

Synthetic Production of Tunable Magnesium Oxide for Thermal Batteries: Drag and Drop

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Abstract

Magnesium oxide (MgO) is a critical binder material in the manufacture of thermal batteries, owing to its stability and ability to immobilize molten electrolyte. Established supply chains can cease production and new sources must be evaluated at great expense in terms of time and revenue. To ensure the supply of this critical material, Qynergy has developed an MgO binder material ("GoMax") for electrolytes. The new MgO binder material is scientifically engineered in such a way that it can be produced from multiple precursors, thus mitigating supply chain risk. The purpose of this work is to demonstrate the benefit of such production from synthetic precursors, as well as the ability to tune the morphological properties to 'drag and drop' the as-synthesized MgO into current separator design without disrupting pellet production or battery performance. In the current work, two forms of the Qynergy GoMax MgO are characterized with electrolyte salt mixtures made in-house by EnerSys Advanced Systems Inc. (EAS) to demonstrate the tunability of the synthesized materials and common morphologies with the currently used material. Tests include morphological characterization of GoMax and Electrolyte/Binder (EB) mix using different iterations of GoMax. EAS and Qynergy have demonstrated common physical characteristics of GoMax and current binder used in our thermal batteries.

Keywords

Thermal batteries; magnesium oxide; binder; separator; particle size distribution; morphology; single cell.

Introduction

Thermal batteries are primary reserve power sources. They are single use, dormant at room temperature and activated on demand. They are primarily used in military applications owing to several attractive characteristics including >20-year shelf life, high rate capability, wide operating temperature range, and little/no self-discharge. The technology has been well-studied and characterized over time owing to such defense applications [1, 2]. The batteries are comprised of primary components: the electrochemical cell which includes the anode, cathode, and EB mix, also known as the separator, and the heat source which ignites upon activation, causing the electrolyte in the separator to heat to a molten state, thus activating the battery through ionic transfer. Figure 1 outlines the structure and sequence of activation of a thermal battery.

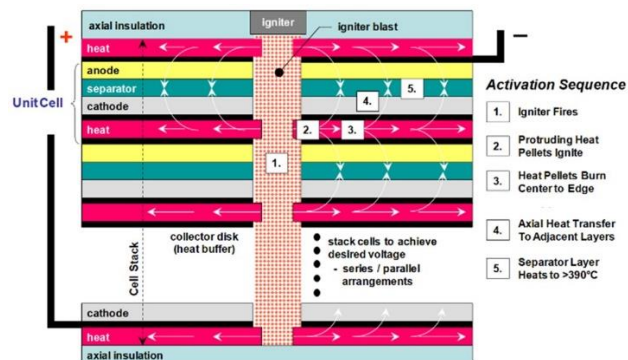


Figure 1: Thermal battery schematic with activation and operation sequence.

MgO is a vital material in thermal battery separators; it is thermally stable, inert, and electrically non-conducting material [3]. It provides physical separation between the anode and the cathode to prevent cell shorting, as well as binding molten electrolyte to prevent leakage. The morphology of MgO is also critical to its performance. Numerous grades are commercially available in large quantities, but not all will function effectively in thermal battery separators. Important properties include purity, powder flow, deformation, and binding characteristics. Current commercial manufacture is not directed towards thermal battery applications and quality control in this environment is not a priority for suppliers. Previous supply chains for qualified MgO have ceased production and new sources had to be evaluated at great expense in terms of time and cost. Thermal battery manufacturers have little control over the quality and future supply of purchased MgO.

MgO synthesis from widely available precursors would allow for vertical integration and strict quality control of this vital material. The goal at Qynergy has been to synthesize MgO materials from widely available precursors to retain or improve powder flow, pellet strength, and binding capability. The MgO synthesis process permits highly tunable material that can be tailored towards a specific electrolyte. In this work, EAS has partnered with Qynergy to evaluate such MgO with electrolyte currently used in commercial thermal batteries developed and manufactured by EAS. The physical characteristics of two iterations of GoMax MgO and EAS electrolyte have been evaluated to establish whether it is possible to 'drag and drop' one of these iterations into EAS's current separator powder. EAS matches characteristics of currently used MgO with the new

GoMax MgO in an attempt to retain the physical and chemical properties of separator in terms of powder flow, particle size distribution, morphology, and electrochemical performance.

Methods and Materials

The importance of evaluating material properties for the MgO separator component has been outlined. Subsequently, several methods of physical and chemical evaluation are employed in this work to evaluate GoMax in EAS's EB mix.

Two iterations of GoMax MgO were evaluated in this study, hereafter referred to as GoMax A and GoMax B. Upon receipt, both materials were tested for moisture using the Karl Fisher Titration method. As received, GoMax A and GoMax B were also evaluated for particle size distribution (www. Microtrac.com).

