Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A

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Electronic Supplementary Information

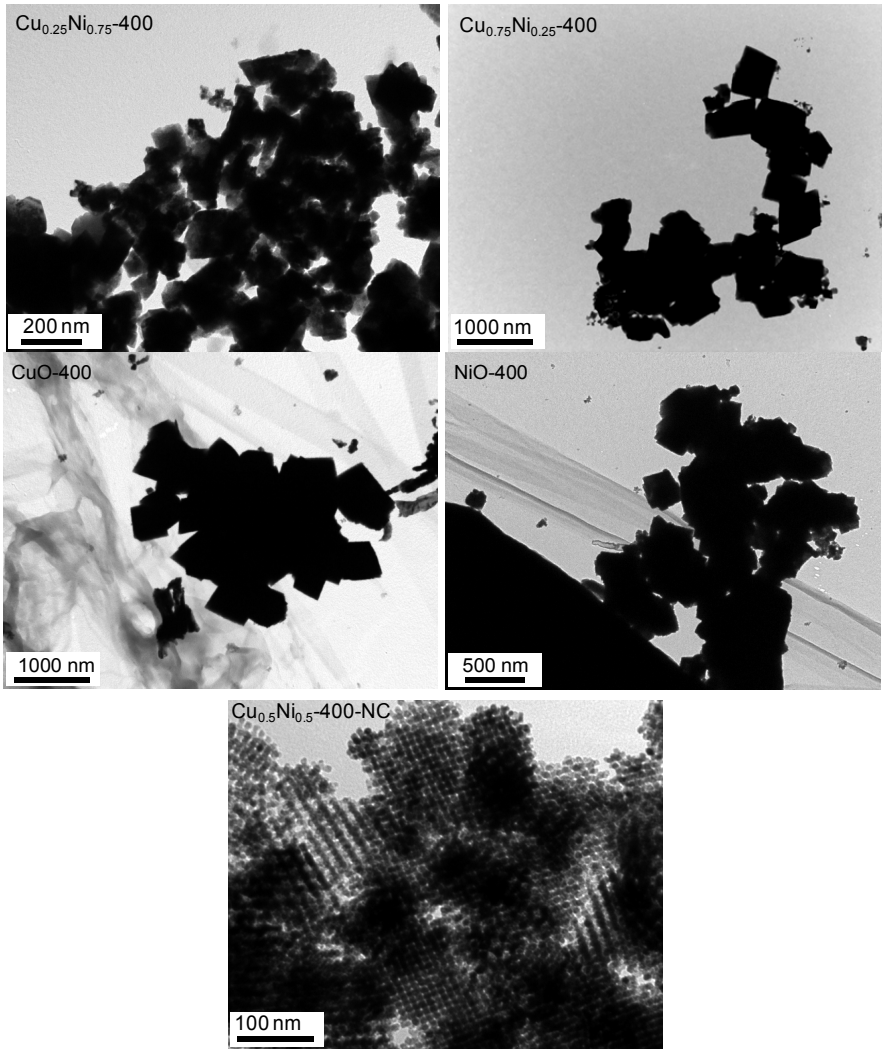


Fig. S1 Representative TEM images of the samples of different compositions (as indicated) prepared by thermal decomposition of metal nitrate precursors and the nanocasting approach.

