Dehydrogenation Performances of Different Al Source Composite Systems of 2LiBH$_4$ + M (M = Al, LiAlH$_4$, Li$_3$AlH$_6$)

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Hydrogen has become a promising energy source due to its efficient and renewable properties. Although promising, hydrogen energy has not been in widespread use due to the lack of high-performance materials for hydrogen storage. Previous studies have shown that the addition of Al-based compounds to LiBH$_4$ can create composites that have good properties for hydrogen storage. In this work, the dehydrogenation performances of different composite systems of 2LiBH$_4$ + M (M = Al, LiAlH$_4$, Li$_3$AlH$_6$) were investigated. The results show that, under a ball to powder ratio of 25:1 and a rotation speed of 300 rpm, the optimum ball milling time is 50 h for synthesizing Li$_3$AlH$_6$ from LiH and LiAlH$_4$. The three studied systems destabilized LiBH$_4$ at relatively low temperatures, and the 2LiBH$_4$-Li$_3$AlH$_6$ composite demonstrated excellent behavior.

Based on the differential scanning calorimetry results, pure LiBH$_4$ released hydrogen at 469°C. The dehydrogenation temperature of LiBH$_4$ is 416°C for 2LiBH$_4$-Li$_3$AlH$_6$ versus 435°C for 2LiBH$_4$-LiAlH$_4$ and 445°C for 2LiBH$_4$-Al. The 2LiBH$_4$-Li$_3$AlH$_6$, 2LiBH$_4$-LiAlH$_4$, and 2LiBH$_4$-Al samples released 9.1, 8, and 5.7 wt.% of H$_2$, respectively. Additionally, the 2LiBH$_4$-Li$_3$AlH$_6$ composite released the 9.1 wt.% H$_2$ within 150 min. An increase in the kinetics was achieved. From the results, it was concluded that 2LiBH$_4$-Li$_3$AlH$_6$ exhibits the best dehydrogenation performance. Therefore, the 2LiBH$_4$-Li$_3$AlH$_6$ composite is considered a promising hydrogen storage material.

Keywords: hydrogen storage materials, LiBH$_4$, composite system, 2LiBH$_4$-Li$_3$AlH$_6$, dehydrogenation performance

INTRODUCTION

Hydrogen energy has become an ideal new energy resource due to its clean, efficient, and renewable properties (Schlapbach and Zuttel, 2001). Although the use of hydrogen energy is promising, widespread use has been hindered by issues in the advancement of high-performance materials for hydrogen storage (Liu et al., 2010). The complex hydride (Ley et al., 2014) LiBH$_4$, which has...
a hydrogen capacity storage of 18.5 wt%, has drawn extensive attention. LiBH₄ decomposes into an intermediate compound Li₃B₂H₁₂, which generates B and releases H₂ as described in Equation (1) (Orimo et al., 2006).

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\text{LiBH}_4 \rightarrow \frac{1}{12}\text{Li}_3\text{B}_2\text{H}_{12} + \frac{5}{6}\text{LiH} + \frac{13}{12}\text{H}_2 \rightarrow \text{LiH} + \text{B} + 3/2\text{H}_2
\]  

However, many factors impede the commercial application of pure LiBH₄, such as high dehydrogenation temperature, slow dehydrogenation rate, and poor cycle reversibility (Lodziana and Vegge, 2004). To solve these issues, researchers have concentrated on modifying LiBH₄ (Vajo and Olson, 2007) through anion/cation substitution (Fang et al., 2011; Lombardo et al., 2019), catalytic modification (Kou et al., 2012; Huang et al., 2016; Zhai et al., 2016), and the application of the confinement effect of nano-materials (Vajo, 2011; Zhang et al., 2017).