To further evaluate GoMax A and GoMax B the material was combined with EAS electrolyte to form two EB mixes, hereafter referred to as Separator A (using GoMax A) and Separator B (using GoMax B). The methods of this material synthesis as well as the nature of the EAS electrolyte are proprietary. Upon synthesis, Separator A and Separator B were evaluated for physical and chemical properties. The morphology of the EB mixes was examined using Morphologi G3 (www. Malvern.com) and compared with current EAS separator. Tap density of both EB mixes was also evaluated. Chemical characteristics of Separator A and Separator B were explored using Differential Scanning Calorimetry (DSC). Finally, electrochemical cell analysis was performed on a single cell setup for Separators A and B.

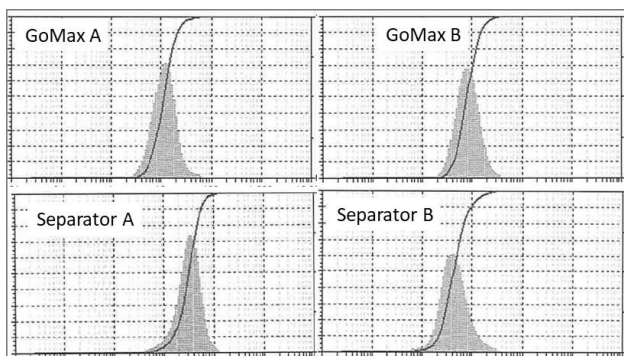


Figure 2: Particle size distribution for GoMax A, GoMax B, Separator A, and Separator B.

Results

In this work, two forms of GoMax MgO are evaluated. MgO was inspected upon arrival for water content and conformed to requirements. Particle size distribution was examined on GoMax MgO alone as well as the resulting EB mix. Figure 2 outlines the particle size distribution for four materials: GoMax A, GoMax B, Separator A, and Separator B. The particle size distribution is relatively narrow, which is a good indication of favorable powder flow.

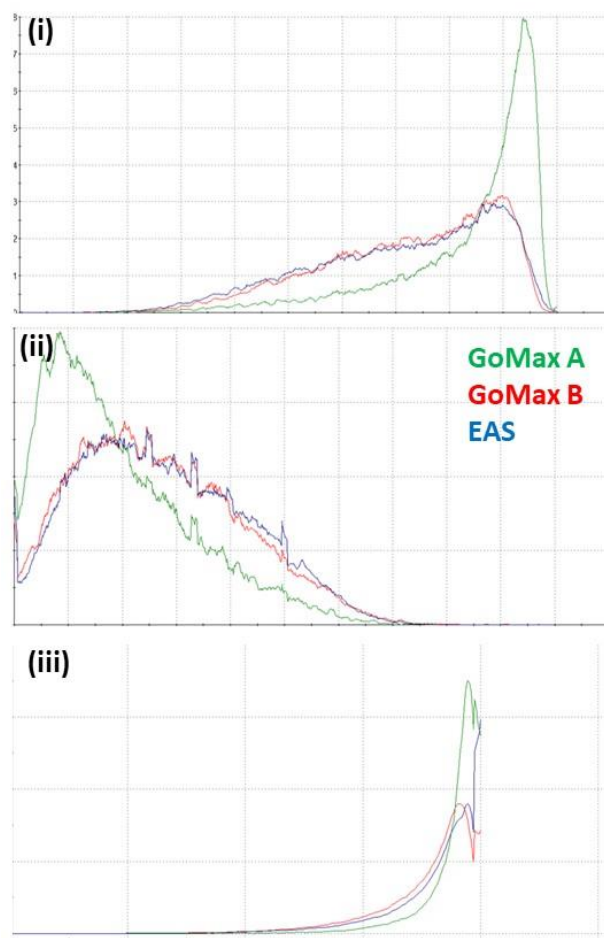


Figure 3: Morphology data for Circularity (i), Elongation (ii), and Convexity (iii) of GoMax A (green), GoMax B (red), and current EAS MgO (blue).

The morphology of the EB mix is also a good indication of pellet manufacturability, as well as performance of the material during battery actuation. Figure 3 outlines a comparison of GoMax A, GoMax B, and EAS MgO for circularity, elongation, and convexity. It is particularly interesting to note that the particle size and shape characteristics of GoMax B agree well with EAS MgO, which may be used to predict ease of manufacture with the new MgO material as well as operational behavior. Separator A and Separator B were also examined with Morphologi G3. Figure 4 outlines a comparison of circularity, elongation, and convexity for each material. It is noteworthy that for the fused separator material, EAS separator is in better agreement with Separator A (utilizing GoMax A), which demonstrates that process reactions play a large part in separator design.