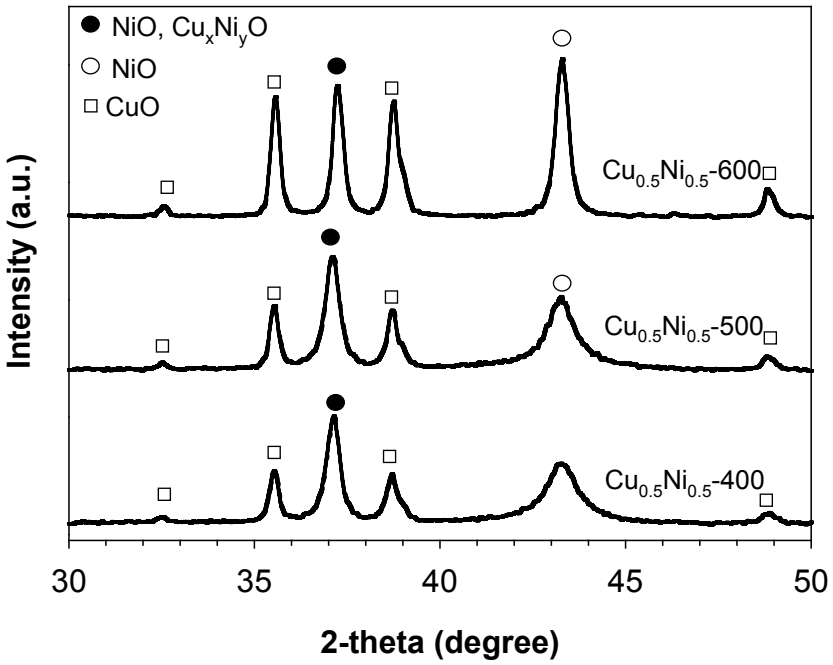


Fig. S2 Wide-angle powder XRD patterns of mixed metal oxides prepared at different temperatures (as indicated) (Bruker SMART APEXII X-ray diffractometer with a Cu K_α radiation).

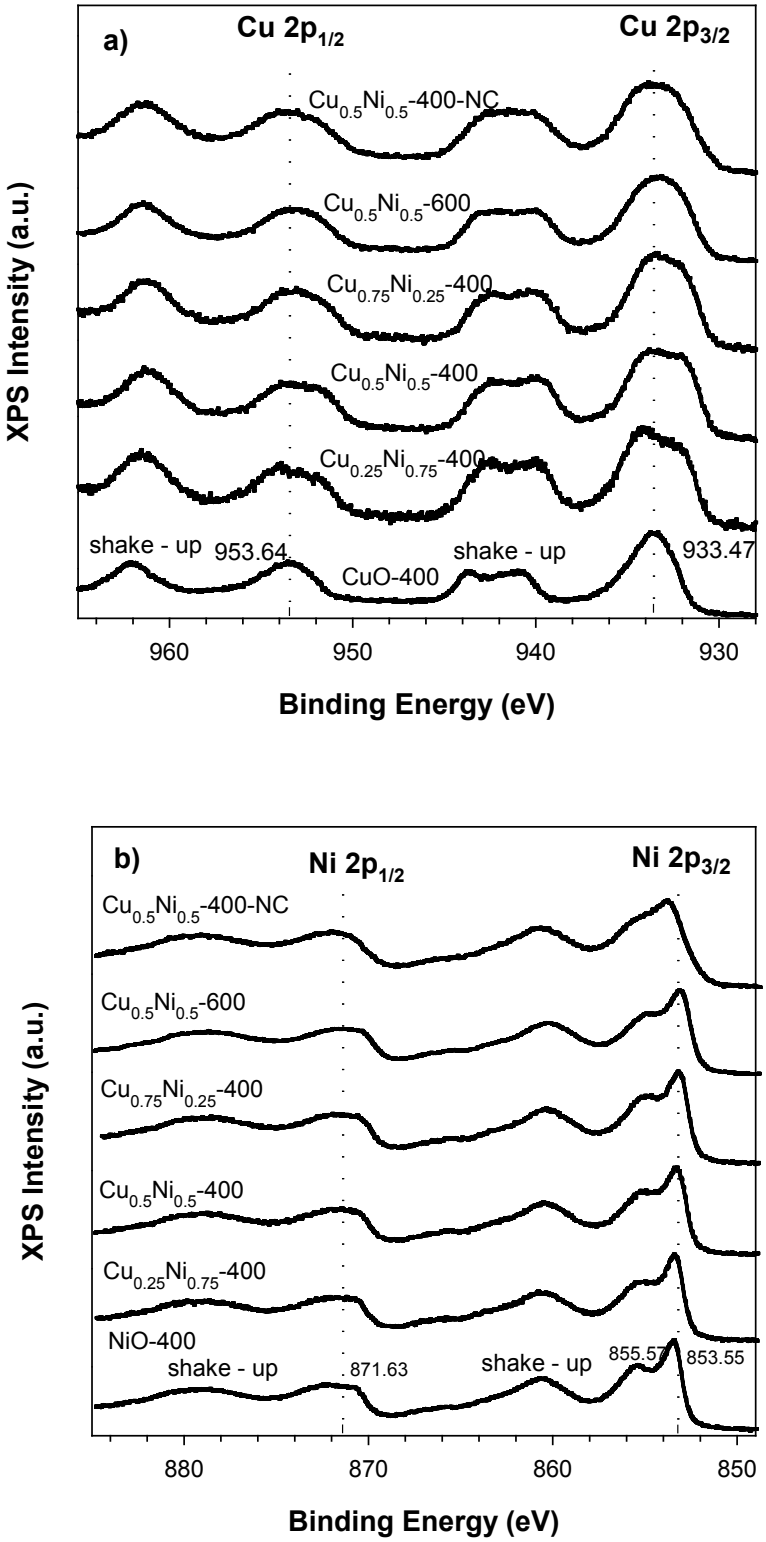


Fig. S3a-b XPS spectra of the prepared catalysts (the spectra are normalized by their peak intensity and energy corrected for adventitious carbon at 284.8eV).

