

## Supporting Information

### **Porous nanorods by stacked NiO nanoparticulate exhibiting corn-like structure for sustainable environmental and energy applications**

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#### **EXPERIMENTAL DETAILS**

##### **1. Synthesis of NiO nanorods**

Without additional purification, all of the precursors, reagents, and solvents were employed as obtained. Nickel chloride ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ), sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ), 5 % Nafion, and Iridium oxide ( $\text{IrO}_2$ ) were purchased from Sigma Aldrich. Potassium hydroxide (KOH) was procured from Loba Chemie Pvt. Ltd. All through the experiment, double Ionized (DI) water was used. The experiment-related glassware was completely inert both physically and chemically.

0.474 g NiCl<sub>2</sub>·6H<sub>2</sub>O and 0.126 g sodium oxalate were dissolved in a mixed solvent containing 18 ml DI water and 30 ml ethylene glycol under constant stirring for 30 min. The resulting homogenous solution was transferred to a Teflon liner and placed in a stainless-steel autoclave, where it reacted for 12 hours at 180 °C. The recovered product was subsequently treated with ethanol and DI water after cooling gradually. To obtain porous NiO nanorods, it was additionally dried at 60 °C for 2 hours and annealed at 400 °C for 2 hours.

## 2. Characterization

The morphology of the synthesized NiO product is analyzed using Field Emission Scanning Electron Microscopy (FESEM, JEOL, JSM-7610 F Plus). The crystal structure of the NiO is investigated from X-ray diffraction (Empyrean-DY2528, Malvern Panalytical,  $\lambda_{\text{Cu}} = 1.5405 \text{ \AA}$ ) and further confirmed from the high-resolution images and selective area electron diffraction (SAED) obtained from Transmission Electron Microscopy (TEM, JEOL JEM-2100 F). The size distribution of nanoparticles and porous morphology of NiO nanorods are confirmed from the Small Angle X-ray Scattering (SAXS, Empyrean-DY2528, Malvern Panalytical) and BET analysis (QUANTACHROME Autosorb iQ2), respectively. The chemical states and electronic structure are analyzed from X-ray photoelectron spectroscopy (XPS, Thermo Scientific Inc. K $\alpha$ ) and Extended X-ray-Absorption Fine Structure (EXAFS, Rigaku R-XAS) analysis.

## 3. Gas adsorption-desorption measurements

Gas adsorption-desorption studies were performed using QUANTACHROME Autosorb iQ2 gas analyzer, and obtained isotherms were analyzed using ASIQwin software. The sample was degassed for 10 h in a high vacuum to eliminate moisture and other adsorbed gases before CO<sub>2</sub> capture. Barrett-Joyner-Halenda (BJH) and Brunauer-Emmett-Teller (BET) models were used to estimate porosity and specific surface area, respectively, in the N<sub>2</sub> gas environment. The equilibrium CO<sub>2</sub> adsorption isotherms were obtained at 273 and 298 K and further used to calculate isosteric heat of adsorption ( $-Q_{\text{st}}$  or  $\Delta H_{\text{ads}}$ ) from the Clausius-Clapeyron equation,[1, 2]

$$\ln\left(\frac{P_2}{P_1}\right) = \frac{-Q_{\text{st}}}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad \text{--(S1)}$$

where  $P_2$  and  $P_1$  are the pressures (bar) corresponding to two different isotherms at temperatures  $T_2$  (i.e., 298 K) and  $T_1$  (i.e., 273 K), respectively, for the same volume of gas adsorbed, and  $R$  is the universal gas constant (i.e., 8.3145 J mol<sup>-1</sup> K<sup>-1</sup>).

## 4. Electrochemical measurements for supercapacitor

Initially, 1×1 cm of nickel foam was ultrasonically treated in concentrated HCl and further washed with DI water and ethanol to remove the oxide contamination on the surface, if any. The homogeneous ink consisting of 10 mg of NiO nanorods and 10 μL of Nafion was drop cast on nickel foam and dried at ambient overnight to prepare the working electrode for the supercapacitor. The energy storage performance is evaluated from cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and cyclic stability measurements performed in 1M KOH aqueous electrolyte in a three-electrode configuration using an electrochemical workstation (Metrohm Autolab PGSTAT204), where NiO/Ni-foam is the working electrode, platinum is the counter electrode, and saturated calomel electrode (SCE) is the reference electrodes. The specific capacitance ( $C_s$ ) of NiO nanorods is estimated from the CV and GCD curves using equations,

