

## Supplementary Information

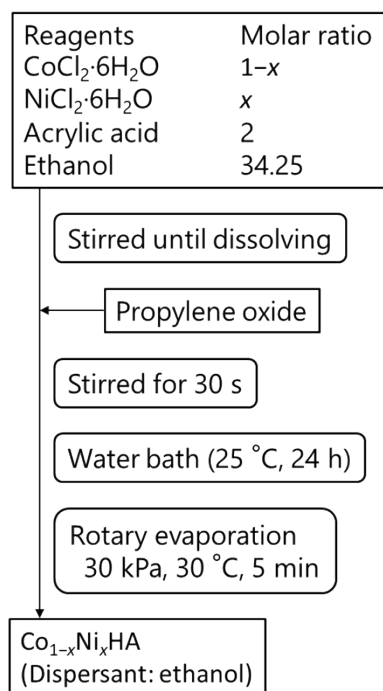
### **Thermal self-reduction of metal hydroxide salt monolayer nanoparticles leads formation of nanoparticulate and porous structured alloys.**

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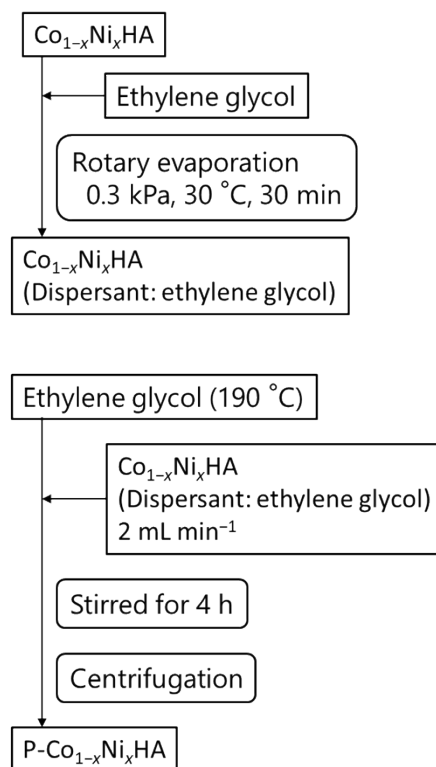
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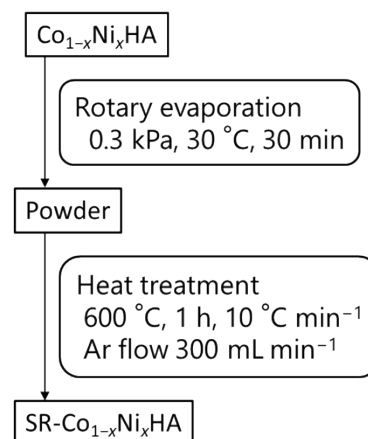
(a) Synthesis of dispersions of MHS monolayer nanoparticles