Al and Al-based composites with LiBH₄ are of interest. Founded on first-principles calculation, Siegel et al. (2007) predicted that LiBH₄ would react with Al to generate AlB₂, LiH, and H₂ at 280°C under a hydrogen pressure of 1 bar. For the LiAlH₄-LiBH₄ system, Mao et al. (2009) found that the addition of TiF₃ decreased the onset temperatures of H₂ release by 64 and 150°C compared with the undoped system. The decomposition enthalpy values of LiBH₄ also reduced to 60.4 kJ/mol. He et al. (2019) studied the dehydrogenation performance of LiBH₄/LiAlH₄ composite, found that 8.7 wt.% of hydrogen was released at 500°C, and defined a “Li-Al-B-H” compound. Soru et al. (2014) focused on the phase structural transformation of the LiAlH₄-LiBH₄ system, which can produce 6.8 wt.% of hydrogen. According to a study by Carrillo-Bucio et al. (2017), a surface-oxidized 2LiBH₄ + Al composite did not release hydrogen until heated to 400°C under a 3 bar initial backpressure. With the catalytic effect of TiF₃, the mixture obtained 9.3 wt.% of hydrogen release as compared to 5.8 wt.% of the undoped mixture. In research by Zhang et al. (2018), a combined mixture of MgH₂, LiBH₄, and LiAlH₄ showed superior performance, starting to release hydrogen at 280°C and maintaining reversibility. In our previous studies (Li et al., 2012), the dehydrogenation temperatures of 2LiBH₄ + Li₃AlH₆ doped with titanium were decreased by 80 and 50°C, respectively, vs. the undoped system. From the previous studies, it can be summarized that Al can enhance the hydrogen storage performance of LiBH₄ to some extent. However, the dehydrogenation behaviors of different Al source composite systems have not been systematically studied. In this work, three Al-based LiBH₄ composite systems, 2LiBH₄-Al, 2LiBH₄-LiAlH₄, and 2LiBH₄-Li₃AlH₆ were prepared, and the hydrogen storage performance was investigated.

**MATERIALS AND METHODS**

Table 1 shows the raw materials used in this study. All powders were carefully stored in a glove box (MIKROUNA), in which noble gas was loaded and the oxygen content and water vapor content were kept below 1 ppm. A planetary mill (QM-3SP2) was utilized to prepare the composites.

The dehydrogenation properties of the materials were tested with a Sievert-type device. The changes in temperature and pressure over time were recorded. The ideal gas state equation was utilized to calculate the dehydrogenation capacity. Thermal analysis was performed using differential scanning calorimetry (DSC, Netzsch 449C Jupiter) combined with thermogravimetric analysis (TG, QMS 403C). To protect the samples from oxidation, high purity argon gas was added at a flow rate of 50 mL/min. An empty aluminum crucible was used as a reference during analysis. The phase compositions of the samples were determined by X-ray powder diffraction (X’Pert-PRO, PANalytical); its scanning range (2θ = 5°-80°). A specific sealed device was used to protect samples from being oxidized or damped during analysis. Fourier transform infrared spectroscopy (FTIR, Tensor 27) was employed to detect some amorphous substances with a scanning rate of 30 cm⁻¹/min and a resolution ratio of 0.5 cm⁻¹.

| TABLE 1 | Raw materials used in the study. |
|-----------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|
| Materials             | LiBH₄ | Al  | LiAlH₄ | LiH  |
| Purity (%)             | ≥95   | ≥99 | ≥95   | ≥98  |
| State                  | Powder         | Powder         | Powder         | Powder         |
| Supplier               | Acros          | Sinopharm      | Sigma-Aldrich  | Sigma-Aldrich  |

**RESULTS AND DISCUSSION**

**Preparation of Li₃AlH₆ Powder**

In this work, Li₃AlH₆ powder was synthesized in-situ by ball milling LiH and LiAlH₄. Approximately 15 g mixed powder of LiH and LiAlH₄ was milled each time with a molar ratio of 2:1, a ball to powder ratio of 25:1, and a milling speed of 300 rpm. To suppress temperature rise in the ball milling, every 30 min was set as an operation cycle, which contained a stop time of 6 min, periodically. After ball milling for 20 h, XRD patterns were obtained and examined to characterize the mixed powder. Figure 1 shows the XRD results of pure LiH powder, LiAlH₄ powder, and the LiH and LiAlH₄ powder after ball milling for 20 h. No impurity phases were observed in any of the samples, which indicates that the samples were highly pure and did not oxidize. As shown in Figure 1C, a few Li₃AlH₆ diffraction peaks are present, while the rest are of LiAlH₄ and LiH. This shows that the synthesis reaction was not completed. Due to long milling time, LiH peaks were much lower than before. The LiH content was lower, and an amorphous structure was formed.

