Effect of Different Amounts of TiF₃ on the Reversible Hydrogen Storage Properties of 2LiBH₄–Li₃AlH₆ Composite

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Hydrogen is a potential green alternative to conventional energy carriers such as oil and coal. Compared with the storage of hydrogen in gaseous or liquid phases, the chemical storage of hydrogen in solid complex hydrides is safer and more effective. In this study, the complex hydride composite 2LiBH₄–Li₃AlH₆ with different amounts of TiF₃ was prepared by simple ball-milling and its hydrogen storage properties were investigated. Temperature programmed desorption and differential scanning calorimetry were used to characterize the de/rehydrogenation performance, and X-ray diffraction and scanning electron microscopy (SEM) were used to explore the phase structure and surface topography of the materials. The dehydrogenation temperature decreased by 48°C in 2LiBH₄–Li₃AlH₆ with 15 wt% TiF₃ composites compared to the composite without additives while the reaction kinetics was accelerated by 20%. In addition, the influence of hydrogen back pressure on the 2LiBH₄–Li₃AlH₆ with 5 wt% TiF₃ composite was also investigated. The results show that hydrogen back pressure between 2.5 and 3.5 bar can improve the reversible performance of the composite to some extent. With a back pressure of 3.5 bar, the second dehydrogenation capacity increased to 4.6 wt% from the 3.3 wt% in the 2LiBH₄–Li₃AlH₆ composite without hydrogen back pressure. However, the dehydrogenation kinetics was hindered. About 150 h, which is 100 times the time required without back pressure, was needed to release 8.7 wt% of hydrogen at 3.5 bar hydrogen back pressure. The SEM results show that aluminum was aggregated after the second cycle of dehydrogenation at the hydrogen back pressure of 3 bar, resulting in the partial reversibility of the 5 wt% TiF₃-added 2LiBH₄–Li₃AlH₆ composite.

Keywords: 2LiBH₄–Li₃AlH₆, hydrogen storage performance, catalytic modification, hydrogen back pressure, reversible performance

INTRODUCTION

As a new energy source, hydrogen has great potential to solve the serious energy depletion and atmospheric pollution caused by excessive utilization of conventional energy sources. Hydrogen is the most abundant element in the Universe. The calorific value of 1 kg of hydrogen is 140 megajoules of energy, which is three times that of the amount of energy released by the same weight of oil.
Moreover, water vapor is the only combustion product of hydrogen, making it a pollution-free energy source. However, the limitations of hydrogen storage technology have hindered its practical application.

As a promising candidate for solid-state hydrogen storage, LiBH₄ has a high theoretical gravimetric hydrogen density of 18.5 wt%, which is the highest among all the solid hydride materials. (Züttel et al., 2003) first studied the desorption properties of LiBH₄. The material released 0.3 wt% hydrogen at 200°C and 1 wt% hydrogen at 320°C. After heating to 500°C, rapid hydrogen desorption was observed, and 9 wt% of hydrogen was released. This capacity meets the requirements of the US Department of Energy (DOE) (Jain et al., 2010), but the desorption temperature is too high for practical applications, and the dehydrogenation kinetics also need to be improved.

Many researchers have focused on the modification of LiBH₄ using various methods, such as composite modification (Xia et al., 2011; Langmi et al., 2012; Bilen et al., 2015; Liu et al., 2015; Sofianos et al., 2017), catalyst modification (Puszkiewicz et al., 2014; Ma et al., 2016; Zhang et al., 2018), and nanoengineering (Zhao et al., 2014; Surrey et al., 2016; Sofianos et al., 2018). Compositing LiBH₄ with other active hydrides is an effective way to enhance its hydrogen storage performance. For example, Liu et al., 2016 milled LiBH₄ with AlH₃ to form a 2LiBH₄ + AlH₃ composite. The dehydrogenation temperature of the 2LiBH₄ + AlH₃ composite was reduced by 30°C compared with pure LiBH₄, and 11.2 wt% hydrogen was obtained. (Ding et al., 2019) found that in a nanosized composite of LiBH₄ and MgH₂ prepared by ball-milling with aerosol spraying (BMAS), the LiBH₄ released hydrogen at 265°C, and approximately 5.0 wt% H₂ was reversible in the first five dehydrogenation and rehydrogenation cycles. Using the shrinking-core model, it was found that a larger interfacial area between LiBH₄ and MgH₂ improves the dynamics of the nano 2LiBH₄ + MgH₂ mixture (Ding et al., 2019).

