Supporting information

Electrochemically induced surface modifications of mesoporous spinels $(Co_3O_{4-\delta}, MnCo_2O_{4-\delta}, NiCo_2O_{4-\delta})$ as the origin of the OER activity and stability in alkaline medium

I. Abidat ^{1,2}, N. Bouchenafa-Saib ², A. Habrioux ^{*,1}, C. Comminges ^{*,1}, C. Canaff ¹, J. Rousseau ¹, T.W. Napporn ¹, D. Dambournet ^{3,4}, O. Borkiewicz ⁵, B. Kokoh ¹.

Author information : aurelien.habrioux@univ-poitiers.fr; clement.comminges@univ-poitiers.fr

1. Université de Poitiers, IC2MP CNRS UMR 7285,4 rue Michel Brunet B27, TSA51106, 86073Poitiers, Cedex 9, France

2. Laboratoire d'Analyse fonctionnelle des procédés chimiques, Faculté de Technologie, Université Blida 1, BP 270 Route de Soumâa Blida, Algeria.

3 Sorbonne Universités, UPMC Univ Paris 06, UMR 8234, PHENIX, F-75005, Paris, France. 4 CNRS, UMR 8234, PHENIX, F-75005, Paris, France.

5 X-ray Science Division, Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439, United States.

Synthesis of mesoporous NiO

0.50 g of SBA-15 were dispersed in 5 mL of ethanol containing nickel nitrate $Ni(NO_3)_2.6H_2O$ (99%, Merck) at a concentration of 0.7 mol L⁻¹. The solution was then stirred for 1 h at room temperature before evaporation of ethanol at 50 °C. The composite was calcined at 300 °C for 6 h in order to decompose nitrate species. A second impregnation was carried under the same experimental conditions. The impregnation step was followed by calcination at 450 °C for 6 h under air. The silica template was then removed using 2 mol L⁻¹ NaOH aqueous solution. Mesoporous NiO powder was then filtered and washed several times with ultra-pure water. The powder was finally dried at 50 °C during 24 h.



Figure S1. (A) Low angle XRD pattern of SBA-15 template; (B) N₂ adsorption-desorption isotherm for SAB-15 template.



Figure S2. PDFs of $Co_3O_{4-\delta}$, $MnCo_2O_{4-\delta}$, $NiCo_2O_{4-\delta}$. In the case of $MnCo_2O_{4-\delta}$, the features in the PDF tend to fall off at around 40 Å which primary indicates a lower particle size as compared to $Co_3O_{4-\delta}$ and $NiCo_2O_{4-\delta}$.



Figure S3. Voltammogram recorded in supporting electrolyte (0.1 M KOH) under N_2 atmosphere at a scan rate of 50 mV s⁻¹ with NiO at room temperature



Figure S4. Overlay of EIS bode plots for $Co_3O_{4-\delta}$ (red) and $NiCo_2O_{4-\delta}$ (blue) at 1.800 V vs. RHE. Solid lines are fitting results.