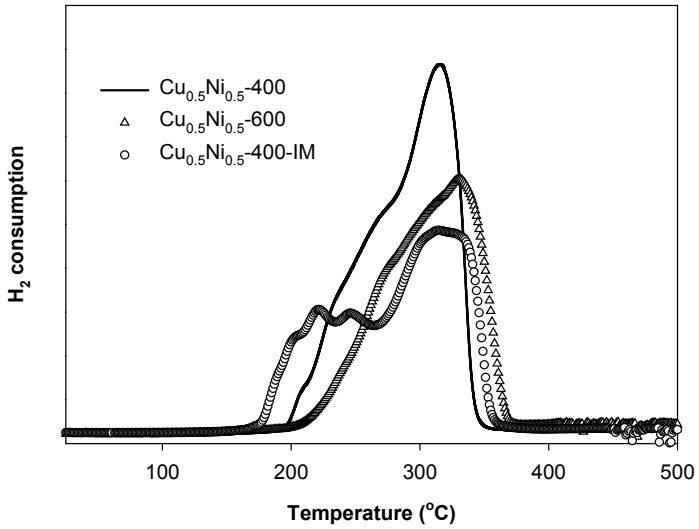


Fig. S4 H_2-TPR profiles of the samples prepared by one-step thermal treatment at 400°C and 600°C, and by post impregnation of copper nitrate on pre-formed NiO (as indicated).

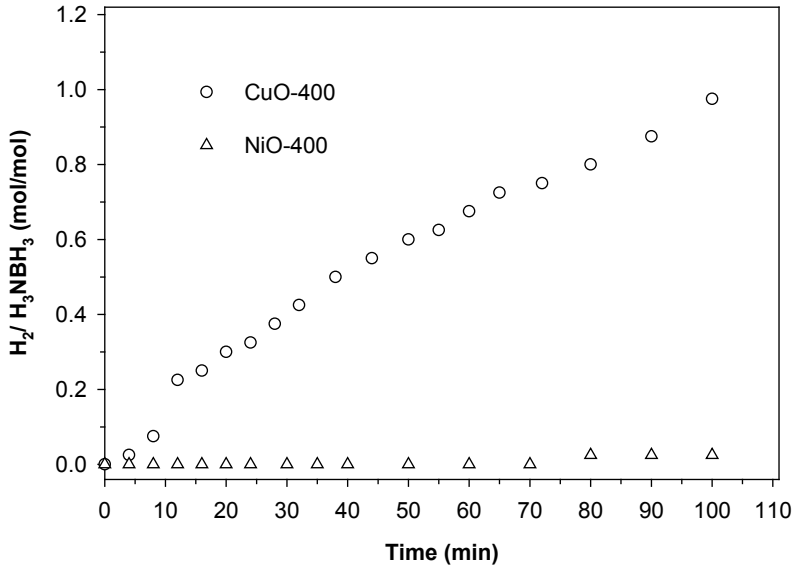


Fig. S5 Volume of hydrogen generated from the hydrolysis of AB catalyzed by single oxides CuO and NiO, as a function of time (H_3NBH_3=1.48mmol, catalyst=10mg, H_2O=10ml, T=25°C).

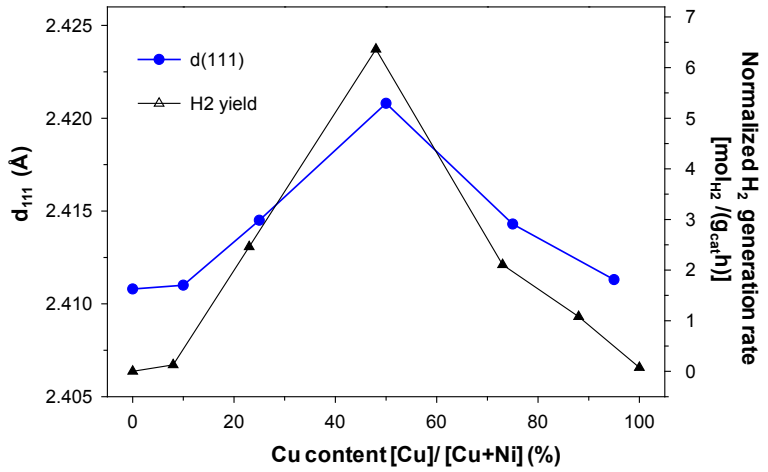


Fig. S6 Plot representing the lattice d-spacing value, corresponding to the (111) plane, and reaction rate at half conversion, as functions of the catalyst composition.

