

Supplementary Materials

Evaluation of Pt deposition onto dye-sensitized NiO photocathodes for light-driven hydrogen generation

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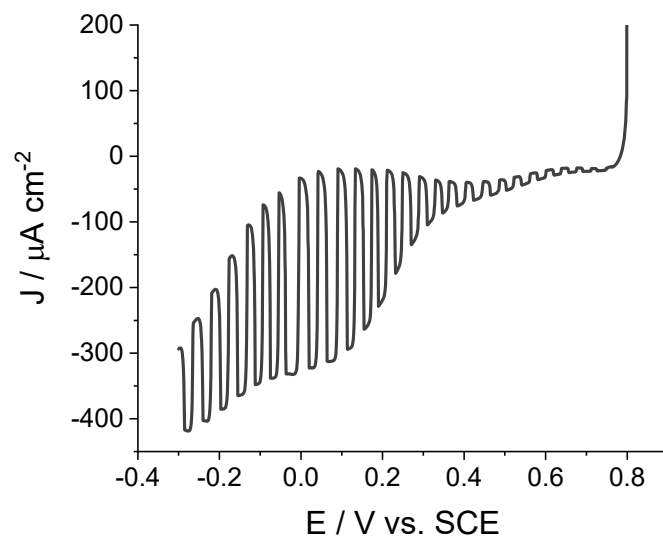


Figure S1. LSV of sensitized NiO electrode of type A in N₂-purged 0.1 M acetate buffer at pH 4 in the presence of 0.01 M [Co(NH₃)₅Cl]Cl₂ as an irreversible electron acceptor.

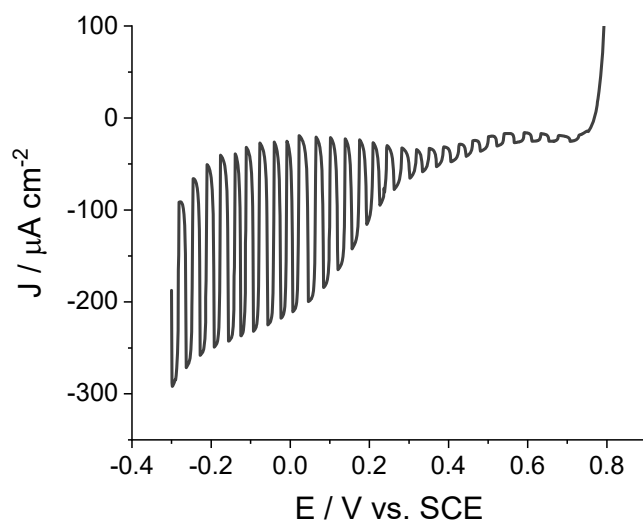


Figure S2. LSV of sensitized NiO electrode of type B in N₂-purged 0.1 M acetate buffer at pH 4 in the presence of 0.01 M [Co(NH₃)₅Cl]Cl₂ as an irreversible electron acceptor.

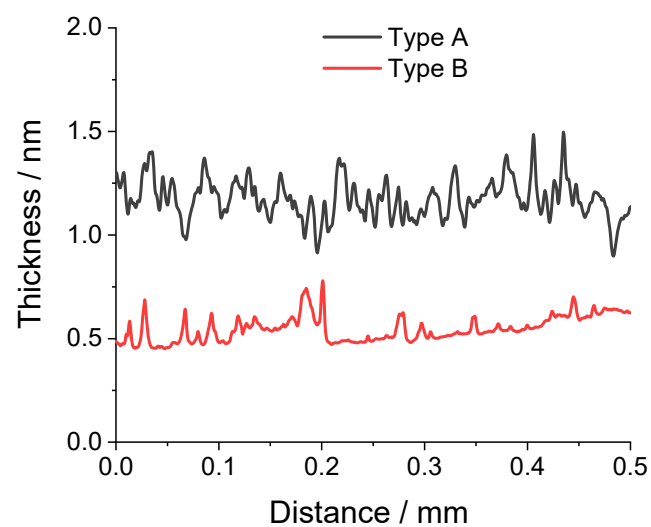


Figure S3. Film thickness of NiO electrodes of type A and type B measured using profilometry.

