5\textsuperscript{(10c)} (0.5 m in THF) 2.2\textsuperscript{(1c)} (0.5 m in THF) N/A</td>
<td></td>
</tr>
<tr>
<td>Mes\textsubscript{2}/(PhMgCl\textsubscript{2})\textsubscript{2}</td>
<td>2.9\textsuperscript{(1d)} (0.5 m in THF)</td>
<td>N/A</td>
</tr>
<tr>
<td>(FMPMC)\textsubscript{2}/AI\textsubscript{2}Cl\textsubscript{3}</td>
<td>2.9\textsuperscript{(1d)} (0.5 m in THF)</td>
<td>N/A</td>
</tr>
</tbody>
</table>

placed by amides,[14,11] and later by alkoxides,[12] aryloxides, or chlorides, often yielding compounds compatible with Mg metal.[13] These could also be hybridized with the first approach (addition of a strong Lewis acid) to further increase their stability against electrochemical oxidation.[12,13]

At the beginning of this century, an important milestone was reported by Aurbach et al. who demonstrated the first, and only, example of a rechargeable Mg battery, enabled largely by the organo(halo)aluminate electrolytes they developed. The prototype provided 65 Wh kg⁻¹ energy density by coupling a Mg metal anode with a Chevrel-phase Mo₆S₈ cathode, and the voltage requirement (2.0 V vs. Mg) was fulfilled with the electrolyte Mg(AlCl₂BuEt)/tetrahydrofuran (THF).[10c] As a further increase in the energy density is desired, creating higher voltage cathodes became of interest and, with that, so did electrolytes with improved electrochemical oxidative stability. Subsequent advancements following the two aforementioned strategies resulted in state-of-the-art electrolytes possessing enhanced electrochemical properties, which was evident from a highly reversible performance, high bulk conductivity, and widened electrochemical windows.[13] Despite these scientific feats, these electrolytes were found to have several drawbacks that need to be overcome and are currently being addressed.

One of these relates to the use of volatile solvents, such as THF, where an optimal performance has been demonstrated in earlier reports.[10a] This solvent was used to study other electrolytes that were developed later; however, owing to its high volatility, less volatile/flammable ethers such as glymes are more desired for liquid electrolytes.[2,16] A recent advancement in this area was enabled by using non-Grignard-type haloalumimates based on amides such as (HMDS)₂Mg/2AlCl₃ and inorganic salts such as MgCl₂/2AlCl₃. These electrolytes were shown to perform well in glymes, which offer a possible path to overcome this challenge.

Another important issue was related to the incompatibility with certain cathodes, such as sulfur. This is particularly prominent in systems containing nucleophilic anions or free Lewis acids.[20] To tackle this issue, less nucleophilic organic amides such as those based on hexamethyldisilazide,[13b,2] or inorganic salts based on MgCl₂[13d,4] were proposed as potential solutions.

However, one standing and common limitation of this family of electrolytes is their recently discovered corrosive nature.[15] Corrosion of non-noble metal electrodes is manifested by parasitic anodic current caused by metal dissolution. As a consequence, when anodic stability was tested on non-noble metal electrodes, a current was observed at potentials below that measured on noble metal and inert electrodes, representative of electrolyte decomposition (Table 1).[16] Therefore, it is critical that the oxidative stability of electrolytes is evaluated on a variety of non-noble metals. As the corrosion of current collectors has been linked to chloride ions,[17] chloride-free electrolytes are deemed necessary. However, this not a trivial task, given that the design of state-of-the-art electrolytes that are compatible with Mg metal hinges on the two working principles previously described, typically involving either organomagnesium halide reagents or halogenated Lewis acids such as AlCl₃. Although one obvious way to mitigate the corrosive behaviour would be to use chloride-free reagents, we chose to follow an approach granted to eliminate the problem at its core by discovering new Mg compatible systems that are halide free. Our quest was to create a new design platform that could be used as basis for electrolyte design, as depicted in Figure 2.


