Supplementary Materials

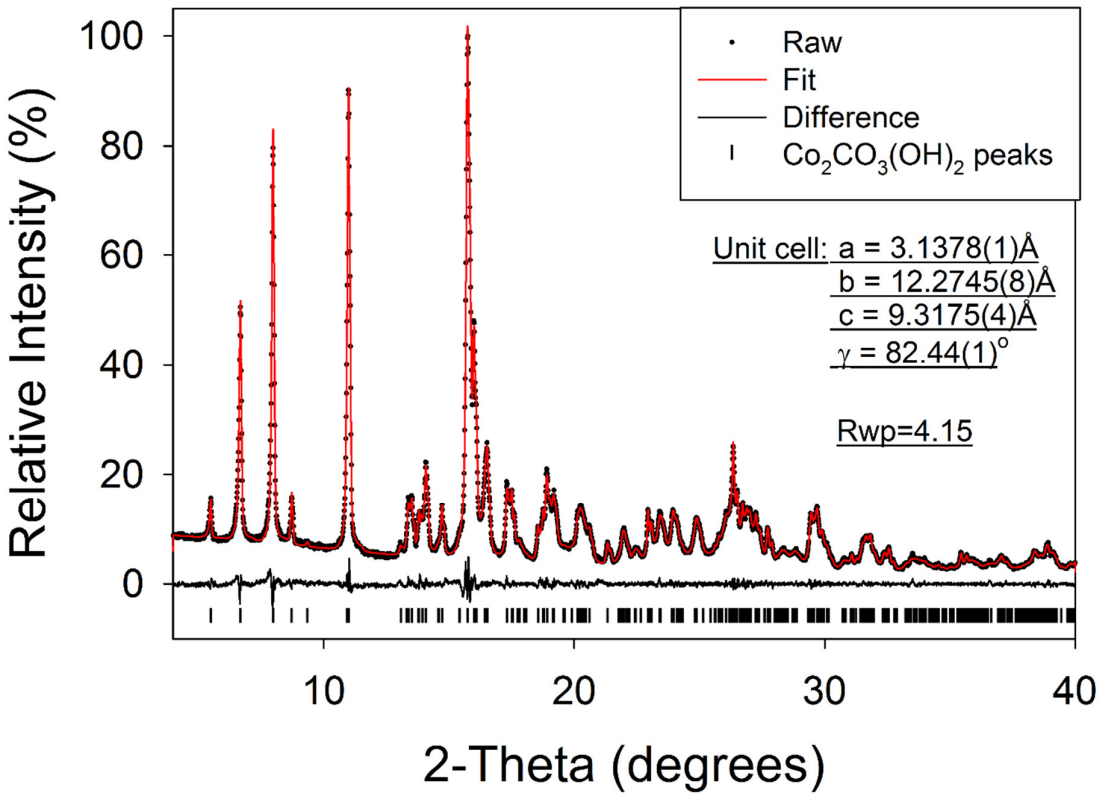

Figure S1. Fitted x-ray diffractogram for the $Co_2(CO_3)OH_2$ intermediate phase. Data were collected in capillary transmission mode using a Mo-K_α source, and were fitted using a structureless "Pawley" type approach. Space group and unit cell values were taken from González-López et. al. (J González-López, J.K. Cockroft, A. Fernández-González, A. Jiménez, R. Grau-Crespo. Acta Cryst., **2017**, B73, 868-873).

