Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.
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# Supporting Information

## Experimental details

The reagents were all purchased from Sinopharm Chemical Reagent Co., Ltd. (shanghai, China) and used without further purification.

**Synthesis of Zn-Co-LDH nanoparticle:** To synthesize the Zn-Co-LDH nanosheets, 0.4462 g of zinc nitrate hexahydrate (1.5 mmol), 0.8731 g of cobalt nitrate hexahydrate (3 mmol) and 1.441 g of urea (24 mmol) were dissolved in 80 mL deionized water. The formed solution was transferred into a 350 mL homemade round-bottomed flask and treated under microwave irradiation in a XH-MC-1 microwave reactor at 900 W. After 20 min of microwave treatment, the reaction was cooled to room temperature naturally. The product was filtered, washed thoroughly with distilled water and absolute ethanol, and dried at 60 ℃ overnight.

**Synthesis of $ZnCo_2O_4$:** $ZnCo_2O_4$ was synthesized via a simple co-precipitation at room temperature. First, 0.4462 g of zinc nitrate hexahydrate (1.5 mmol), 0.8731 g of cobalt nitrate hexahydrate (3 mmol) were mixed with deionized water (80mL) at room temperature under vigorous stirring. The solution was stirred for 8 h .The pH of the solution was then adjusted to 9.0 with aqueous NaOH solution (1 mol/L) at room temperature. After stirring for four more hours, a solid product was formed. After being retrieved by centrifugation and washed several times with distilled water and absolute ethanol, the resulting product was dried in vacuum at 80 ℃ for 12 h. The final $ZnCo_2O_4$ was obtained

through heat treatment of the intermediate precursor at 300 ℃ for 2 h.

**Synthesis of $Co_3O_4$:** To synthesize $Co_3O_4$, 1 g of $Co(NO_3)_2 \cdot 6H_2O$ was ground for 4 hours at room temperature. The final $Co_3O_4$ was obtained through heat treatment of the intermediate precursor at 450 ℃ for 2 h.

**Electrochemical Measurements.** The electrocatalytic properties of the materials were studied with a three-electrode system. A saturated calomel electrode (SCE) and a platinum wire (diameter: 0.25mm) were used as the reference and the counter electrodes, respectively. A glassy carbon electrode of 3 mm in diameter was polished sequentially with 0.3 and 0.05 μm Alumina powder, sonicated for 5 min each in 50 wt% nitric acid, ethanol and de-ionized water, and then dried in air. 2 mg of the catalyst and 40 μL of Nafion solution (5 wt%) were dispersed in 360 μL ethanol by at least 30 min sonication to form a homogeneous ink. Then, 3 μL of the as-prepared catalyst ink was loaded on the clean glassy carbon electrode (GCE), with a loading amount of about 0.212mg/cm$^2$.

**Turnover frequency (TOF) calculation of the catalysts**: The TOF Value is calculated from the equation:

$$TOF = \frac{J \times A}{4 \times F \times m}$$

J is the current density at overpotential of 0.3V in A/cm$^2$.A is the area of the carbon fiber paper electrode.F is the faraday constant (a value of 96485 C/mol). M is the number of

moles of the active materials that are deposited onto the glassy carbon electrode (GCE).

