Method

To electrochemical Connections to Ni(OH)₅ air point, capped 0 temperature •

Motivation

Finely both Ni(OH)₅ NiOOH aqueous methods then reaction:

- BET, 2 O₃
- Oxygen evolution reaction (OER):
- Ni(OH)₂ NiOOH Ni(OH)₂ + OH⁻
- NaOH dried, and pellets and room then precipitated precursor rinsed, dried, and calcined in air at 500°C.

Method B: Room temp. NaOH-induced precipitation
- Finely-ground NaOH pellets gradually added to 0.5M Ni(NO₃)₂ in water at room temperature, then capped at room temperature for 24hrs. Precipitated precursor rinsed, dried, and calcined in air at 500°C.

Method C: Boiling temp. NaOH-induced precipitation
- Finely-ground NaOH pellets gradually added to 0.5M Ni(NO₃)₂ in water at the solution’s boiling point, then capped at room temperature for 24hrs. Precipitated precursor rinsed, dried, and calcined in air at 500°C.

Three aqueous-phase methods were investigated:

Method A: NaOH-induced reflux precipitation
- 0.5M Ni(NO₃)₂ in 10M NaOH solution boiled under reflux for 24hrs, then capped at room temperature for 24hrs. Precipitated precursor (Ni(OH)₂) rinsed, dried, and calcined in air at 500°C.

To determine an optimum synthesis method for maximum electrocatalytic activity and stability.

3. Synthesis Methods

Figure 1. Scanning Electron Microscopy (SEM) micrographs of Method A (A) Ni(OH)₂ and (B) NiO, and Method B (C) NiOOH, and (D) NiO.

Figure 2. (A) Differential Scanning Calorimetry (DSC) and (B) Thermogravimetric Analysis (TGA) for Methods A and B NiOOH nanoparticles. Heating and cooling rates 10°C/min, temperature range 25-1000°C.

Figure 3. X-ray Diffraction (XRD) patterns for (A) Method A, (B) Method B, and (C) Method C precursor Ni(OH)₂ nanoparticles. Inset shows Scherrer Equation average grain boundary sizes.

Figure 4. XRD Specific Surface Areas (m²/g)
Method A Method B Method C
Method A 17 140 212
Method B 42 22 31
Method C 40 10 18

Ni(OH)₂ BET Specific Surface Areas (m²/g)

Figure 5. X-ray diffraction (XRD) patterns for (A) Method A, (B) Method B, and (C) Method C precursor Ni(OH)₂ nanoparticles. Inset shows Scherrer Equation average grain boundary sizes.

Figure 6. CVs for Methods A and B NiO in (A) 0.005M KOH/0.1M CH₃OH vs. SCE at 20mV/s. 200 scans performed for each.

Figure 7. Stability CVs in 0.1M Na₂CO₃/1M CH₃OH for Method A NiO (A) current build-up and (B) current deterioration, and Method B NiO (C) current build-up and (D) current deterioration. Range from 1.0 to 1.1V vs. SCE at 20mV/s. 200 scans performed for each.

Figure 8. IR-corrected electrochemical impedance spectroscopy (EIS) Nyquist plots for (A) Method A and (B) Method B NiO NiOOH in 0.1M Na₂CO₃/1M CH₃OH, and (C) Method A and (D) Method B NiO NiOOH Frequency range from 100Hz to 100kHz.

Figure 9. XRD patterns for (A) Method A, (B) Method B, and (C) Method C precursor Ni(OH)₂ nanoparticles. Inset shows Scherrer Equation average grain boundary sizes.

Figure 10. SEM micrographs of Method A (A) Ni(OH)₂ and (B) NiO, and Method B (C) NiOOH, and (D) NiO.

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