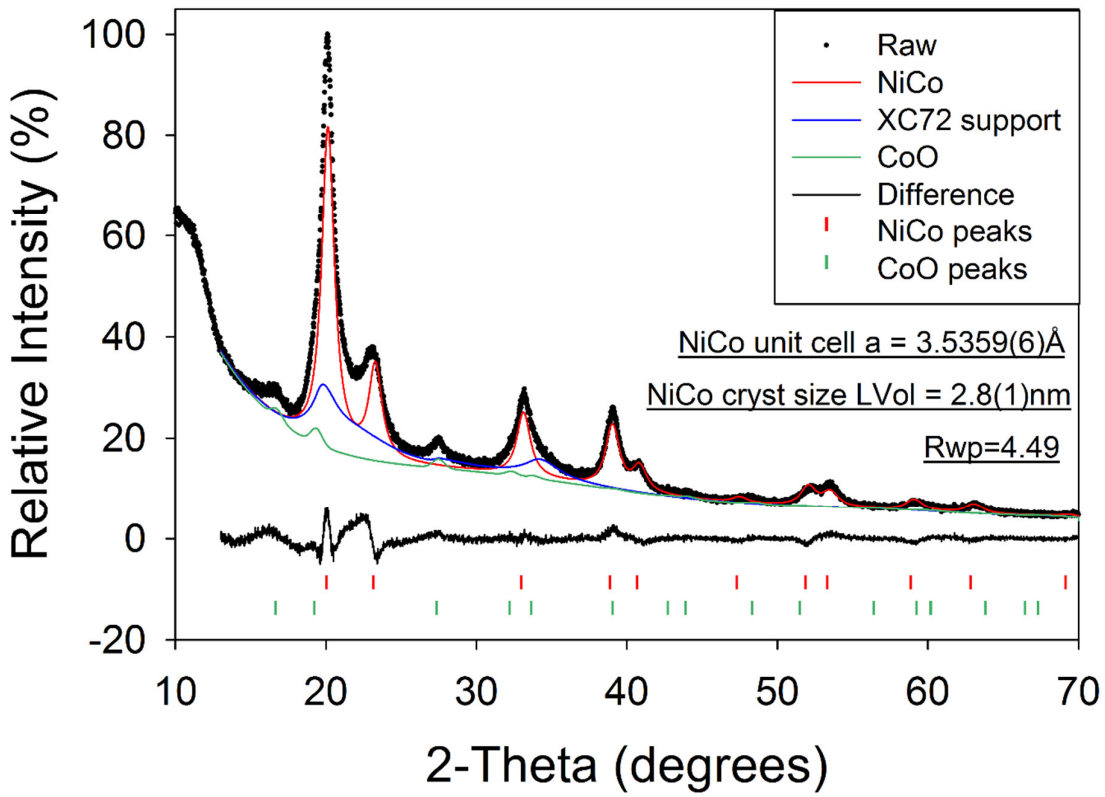

Figure S2. Fitted x-ray diffraction pattern for NiCo/C, as-prepared. Data were collected in capillary transmission mode using a Mo-K_α source, and modelled using a Rietveld type approach. The background contribution from XC72 carbon was modelled from a separate scan.