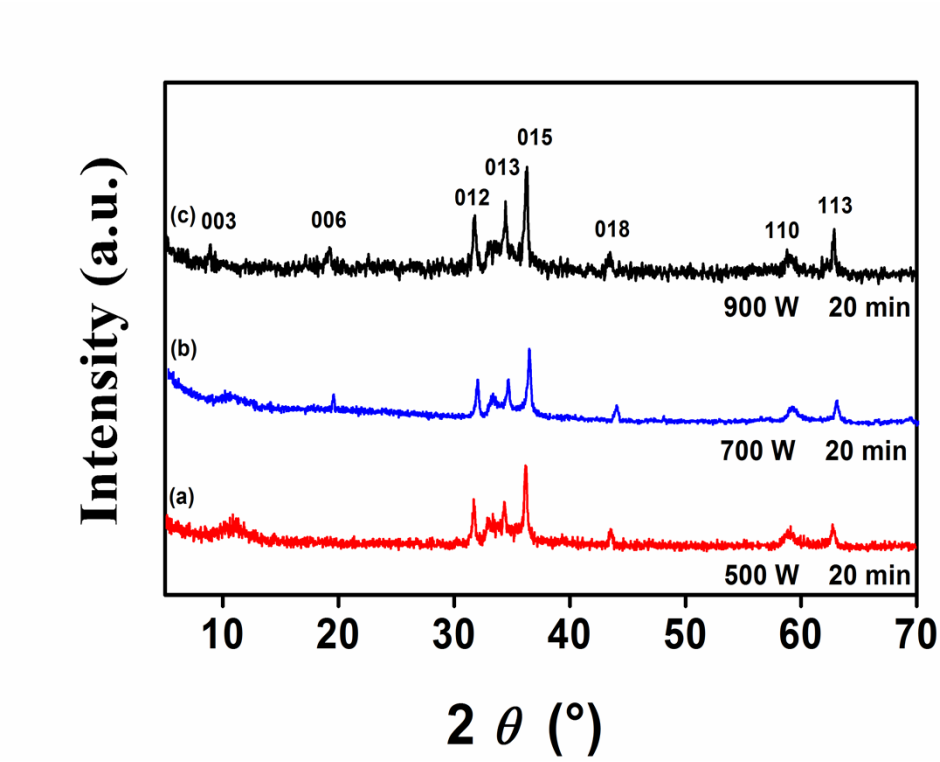


**Fig. S1** XRD patterns of the samples obtained under different microwave power at 20 min: (a) 500W, (b) 700 W, (c) 900W.

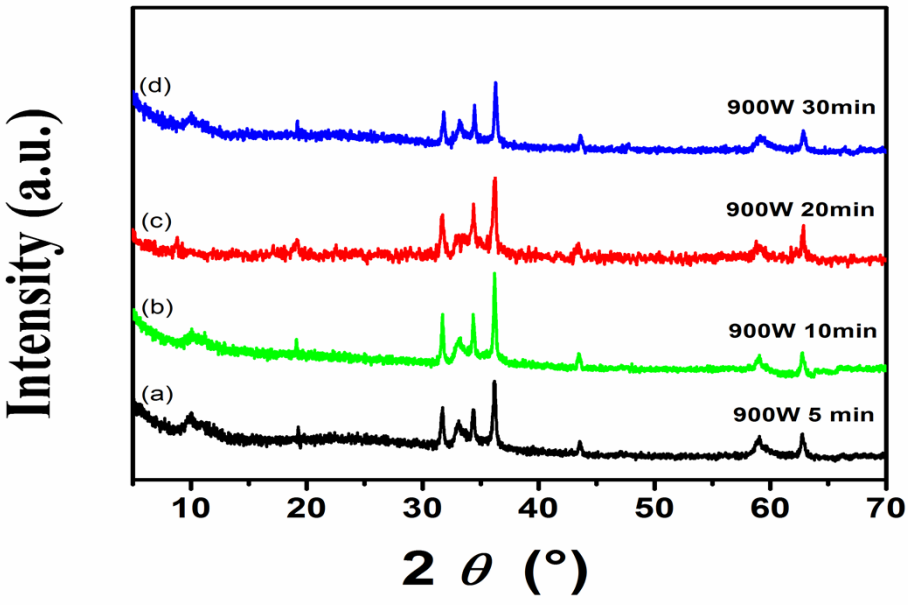


**Fig. S2** XRD patterns of the samples obtained after different microwave irradiation time at 900W: (a) 5 min, (b) 10 min, (c) 20 min, and (d) 30 min.