Other researchers have prepared binary (Mao et al., 2009b; Yu et al., 2010; Hansen et al., 2013; Li et al., 2013; Mustafa et al., 2018; Yahya et al., 2019) or ternary (Yang et al., 2012; Liu et al., 2021) composite systems of LiBH₄ and other complex hydrides (Halim et al., 2018; Halim and Ismail, 2018), such as LiBH₄-LiAlH₄ (Thaweelap and Utke, 2016; Meethom et al., 2020), LiBH₄-Li₃AlH₆ (Choi et al., 2011b), LiBH₄-NaBH₄ (Dematteis et al., 2016), LiBH₄-NaAlH₄, LiBH₄-Mg(BH₄)₂ (Zheng et al., 2020), LiBH₄-Ca(BH₄)₂ (Ampounogli et al., 2015), LiBH₄-Mg₂NiH₄ (Bergemann et al., 2019), LiBH₄-NaBH₄-MgH₂ (Xiang et al., 2018), and LiBH₄-Li₃AlH₆-MgH₂ (Lin et al., 2020). Bargeman et al. (Bergemann, et al. 2019) investigated the reaction mechanism of a LiBH₄-Mg₂NiH₄ composite. They found that the dehydrogenation path was significantly influenced by the back pressure and temperature. When the back pressure was increased to 50 bar, Mg₂NiH₄ reacted with solid LiBH₄ below 270°C, and the reaction enthalpy was reduced by 13 kJ/mol H₂. In Thaweelap’s work (Thaweelap and Utke, 2016), LiBH₄ and LiAlH₄ were synthesized together with Ti-based catalysts or carbon nanotubes to achieve a synergistic effect between the catalysts and composites. Both the kinetics and thermodynamics of the samples were improved. In addition, the reversibility of the LiBH₄-LiAlH₄-MWCNT composite was increased to 3.7 wt% in the second cycle compared to 2.8 wt% in the LiBH₄-LiAlH₄ composite. Wu (Wu et al., 2012) revealed the reaction mechanism of a LiBH₄-Li₃AlH₆ composite, which received 8.5 wt% hydrogen in the dehydrogenation process. Zhou (Zhou et al., 2017) prepared a 2LiBH₄-LiAlH₄ composite nanoconfined in an RFC aerogel, and found that both the thermodynamic and kinetic properties were enhanced.

Some catalysts such as CaH₂ (Jiang et al., 2012), NbF₅ (Mao et al., 2013), TiF₅ (Mao et al., 2010) and NbCl₅ (Tu et al., 2015) destabilize LiBH₄ to decompose H₂ under moderate conditions. Ming mixed LiBH₄ with several additives using the ball-milling method (Au and Jurgensen, 2006). The modified mixtures desorbed hydrogen at a lower temperature and higher rate. Among the various additives, Ti-based catalysts had an obvious effect. Choi (Choi et al., 2011a) demonstrated that TiCl₃ could promote desorption in a Li₃AlH₆/LiBH₄ composite at a lower temperature. Because of the catalyst effect of TiCl₃, Li₃AlH₆ started to release hydrogen at 81°C, and LiBH₄ released hydrogen at 226°C, which are 130 and 200°C lower than the temperatures at which hydrogen was released by pure Li₃AlH₆ and LiBH₄, respectively. A novel two-dimensional (2D) layered Ti₃C₂ material was added to the LiBH₄-THF solution to synthesize a series of LiBH₄-χTi₃C₂ composites with different mass ratios. The LiBH₄-2Ti₃C₂ composite released hydrogen at 172.6°C, and its activation energy was reduced by 50% compared to that of pure LiBH₄ (Zang et al., 2018). TiF₅ is a well-known catalyst for solid-state hydrogen storage materials. It effectively improved the hydrogen storage performance of LiAlH₄-MgH₂ (Mao et al., 2011), MgH₂-NaAlH₄ (Ismail et al., 2012) and 2NaAlH₄+Ca(BH₄)₂ (Mustafa and Ismail, 2019) binary composite.

In this study, a ternary composite of 2LiBH₄-Li₃AlH₆ with different amounts of TiF₅ (the mass percent of TiF₅ was 5, 10, and, 15%) was prepared by a high-energy ball milling method. The synergistic effects between the composite and the catalyst were investigated together with the influence of the hydrogen back pressure on the reversibility.

