Electronic Supplementary Material (ESI) for Dalton Transactions

Increased air stability and decreased dehydrogenation temperature of $LiBH_4$ via modification within poly(methylmethacrylate)

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Experimental Section

Preparation of $LiBH_4$@PMMA composite.

The air-stable $LiBH_4$@PMMA composite was prepared by adding the $LiBH_4$/THF solution (2.0 M $LiBH_4$ in tetrahydrofuran) into the PMMA that swelling in the DMAC (dimethylacetamide) solvent. The obtained solution was vigorously stirred and slowly heated up to 35^{o}C and kept at that temperature for 2 h to obtain the gelatinous product. The gelatinous product was dried by vacuum in room temperature at least for 96 h to remove all the solvent. The composite after exposure to air was dried by freeze drying for 72 h to remove the moisture on the sample surface. Pure $LiBH_4$ used in this work was dried from the $LiBH_4$/THF solution for better comparison.

Instrument and Analysis

The phase structure of sample was characterized on rotation anode X-ray diffractometer (a Philips X'Pert MPD). Zeiss-Supra 40 scanning electron microscope (SEM) and JEOL-2100 transmission electron microscopy (TEM) with an operating voltage of 200 kV were employed to characterize morphology and structure of the $LiBH_4$@PMMA composite. The chemical bondings of the species were identified via a Fourier transform infrared spectrometer (FT-IR, Vector 33). The hydrogen evolution was determined by a mass-spectrometer (MS, Hiden, Qic 20) at a heating rate of 4 °C min^{-1} and the inert Ar was used as the carrier gas with flow rate 60 ml min^{-1}. Differential scanning calorimetry (DSC) measurements were performed by NETZSCH 400 under argon with a gas flow of 25 mL Ar min^{-1} at a heating rate of 4 °C min^{-1}. Isothermal dehydrogenation properties of samples were measured by Setaram PCT-Pro 2000 apparatus at different temperatures. Sample was heated to the target temperature by a rate of 30 °C min^{-1} and the desorbed hydrogen amount was measured to determine the dehydrogenation capacity and kinetic.

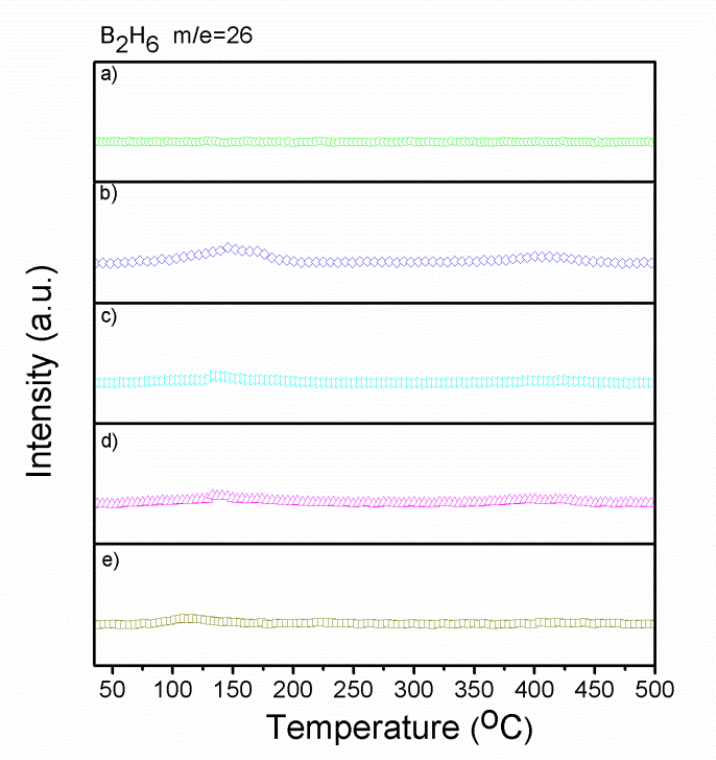

Figure S1. MS borane signals of pure $LiBH_4$ (a), $LiBH_4$@PMMA composite (b) and after exposure the $LiBH_4$@PMMA composite to air for 1h (c), 4h (d), 8h (e).

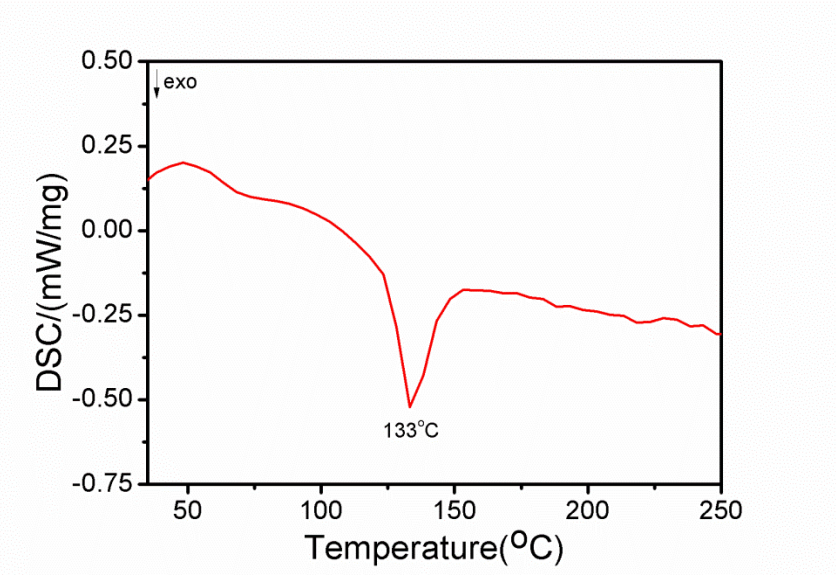

Figure S2. DSC plots of $LiBH_4$@PMMA composite.

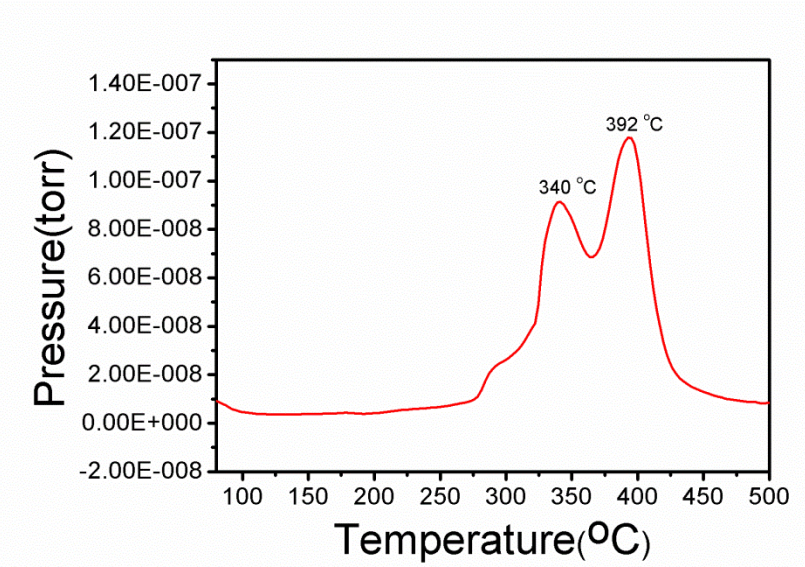

Figure S3. MS hydrogen signal of bulk $LiBH_4$ after ball milling with PMMA .