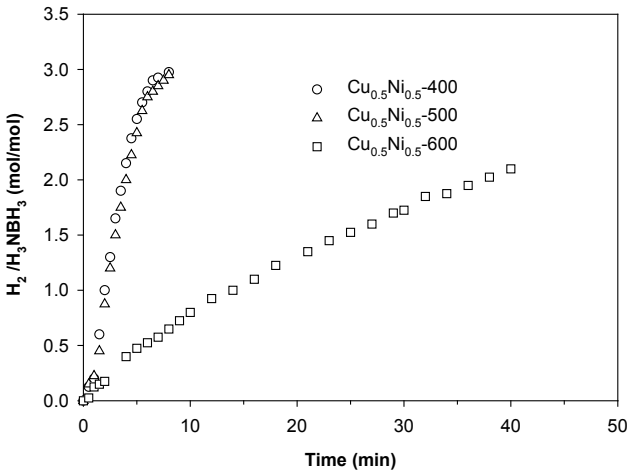


Fig. S7 Time versus volume of hydrogen generated from the hydrolysis of AB catalyzed by binary CuO-NiO oxides prepared at different thermal treatment temperatures (H_3NBH_3=1.48mmol, catalyst=10mg, H_2O=10ml, T=25^oC).

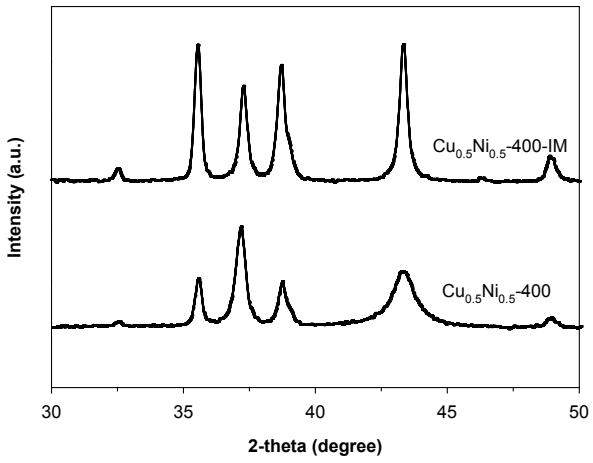


Fig. S8 Comparison of the XRD patterns of the samples obtained by one-step thermal treatment and post-impregnation (as indicated).

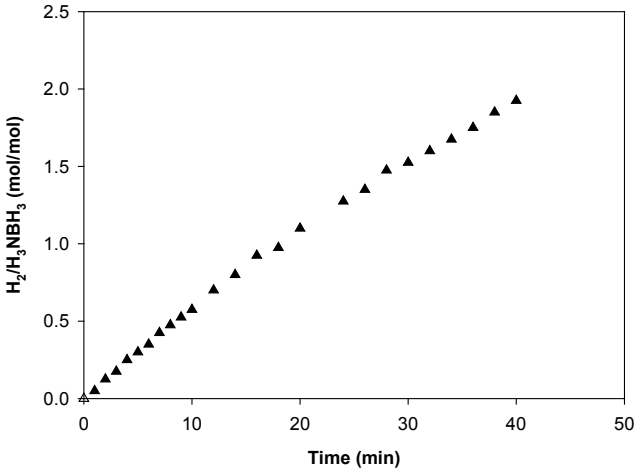


Fig. S9 Time versus volume of hydrogen generated from the hydrolysis of AB catalyzed by $Cu_{0.5}Ni_{0.5}$-400-IM sample (H_3NBH_3=1.48mmol, catalyst=10mg, H_2O=10ml, T=25°C).