Going beyond the restraints imposed by halogen-based electrolytes required us to set forth in a new direction. It became necessary to explore a bottom-up approach based on new Mg-compatible systems. This would serve as an alternative platform towards the design of novel electrolytes. Given that Mg metal passivation in inorganic electrolytes was attributed to the insufficient reductive stability of the anion,[20] we hypothesized that Mg salts of reducing anions should display compatibility with Mg metal. We were interested in reducing agents commonly used in chemical synthesis, which include complex metal hydrides, among which are borohydride salts.[19] Our density functional calculations supported the possibility that [BH₄]⁻ was of higher reductive stability than [BF₄]⁻, which is known to passivate Mg metal.[20] To obtain a proof of concept, the salt was initially studied in THF, the solvent of choice for all other available Mg electrolytes reported at that time. Satisfyingly, cycling voltammetry experiments showed reversible Mg deposition/stripping with about 40% coulombic efficiency (Figure 3a), whereas galvanostatic experiments confirmed the composition of the deposited material as Mg metal.[20] This accomplishment constituted a breakthrough, as it represented the first inorganic salt that is compatible with Mg metal.

Figure 2. Design strategies for the development of electrolytes for rechargeable Mg battery. The ovals with question marks symbolize alternate design paths that do not rely on halo and organo reagents as a basis for further development.
Our first goal was to improve the electrochemical performance of Mg(BH$_4$)$_2$, focusing on increasing the coulombic efficiency and current density while lowering the deposition overpotential. In the state-of-the-art organomagnesium and related electrolytes, that is typically achieved by addition of aluminium- or boron-based Lewis acids; however, as the borohydride structure is not amenable to such methodologies, the development of alternate strategies is deemed necessary. We investigated the nature of species in the electrolyte to get information on the cation coordination sphere. Spectroscopic features of Mg(BH$_4$)$_2$/THF solutions resembled those of other analogous compositions. For example, 3:1 NaBH$_4$/Mg(BH$_4$)$_2$ in DME displayed a lower coulombic efficiency and current density, whereas at higher denticity, coupled with high coulombic efficiencies in glymes, such as diglyme, had an adverse effect on the coulombic efficiency, and Mg oxidation peak current density.

Effect of LiBH$_4$ addition to 0.18 M Mg(BH$_4$)$_2$/DME on 

- the coulombic efficiency, and Mg oxidation peak current density (Figure 3a).
- the cyclic voltammograms in THF and in DME. Adapted from Ref. [21] with permission. Copyright 2012, Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim.

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The presence of the LiBH$_4$ additive also displayed other effects, such as an increased solubility of Mg(BH$_4$)$_2$ in glymes with higher denticity, coupled with high coulombic efficiencies in these solvents. In a later study, Shao et al. also applied our strategy to further optimize the Mg(BH$_4$)$_2$/LiBH$_4$ electrolyte composition. Notably, using diglyme as solvent and increasing LiBH$_4$ content provided a coulombic efficiency for Mg deposition/stripping close to 100%, and these studies further supported the non-trivial role of LiBH$_4$ as an additive; for instance, different coulombic efficiencies were obtained from DME and diglyme electrolyte solutions, despite their similar ionic conductivity. Even more intriguing is the role of the cation. Whereas we observed enhancement by using other borohydrides such as NaBH$_4$, it was not as pronounced as those found with LiBH$_4$ for analogues compositions. For example, 3:1 NaBH$_4$/Mg(BH$_4$)$_2$ in DME displayed a lower coulombic efficiency and current density by 5 and 60%, respectively (Figure S2). Further optimizations and understanding of the borohydride electrolytes are currently under investigation in our labs.

Following the demonstration of a highly reversible electrolyte, we examined the electrolyte stability against the electrochemical performance, owing to reduced solubility of Mg(BH$_4$)$_2$ (<0.05 M), establishing DME as the optimum solvent for this system.