**MATERIALS AND METHODS**

LiBH₄ powder (95% purity) and TiF₅ powder (99% purity) were purchased from Acros Organics and Sigma Aldrich, respectively. Li₃AlH₆ powder was obtained by mixing LiAlH₄ (95% purity, Sigma Aldrich) and LiH (98% purity, Alfa Aesar) using a high-energy ball-milling method at a ball-to-powder ratio of 40:1 (Li et al., 2020). All the materials used in this work were stored in a glove box purchased from Mikrouna. The glove box was filled with high-purity argon to purge the atmosphere, and the water vapor content was less than 1 ppm. The 2LiBH₄-Li₃AlH₆ series with and without TiF₅ composites was weighed and mixed in this glove box, and then loaded in a sealed stainless ball-milling tank. Planetary ball-milling equipment (QM-3SP4) was used to grind the materials at 300 revolutions per minute for 1 h. A Sievert-type instrument which recorded the pressure change of the hydrogen released during the heating process was used to...
evaluate the dehydrogenation/rehydrogenation performance of the materials. Approximately 200 mg of each sample was charged into a thin stainless-steel tube reactor. The reactor was then heated from ambient temperature to the set temperature at a constant heating rate. The time, temperature, and pressure data were recorded every ten seconds. The amount of hydrogen released or absorbed during the heating process was calculated using the ideal gas state equation. Differential scanning calorimetry (DSC, Netzsch STA 449F3/Netzsch Q403C) was used to evaluate the thermal effects of the materials. In the glove box, about 1–1.5 mg of samples were loaded each time and sealed in an aluminum crucible. Both the sample crucible and the reference crucible were placed in the reaction chamber of the DSC equipment, which was filled with flowing high purity argon at a flow rate of 50 ml/min.

X-ray diffraction (XRD, X’Pert-PRO) and scanning electron microscopy (SEM, FEI FSEM SIRION-100) were used to analyze the phase structure and observe the surface topography of the material. The XRD K-Alpha1 and K-Alpha2 wavelengths were 1.540598 Å and 1.544426 Å, respectively. The samples were sealed in a transparent macromolecular container to prevent oxidation. Fourier transform infrared spectroscopy (FT-IR, Bruker, Tensor 27) at the scan rate of 30 cm⁻¹/min and resolution of 0.5 cm⁻¹ was used for chemical bond analysis. The crucibles used in the experiments were aluminum crucibles.

RESULTS AND DISCUSSION

DSC Traces of LiBH₄, Li₃AlH₆, and LiBH₄–Li₃AlH₆ Samples

The DSC traces of the Li₃AlH₆, LiBH₄, and 2LiBH₄–Li₃AlH₆ samples at the heating rate of 5°C/min from ambient temperature to 500°C are shown in Figure 1. In the Li₃AlH₆ sample (Figure 1A), there is an endothermic peak at 214°C due to the decomposition process of Li₃AlH₆. The last peak in the DSC trace of Li₃AlH₆ was the decomposition of LiH (Aresfarenandez, 2007). The decomposition reaction of Li₃AlH₆ and the resulting products are described by Eq. 1 (Zang, et al., 2018):

\[
\text{Li}_3\text{AlH}_6 \rightarrow 3\text{LiH} + \frac{3}{2}\text{H}_2 \tag{1}
\]

There are three endothermic peaks in the DSC trace of LiBH₄ shown in Figure 1B. The first peak at 111°C is the lattice transition peak of LiBH₄, and the second peak at 287°C corresponds to the melting of LiBH₄ (Züttel, et al., 2003). The third peak is wider than the first two and indicates the decomposition of LiBH₄ (Li et al., 2017). The decomposition process is shown in Eq. 2 (Mao et al., 2009a):

\[
\text{LiBH}_4 \rightarrow \text{LiH} + \text{B} + \frac{3}{2}\text{H}_2 \tag{2}
\]

Four endothermal peaks corresponding to the characteristic peaks of the components were observed for the 2LiBH₄–Li₃AlH₆ composite. The LiBH₄ in the 2LiBH₄–Li₃AlH₆ composite melted at 274°C, which is 13°C lower than the melting point of pure LiBH₄. The second and the forth endothermal peaks mean a rapid dehydrogenation temperature of Li₃AlH₆ and LiBH₄, were 199 and 417°C (see Figure 1C), which were lower than pure Li₃AlH₆ and LiBH₄, indicating that the dehydrogenation performance of the 2LiBH₄–Li₃AlH₆ composite obtained a better thermal performance.