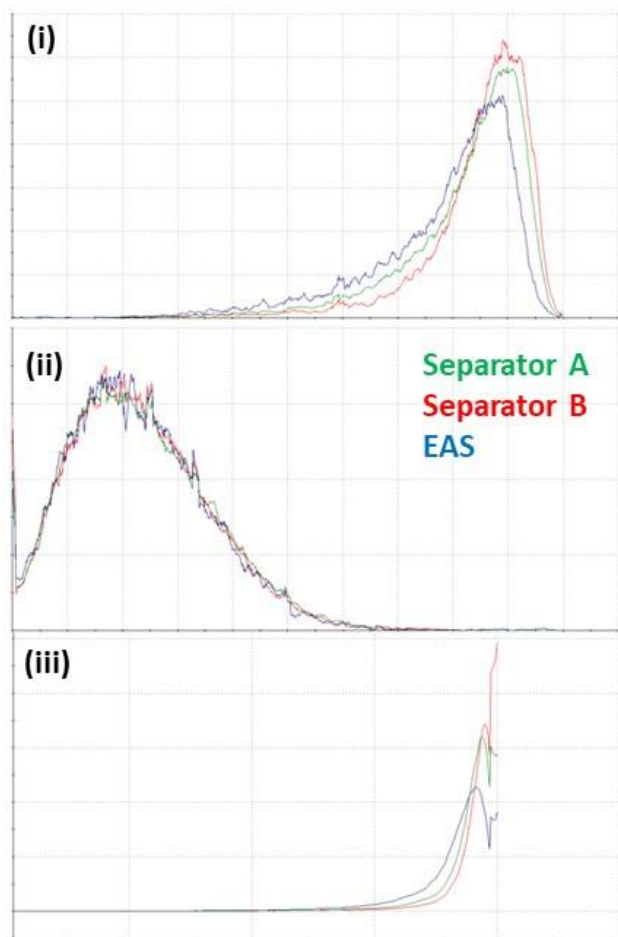


Figure 4: Morphology data for Circularity (i), Elongation (ii), and Convexity (iii) of Separator A (green), Separator B (red), and current EAS EB (blue).

The Hausner ratio of powders is a good indicator of powder flow in manufacturing. Table 1 outlines the Hausner ratio for Separator A and Separator B.

Material	Hausner Ratio
Separator A	1.44
Separator B	1.40

Table 1: Hausner Ratio for Separator A and Separator B.

While the values deviate somewhat from the 'ideal 1.1' value, it should be noted that for pellet manufacture, it is important to find a compromise between powder flow and ease of pellet pressing, as the two can sometimes be inversely proportional. Therefore, while a powder may flow well according to this ratio, it may be difficult to form a rugged pellet with the same material.

The chemical properties of Separator A and Separator B were evaluated with DSC, which outlines melting point and heat of fusion. These parameters allow us to inspect the performance of the material at temperature to ensure that the separator functions in a manner that is cohesive with the

temperature of operation. It is noteworthy that both mixes display a very clean profile which is an indication of the purity of the GoMax MgO for both iterations examined (A and B). This allows for more predictable behavior and greater quality control of separator materials.

Upon completion of Separator A and Separator B powder evaluation, each material was pressed into pellets and formed into a cell with anode and cathode pellets to probe behavior under heat and pressure at open circuit voltage (OCV). A predefined operating temperature and pressure were applied to a single cell each of Separator A and Separator B and the resulting voltage output at OCV was observed. Figure 5 outlines the curves for each cell at OCV over several minutes. The consistency of the output voltage upon melting of the electrolyte is clear and demonstrates the viability of GoMax MgO as a binder material in thermal batteries.

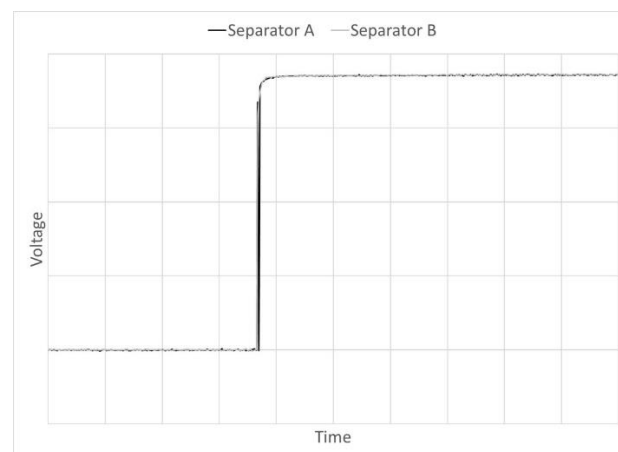


Figure 5: Single cell tests on Separator A and B at open circuit voltage.

Next steps in this study must include examination of deformation and leakage of these pellets. This will help to evaluate the behavior of the new EB mix material in an operating thermal battery.

Conclusions

Synthesis of MgO from widely available precursors is very attractive in thermal battery manufacturing with respect to quality control and vertical integration. In this work EAS demonstrated initial testing of Qynergy GoMax MgO in EAS EB mix. Physical and morphological studies demonstrated that GoMax can be tailored to fit a particular electrolyte mix owing to the tunability of the synthesis process. GoMax EB mixes exhibited comparable electrochemical behavior to currently used materials which may allow us to 'drag and drop' this material into existing separator pellets without the necessity of modification of the manufacturing process. Further testing and examination will provide an enhanced understanding of this material and its viability in thermal batteries.

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