

# Nanostructured Hybrid NiFeOOH/CNT Electrocatalysts for Oxygen Evolution Reaction with Low Overpotential

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Control sample, NiFeOOH-5, was prepared with KOH or NaOH as the alkaline source. Though the materials possess LDH structure, the TEM images show that these control samples trend to aggregate together for the formation of micrometer sized particles (Fig. S1 and S2). This may be related with the fast reaction rate using KOH or NaOH as alkaline source. The employment of TMAOH which could release OH<sup>-</sup> slowly is beneficial for the formation of nanostructured material.

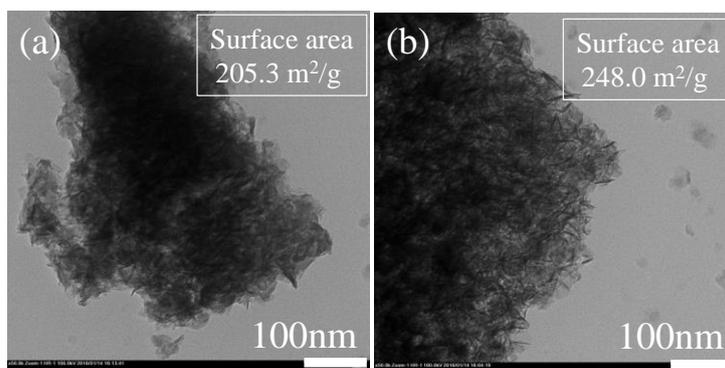


Fig. S1. TEM images of NiFeOOH-5 obtained using other alkali source (a) KOH (b) NaOH instead of TMAOH in the initial mixture.

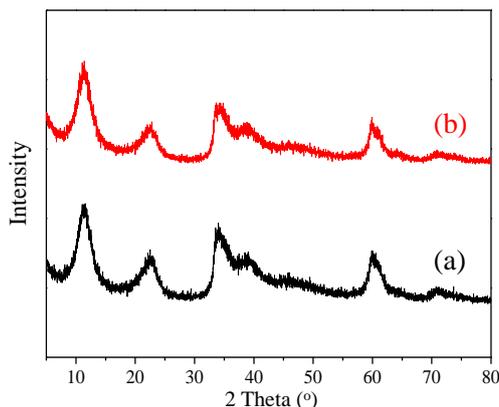


Fig. S2. XRD pattern of NiFeOOH-5 obtained using other alkali source (a) KOH (b) NaOH instead of TMAOH in the initial mixture.

Another control sample, NiCoOOH-5, was also prepared without the addition of H<sub>2</sub>O<sub>2</sub>. The X-ray diffraction (XRD) characterization shows that LDH structure cannot be obtained for NiCoOOH-5 without using H<sub>2</sub>O<sub>2</sub> in the synthetic system and the particle size is very large (Fig. S3). The above results show that employment of TMAOH and H<sub>2</sub>O<sub>2</sub> is quite important for the formation of nanostructured LDH under mild reaction conditions.

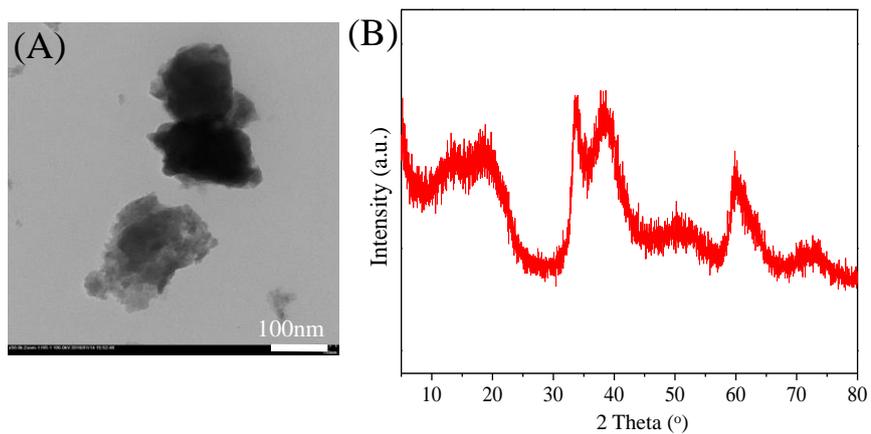


Fig. S3. (A) TEM images and (B) XRD pattern of NiCoOOH-5 synthesized without  $\text{H}_2\text{O}_2$  in the initial mixture.