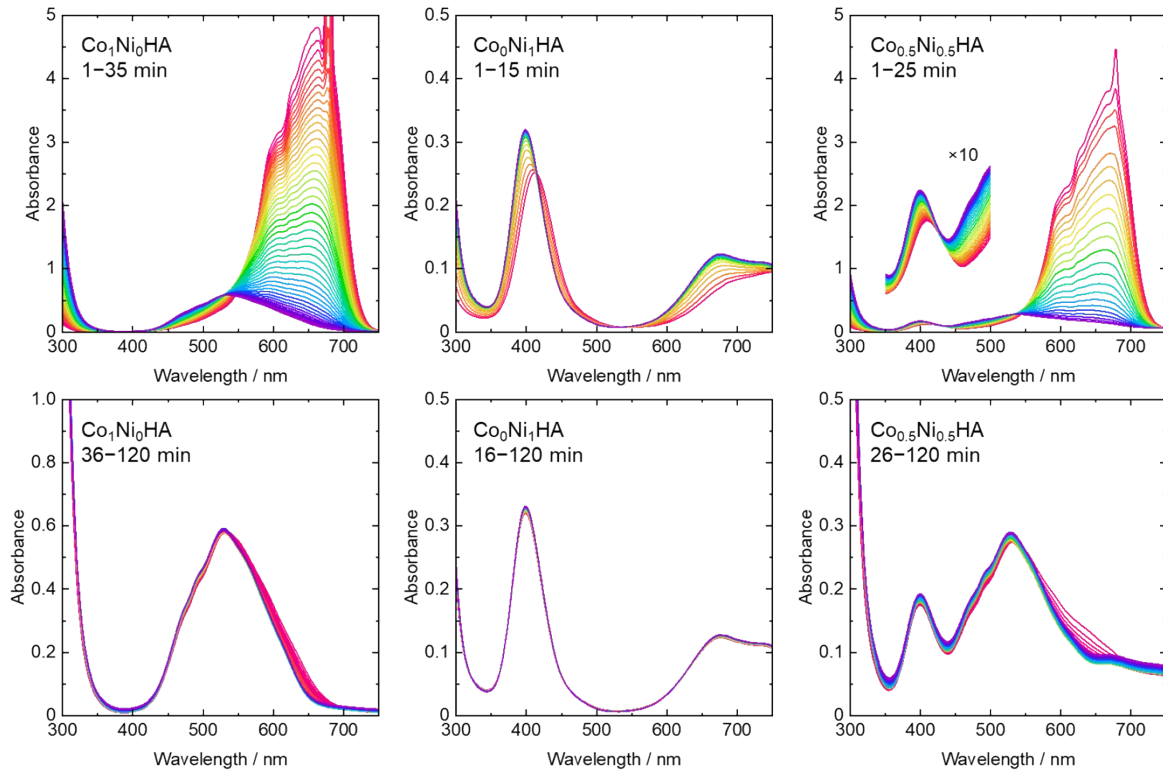
(b) Reduction of MHS nanoparticles through polyol process