Effect of LiBH$_4$ addition to 0.18 M Mg(BH$_4$)$_2$/DME on 

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chemical oxidation on different materials. Surprisingly, the stability on platinum (1.7 V vs. Mg) was lower than those on stainless steel and glassy carbon (2.2 and 2.3 V vs. Mg, respectively), which is likely caused by the catalytic effects of Pt metal on BH₄⁻ decomposition through strong dissociative adsorption; this indicates the importance of BH₄⁻ – electrode surface interactions.[25] Notably, as the borohydride electrolytes are not corrosive, the stability on steel is similar to that observed for other electrolytes that are twice as stable on Pt. Most importantly, borohydride electrolytes remain the only relatively ionic and halide free salts that are highly compatible with Mg metal.

4. Aromatic Borohydrides: The Next Step to Stability

Although some of the electrochemical properties of Mg(BH₄)₂ electrolyte solutions can be adjusted by the judicious choice of solvent and additives, improving its oxidative stability requires more profound modifications. Mg(BH₄)₂ has been extensively studied as a potential candidate for hydrogen storage, because of its high theoretical H₂ content.[26] Several studies have reported the undesired formation of higher boranes during thermal decomposition of Mg(BH₄)₂, which are detrimental for reversible H₂ storage.[27] Among them, magnesium dodecaborate (MgB₁₂H₁₂) has been identified as the major by-product.[28] The [B₁₂H₁₂]²⁻ dianion is the most stable representative of the closo-borane family, a group of higher borane clusters with stoichiometry [BₓHₓ]²⁻ (n = 6-12) and closed deltahedral structures (polyhedral with triangular faces).[29] Such compounds display an unusual stability relative to neutral binary boron hydrides with open structures originally discovered by Stock.[29] The source of their stability arises from the tangentially delocalized sigma bonding present in the boron framework and are, therefore, regarded as three-dimensional aromatic species.[30] Indeed, the two negative charges in [BₓHₓ]²⁻ are required to fulfill aromatic electron count and are, thus, delocalized over the cluster. As a consequence, the oxidation potential for the [BₓHₓ]²⁻ dianion has been reported to occur above 4 V (vs. Mg), whereas no accounts of its electrochemical reduction have been found.[31] In a broad sense, [BₓHₓ]²⁻ can be regarded as the aromatic analogue of [BH₄]⁻, the same as benzene is to methane.

In a first approach towards exploring boron clusters as Mg electrolytes, MgB₁₂H₁₂ was targeted.[20] Even though the compound was successfully synthesized, its insolubility in ethers hampered electrochemical studies. It is likely that the high charge associated with both the anion and the cation is the main reason behind the low solubility. Isostuctural and isoelectronic families of closo-carboranes exist, in which one or more [BH₄]⁻ units are formally replaced by isoelectronic [CH]⁻ units, providing a route to reduce the total cluster charge. Among them, neutral m-carborane (1,7-C₂B₁₀H₁₂) has previously been reported to possess comparable electrochemical stability to that of [BₓHₓ]²⁻ anion and was chosen as the entry point to soluble Mg salts containing boron clusters.[31] For a proof of concept, we selected m-carbonyl magnesium chloride to enable high solubility (>1 M), aided by the Mg–C covalent bond character.[20] A relatively high bulk conductivity of 0.6 mS cm⁻¹ (at 0.1 g mL⁻¹) was measured, indicative of salt dissociation. In addition, in contrast with other electrolytes based on the [Mg₂Cl₃]⁻ cation, recrystallized m-carbonyl magnesium chloride salt (1) displayed a complex anion containing a Mg atom (Figure 4a). Solutions of Mg(1,7-C₂B₁₀H₁₂)Cl exhibit a reversible redox couple (coulombic efficiency >98%) with an onset potential and peak shape consistent with Mg deposition and stripping (Figure 4b). Note that 1 was confirmed to be one of the electroactive species, as a similar reversible performance as the bulk was found.[20] Additionally, a high anodic stability of 3.2 V (vs. Mg) was observed, remarkably higher than the 2.4 V displayed by 4-F-PhMgBr/THF,[31] which was considered the best Grignard compound until that date. It is also comparable with electrolytes generated by addition of Lewis acids such as AlCl₃ or Br₂ to Grignard reagents, amidomagnesium compounds, or MgCl₂.[31]