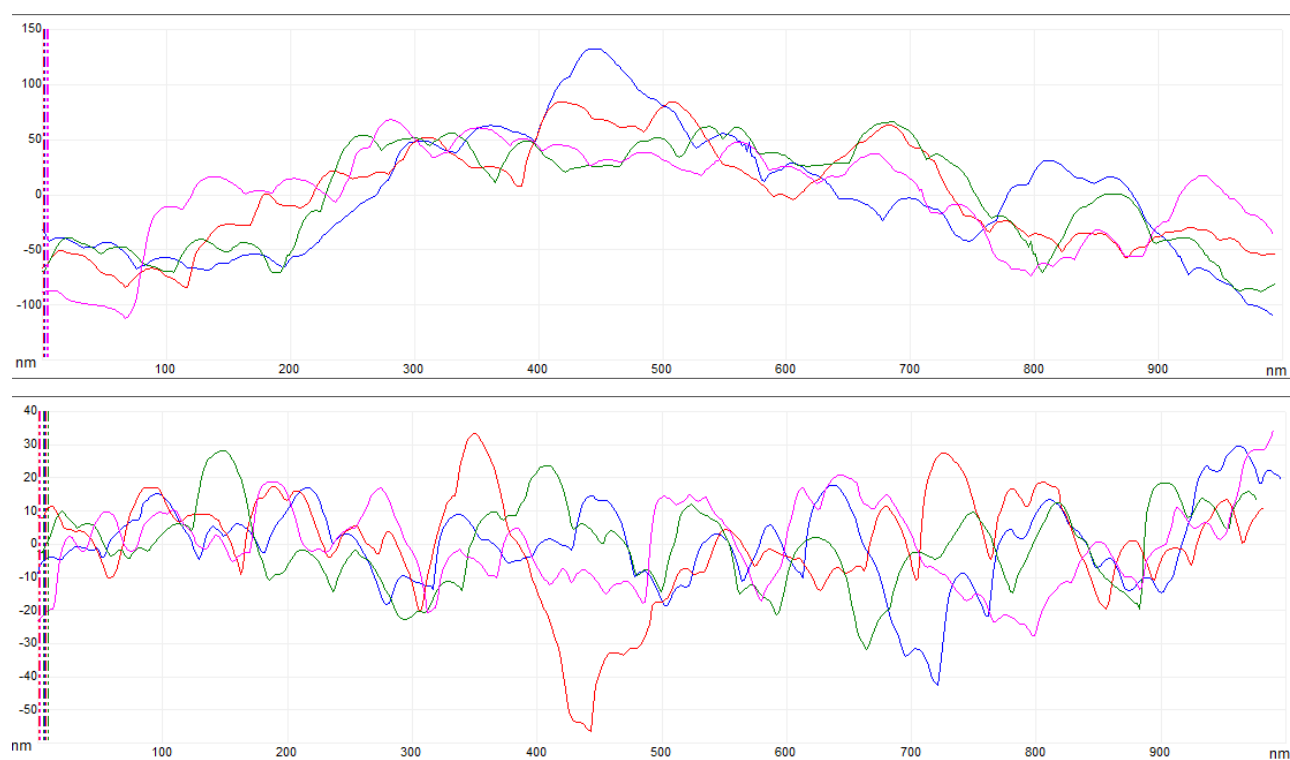


Figure S4. Section analyses of NiO electrodes of type A (top) and type B (bottom) measured using AFM.

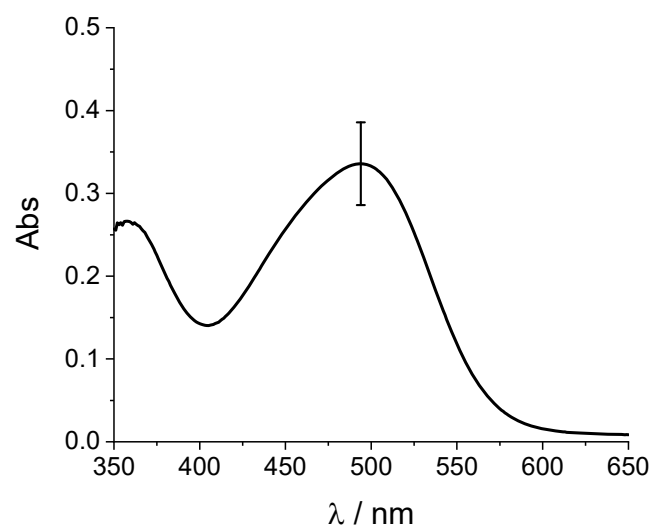


Figure S5. Absorption spectrum of a solution (optical pathlength of 1 cm) obtained after desorption of a sensitized NiO electrode of type A (average of three samples, surface area of 1.5 cm²) with 20 μ L TBAOH 1 M in methanol and further dilution with methanol to a total volume of 3 mL.