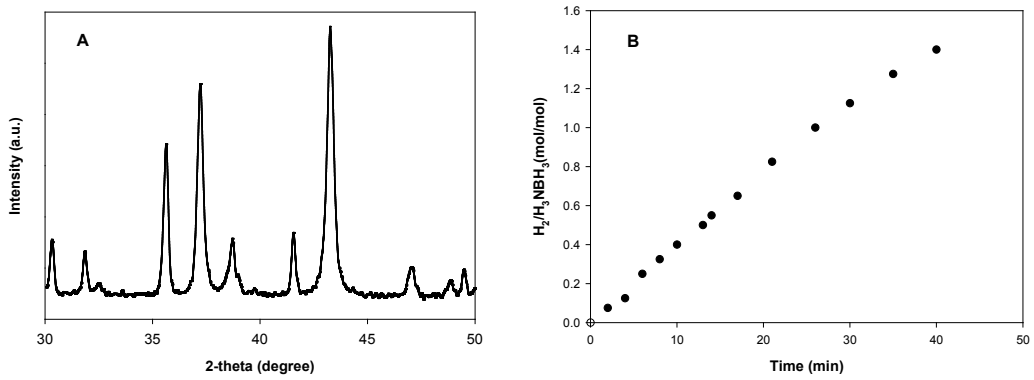


Fig. S10 (A) XRD pattern and (B) Time versus volume of hydrogen generated from hydrolysis of AB catalyzed by binary CuO-NiO oxides sample prepared using copper chloride precursor (H_3NBH_3=1.48mmol, catalyst=10mg, H_2O=10ml, T=25°C).

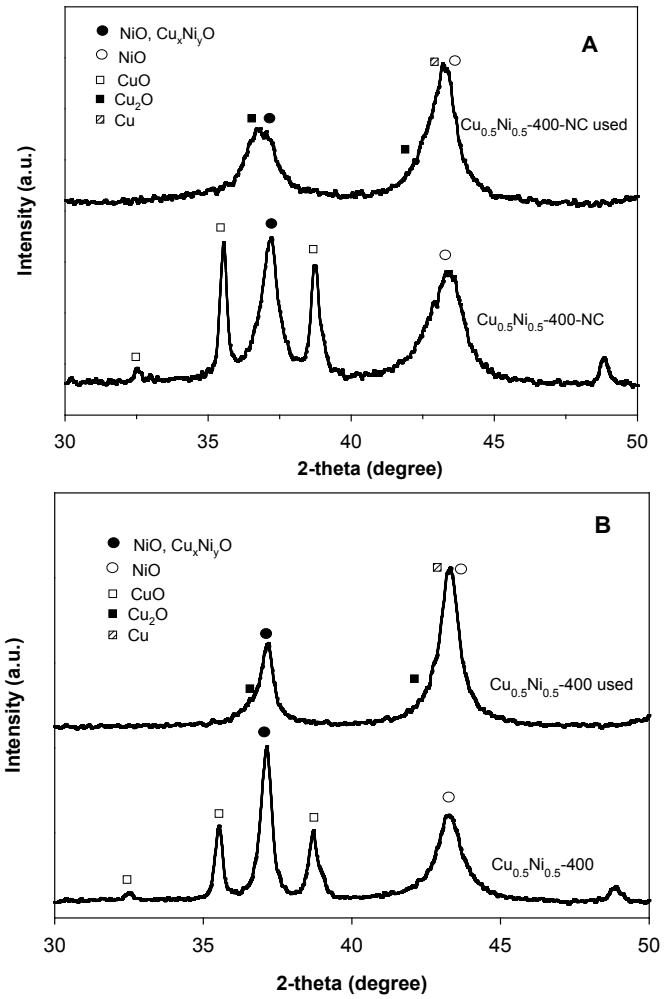
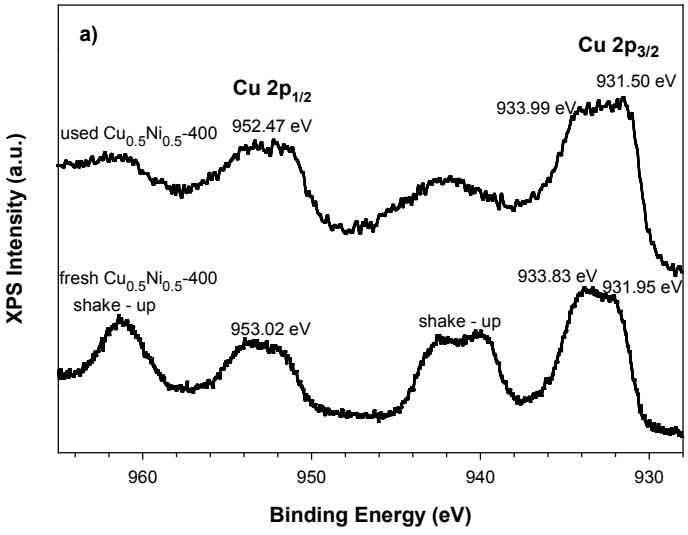


Fig. S11 Wide-angle XRD patterns of the fresh and spent catalysts: (A) nanocast material, (B) material by thermal decomposition of nitrate salts.



