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# Highly Dispersed Core–Shell Ni@NiO Nanoparticles Embedded on Carbon–Nitrogen Nanotubes as Efficient Electrocatalysts for Enhancing Urea Oxidation Reaction

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✓ **Cite this:** *Energy Fuels* 2023, 37, 6, 4616–4623

Publication Date: February 27, 2023 ▾

<https://doi.org/10.1021/acs.energyfuels.2c04377>

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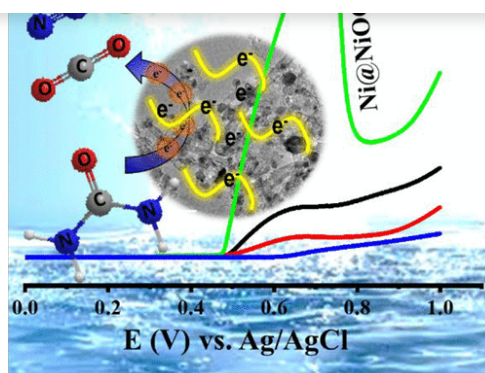
## Abstract

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Wastewater treatment and energy production are important fields of research to meet the current requirements of sustainable energy development and wastewater restoration. The urea oxidation reaction (UOR) can be used for simultaneous environmental remediation and energy production on a priority basis. Nickel–nickel oxide (Ni@NiO) core–shell NPs on carbon–nitrogen nanotubes (CNNTBs) have been synthesized as electrocatalysts by using a simple and easy aerial annealing method. Morphological analysis by high resolution–transmission electron microscopy (HR-TEM) confirms the formation of Ni@NiO NPs on CNNTBs with an average size of  $\sim 40$  nm. Powder X-ray diffraction (XRD) confirms Ni@NiOCN-X with a face-centered cubic (FCC) crystal structure. A BET surface area measurement suggests that annealing at  $400$  °C (Ni@NiOCN-4) creates a larger surface area than that of carbon–nitrogen material (CN). The electrochemical studies of the Ni@NiOCN-4 nanocomposite reveal that it has a better electrochemical activity and ultralow onset potential with an  $E_{\text{onset}} = 0.42$  V<sub>Ag/AgCl</sub> and current density of  $34$  mA/cm<sup>2</sup> for a potential of  $0.71$  V<sub>Ag/AgCl</sub> toward urea oxidation reactions. The annealing temperature-dependent studies were found to be effective toward the morphology tuning and hence tunable catalytic performance toward urea oxidation reactions. In urea oxidation studies, chronoamperometry ( $i-t$ ) and EIS analysis show that the proposed Ni@NiOCN-4 system has long-term stability and ultrafast electron transfer. This work showed a simple way for temperature-dependent morphology tuning of a catalyst with enhanced electrochemical activity for urea oxidation, which is useful for sustainable energy production utilizing urea-rich wastewater.

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## Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.energyfuels.2c04377>.

- Materials, synthesis of carbon–nitrogen material (CN), instrumentation details, and electrochemical measurements. S1: Electrode preparation. S2: TEM image and SEAD pattern of the catalyst. S3: EDAX analysis. S4: Elemental mapping and EDAX analysis of Ni@NiOCN-4. S5: FTIR spectra. S6: TGA analysis. S7: XPS analysis. S8: ESR spectra. S9: CV and EIS of catalyst in 1 M KOH. S10: FE-SEM image of Ni@NiOCN-4. S11: Electrochemical surface area (ECSA). S12: Precipitation test of  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ . S13:  $^{13}\text{C}$ -NMR. S14: Enhancement factor. S15: Inverted buret setup. Tables S-1 and S-2. ([PDF](#))
- Video S1 ([MP4](#))
- Video S2 ([MP4](#))

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## List of supporting information

### Materials



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