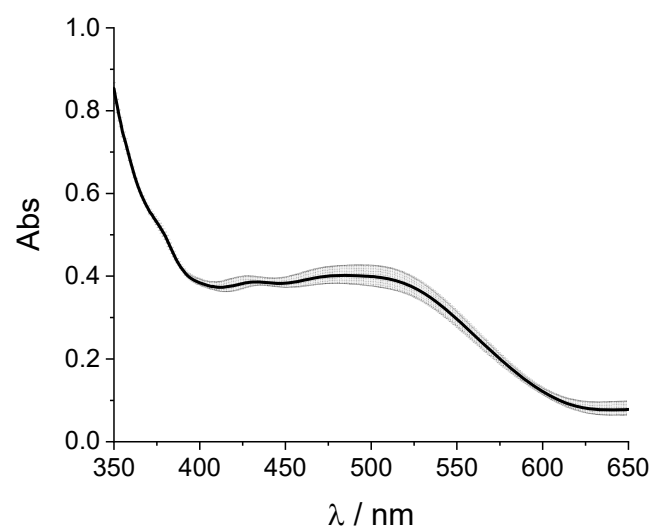


Figure S6. Absorption spectrum (average of three samples) of a NiO electrode of type B sensitized after overnight soaking in a 0.5 mM acetonitrile solution of the **P1** dye.

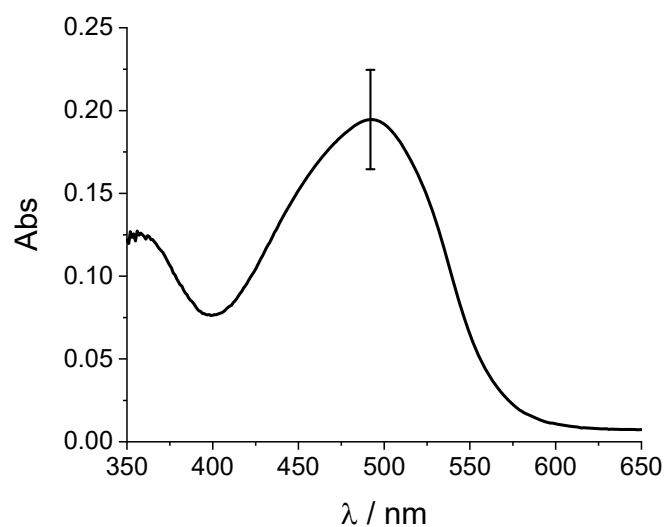


Figure S7. Absorption spectrum of a solution (optical pathlength of 1 cm) obtained after desorption of a sensitized NiO electrode of type B (average of three samples, surface area of 1.5 cm^2) with $20 \text{ }\mu\text{L}$ TBAOH 1 M in methanol and further dilution with methanol to a total volume of 3 mL.

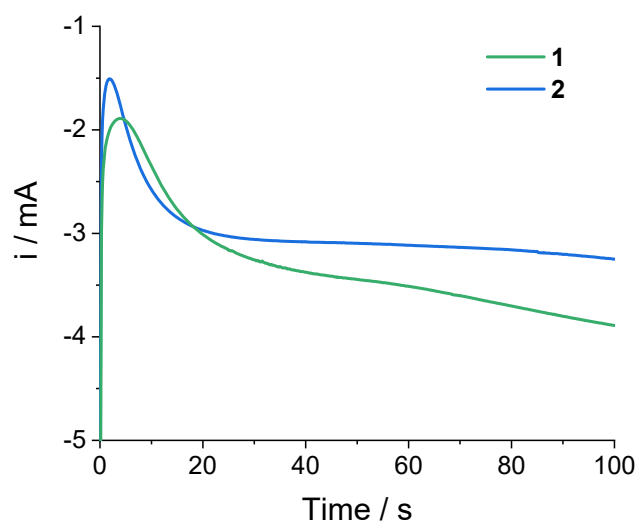


Figure S8. Exemplars of current vs. time trace recorded during the deposition with Method 1 of sensitized NiO electrodes of type A.

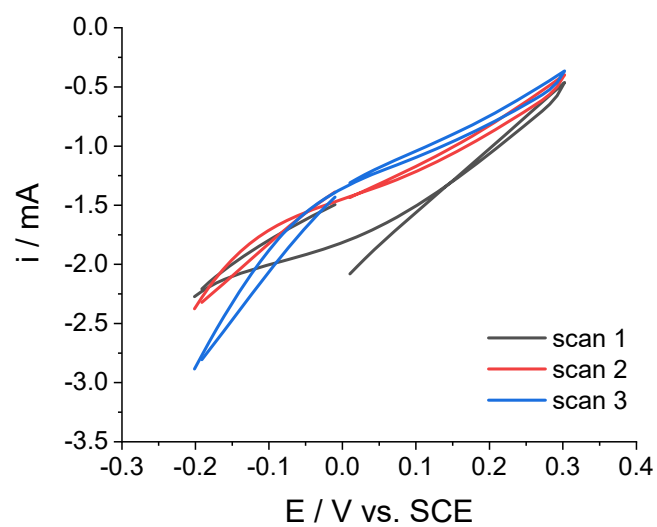


Figure S9. Exemplar of current vs. voltage traces recorded during the deposition with Method 3 of sensitized NiO electrodes of type A.

