

Unraveling the Intermediate Species of Co_3O_4 Hollow Spheres for CO_2 Photoreduction by In Situ X-ray Absorption Spectroscopy

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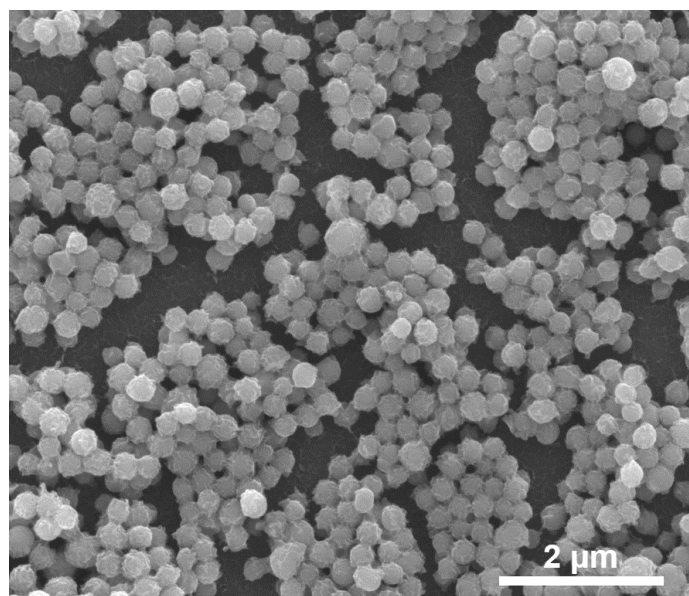


Figure S1. SEM image of the Co-glycolate spheres.

Table S1. The fitting parameters of EXAFS spectrum of Co₃O₄ hollow spheres.

Vector	N	Before Catalysis		At Equilibrium	
		R(Å)	$\sigma^2 \times 10^{-3} (\text{\AA}^2)$	R(Å)	$\sigma^2 \times 10^{-3} (\text{\AA}^2)$
Co-O	4.6	1.93±0.02	2.7	1.91±0.02	4.8
Co-Co ₁	4	2.86±0.02	2.9	2.87±0.02	4.0
Co-Co ₂	8	3.36±0.02	6.6	3.36±0.02	8.1

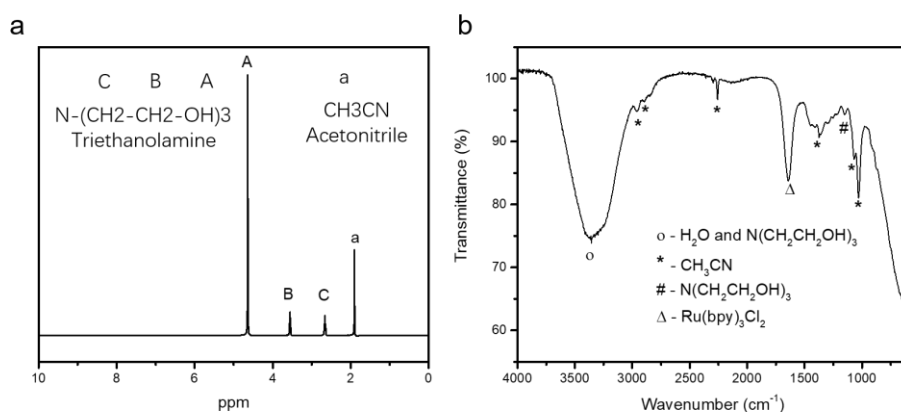


Figure S2. (a) ¹H NMR spectrum (in D₂O) and (b) FTIR spectrum of the product.

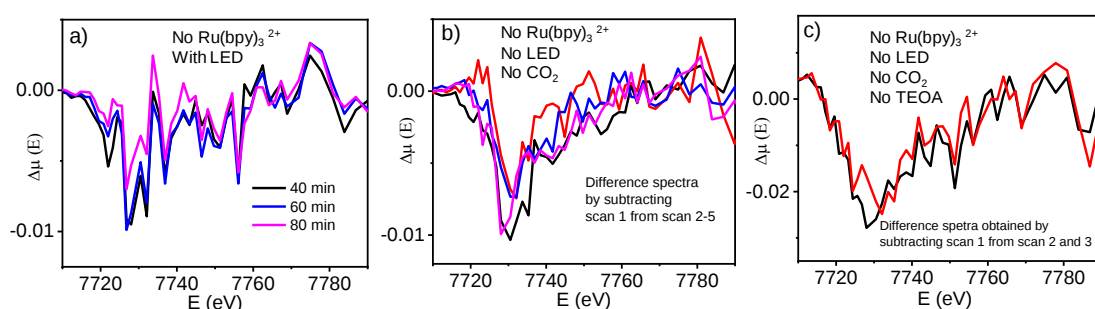


Figure S3. The difference spectra of in situ XAS of Co₃O₄ hollow spheres under different conditions. a) LED on, no Ru(bpy)₃²⁺; b) LED off, no Ru(bpy)₃²⁺, no CO₂; c) LED off, no Ru(bpy)₃²⁺, no CO₂; no TEOA.

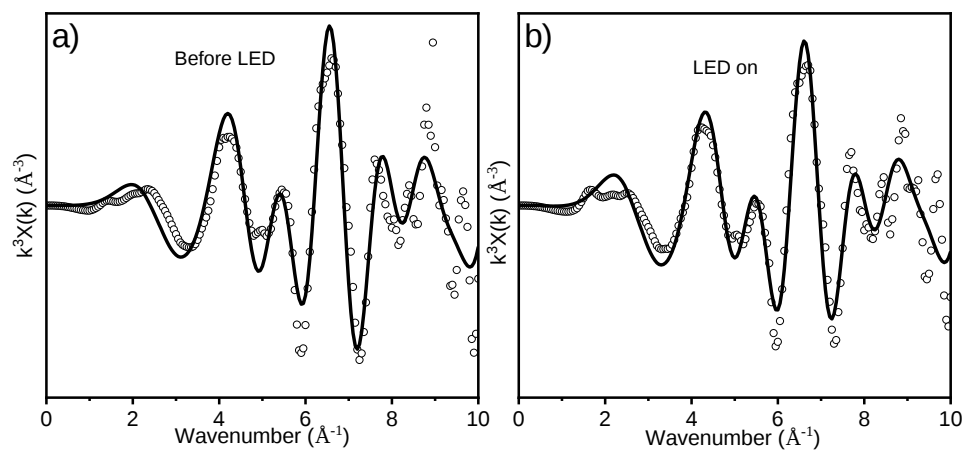


Figure S4. EXAFS of Co_3O_4 hollow spheres before LED illumination (a) and during LED illumination (b) in k -space. The open dots and solid lines represent the experimental and fitting results.