Further ball milling was carried out since the synthesis reaction to Li₃AlH₆ was not completed. The milling samples were taken for XRD analysis after every 10 h. Figure 2 shows the XRD results of the milled powders after 20, 30, 40, and 50 h. Strong double peaks at 22° and 23° are detected, which are the characteristic peaks of Li₃AlH₆. After ball milling for 30 h, most of the diffraction peaks are Li₃AlH₆; however, some LiAlH₄ and
LiH peaks can still be found at 27°, 43°, and 46°. As the ball milling time increased, the LiH and LiAlH₄ content decreased, which shows that the synthesis reaction proceeds as the milling time increases. Nevertheless, the diffraction intensity of Li₃AlH₆ decreased, which shows that the longer milling time, the more likely the production of an amorphous phase becomes.

FTIR analysis was used to investigate the synthesized powder. In pure LiAlH₄ (Figure 3A), the bending modes around 1,780 and 1,610 cm⁻¹ correspond to the A-H stretching band, as was previously shown by Chen et al. (2001). After 50 h ball milling, this A-H stretching band disappeared in the 2LiBH₄-LiAlH₄ composite (Figure 3B). A new A-H stretching band was generated around 1,380 cm⁻¹, which belongs to Li₃AlH₆. This indicates the completion of the synthesis reaction.

Characterization of the Composite Samples
The 2LiBH₄ + M (M = Al, LiAlH₄, Li₃AlH₆) composite systems were prepared by ball milling, respectively. The ball-milling time was 1 h, with a mole ratio of 2:1. The as-milled mixtures are presented in Figure 4. In the 2LiBH₄-Al composite, the narrow and sharp diffraction peaks are of Al, while the other diffraction peaks are of LiBH₄. The intensity of the LiAlH₄ peaks in the 2LiBH₄-LiAlH₄ composite are also strong, but both LiBH₄ and Li₃AlH₆ have attenuated diffraction peaks in 2LiBH₄-Li₃AlH₆. The absence of other peaks indicated that there were no side reactions that could generate impurities or by-products during ball milling.
Dehydrogenation Performances

The dehydrogenation performances of the samples were investigated. The four samples were heated to 550 °C at a heating rate of 5 °C/min in an argon atmosphere. The DSC/TG results are shown in Figure 5. In the pure LiBH$_4$ sample (Figure 5A), the endothermic peak at 120 °C denotes that LiBH$_4$ transformed from an orthorhombic to a hexagonal crystal system. The endothermic peak at 286 °C is attributed to the melting of LiBH$_4$. According to the TG curve, LiBH$_4$ began to release hydrogen and lose weight at 400 °C. There is a dehydrogenation peak at around 469 °C, and the rate of dehydrogenation slowed down after that. Both the transformation and the melting peak of LiBH$_4$ for each composite sample are shown in Figures 5B–D. All samples have lower transformation and melting temperatures than pure LiBH$_4$ (transformation temperature: $T_d < T_c < T_b < T_a$; melting temperature: $T_c < T_d < T_b < T_a$). In the as-milled 2LiBH$_4$-Al sample (Figure 5B), the wide endothermic range around 450 °C represents the decomposition of LiBH$_4$. The last peak at 529 °C is the decomposition of LiH. As shown in Figure 5C, peaks at 190 and 198 °C indicated the dehydrogenation of LiAlH$_4$, which were verified in our previous study (Li et al., 2012). LiAlH$_4$ decomposed into Li$_3$AlH$_6$ at 190 °C, and Li$_3$AlH$_6$ started to generate LiH, Al, and release H$_2$ at 198 °C. LiBH$_4$ and LiH started to decompose at 435 and 472°C, respectively. Figure 5D shows that the decomposition peak of Li$_3$AlH$_6$ is at 198°C, which is consistent with the 2LiBH$_4$-LiAlH$_4$ sample. The decomposition peak of LiBH$_4$ (416°C) is lower than that of the 2LiBH$_4$-LiAlH$_4$ (435°C) or 2LiBH$_4$-Al samples (445°C). In addition, the decomposition peak of LiH, which is around 452°C, is also lower than that of the other composite systems. These results showed that the LiBH$_4$ became more unstable due to the addition of Li$_3$AlH$_6$ and that hydrogen was released at a lower temperature. The main reason for this is that the Al present in Li$_3$AlH$_6$ was active enough to stimulate the dehydrogenation reaction.