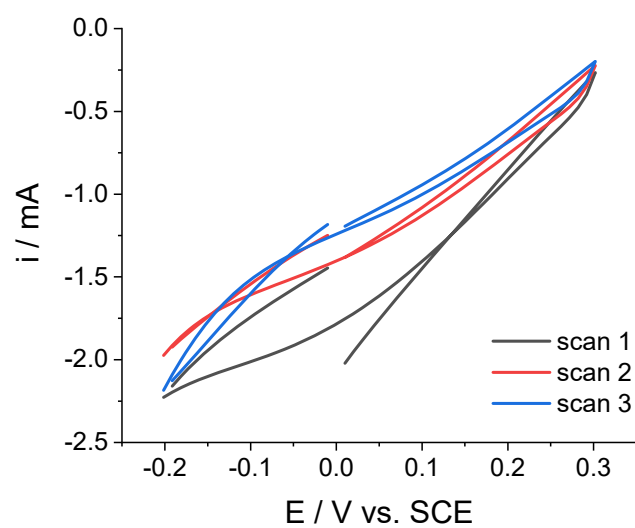


Figure S10. Exemplar of current vs. voltage traces recorded during the deposition with Method 4 of sensitized NiO electrodes of type A.

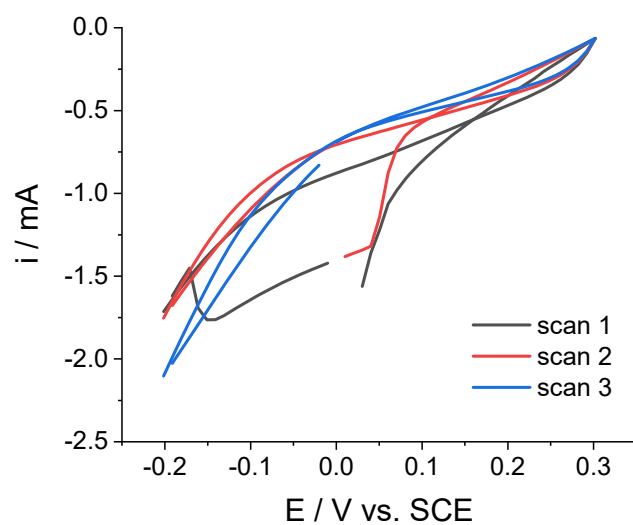


Figure S11. Exemplar of current vs. voltage traces recorded during the deposition with Method 5 of sensitized NiO electrodes of type A.

