

Supporting Information for

**Binary Cu-Co Catalysts Derived from Hydrotalcites with Excellent  
Activity and Recyclability towards  $\text{NH}_3\text{BH}_3$  Dehydrogenation**

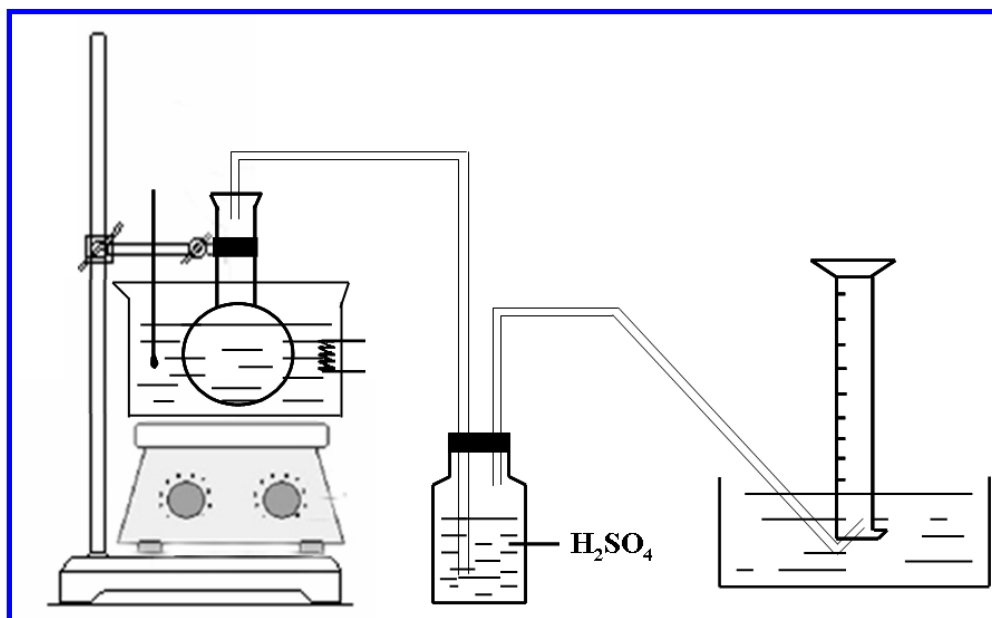
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G. Evans and Xue Duan