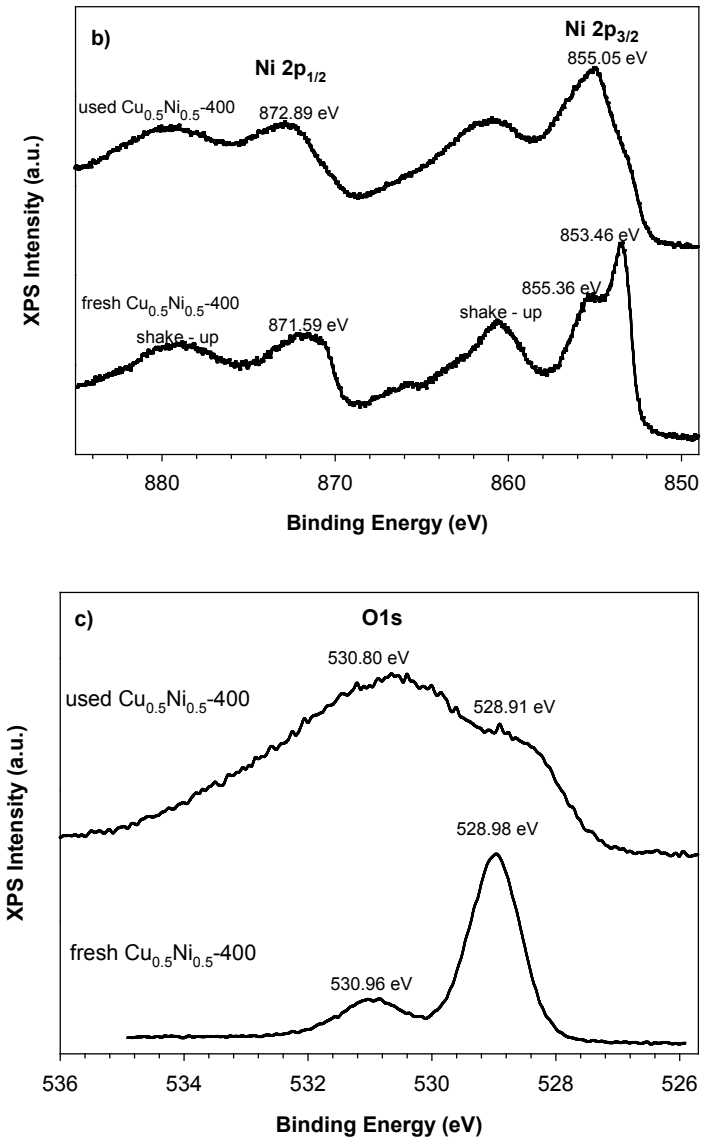


Fig. S12a-c XPS spectra of the fresh and used catalysts prepared via thermal decomposition (the spectra are normalized by their peak intensity and energy corrected for adventitious carbon at 284.8eV)

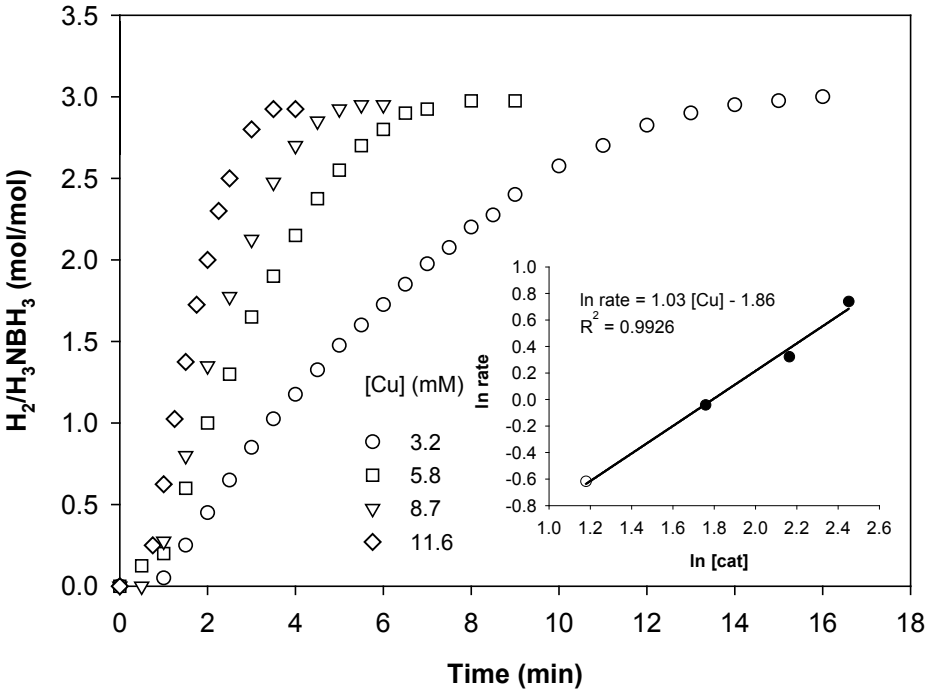


Fig. S13 Plot of time versus volume of hydrogen generated using $Cu_{0.5}Ni_{0.5}$-400 at different catalyst concentrations ([AB] = 148 mmol, T = 25°C). (Inset: ln [Cu] *vs* ln rate plot).