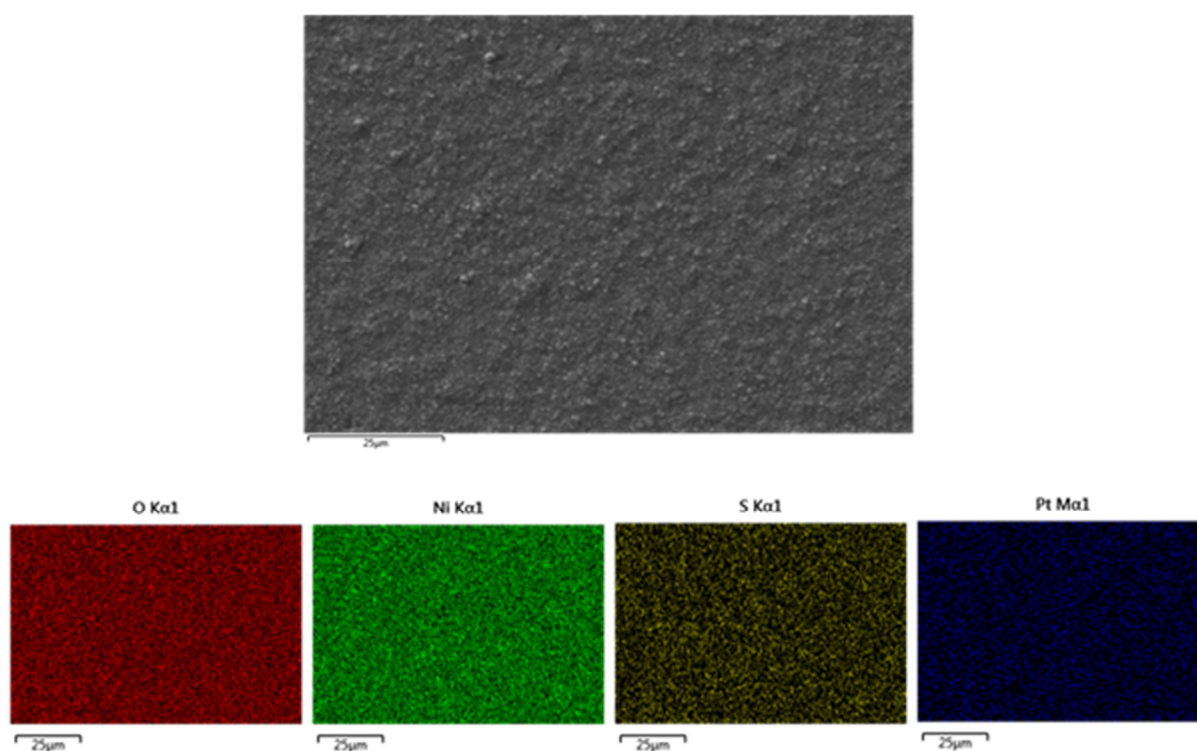


Figure S12. SEM/EDS analysis of NiO electrodes of type A prepared using Method 2 at 20 s.

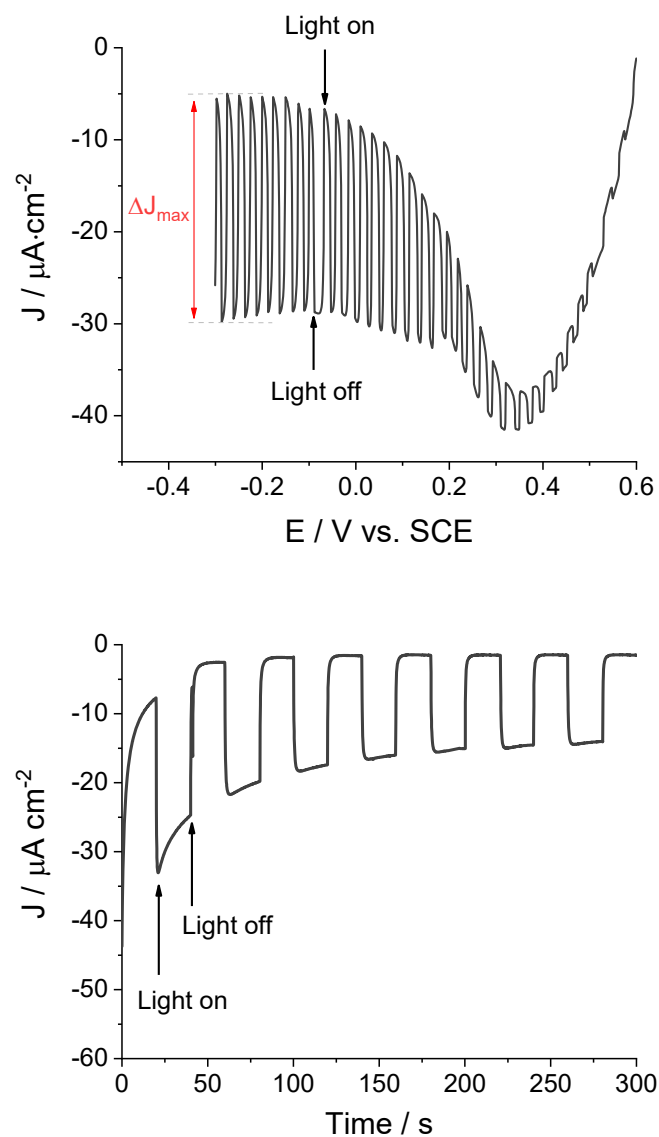


Figure S13. Representative LSV (top) and CA (bottom) of NiO-A|**P1**|Pt-**1** at 20 s in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation to highlight on-off light cycles and the procedure to estimate the maximum photocurrent density (ΔJ_{max}).

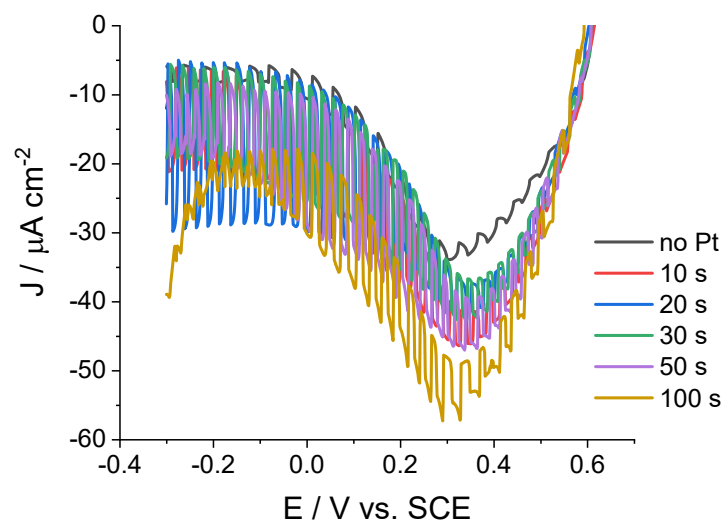


Figure S14. LSV of NiO-A|**P1**|Pt-**1** at different deposition times in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation.