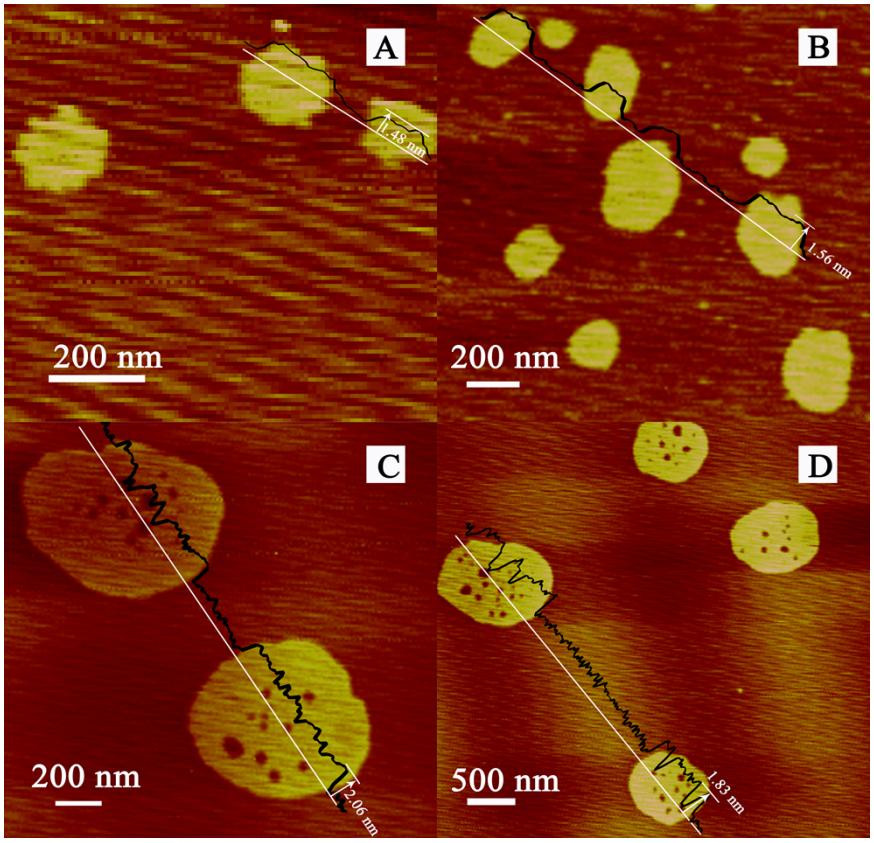


**Fig. S3** AFM images of the samples obtained after different microwave irradiation time at 900W: (a) 5 min, (b) 10 min, (c) 20 min, (d) 30 min.

