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Supplementary Information

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

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Experimental Details

Materials

$CoCl_2.6H_2O$, $NiCl_2.6H_2O$ and $CuCl_2.2H_2O$ 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 $(NH_4)_2SO_4$ 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.

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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

1. P. V. Ramachandran and P. D. Gagare, *Inorg. Chem.*, 2007, **46**, 7810.

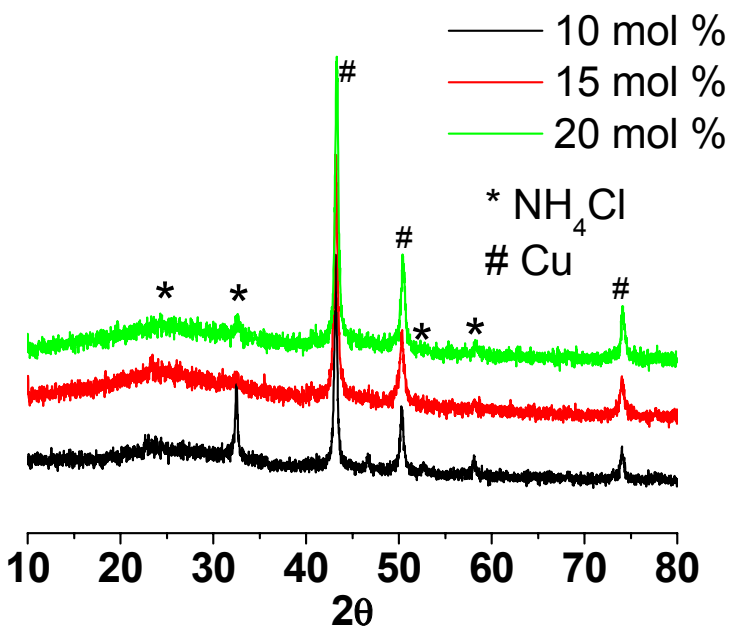
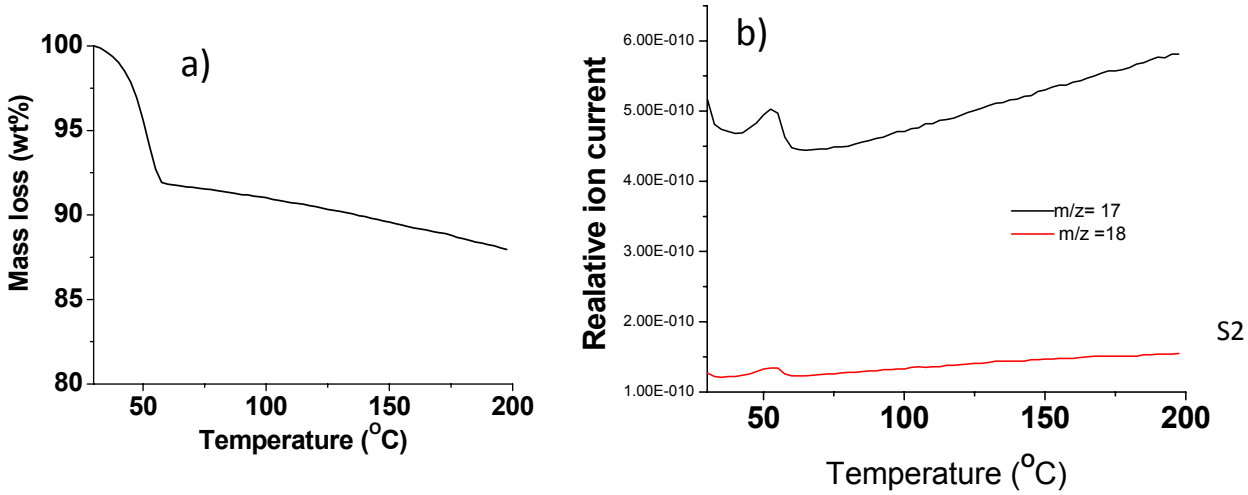


Fig. S1. Powder XRD pattern of the final products of Cu^{2+}-induced hydrogen release from AB at



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60 °C

Fig. S2. a) TG profiles of $CuCl_2$; b) Synchronous MS Profiles of m/z =17, 18 (water). The ramp rate is 2 °C min^{-1}

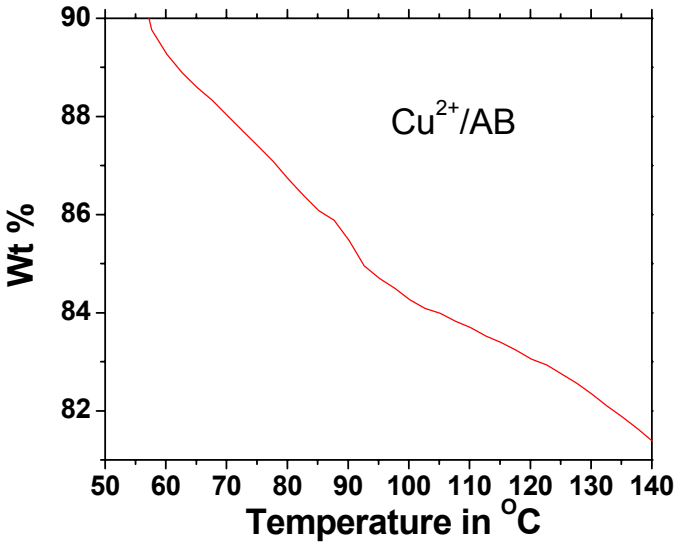

Fig. S3. Enlarged view of TG profile of Cu^{2+}/AB composite at a ramp rate of 2 °C/min.