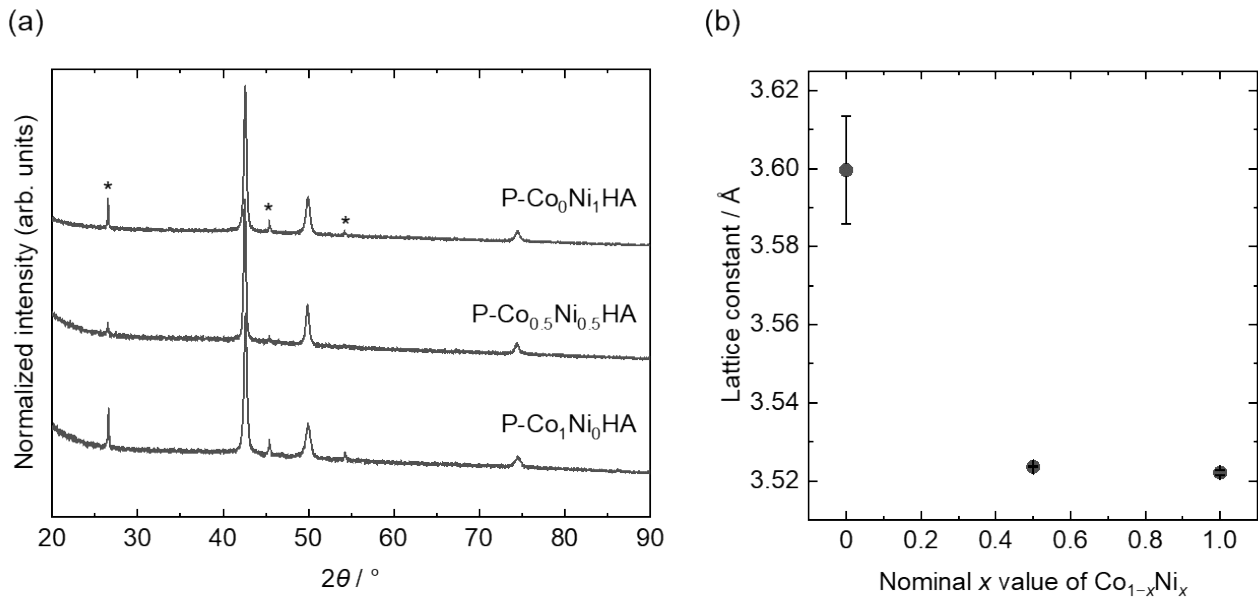
(c) Self-reduction of MHS nanoparticles



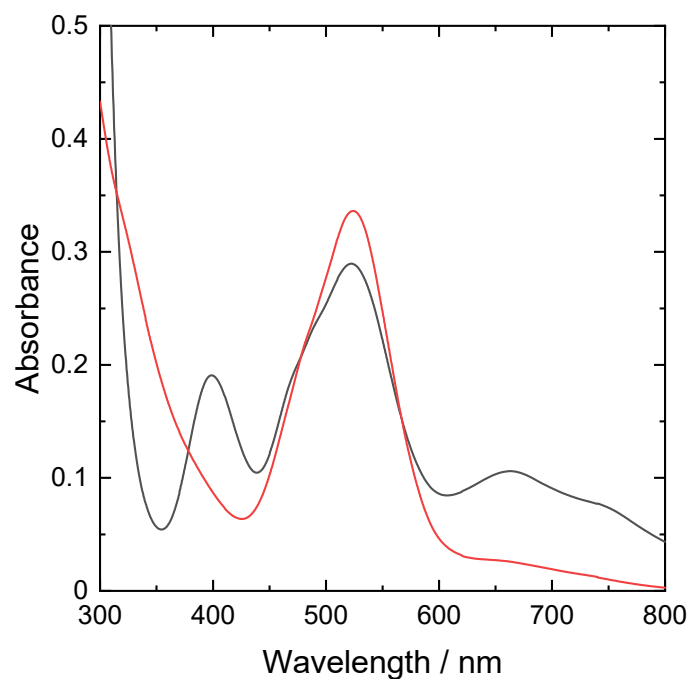
**Scheme S1** Synthetic steps of (a) dispersions of MHS nanoparticles, (b) reduction of MHS nanoparticles through polyol process, and (c) self-reduction of MHS nanoparticles.



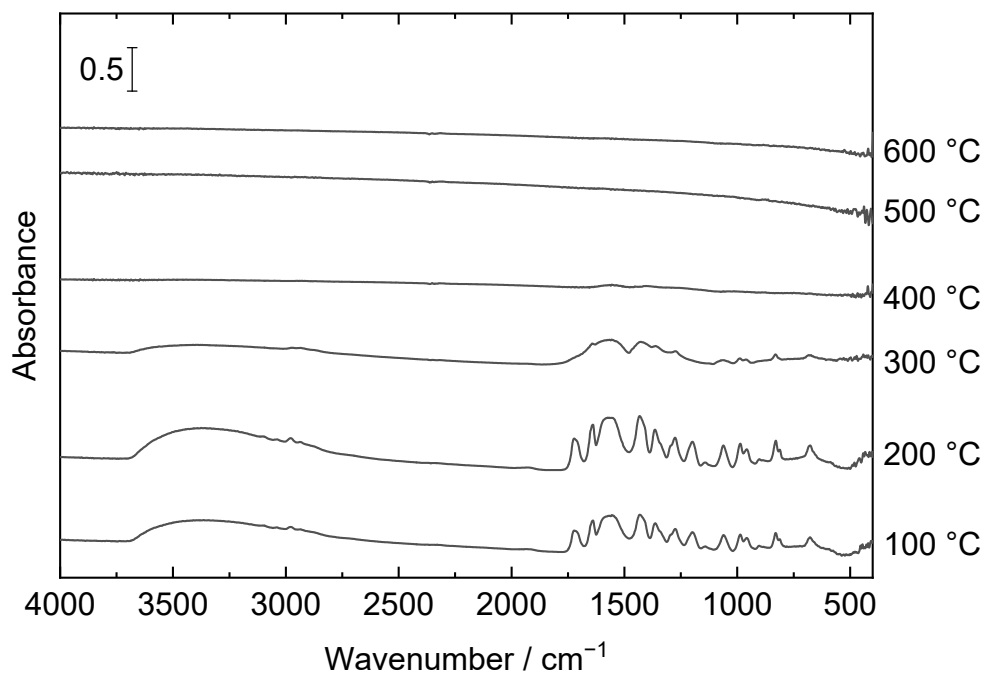
**Fig. S1** UV-Vis spectra of  $\text{Co}_1\text{Ni}_0\text{HA}$ ,  $\text{Co}_0\text{Ni}_1\text{HA}$ , and  $\text{Co}_{0.5}\text{Ni}_{0.5}\text{HA}$  after addition of PO (time resolution of 1 min).