Influence of TiF₃ on Hydrogen Desorption Performance of 2LiBH₄–Li₃AlH₆ Composite

The hydrogen storage properties of the 2LiBH₄–Li₃AlH₆ composite both in its original state and in the presence of TiF₃ were investigated in this study. The samples were weighed and loaded in a glove box filled with pure argon. The

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**FIGURE 1** | DSC traces of (A) Li₃AlH₆, (B) LiBH₄, and (C) 2LiBH₄–Li₃AlH₆ composite.

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**FIGURE 2** | XRD patterns of 2LiBH₄–Li₃AlH₆ samples with different amounts of TiF₃ after ball-milling: (A) 0 wt%, (B) 5 wt%, (C) 10 wt%, and (D) 15 wt%.
mass percentages of TiF₃ in the four samples were 0, 5, 10, and 15 wt%. All the samples were prepared by ball-milling for 1 h, and XRD was used to determine the composition of the samples. LiBH₄ and Li₃AlH₆ peaks were observed in the 2LiBH₄–Li₃AlH₆ sample without the TiF₃ additive (Figure 2A). The main diffraction peaks of the three 2LiBH₄–Li₃AlH₆ composites with different amount of TiF₃ (Figures 2B–D) correspond to those of the 2LiBH₄–Li₃AlH₆ sample without additive. The weak diffraction peaks at 23° or 70° in Figure 2C and Figure 2D could be attributed to TiF₃. The absence of a TiF₃ peak in Figure 2B may possibly be due to the small amount of highly dispersed TiF₃. As the TiF₃ content increased, the diffraction peak of Al in the 2LiBH₄–Li₃AlH₆ with TiF₃ composites gradually became obvious, and weak LiH peaks appeared at 38° and 44°. These results indicate that TiF₃ destabilized Li₃AlH₆ by promoting its decomposition into LiH and Al during the ball-milling process (Ares, 2008). Approximately 1 mg of the prepared samples was loaded into an aluminum oxide crucible for the DSC test each time. The results are shown in Figure 3. Comparing the 2LiBH₄–Li₃AlH₆ samples with TiF₃ (Figures 3B–D) or without TiF₃ (Figure 3A), the lattice transition peaks of LiBH₄, which were between 112 and 115°C, were almost unchanged. The melting peak of LiBH₄ decreased from 274 to 257°C as the amount of TiF₃ increased. In addition, compared with the 2LiBH₄–Li₃AlH₆ sample, the Li₃AlH₆ decomposition peak shifted obviously to lower temperatures and became wider as the amount of TiF₃ increased. As the amount of TiF₃ increased, the weight percentage of H in 1 g of ball-milled sample decreased from 9.24 wt% to 7.85 wt%, as shown in Table 1.

A temperature-programmed desorption (TPD) test was performed to study the dehydrogenation properties with and without TiF₃ in the 2LiBH₄–Li₃AlH₆ samples. Table 1 shows the theoretical hydrogen storage capacities of the 2LiBH₄–Li₃AlH₆ samples with different amounts of TiF₃. As the amount of TiF₃ increased, the weight percentage of H in 1 g of ball-milled sample decreased from 9.24 wt% to 7.85 wt%, as shown in Table 1. Approximately 200 mg of each sample was loaded into a stainless-steel reactor. The reactor was heated from room temperature to 440°C at a heating rate of 5 °C/min and maintained at 440°C for half an hour. The hydrogen desorption curves of the four samples are shown in Figure 4. The 2LiBH₄–Li₃AlH₆ sample underwent a two-step dehydrogenation process in which the release of hydrogen began at 190°C and approximately 3 wt% of hydrogen was released in the first step. The second step began at approximately 330°C and the remaining 6.15 wt% of hydrogen was released. The total dehydrogenation capacity of 9.15 wt% is consistent with the theoretical hydrogen storage capacity. In the 2LiBH₄–Li₃AlH₆ with 5 wt% TiF₃ composite, the initial dehydrogenation temperatures of the two-step process decreased to 110 and 270°C. As the amount of TiF₃ increased, the decomposition temperatures of Li₃AlH₆ and LiBH₄ tended to

### Table 1: Theoretical hydrogen storage capacity and actual hydrogen desorption capacity of 2LiBH₄–Li₃AlH₆ samples with different amounts of TiF₃.

| TiF₃ content (wt%) | 0  | 5  | 10 | 15 |
|-------------------|----|----|----|----|
| Theoretical hydrogen storage capacity (wt%) | 9.24 | 8.77 | 8.31 | 7.85 |
| Actual hydrogen desorption capacity (wt%) | 9.15 | 8.67 | 8.27 | 7.71 |

![Figure 3](image3.png) DSC traces of 2LiBH₄–Li₃AlH₆ samples with different amounts of added TiF₃: (A) 0 wt%, (B) 5 wt%, (C) 10 wt%, and (D) 15 wt%.