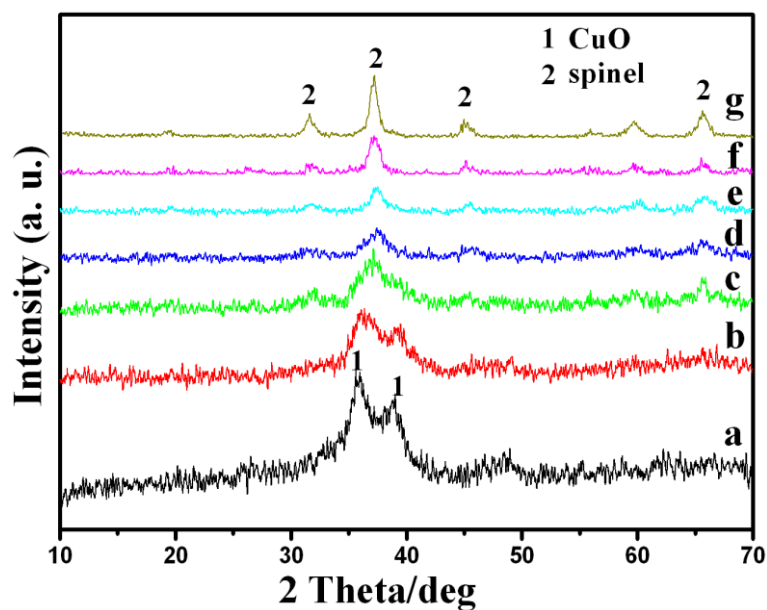
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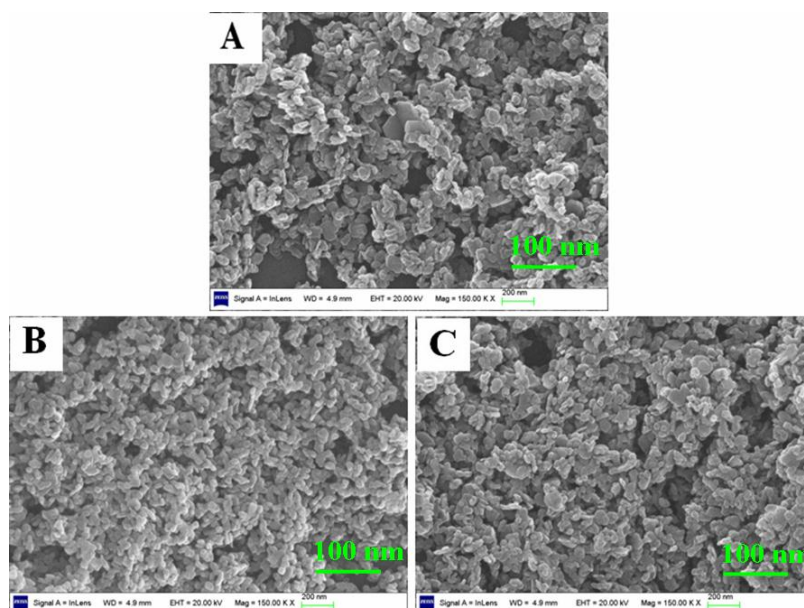
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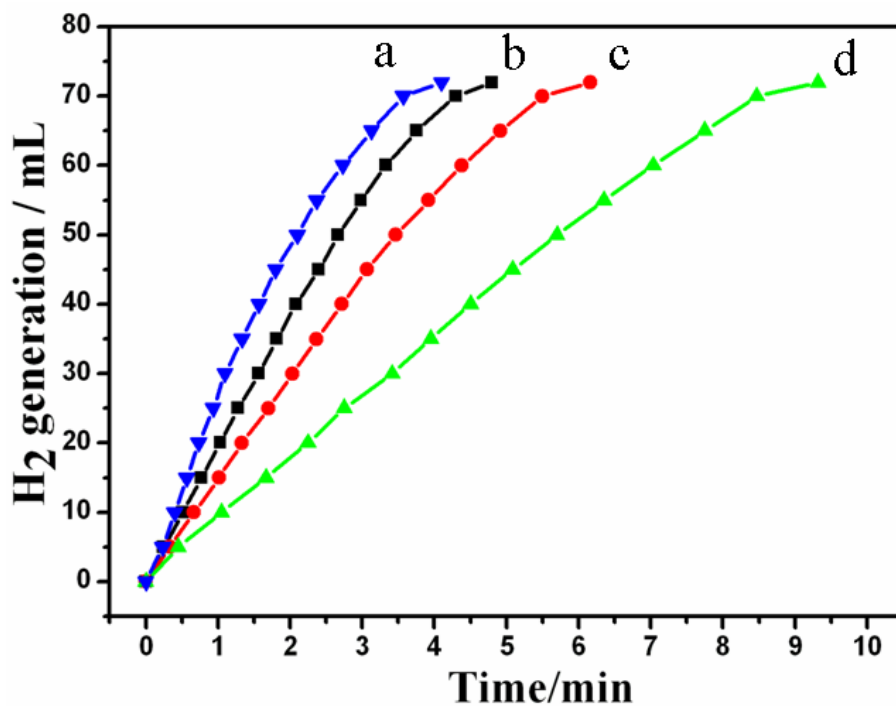
**Figure S1.** The experimental setup for hydrogen generation from the  $\text{NH}_3\text{BH}_3$  dehydrogenation.



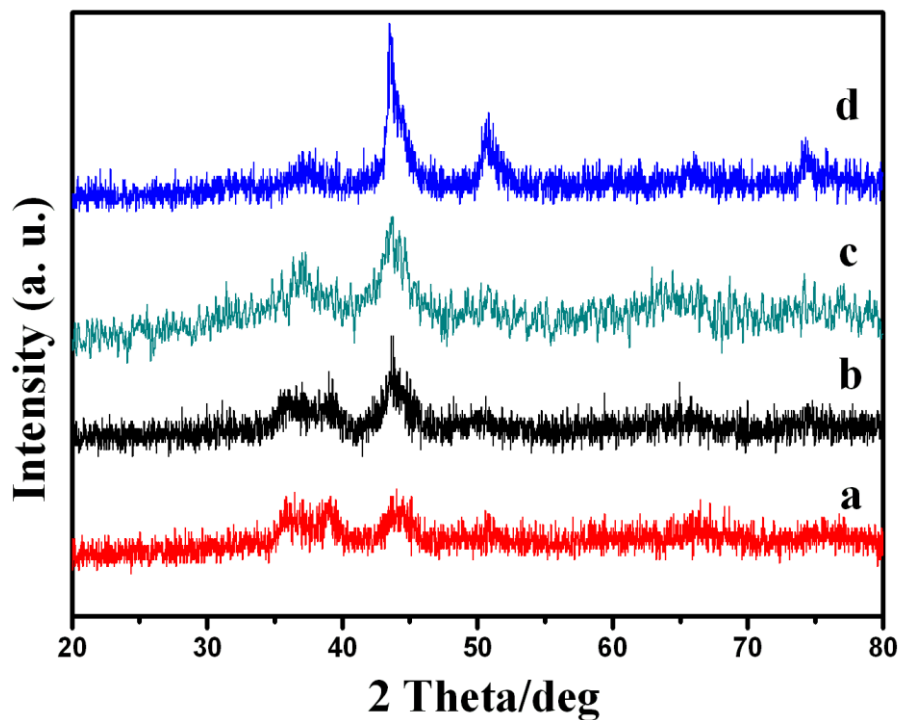
**Figure S2.** XRD patterns of the  $(\text{Cu}_x\text{Co}_y)_2\text{Al-MMO}$  samples with various ratios of  $x(\text{Cu}) : y(\text{Co})$ :  
a) 1:0, b) 0.87:0.13, c) 0.75:0.25, d) 0.50:0.50, e) 0.25:0.75, f) 0.13:0.87, g) 0:1.