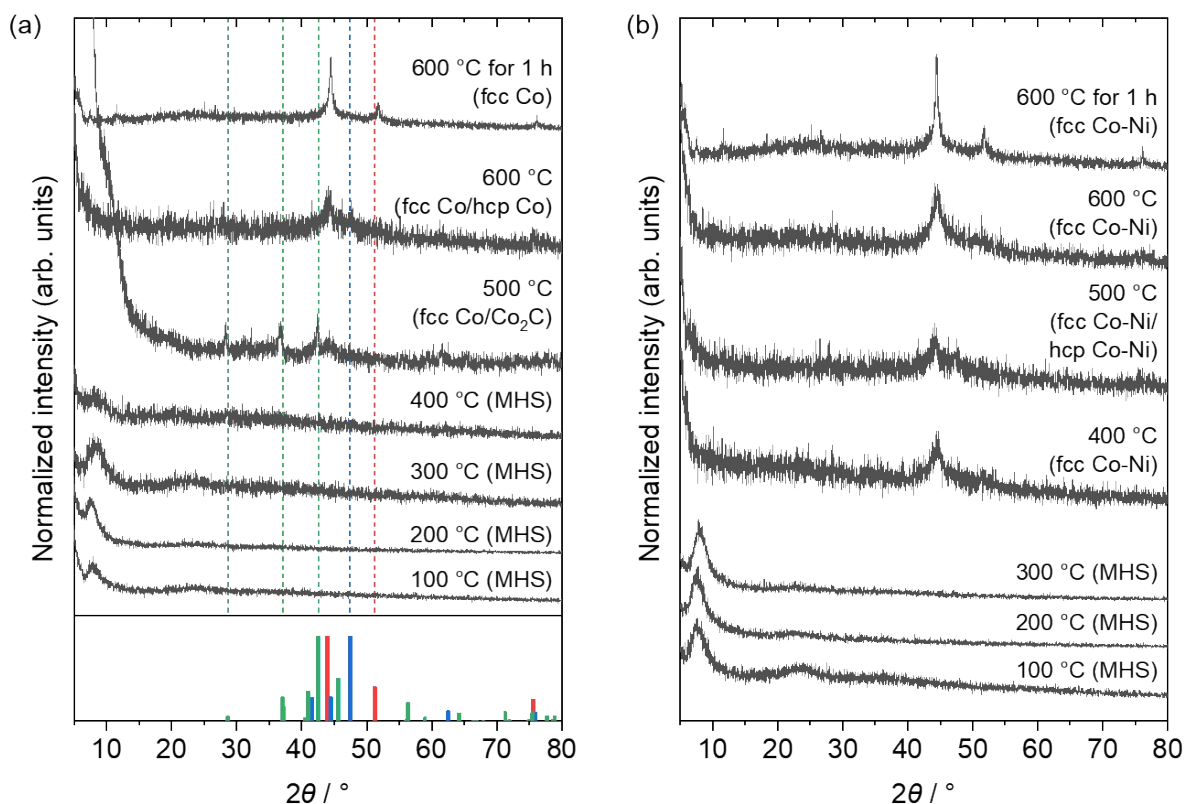
**Fig. S2** (a) XRD patterns and (b) calculated lattice constants of  $\text{P-Co}_1\text{Ni}_0\text{HA}$ ,  $\text{P-Co}_0\text{Ni}_1\text{HA}$ , and  $\text{P-Co}_{0.5}\text{Ni}_{0.5}\text{HA}$ . \* Si reference.