![Figure 4](image4.png) Hydrogen desorption curves of 2LiBH₄–Li₃AlH₆ samples with different amounts of added TiF₃.
decrease. The Li₃AlH₆ decomposed at 88°C in the 10 wt% TiF₃-added 2LiBH₄–Li₃AlH₆ composite, compared to 85°C in the 15 wt% TiF₃-added sample. LiBH₄ started to release hydrogen at 238°C in the 10 wt% TiF₃-added sample and at 225°C in the 15 wt% TiF₃-added sample. These results indicate that the addition of TiF₃ improved the thermodynamic properties of the 2LiBH₄–Li₃AlH₆ composites. In contrast to the 2LiBH₄–Li₃AlH₆ composite, the absence of a "platform area" between the first and second steps of the composites with TiF₃ indicated that LiBH₄ tended to decompose at a lower temperature and the rate of hydrogen desorption were accelerated by the catalytic effect of TiF₃. The time required for total decomposition decreased to 82 min when the TiF₃ amount was 15 wt% compared to the 95 min required for the 2LiBH₄–Li₃AlH₆ composite without TiF₃. Clearly, the addition of TiF₃ enhanced both the thermodynamic and kinetic properties of the 2LiBH₄–Li₃AlH₆ composite system. However, the theoretical hydrogen storage capacity and the actual hydrogen desorption capacity of the composites with TiF₃ decreased as the amount of TiF₃ increased (shown in Table 1).

Reversible Performance of TiF₃ Added 2LiBH₄–Li₃AlH₆ Composite with Different Back Pressures

The reversible performance of the 2LiBH₄–Li₃AlH₆ composite was investigated in this study. Approximately 200 mg samples of 2LiBH₄–Li₃AlH₆ composite were loaded in a tubular reactor and heated to 450°C at a heating rate of 5°C/min. The dehydrogenated samples were maintained at 450°C for 250 min and then cooled down to ambient temperature naturally. The hydrogen adsorption process was performed at the hydrogen pressure of 100 bar at 450°C for 10 h. The dehydrogenation curves of the 2LiBH₄–Li₃AlH₆ composite are shown in Figure 6. The hydrogen release capacity of the 2LiBH₄–Li₃AlH₆ composite was 9.2 wt% in the first cycle, and 3.3 wt% and 2.4 wt% in the second and third cycles, respectively. Unlike the two-step desorption reactions of the first cycle, only one-step reactions occurred in the second and third cycles, and the initial dehydrogenation temperature reached as high as 400°C.

The observation of the B–H bond of LiBH₄ at 2,200–2,400 cm⁻¹ in the FT–IR curves of the ball-milled sample and the resorption sample (Figure 7) indicates that the 2LiBH₄–Li₃AlH₆ composite was partially reversible, and that the
reversible component was LiBH₄. The formation process of LiBH₄ is given by Eq. 5 (Li et al., 2012):

\[ 2\text{LiH} + \text{AlB}_2 + 3\text{H}_2 \rightarrow 2\text{LiBH}_4 + \text{Al}. \]  

(5)