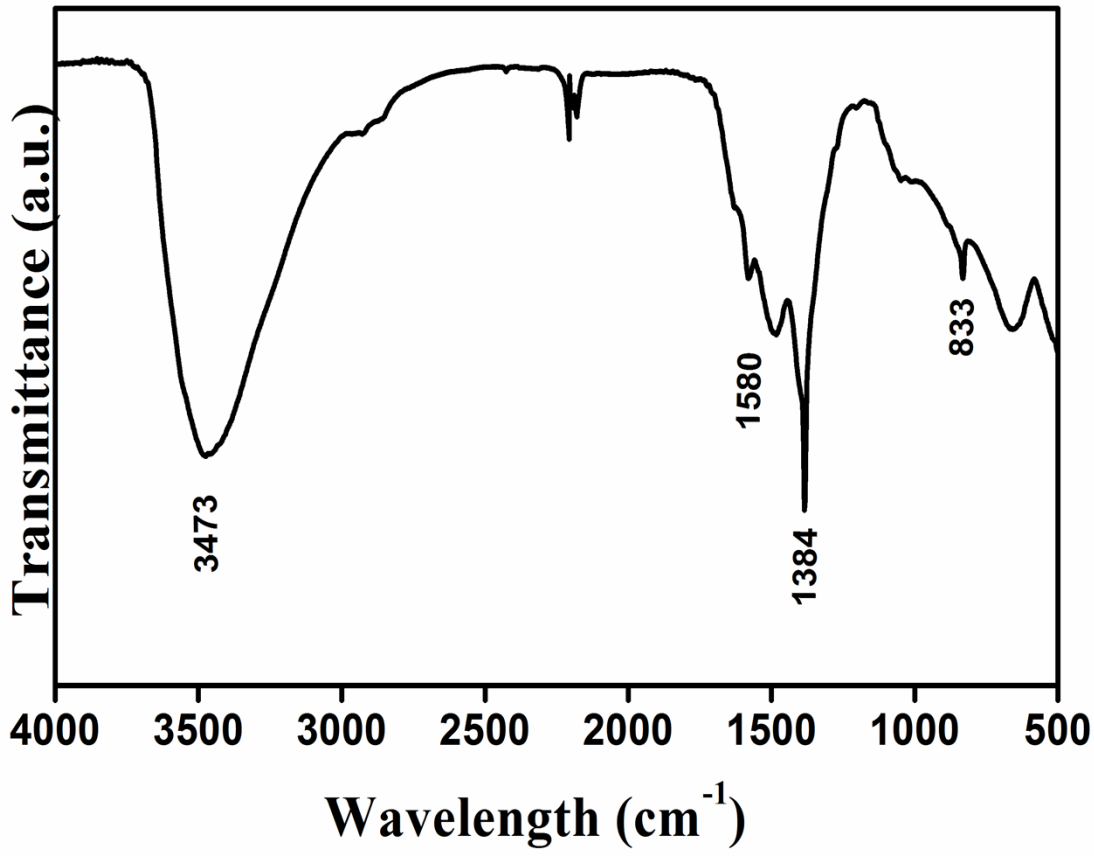


**Fig.S4** FTIR spectrum of the Zn-Co-LDH nanosheets

The FTIR spectrum of Zn-Co-LDH nanosheets shows four obvious absorption bands in the region from 500 to 4000 $cm^{-1}$. A strong and broad IR absorption due to stretching vibrations of OH groups and a relatively weak absorption due to bending vibration of OH groups are observed at ~3473 and ~1580 $cm^{-1}$, respectively; this indicates that a large number of OH groups and water molecules exist in the Zn-Co-LDH material. The other two absorption bands at ~1348 and ~833 $cm^{-1}$ are characteristic of $CO_3^{2-}$.