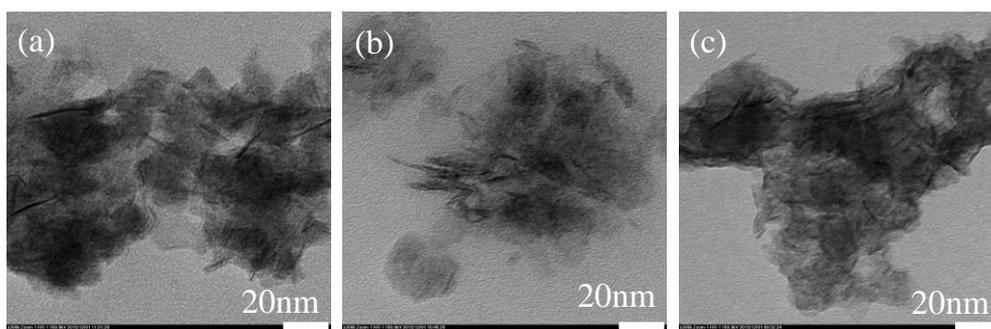


Fig. S4. TEM images of (a) NiCrOOH-8, (b) NiFeOOH-5, (c) NiCoOOH-5.

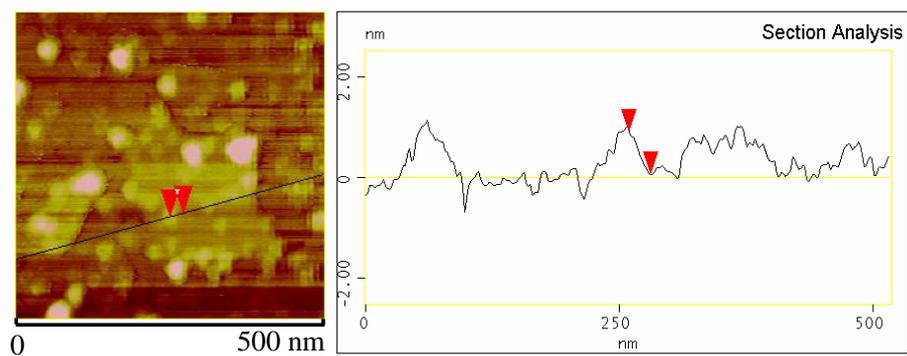


Fig. S5. Atomic force microscopy (AFM) image and height profile of single NiFeOOH-5.

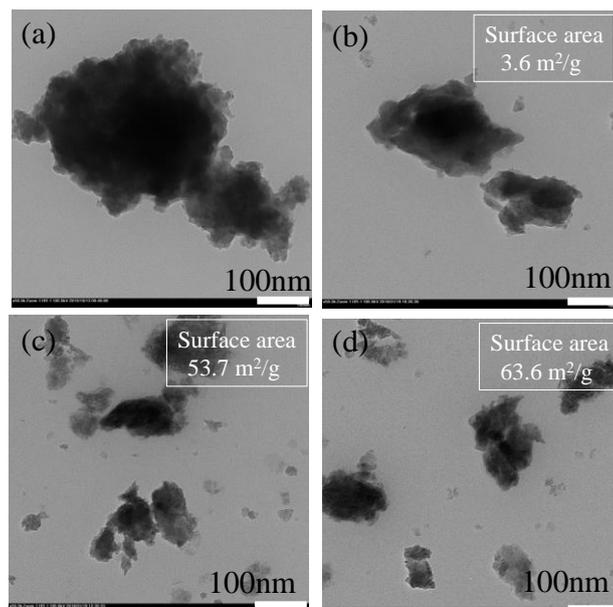


Fig. S6. TEM images of NiFeOOH-5 obtained using variable reaction durations (a) 25 min, (b) 4 h, (c) 8 h, (d) 12 h.

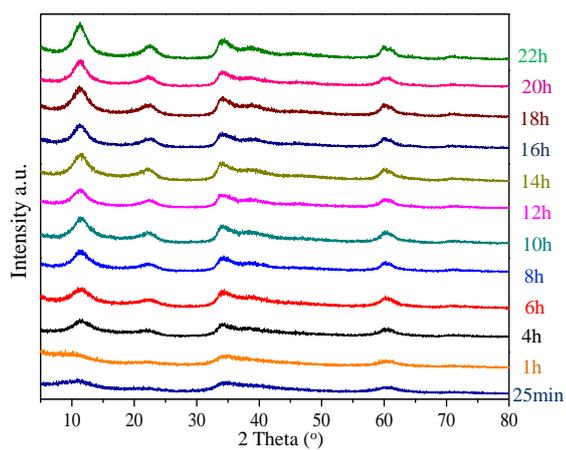


Fig. S7. XRD patterns of NiFeOOH-5 obtained using variable reaction durations.