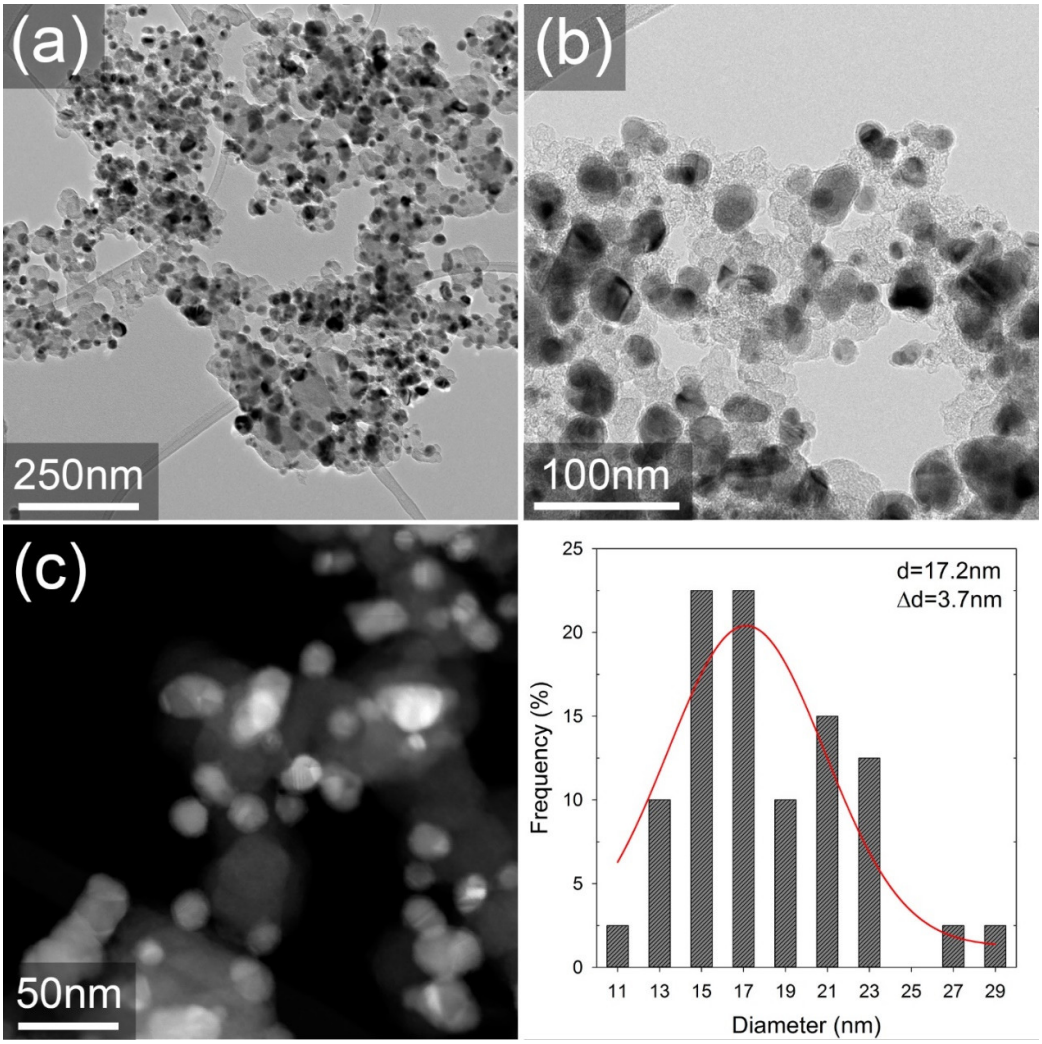

Figure S3. (**a**,**b**) Bright Field and (**c**) Dark Field images of annealed NiCo/C- nanoparticles at various magnifications. (**d**) Size histogram of the annealed particles from image analysis.

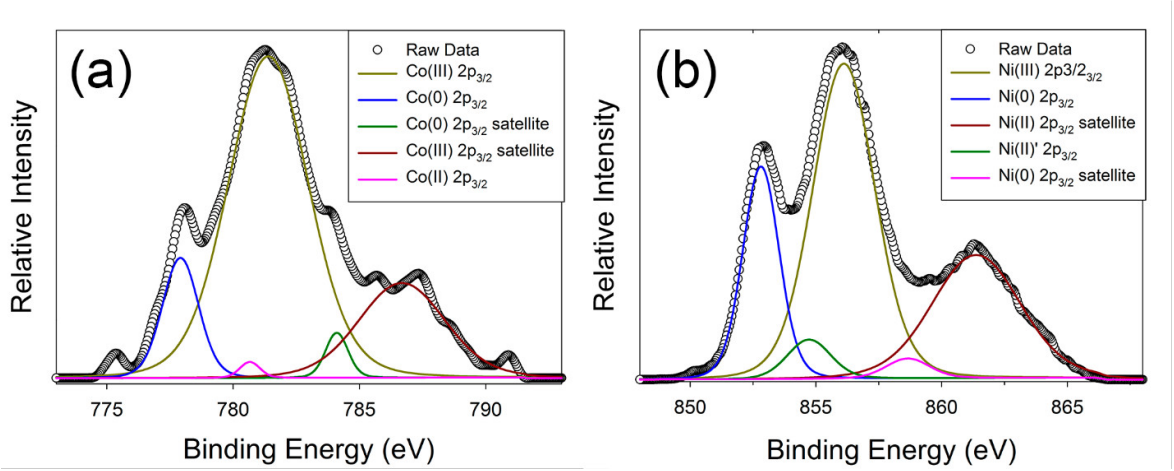

Figure S4. Fitted XPS spectra of (**a**) Co2p and (**b**) Ni2p regions.