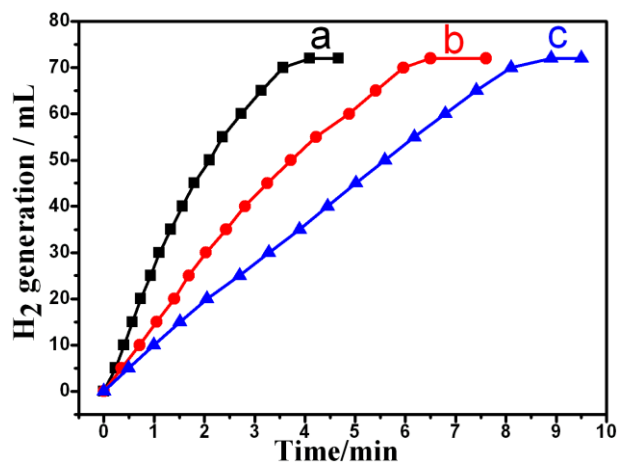
**Figure S3.** SEM images of the MMO samples: A)  $\text{Cu}_2\text{Al-MMO}$ , B)  $(\text{Cu}_{0.50}\text{Co}_{0.50})_2\text{Al-MMO}$ , C)  $\text{Co}_2\text{Al-MMO}$ .



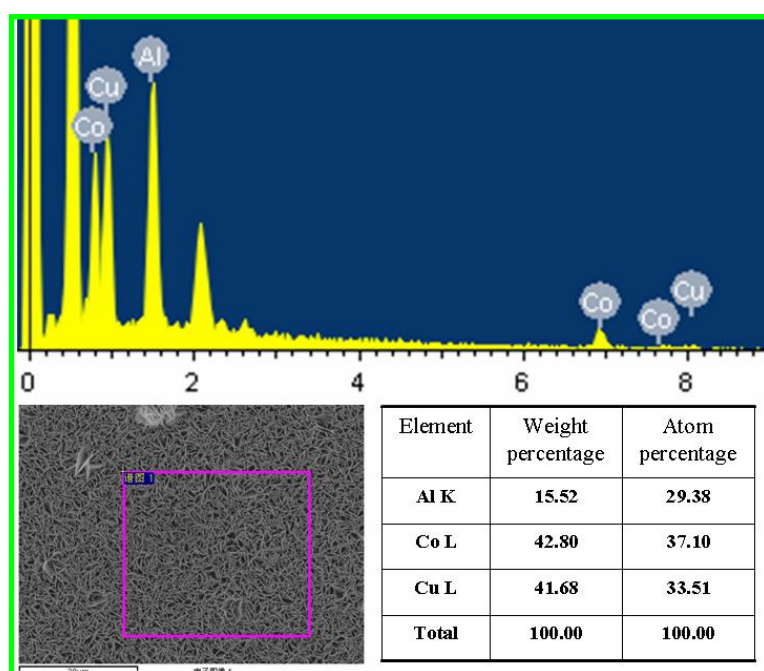
**Figure S4.** Plot of  $\text{H}_2$  volume generated from AB hydrolysis vs. time catalyzed by (a)  $(\text{Cu}_{0.50}\text{Co}_{0.50})_2\text{Al-300}$ , (b)  $(\text{Cu}_{0.50}\text{Co}_{0.50})_2\text{Al-400}$ , (c)  $(\text{Cu}_{0.50}\text{Co}_{0.50})_2\text{Al-500}$ , and (d)  $(\text{Cu}_{0.50}\text{Co}_{0.50})_2\text{Al-600}$  ( $w_{\text{cat.}} = 20$  mg,  $[\text{AB}] = 50$  mM,  $(\text{Cu}+\text{Co})/\text{AB}=0.09$ ,  $T = 25 \pm 1$  °C).



**Figure S5.** XRD patterns of the  $(\text{Cu}_{0.50}\text{Co}_{0.50})_2\text{Al-Cat}$  samples obtained by reduction in  $\text{H}_2$  at different temperatures: (a) 300 °C, (b) 400 °C, (c) 500 °C, (d) 600 °C.



**Figure S6.** The recyclability for the  $(\text{Cu}_{0.50}\text{Co}_{0.50})_2\text{Al}$ -Cat powdered catalyst towards  $\text{NH}_3\text{BH}_3$  dehydrogenation in three consecutive cycles from curve a to c.



**Figure S7.** SEM-EDS analysis for the  $\text{CuCoAl}$ -LDHs film precursor on Al substrate prepared by the *in situ* growth method.