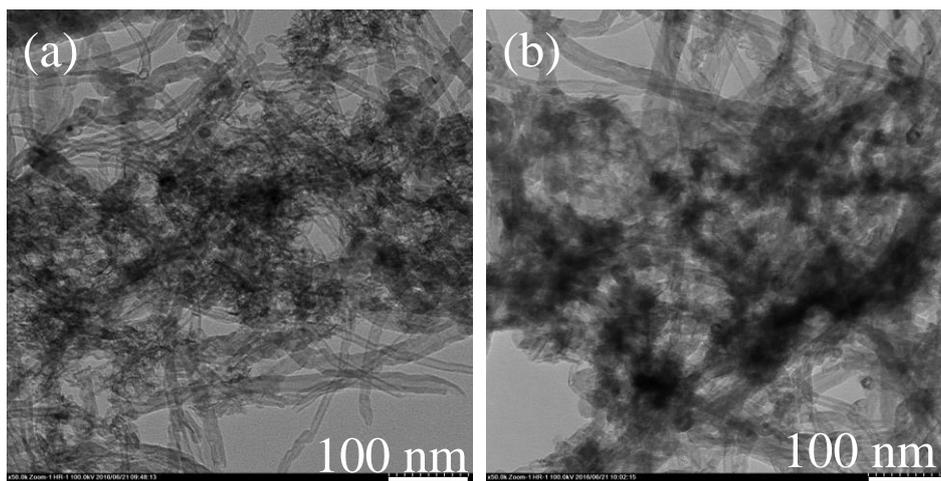


Fig. S8. TEM images of (a) NiCoOOH/CNT-48% and (b) NiCrOOH/CNT-48%.

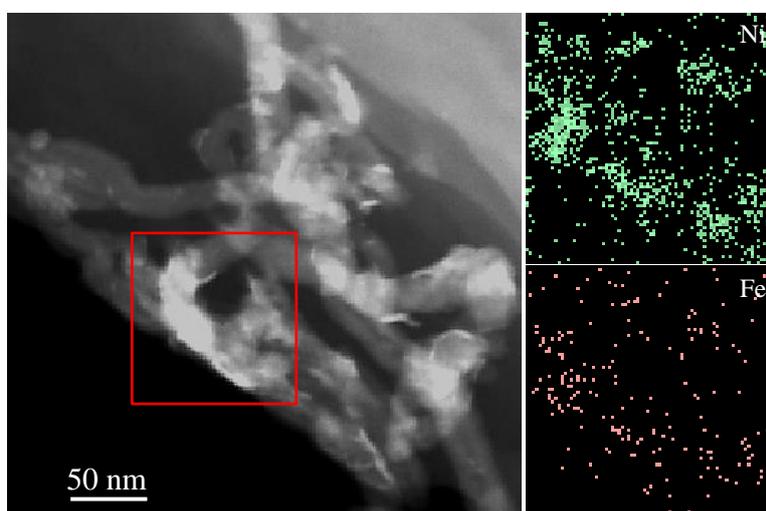


Fig. S9. Elemental mapping of Ni and Fe in NiFeOOH/CNT-47%.

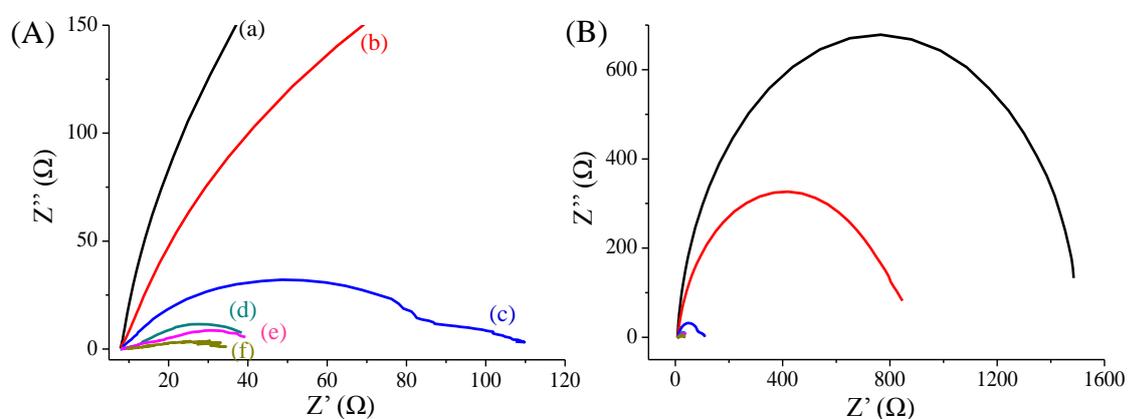


Fig. S10. EIS spectra of (a) NiCrOOH, (b) NiCoOOH, (c) NiFeOOH, (d) NiCrOOH-CNT-48%, (e) NiCoOOH/CNT-48% and (f) NiFeOOH/CNT-47%, (A) is the enlarged part of (B).

The EIS measurement was carried out from 100 MHz to 1 Hz at overpotential of 387 mV. The charge transfer resistance of the materials is significantly decreased after hybridizing with CNT.