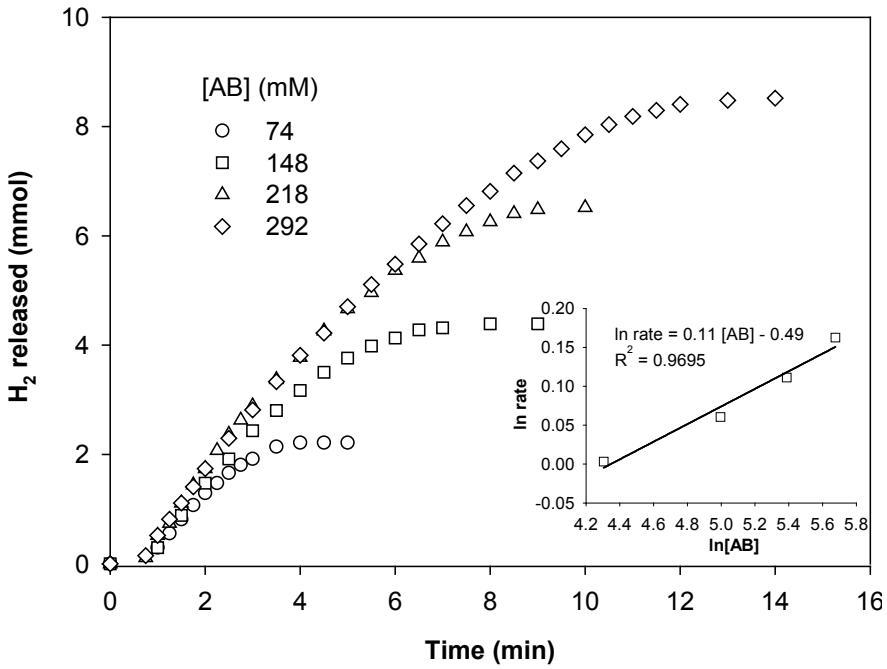


Fig. S14 Plot of time versus volume of hydrogen generated from the hydrolysis of AB catalyzed by $Cu_{0.5}Ni_{0.5}$-400 at different AB concentrations ([Cu] = 5.8 mmol, T = 25°C). (Inset: ln[AB] *vs* ln rate plot)