The temperature-programmed desorption (TPD) method was implemented to study the dehydrogenation performance of pure LiBH$_4$, 2LiBH$_4$-Al, 2LiBH$_4$-LiAlH$_4$, and 2LiBH$_4$-Li$_3$AlH$_6$; the results are shown in Figure 6. The samples were heated to 400°C and held for 5 h. The pure LiBH$_4$ began to release H$_2$ at around 400°C. Its desorption rate slowed down when 5 wt.% of H$_2$ had been released. The 2LiBH$_4$-Li$_3$AlH$_6$ sample released 9.1 wt.% H$_2$ within 150 min, which is the fastest reaction kinetics in this work. Its decomposition process includes two main steps, with the release of 3 and 6.1 wt.% H$_2$ as the first and second steps, respectively. In the 2LiBH$_4$-LiAlH$_4$ sample, LiAlH$_4$ began to decompose at ~130°C and released 3.9 wt.% H$_2$. Subsequently,
as its diffraction intensity is relatively weaker. In Figure 7B, residual peaks of LiBH$_4$ are observed, which demonstrates incomplete dehydrogenation. In Figure 7C, besides LiH and Al, the dehydrogenation products contain AlB$_2$, which was reported as the reversible phase (Friedrichs et al., 2009). AlB$_2$ can accelerate the formation of LiBH$_4$ in the reverse reaction. The AlB$_2$ content was higher in 2LiBH$_4$-Li$_3$AlH$_6$ than in the other samples, which signifies higher reversibility of 2LiBH$_4$-Li$_3$AlH$_6$. There is an unexpected peak near 50° both in the 2LiBH$_4$-LiAlH$_4$ and 2LiBH$_4$-Li$_3$AlH$_6$ samples, which was not identified but was also detected in previous reports (Yang et al., 2007).

**CONCLUSIONS**

In this work, the dehydrogenation performance of three different Al source composite systems, 2LiBH$_4$-Al, 2LiBH$_4$-LiAlH$_4$, and 2LiBH$_4$-Li$_3$AlH$_6$, was analyzed. Elemental Al was the raw material and was used without further purification in the 2LiBH$_4$-Al composite, in contrast to the other two samples, where it was decomposed from LiAlH$_4$ or Li$_3$AlH$_6$. Li$_3$AlH$_6$ powder was prepared from LiH and LiAlH$_4$. It can be concluded that the optimum synthetic conditions for ball milling are 50 h with a 25:1 ball to powder ratio at 300 rpm. The results demonstrate that Al, LiAlH$_4$, and Li$_3$AlH$_6$ had a stimulative effect on LiBH$_4$, allowing dehydrogenation at a relatively lower temperature. Additionally, 2LiBH$_4$-Li$_3$AlH$_6$ was shown to have the best performance, with the endothermic peak of LiBH$_4$ at 416°C, 53°C lower than that of the pure LiBH$_4$ sample (469°C). The TPD results also verified the superior results of 2LiBH$_4$-Li$_3$AlH$_6$, which showed the best kinetics performance among the composite samples. 2LiBH$_4$-Li$_3$AlH$_6$ released 9.1 wt.% H$_2$ in only 150 min, which is over 95% of its theoretical hydrogen storage capacity. Its dehydrogenation product, AlB$_2$, was reported as a reversible phase by researchers (Friedrichs et al., 2009), which could promote the reverse reaction of producing LiBH$_4$. Further studies are needed to research the reversibility of the 2LiBH$_4$-Li$_3$AlH$_6$ composite.

**DATA AVAILABILITY STATEMENT**

All datasets generated for this study are included in the article/supplementary material.

**AUTHOR CONTRIBUTIONS**

LC and YL: conception and design of study. YL and JH: acquisition of data. YL and XX: analysis and/or interpretation of data. YL: drafting the manuscript. DZ and SW: revising the manuscript critically for important intellectual content. YL and XX: approval of the version of the manuscript to be published.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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