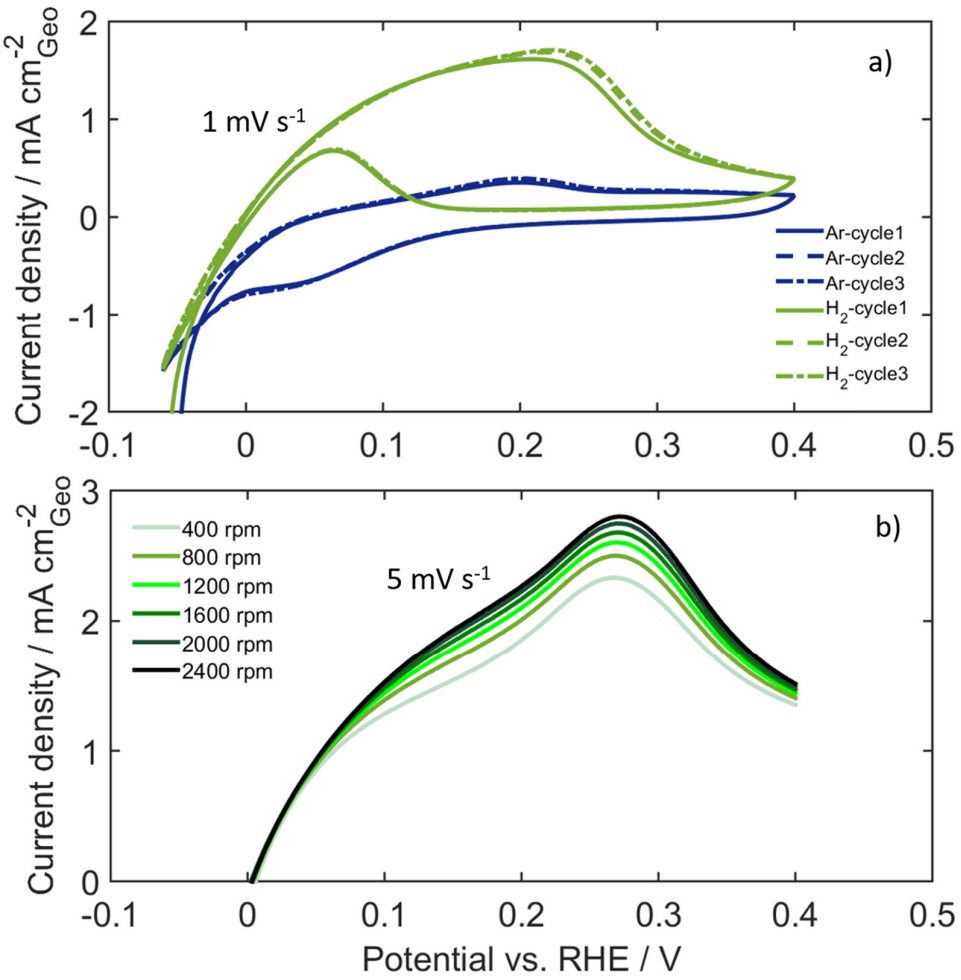

Figure S5. (**a**) Cyclic voltammograms of NiCo/C in both Ar-saturated and H_2-saturated 0.1M KOH, Scan rate: 1 mV/s and rotating speed 1600 rpm. (**b**) LSVs of NiCo/C in H_2- saturated 0.1 M KOH. Scan rate: 5 mV/s at different rotating speeds. The catalyst loading was 500 μg metal.