$$C_s = \frac{\int idV}{m\Delta V} \quad \text{for CV} \quad \text{--(S2)}$$

$$C_s = \frac{I}{m} \int \frac{dt}{V} \quad \text{for GCD} \quad \text{--(S3)}$$

where  $C_s$  is the specific capacitance of the NiO nanorods (F/g),  $m$  is the mass of the active electrode materials (g),  $v$  is the scan rate (mV/s),  $\Delta V$  is the applied potential range (V),  $I$  is applied current (A), and  $V$  is applied potential in the GCD.

Further, the charge storage mechanism is evaluated from the current density variation to the scan rate of porous NiO nanorods expressed from Eq. S4.

$$i_p = av^b \quad \text{or} \quad \log(i_p) = \log(a) + b \log(v) \quad \text{--(S4)}$$

where,  $a$  and  $b$  are constants, and  $i_p$  is the current density. The estimated value of  $b$  represents the diffusion (if  $b = 0.5$ ) and surface- (if  $b = 1$ ) controlled process.

## 5. Electrocatalytic water splitting

The homogeneous ink is prepared using 3.5 mg NiO catalyst, 600 μL DI water, 380 μL ethanol, and 20 μL 5 % Nafion solution. The as-prepared ink is drop-cast on a glassy carbon electrode and dried overnight under ambient conditions. Typically 0.25 mg/cm<sup>2</sup> catalyst loaded over 3 mm diameter was used as the working electrode to study water splitting performance. The electrocatalytic performance was investigated in a standard three-electrode system using an electrochemical workstation equipped with Nova 2.1.4 software (Metrohm Autolab, Multichannel-204). The OER and HER performance were determined using electrolyte solutions of 1 M KOH and 0.5 M H<sub>2</sub>SO<sub>4</sub> degassed with nitrogen gas for 30 min, respectively. OER was performed in a conventional three-electrode system of Hg/HgO reference and platinum counter electrode. OER

activity was examined from the linear sweep voltammetry (LSV) and CV measurements performed in the optimized potential range of 0 to 1 V at a scan rate of 10 mV/s and 0.15 to 0.25 V at various scan rates between 20 to 100 mV/s, respectively. However, HER activity is evaluated using Ag/AgCl (3 M KCl) reference electrode along with a graphite rod as a counter electrode instead of platinum to avoid deposition on the electrode surface, which can mislead the hydrogen evolution. HER activity was investigated from LSV in the potential range of 0.2 V to -0.8 V vs. Ag/AgCl at a scan rate of 10 mV/s. The long-term stability of NiO porous nanorods for 25 h is tested using chronoamperometry (CA) at a constant overpotential of 441 and 345 mV for OER and HER activity, respectively. The change in overpotential after 3000 LSV cycles at a constant scan rate of 50 mV/s was investigated to estimate the stability of NiO nanorods for both OER and HER activity. The potentials were calibrated versus RHE using the Nernst equation for HER (Eq. S5) and OER (Eq. S6)

$$E_{(RHE)} = E_{(Ag/AgCl)} + 0.059 \text{ pH} + E_{Ref} \quad \text{--(S5)}$$

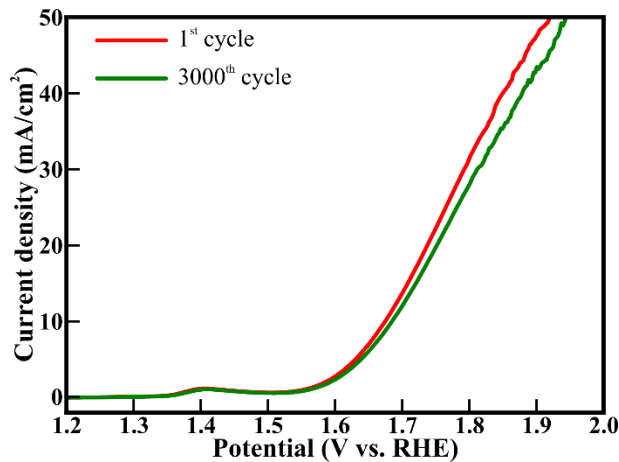
$$E_{(RHE)} = E_{(Hg/HgO)} + 0.059 \text{ pH} + E_{Ref} \quad \text{--(S6)}$$