Most importantly, the oxidation onset potential determined by linear-sweep voltammetry experiments on a variety of working electrodes was similar (3.2 V vs. Mg) for Pt, 316-SS, and Al (Figure S3).[20] In previous studies with other halide-containing electrolyte solutions, the oxidation onset was always...
highly dependent on the electrode material. Chronoamperometry experiments further supported the measured oxidative stability (Figure S3). Interestingly, this result constitutes the highest reported stability on a steel electrode to date, suggesting that the closo-borane scaffold does indeed offer unique stability when incorporated into a Mg-battery electrolyte material. This electrolyte also supported Mg-ion insertion into a host cathode material such as Chevrel-phase Mo₆S₈, which allowed for the demonstration of a battery. The battery was cycled and a stable reversible specific capacity was achieved (ca. 90 mAh g⁻¹ at 0.05 C; Figure S4).²⁰

Despite obtaining increased compatibility toward non-noble metals by using a closo-borane electrolyte, further improvements are still needed. That is, although the corrosivity has been lowered, it has not been eliminated; furthermore, as discussed before, excellent electrochemical performance needs to be achieved in a solvent other than THF. However, the results provided a proof of concept that boron clusters offer an opportunity for effectively tackling the existing challenges with current electrolytes. Further steps should be aimed at designing chloride-free boron cluster electrolytes capable of supporting excellent Mg plating/deposition performance in THF free solvents and are compatible with non-noble metals.

5. Conclusions and Outlook

Rechargeable batteries are one of the most effective means of storing energy, and rechargeable Mg batteries hold promise for increasing the battery energy density (i.e. versus Li-ion batteries). However, this technology is currently in its infancy and facing substantial hurdles caused by the absence of practical electrolytes and cathodes. In particular, the development of electrolyte systems with wide electrochemical windows and high compatibility with battery components is crucial to discovering high-voltage cathodes.

Upon examining the progress made in the developments of Mg battery electrolytes over the past two decades, we felt that the design toolbox has been used exhaustively and may have been lowered, it has not been eliminated; furthermore, as discussed before, excellent electrochemical performance needs to be achieved in a solvent other than THF. However, the results provided a proof of concept that boron clusters offer an opportunity for effectively tackling the existing challenges with current electrolytes. Further steps should be aimed at designing chloride-free boron cluster electrolytes capable of supporting excellent Mg plating/deposition performance in THF free solvents and are compatible with non-noble metals.

lowest corrosivity towards non-noble metals to date. Nonetheless, this is just the beginning of the journey, and making the boron cluster electrolytes soluble in the absence of chloride and demonstrating high performance in a non-THF solvent is the next big challenge. Certainly, anions based on the B–H motif offer a new landscape in the universe of rechargeable Mg battery electrolytes and pave the way for designing practical rechargeable Mg batteries.

Experimental Section

Experimental details are shown in the Supporting Information.

Acknowledgements

R.M. and O.T. would like to thank Dr. T.S. Arthur and Dr. Koji Suto for their insightful suggestions. Special thanks for Prof. Nathaniel Szymczak for his insightful suggestions. Funding for O.T. was provided from the Chemistry Department at the University of Michigan. Funding for meeting the cost of this article was provided by Toyota Motor Engineering and Manufacturing North America TEMA.

Keywords: batteries · boron · electrolyte · energy storage · magnesium


See table 1.

Received: June 27, 2014
Revised: August 22, 2014
Published online on October 1, 2014