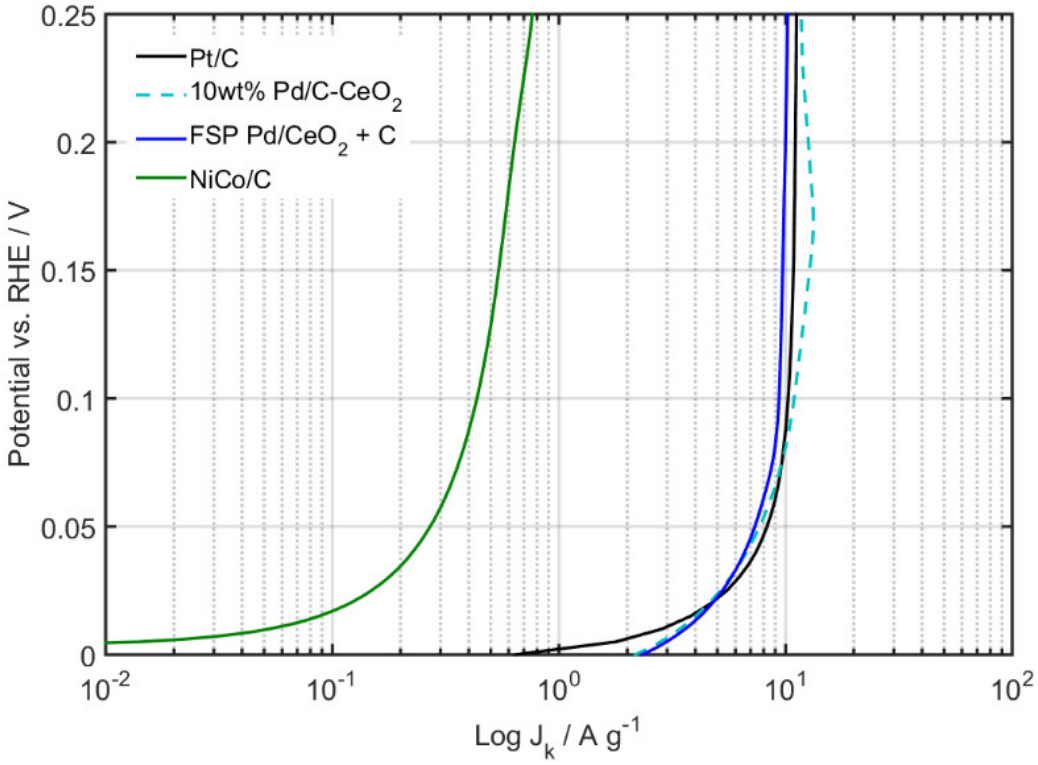

Figure S6. Mass activities of HOR catalysts in RDE. H_2- saturated 0.1 M KOH. Scan rate: 5 mV/s, the catalyst loading was 500 μg based on Ni and for the PGM catalysts 50 μg. rotating speed 1600 rpm.

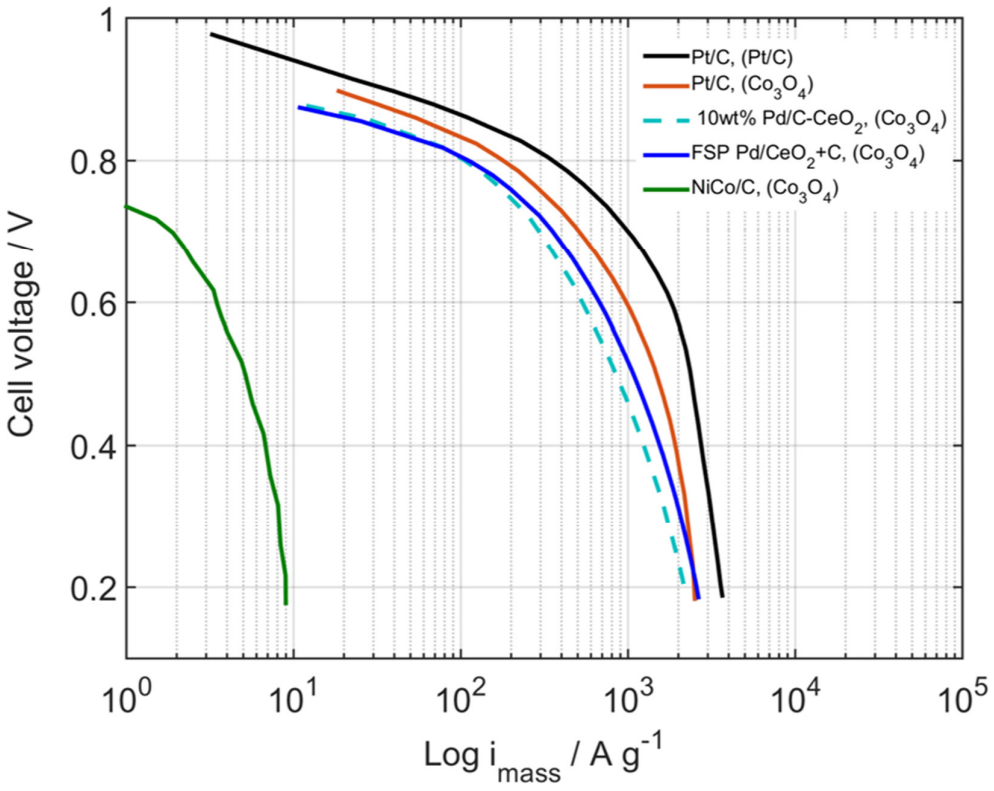

Figure S7. AEMFC polarisation curves normalised to anode catalyst mass (cathodes are denoted within parenthesis in the legend). Measurements taken at 70 °C, H_2 and O_2 on anode and cathode, using 1.0 and 0.5 slpm, respectively. Gas dew point 68 and 73 °C for the anode and cathode, respectively.