Moreover, the electrochemically active surface area (ECSA) is estimated from Eq. S7.[3]

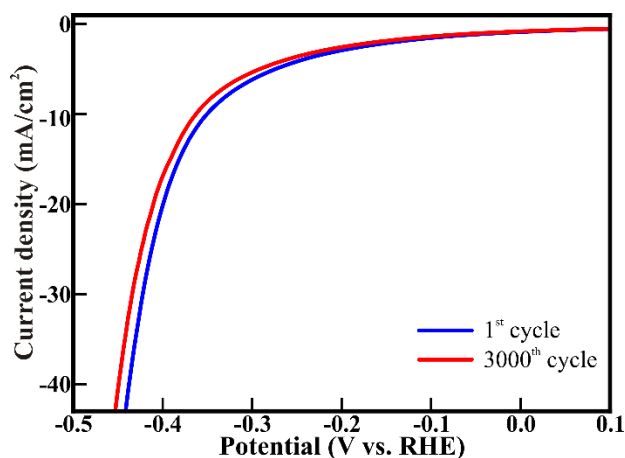
$$ECSA = \frac{C_{dl}}{C_s} \quad \text{--(S7)}$$

where  $C_s$  is the specific capacitance of a flat working electrode (i.e., 40  $\mu\text{F}/\text{cm}^2$ ) and  $C_{dl}$  is determined from the slope of  $J_a$  or  $J_c$  vs. scan rate.  $J_a$  and  $J_c$  are anodic and cathodic current densities, respectively.

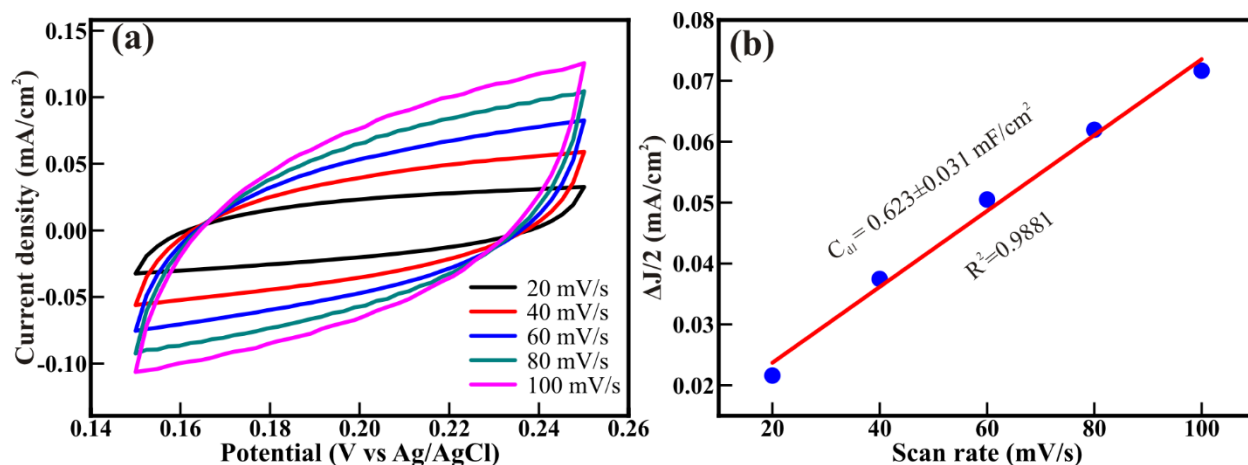
## RESULTS



**Figure S1** Polarization curves (LSV) of porous NiO nanorods for 1<sup>st</sup> and 3000<sup>th</sup> cycles of continuous operation in 1 M KOH towards OER activity.



**Figure S2** Polarization curves (LSV) of porous NiO nanorods for 1<sup>st</sup> and 3000<sup>th</sup> cycles of continuous operation in 0.5 M H<sub>2</sub>SO<sub>4</sub> towards HER activity.



**Figure S3** (a) Cyclic voltammetry curves of porous NiO nanorods, and (b) corresponding plot of  $\Delta J/2$  vs. scan rate at a static potential of 0.2 V (vs. Hg/HgO) to determine double layer capacitance ( $C_{dl}$ ).

## REFERENCES

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