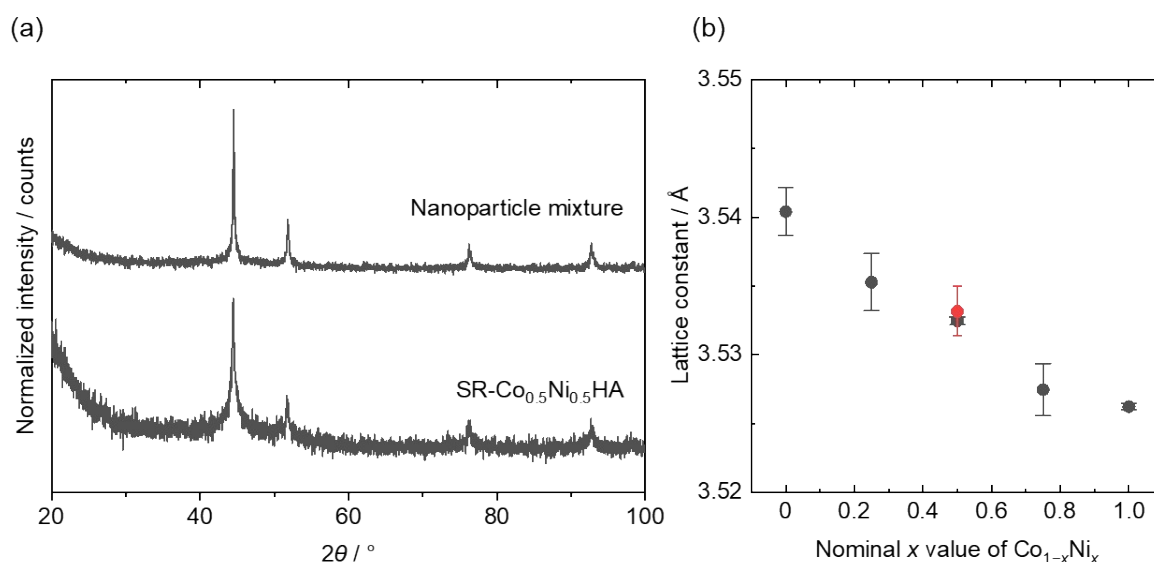
**Fig. S3** UV-Vis spectra of patterns of  $\text{Co}_{0.5}\text{Ni}_{0.5}\text{HA}$  dispersed solution before (black) and after (red) polyol process.



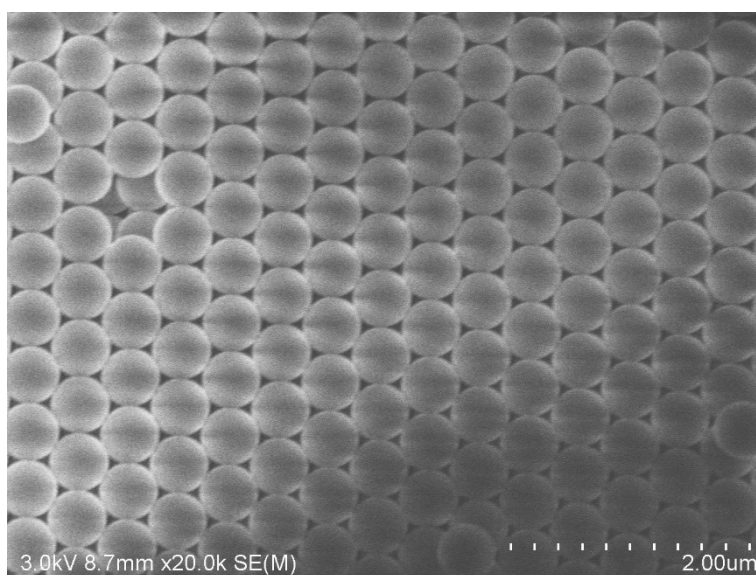
**Fig. S4** IR spectra of  $\text{Co}_0\text{Ni}_1\text{HA}$  after heat-treatment at 100–600 °C without holding the temperature.



**Fig. S5** XRD patterns of (a)  $\text{Co}_0\text{Ni}_1\text{HA}$  and (b)  $\text{Co}_{0.5}\text{Ni}_{0.5}\text{HA}$  after heat-treatment at 100–600 °C without holding the temperature. The red, blue, and green bars in (a) are fcc Co (JCPDS #15-0806), hcp Co (#89-7373), and  $\text{Co}_2\text{C}$  (# 65-8206).



**Fig. S6** (a) XRD patterns of  $\text{SR-Co}_{0.5}\text{Ni}_{0.5}\text{HA}$  and heat-treated nanoparticle mixture of  $\text{Co}_1\text{Ni}_0\text{HA}$  and  $\text{Co}_0\text{Ni}_1\text{HA}$ . (b) Lattice constants of  $\text{SR-Co}_{1-x}\text{Ni}_x\text{HA}$  ( $x = 0, 0.25, 0.5, 0.75, 1$ ) (black) and heat-treated nanoparticle mixture of  $\text{Co}_1\text{Ni}_0\text{HA}$  and  $\text{Co}_0\text{Ni}_1\text{HA}$  (red).



**Fig. S7** SEM image of polystyrene microparticle template.