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Electronic Supporting Information (ESI) for the following paper:

Facile Synthesis of Ni/NiO Nanocomposites: The Effect of Ni Content in NiO upon the Oxygen Evolution Reaction within Alkaline Media

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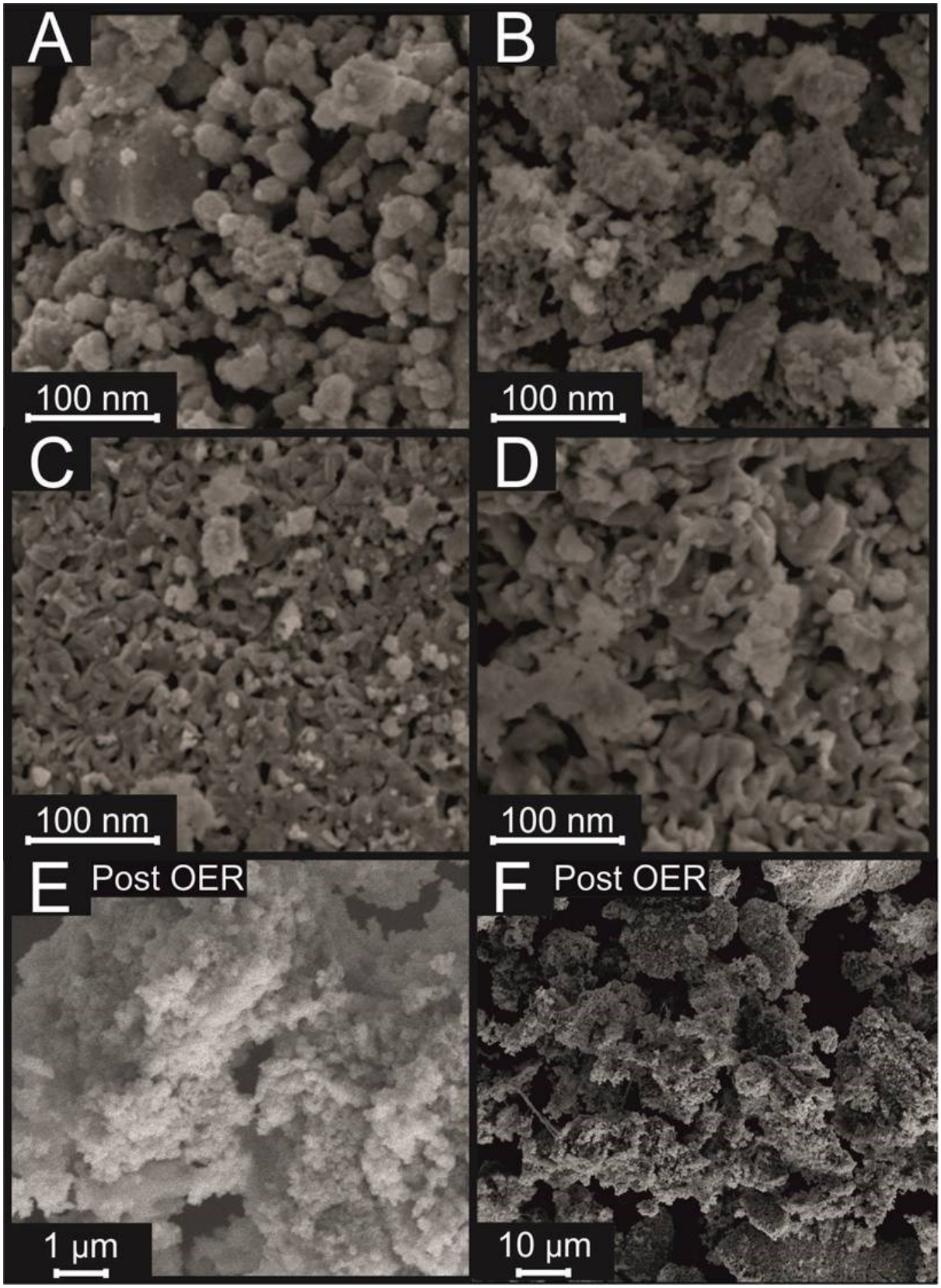
Physicochemical characterisation equipment