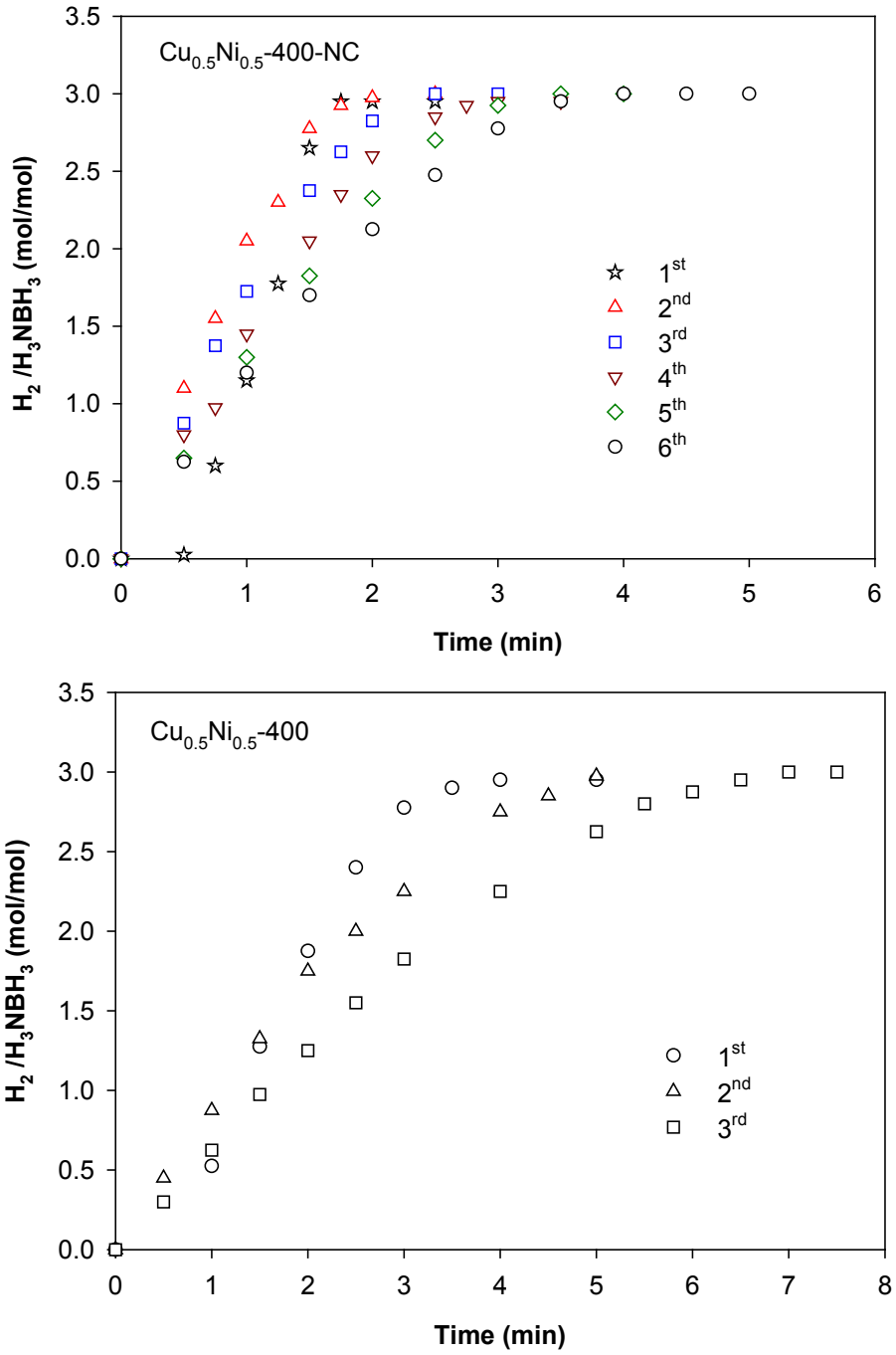


Fig. S15 Time versus volume of hydrogen generated from the hydrolysis of AB catalyzed by $Cu_{0.5}Ni_{0.5}$-400-NC and $Cu_{0.5}Ni_{0.5}$-400 over several cycles for reusability test. ([Cu] = 11.6mmol, [AB] = 148mmol, T = 25°C).

Table S1 Textural and structural parameters of the prepared samples derived from nitrogen physisorption measurements at -196°C and X-ray diffraction.

Samples	S_{BET} [a] (m^2g^{-1})	d spacing of NiO (111) plane[b]
$Cu_{0.1}Ni_{0.9}$-400	12	2.4119
$Cu_{0.25}Ni_{0.75}$-400	11	2.4145
$Cu_{0.5}Ni_{0.5}$-400	10	2.4208
$Cu_{0.75}Ni_{0.25}$-400	3	2.4143
$Cu_{0.9}Ni_{0.1}$-400	3	2.4113
$Cu_{0.5}Ni_{0.5}$-500	8	2.4233
$Cu_{0.5}Ni_{0.5}$-600	7	2.4145
$Cu_{0.5}Ni_{0.5}$-400-IM	12	2.4120
$Cu_{0.5}Ni_{0.5}$-400-NC	73	2.4162
CuO-400	3	-
NiO-400	28	2.4108

[a] S_{BET}, apparent BET specific surface area deduced from the isotherm analysis in the relative pressure range from 0.05 to 0.20; [b] Obtained from XRD.

Table S2 Nominal and actual molar Cu/Ni ratios of the prepared composite oxides.

Samples	Nominal Cu/Ni ratio	Actual Cu/Ni ratio in the bulk	Surface Cu/Ni ratio
$Cu_{0.25}Ni_{0.75}$-400	0.33	0.30	0.21
$Cu_{0.5}Ni_{0.5}$-400	1.00	0.89	0.23
$Cu_{0.75}Ni_{0.25}$-400	3.00	2.56	0.21
$Cu_{0.5}Ni_{0.5}$-400-NC*	1.00	0.85	0.55
$Cu_{0.5}Ni_{0.5}$-600	1.00	0.87	0.57

**The Si content determined by XPS is about 2.78 at% in the nanocast $Cu_{0.5}Ni_{0.5}$-400-NC sample.*