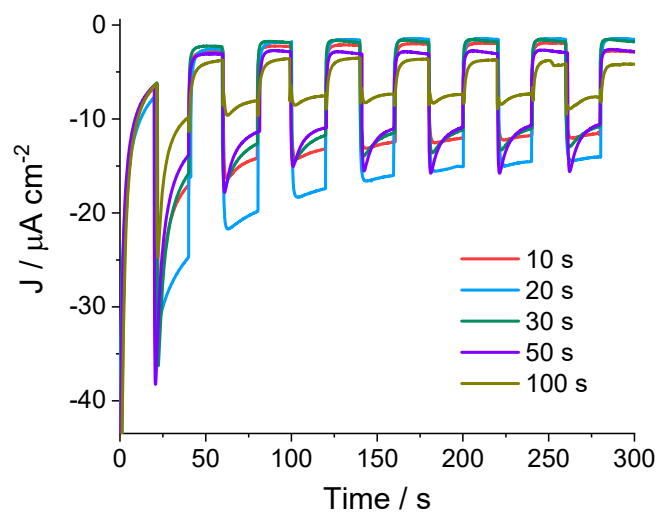


Figure S15. CA at -0.2 V vs. SCE of NiO-A|**P1**|Pt-**1** at different deposition times in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation.

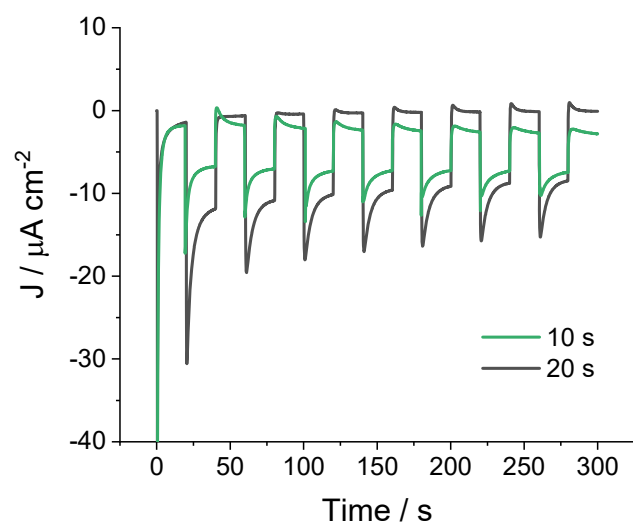


Figure S16. CA at -0.2 V vs. SCE of NiO-A|**P1**|Pt-2 at different deposition times in N_2 -purged 0.1 M acetate buffer at pH 4 under chopped irradiation.

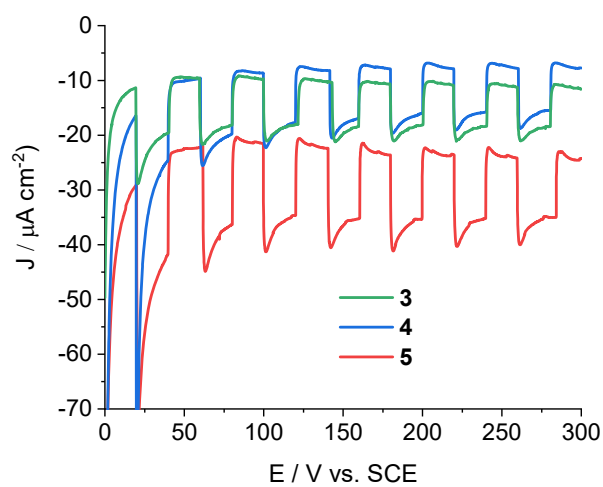


Figure S17. CA at -0.2 V vs. SCE of NiO-A|**P1**|Pt-*n* with *n* = 3-5 in N_2 -purged 0.1 M acetate buffer at pH 4 under chopped irradiation.

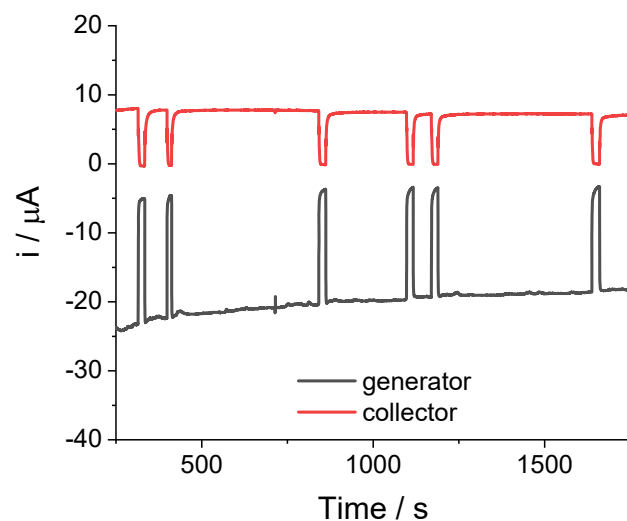


Figure S18. Faradaic efficiency determination for NiO-A|**P1**|Pt-1 at 20 s in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation: the NiO-based photocathode generator was held at -0.2 V vs. SCE while the Pt-based collector was held at $+0.2$ V vs. SCE.