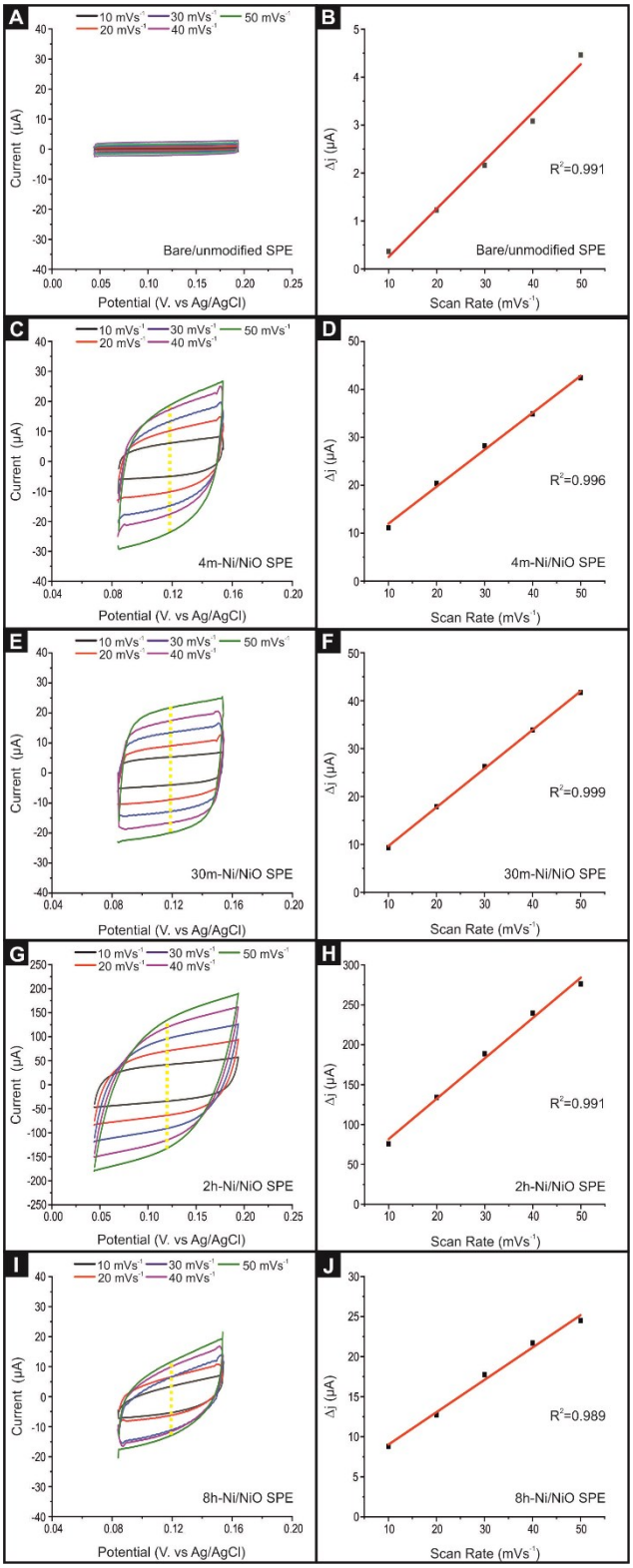
The phase purity and crystalline structure of the synthesized samples were characterized using X'pert PANALYTICAL X-ray powder diffraction (XRD) with a graphite monochromatized Cu Kα radiation source. X-ray photoelectron spectroscopy (XPS) data was acquired using a bespoke ultra-high vacuum system fitted with a Specs GmbH Focus 500 monochromated Al Kα X-ray source, Specs GmbH Phoibos 150 mm mean radius hemispherical analyser with 9-channeltron detection, and a Specs GmbH FG20 charge neutralising electron gun. Survey spectra were acquired over the binding energy range 1400 – 0 eV using a pass energy of 50 eV and high resolution scans were made over the C 1s and O 1s lines using a pass energy of 20 eV. Under these conditions the full width at half maximum of the Ag 3d5/2 reference line is *ca.* 0.7 eV. In each case, the analysis was an area-average over a region approximately 1.4 mm in diameter on the sample surface, using the 7 mm diameter aperture and lens magnification of ×5. The energy scale of the instrument is calibrated according to ISO 15472, and the intensity scale is calibrated using an in-house method traceable to the UK National Physical Laboratory. Data were quantified using Scofield cross sections corrected for the energy dependencies of the electron attenuation lengths and the instrument transmission. Data interpretation was carried out using CasaXPS software v2.3.16.

