Supplementary Information

Cu$^{2+}$-Induced room temperature hydrogen release from ammonia borane

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Experimental Details

Materials

CoCl$_2$.6H$_2$O, NiCl$_2$.6H$_2$O and CuCl$_2$.2H$_2$O were purchased from S. D. Fine Chemicals, India. Metal salts were dehydrated by heating around 120 °C for 12-14 h and stored in a dessicator. Ammonia–borane (AB) was synthesized from (NH$_4$)$_2$SO$_4$ and NaBH$_4$ using the procedure described by Ramachandran and Gagare.$^1$ The purity of AB was established by NMR and IR spectroscopies before use.

Co$^{2+}$-, Ni$^{2+}$-, and Cu$^{2+}$-Induced release of H$_2$ from AB

1 mmol of AB and a fixed quantity of metal chloride salt were placed in a reaction glass tube equipped with a stir bar. The reaction tube was connected to a gas burette via a water trap. The tube was placed in an oil bath that was pre-heated to 60 °C. The two powders were mixed inside the tube by mechanical stirring. In the case of Cu$^{2+}$, reaction started almost instantaneously whereas in the case of Co$^{2+}$ and Ni$^{2+}$, 15-30 min induction periods were observed. The course of the reaction (H$_2$ evolution) was followed by monitoring the change in water level with time and the time taken for 1 mL change in the gas burette reading was noted down. The reactions were monitored up to 400 min. After that water levels in the burette and the reservoir were leveled and the final reading was noted down. The powder obtained after the reaction was isolated and characterized using powder XRD, $^{11}$B MAS-NMR spectroscopy techniques.
The thermolysis reactions were carried out for various Cu\(^{2+}/\)AB ratios: 0.05, 0.10, 0.15, and 0.20. Further increase in Cu\(^{2+}\) concentration did not show much improvement in H\(_2\) releasing rates. Thermolysis of Cu\(^{2+}\)-AB (Cu\(^{2+}/\)AB = 0.05) combination was also carried out at different temperatures i.e., at 50, 55, 60 °C.

Reference

60 °C

**Fig. S2.** a) TG profiles of CuCl₂; b) Synchronous MS Profiles of m/z = 17, 18 (water). The ramp rate is 2 °C min⁻¹.

**Fig. S3.** Enlarged view of TG profile of Cu²⁺/AB composite at a ramp rate of 2 °C/min.