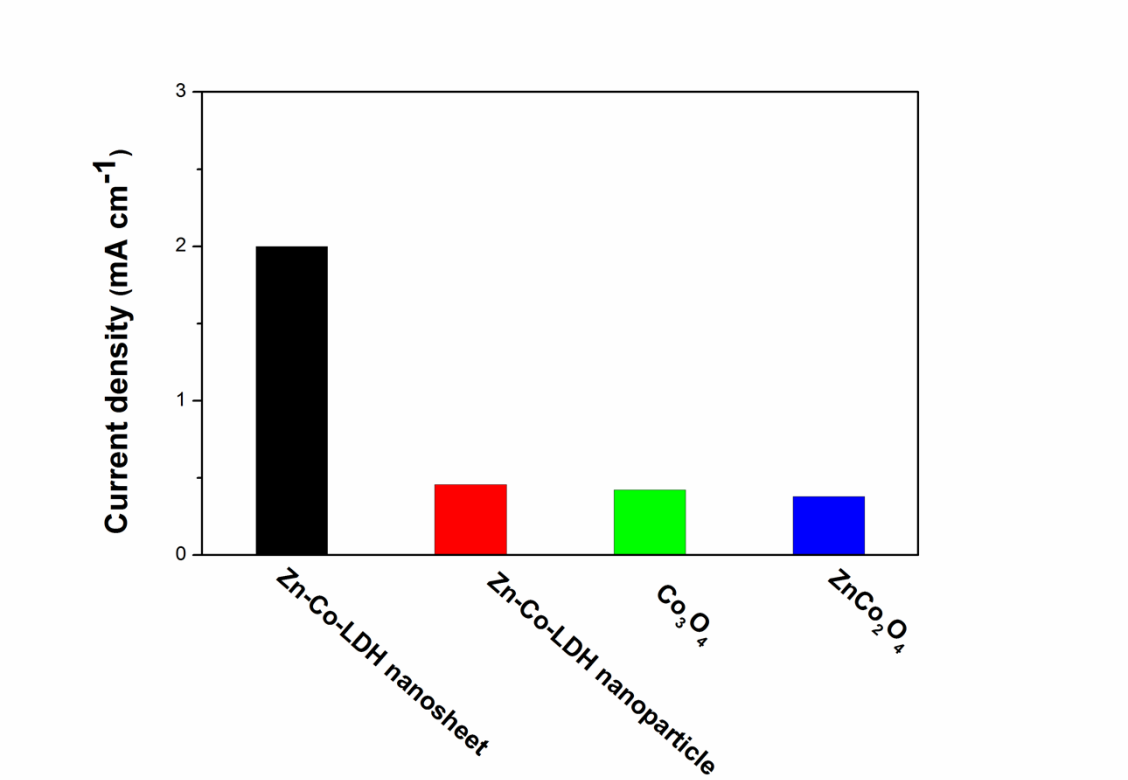


**Fig. S5** Current densities of a series of materials at a fixed overpotential of 0.375 V (0.6 V vs. SCE).

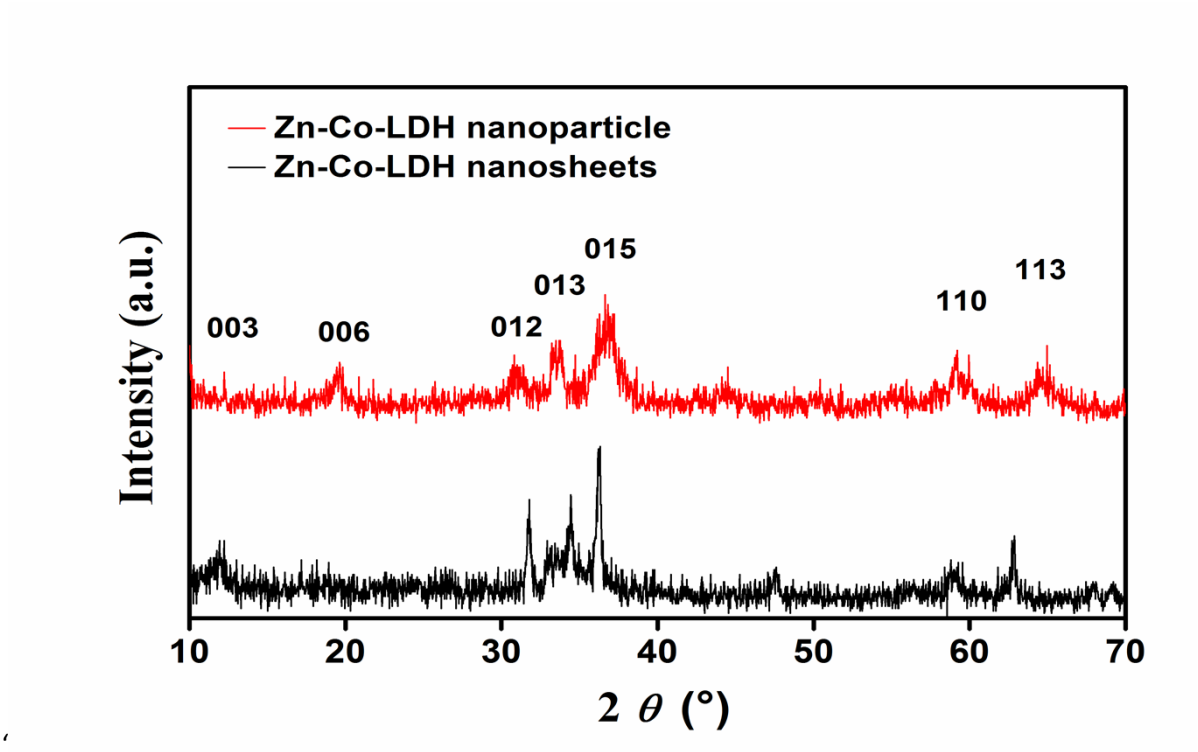


**Fig. S6** XRD pattern of Zn-Co-LDH nanoparticles and nanosheets.

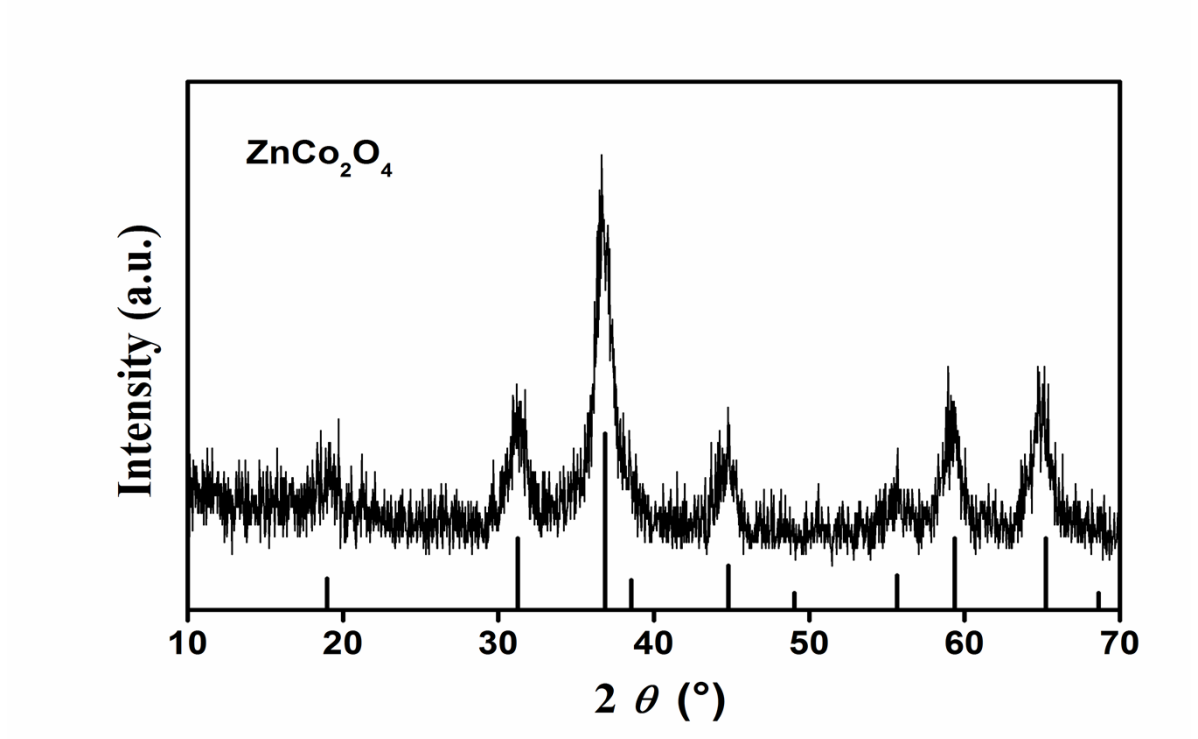