The electrochemically active surface area (ECSA) was determined by the specific capacitance method, as advocated within the academic literature via the following equation: $ECSA = C_d/C_s$ where a literature value of 0.04 mF/cm^2 is used for C_s, while the value of C_d is determined experimentally. The latter is determined for the various Ni/NiO nanocomposites with cyclic voltammograms recorded in 0.1M KOH within a non-Faradaic region (*i.e.* 0.04 to 0.15 V (vs Ag/AgCl)) over the range of scan rates from 10 mV s^{-1} to 50 mV s^{-1}. Consequently, ECSA values of 272.5, 312.5, 1707.4, and 165.0 were determined for 4m-Ni/NiO SPE, 30m-Ni/NiO SPE, 2h-Ni/NiO SPE and 8h-Ni/NiO SPE respectively.

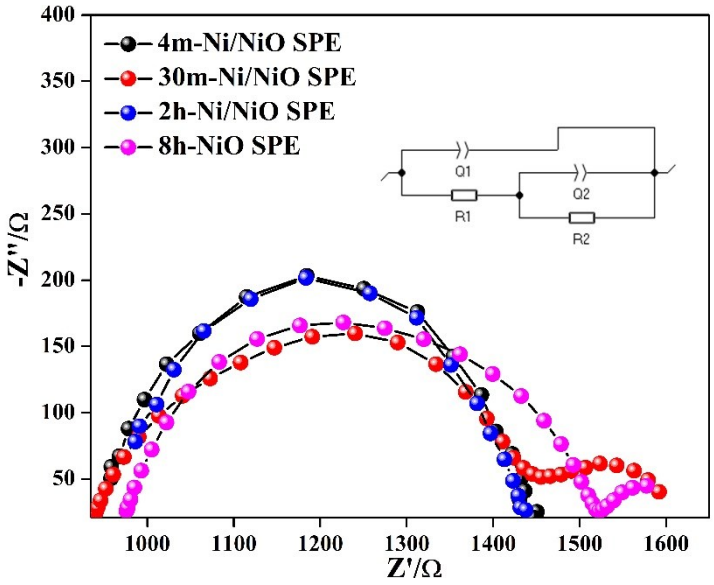
ESI Figure S1. The SEM images of the Ni/NiO nanocomposites annealed at 500 °C for (A) 4 mins, (B) 30 mins, (C) 2 hrs and (D) 8hrs. (E) and (F) are images of the 2h-Ni/NiO SPE post ORR (chronoamperometry for 26 hrs).



ESI Figure S2. Double layer capacitance measurement for determining electrochemically active surface area for the synthesized Ni-based catalysts in 1M KOH solution ;(A, C, E, G and I) CV s were measured in the non-faradic region of 0.04 to 0.15 V (vs Ag/AgCl) with different scan rates from 10 mV s^{-1} to 50 mV s^{-1} ; (B, D, F, H and J) scan rate dependence of the current.



ESI Figure S3. Electrochemical impedance Nyquist plots of Ni/NiO nanocomposite in 1.0 M KOH solution, recorded at +1.50 V with frequency range 0.01 Hz to 100 kHz; inset: equivalent circuit.



ESI Figure S4. XRD patterns of the post- 2h-Ni/NiO SPE post OER. (chronoamperometry for 26 hrs). The peak before 50° that is not labelled is from the underlying supporting SPEs.