

**Unveiling the influence of alkaline modifiers in CuO synthesis on its photocatalytic activity  
for CO<sub>2</sub> reduction**

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## Experimental section

### *Characterization*

X-ray diffraction (XRD) The crystalline phases were characterized by X-ray diffraction (XRD) over the  $2\theta$  range from 20 to 80° using a Shimadzu XRD-6000 diffractometer operating with  $\text{CuK}\alpha$  radiation. A Fourier Transform Infrared spectrometer (FTIR) (Bruker VERTEX 70) was used to investigate surface changes using sample-containing KBr disks with 64 scans and  $4\text{ cm}^{-1}$  resolution in the  $4000 - 400\text{ cm}^{-1}$ . Sample morphology and particle size were analyzed by field emission gun scanning electron microscopy (FEGSEM) JEOL JSM 6701 F.

X-ray photoelectron spectroscopy (XPS) was employed to analyze the surface chemical composition of the catalyst materials. The analysis was performed using a Phi 5000 VersaProbe II instrument (ULVAC-PHI Inc., USA). The X-ray source was monochromatic Al  $\text{K}\alpha$  radiation (1.486 keV) with operating settings of 50 W power, 15 kV accelerating voltage, and a 200  $\mu\text{m}$  spot size. Survey spectra were acquired using a pass energy of 187.5 eV, a step size of 0.8 eV, and a dwell time of 100 ms per step. Detailed high-resolution spectra were obtained using a pass energy of 23.5 eV, a step size of 0.1 eV, and a dwell time of 100 ms per step.

$\text{N}_2$  adsorption-desorption isotherms were measured with a Micromeritics ASAP 2020 analyzer at 77 K. Samples were previously degassed at 80 °C under vacuum until a degassing pressure  $<10\ \mu\text{mmHg}$ . The Brunauer–Emmett–Teller (BET) method was used to calculate the specific surface area (SSA)The zeta potential was measured at room temperature using a Zetasizer Nano-ZS analyzer (Malvern Instruments, UK), at natural pH.

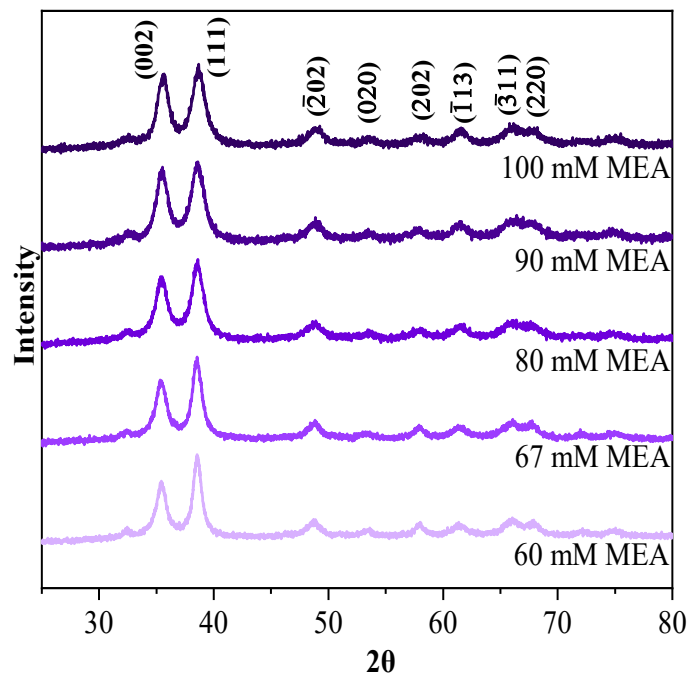
The diffuse reflectance (DRS) spectra (Shimadzu UV-2600) were used to calculate the band gaps using the Tauc technique in the ultraviolet-visible spectrum.

Field emission scanning electron microscopy (JEOL JSM 6510) was used to examine the morphology of the materials.

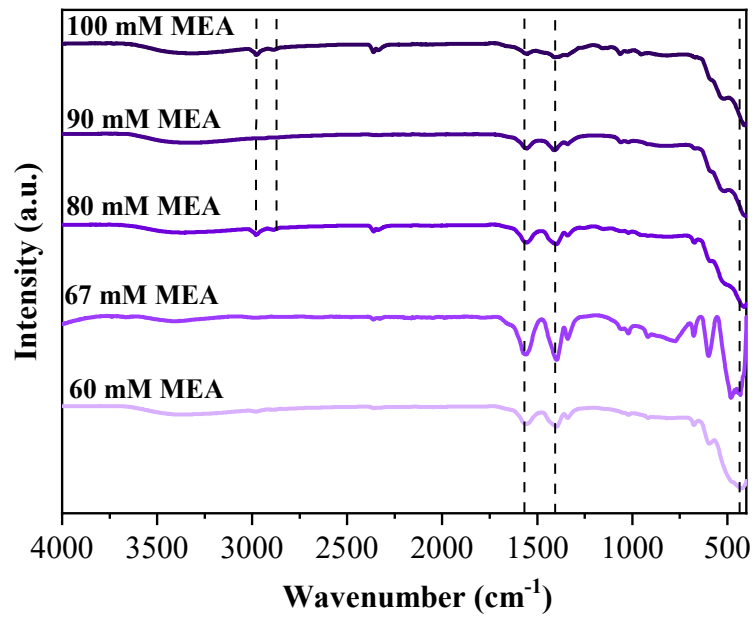
High-resolution transmission electron microscopy (HRTEM) images were obtained using a JEOL microscope (Model JEM2100 LaB6) operating at 200 kV.