**Fig. S7** XRD pattern of $ZnCo_2O_4$

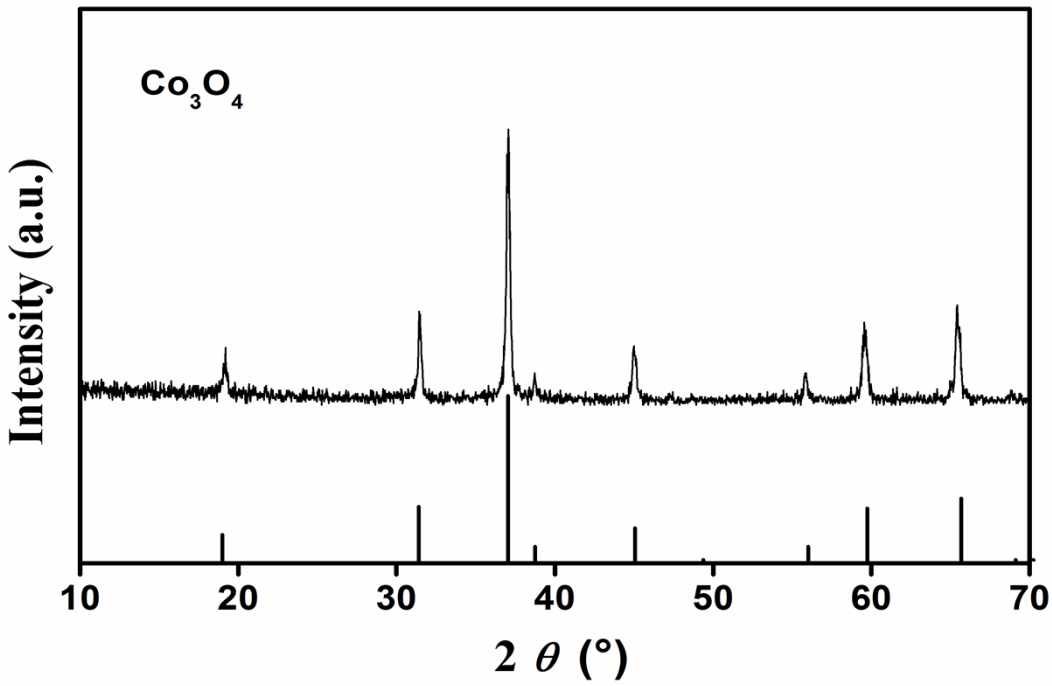


**Fig. S8** XRD pattern of $Co_3O_4$

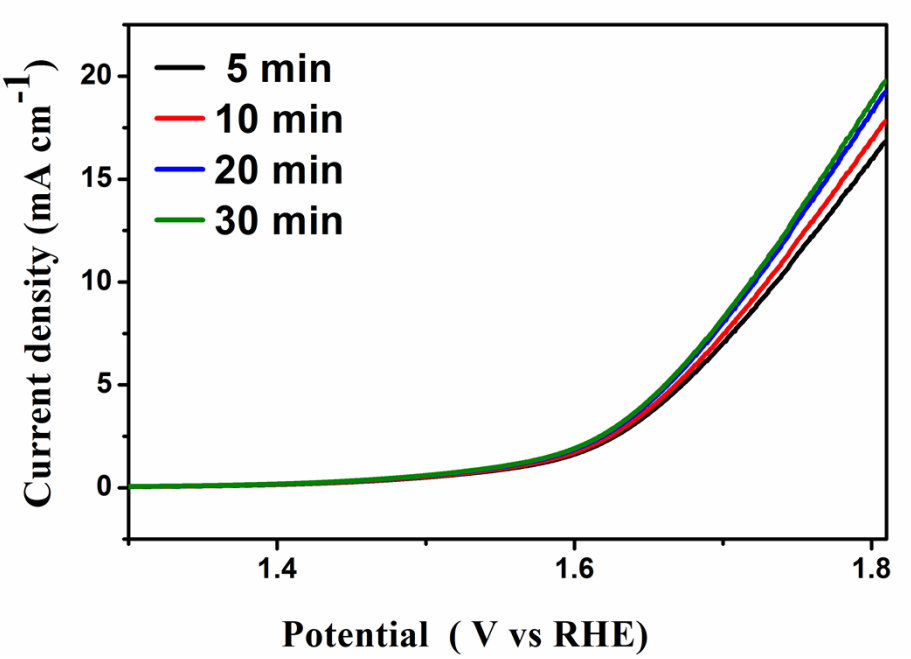


**Fig. S9** Linear sweep voltammograms of samples obtained at different reaction time in KOH (pH 13.0). The scanning rate was 5 mV s-1.