In the second and third dehydrogenation cycles, the hydrogen releasing capacities were lower than 3.5 wt%, which is significantly lower than the first one. Therefore, further research is required to improve the reversible performance. In a previous work, Mao (Mao et al., 2013) reported that hydrogen back pressure could improve the cyclic performance of LiBH₄-based composites. As mentioned earlier, TiF₃ is a superior catalyst for improving the hydrogen storage performance of the 2LiBH₄–Li₃AlH₆ composite. Therefore, the studies that follow focus on the influence of hydrogen back pressure on the dehydrogenation and cyclic performance of TiF₃-added 2LiBH₄–Li₃AlH₆ composites. The 2LiBH₄–Li₃AlH₆ with 5 wt% TiF₃ composite was dehydrogenated at 425 °C with initial hydrogen back pressures of 2.5, 3.0, and, 3.5 bar. The dehydrogenated samples then began to absorb hydrogen at 100 bar hydrogen pressure for 10 h. The dehydrogenation curves at different hydrogen back pressures in the first and second cycles are shown in Figure 8. The dehydrogenation capacity in the first cycle at the different hydrogen back pressures was about 8.2 wt% to 9.0 wt% in the 2LiBH₄–Li₃AlH₆ with 5 wt% TiF₃ composite, which is lower than its theoretical capacity. In the second dehydrogenation cycle, the amount of hydrogen released was 4.0 wt% or 4.6 wt%, which are higher than that of 2LiBH₄–Li₃AlH₆ composite (3.3 wt%). From these results, it could be concluded that a hydrogen back pressure of between 2.5 and 3.5 bar and the addition of TiF₃ could improve the reversible performance of the 2LiBH₄–Li₃AlH₆ composite to some extent. However, the dehydrogenation kinetics of the 2LiBH₄–Li₃AlH₆ with TiF₃ composite was hindered by the hydrogen back pressure. It took more than 150 h to release 8.7 wt% hydrogen with 3.5 bar hydrogen back pressure in the TiF₃-added 2LiBH₄–Li₃AlH₆ composite, compared to 1.5 h in the composite without back pressure.

The XRD patterns of the second-cycle dehydrogenation products are shown in Figure 9. Three phases, namely, AlB₂, LiH, and Al, were detected in all the curves. Compared with Figure 5B, the diffraction intensity of AlB₂ increased in Figures 9A–C, which resulted in the improved reversible performance.

The surface topography of the 2LiBH₄–Li₃AlH₆ composites under different conditions was observed by SEM. The ball-milled sample (Figure 10A) displayed a coral-like structure. At 50,000 × magnification (inset of Figure 10A), the particles had a rod-shaped appearance with a length of approximately 3 μm and a width of 1 μm. After the first cycle of dehydrogenation, the particles agglomerated into aggregates (Figure 10B) instead of rod-shaped structures. After the second cycle of dehydrogenation at the back pressure of 3 bar, some spherical particles were formed, as shown in Figure 10C. Using the energy dispersive spectrometer, these particles were found to consist of metallic aluminum. The aggregation and growth of metallic aluminum led to a poor dispersion and reduced active surface area of the dehydrogenation products, which may explain the partial reversibility of the 2LiBH₄–Li₃AlH₆ composites (Meethom et al., 2020).
CONCLUSION

In this study, the dehydrogenation performance and influence of back pressure on the 2LiBH₄–Li₃AlH₆ with/without TiF₃ composites were investigated. TiF₃ enabled Li₃AlH₆ and LiBH₄ to desorb hydrogen at lower temperatures. With an increase in the amount of TiF₃, the dehydrogenation temperature decreased significantly. In the 15% TiF₃-added composite, Li₃AlH₆ decomposed at 85°C and LiBH₄ at 225°C, that is, at 105°C lower for each component compared with the undoped sample. However, when the TiF₃ content was increased to 15 wt%, the hydrogen capacity of the composite was reduced to 7.85 wt%, which is 15% lower than that without the 2LiBH₄–Li₃AlH₆ composite. In the reversibility evaluation, the hydrogen back pressures of 2.5 bar, 3.0 bar, and 3.5 bar were adopted. The results indicate that the back pressure could enhance the reversible performance of 2LiBH₄–Li₃AlH₆ composite to some extent. A reversible hydrogen capacity of 4.6 wt% was obtained with a back pressure of 3.5 bar in the TiF₃-added 2LiBH₄–Li₃AlH₆ composites. However, the kinetics of the TiF₃-added 2LiBH₄–Li₃AlH₆ composites were hindered. More
than 150 h were required to complete the hydrogen release process at the hydrogen back pressure of 3.5 bar. The SEM results showed that metallic aluminum was aggregated, resulting in poor dispersion of the dehydrogenated phases and reduced active surface area. Further research is needed to improve the desorption and absorption performance of LiBH₄.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding author.

AUTHOR CONTRIBUTIONS

YL and LC: conception and design of study. YL and YZ: acquisition of data. YL analysis and/or interpretation of data. YL: drafting the manuscript. YZ and LC revising the manuscript critically for important intellectual content. YL, YZ, and LC 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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