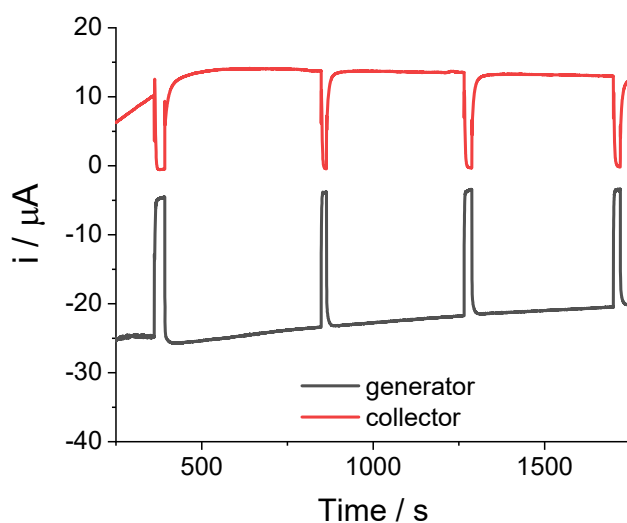


Figure S19. Faradaic efficiency determination for NiO-A|**P1**|Pt-5 in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation: the NiO-based photocathode generator was held at -0.2 V vs. SCE while the Pt-based collector was held at $+0.2$ V vs. SCE.

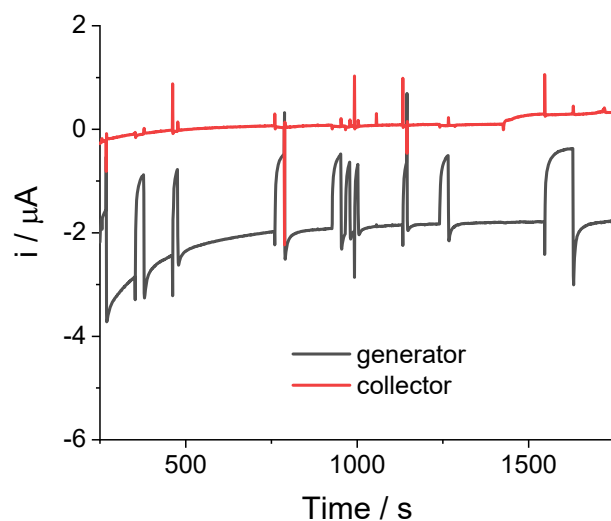


Figure S20. Faradaic efficiency determination for the non-platinized NiO electrode of type A in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation: the NiO-based photocathode generator was held at -0.2 V vs. SCE while the Pt-based collector was held at $+0.2$ V vs. SCE.

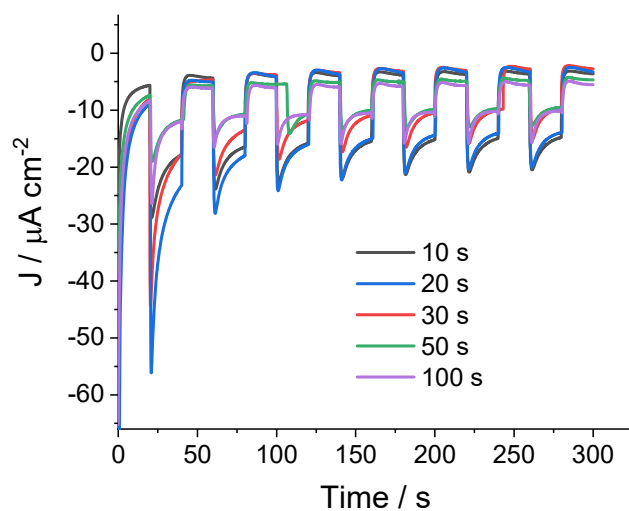


Figure S21. CA at -0.2 V vs. SCE of NiO-B|**P1**|Pt-**1** at different deposition times in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation.