We also investigated the OER activity of samples obtained at different reaction time. Form Fig. S9, the OER activity of the sample obtained at 20 min was almost same as that of sample obtained at 30 min, which was much better than of the samples obtained at 5 min or

10 min.

According to the morphology analysis results and OER activities of different samples, the reaction conditions of microwave-assisted approach were optimized at 20 min of microwave irradiation and 900 W of microwave power.

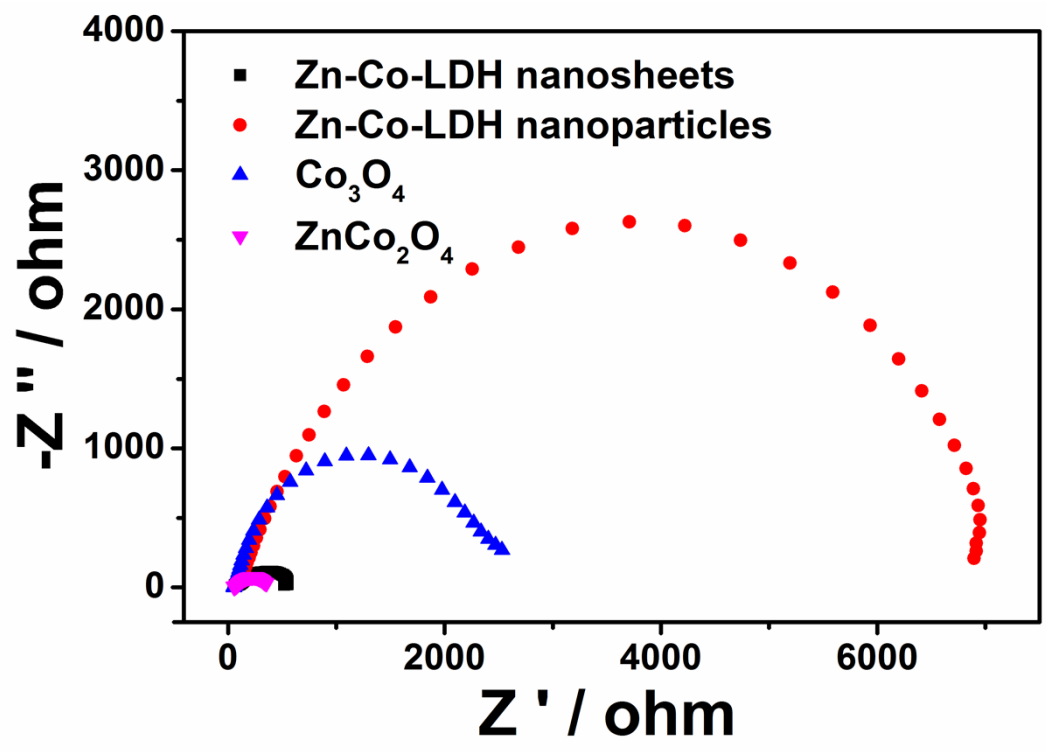


**Fig. S10** Electrochemical impedance spectra (EIS) of ZnCo-LDH nanosheets and Zn-Co-LDH nanoparticles at a potential of 0.6 V (vs. SCE) in 0.1 M KOH solution.