## Results and discussion

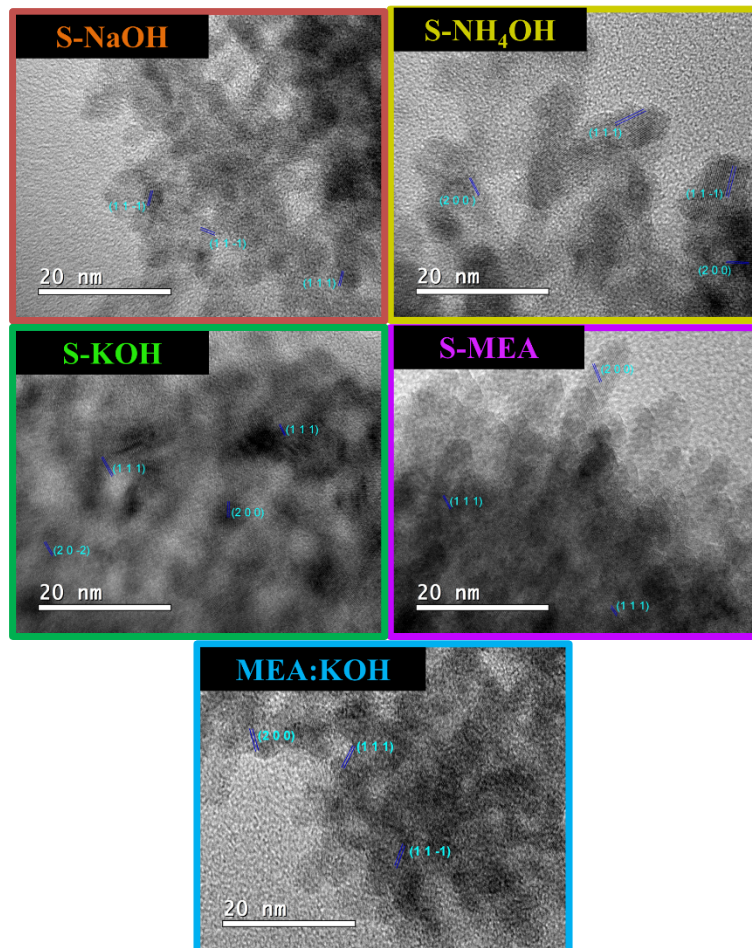
### *Characterization*



**Figure S1.** X-ray diffractograms of samples prepared with 60 to 100 mM of MEA.



**Figure S2.** FTIR spectra of sample S-MEA in concentrations from 60 to 100 mM MEA concentration in (b).

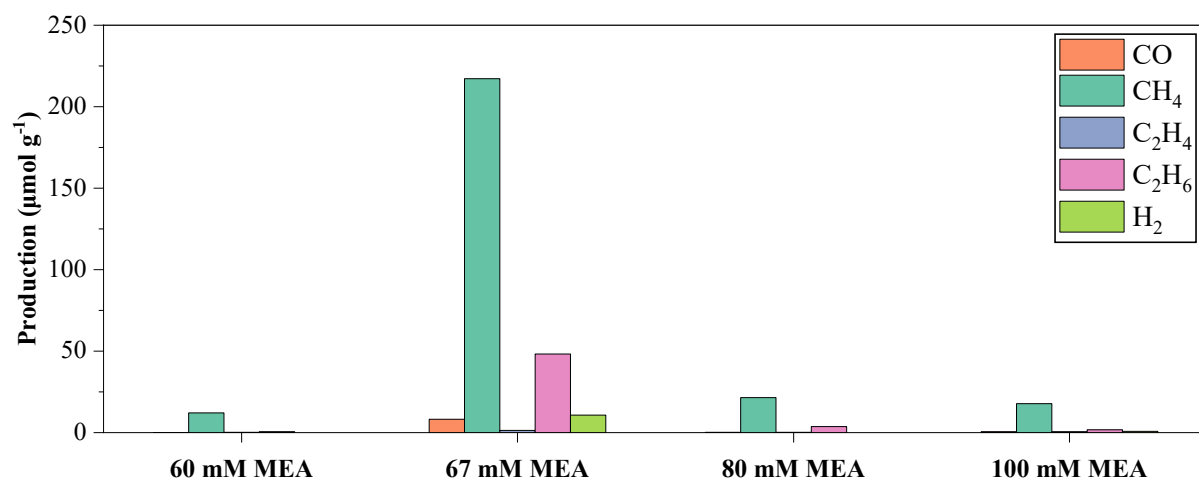


**Figure S3.** HRTEM images of the samples S-NaOH, S-NH<sub>4</sub>OH, S-KOH, S-MEA, and MEA-KOH with the CuO interplanar spaces indicated.

### *CO<sub>2</sub> Photoreduction Performance*

Varying the MEA concentration during catalyst synthesis revealed 67 mM to be the optimal condition for maximizing production rates. This observation is attributed to the influence of MEA concentration on the final pH of the reaction solution. The initial pH of the acetate solution was the same across all samples, consequently, the addition of different MEA concentrations resulted in varying final pH values, which likely influenced both the surface functionalization and

crystallinity of the samples. These findings underscore the critical role of pH in determining the physicochemical properties and catalytic performance of the CuO nanostructures.



**Figure S4.** Production rates after 5 hours of photocatalytic reduction tests of sample S-MEA in concentrations from 60 to 100 mM MEA