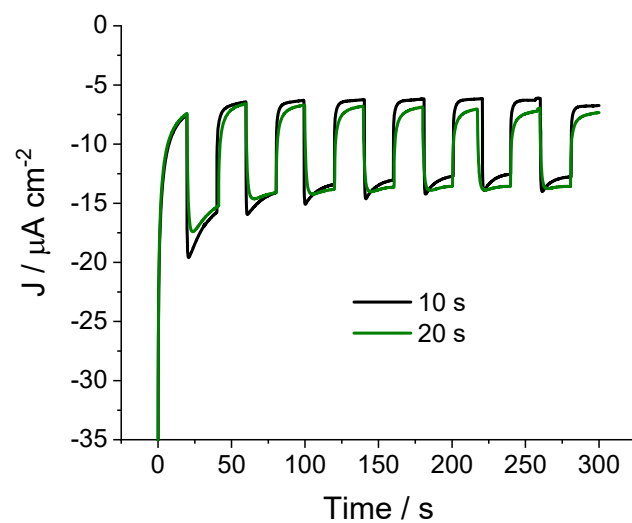


Figure S22. CA at -0.2 V vs. SCE of NiO-B|**P1**|Pt-**2** at different deposition times in N_2 -purged 0.1 M acetate buffer at pH 4 under chopped irradiation.

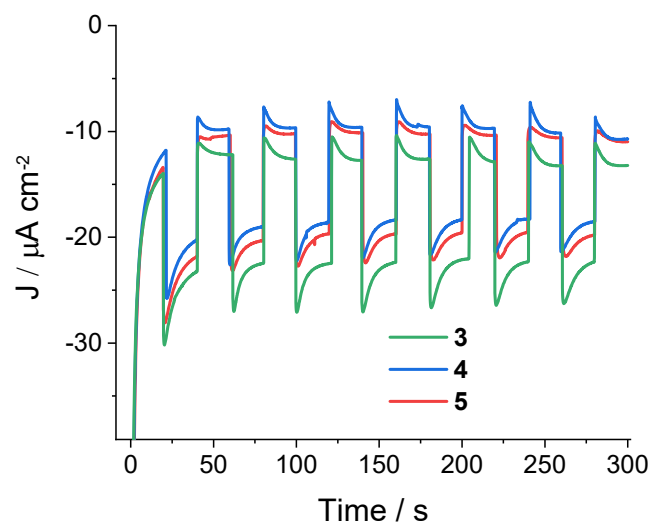


Figure S23. CA at -0.2 V vs. SCE of NiO-B|**P1**|Pt-**n** with $n = 3-5$ in N_2 -purged 0.1 M acetate buffer at pH 4 under chopped irradiation.

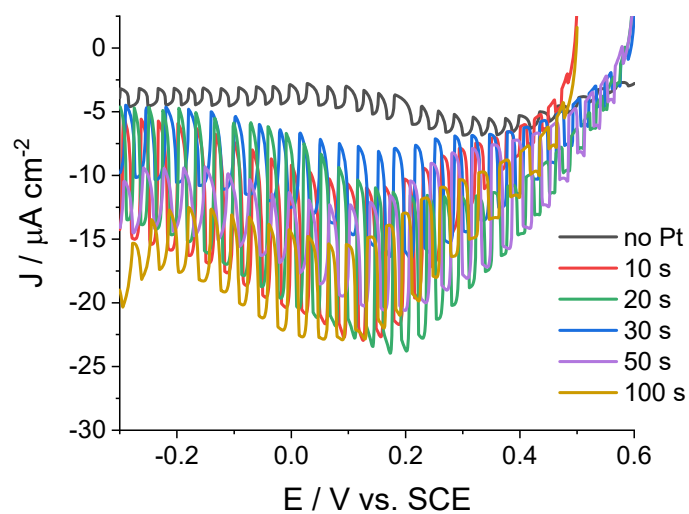


Figure S24. LSV of NiO-B|**P1**|Pt-**1** at different deposition times in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation.

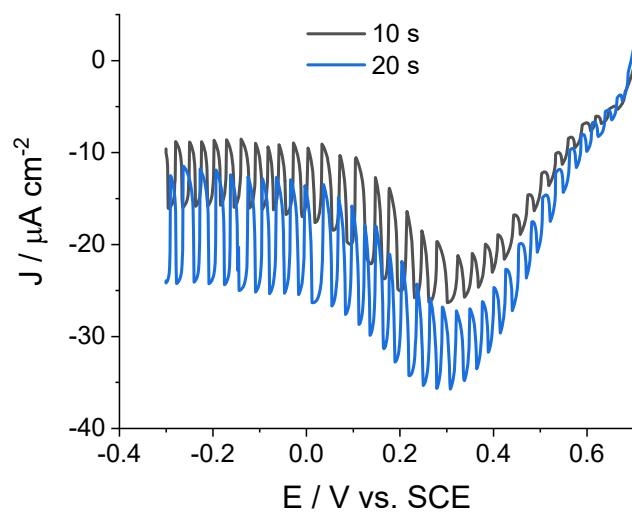


Figure S25. LSV of NiO-B|**P1**|Pt-**2** at different deposition times in N₂-purged 0.1 M acetate buffer at pH 4 under chopped irradiation.