Supporting Information

Electrochemical Nitrate Reduction to NH_3 by Faceted Cu_2O Nanostructures in Acidic Medium

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Analysis of Electrochemical NO_3^- Reduction Product and Quantification

Preparation of standard NH_3 solution: To prepare a standard NH_4Cl solution 38.20 mg of NH_4Cl was dissolved in 100 mL DI water and further diluted as per requirement.

Preparation of standard phenol solution: 5 mL phenol was added to 45 mL of ethanol to obtain 50 mL of 1.0 M phenol solution.

Preparation of standard sodium nitroprusside solution: To prepare a 17 mM 50 mL solution of sodium nitroprusside, 200 mg sodium nitroprusside was into 50 mL water.

Preparation of standard sodium hypochlorite solution: An alkaline solution of tri-sodium citrate and sodium hydroxide was prepared by dissolving 20 g sodium citrate and 1 g NaOH in 100 mL DI water. Finally, this alkaline solution and sodium hypochlorite was added in 4:1 ratio to obtain alkaline sodium hypochlorite solution for colorimetric NH_3 detection.

Detection and quantification of NH_3 in the reaction mixture: NH_3 produced in electrochemical NO_3^- reduction by the Cu_2O based NCs was detected by the IPB method using phenol, sodium nitroprusside and alkaline sodium hypochlorite

solution. First, after electrochemical NO_3RR for 3h, 2 mL aliquot of the electrolyte was taken out and 3 mL of 1.0 M NaOH solution was added into it to make the solution alkaline. From this alkaline electrolyte solution, 1 mL diluted electrolyte solution was taken up and mixed with 7 mL DI water. To this solution, 500 µL, 500 µL and 1 mL of previously prepared phenol, sodium nitroprusside and sodium hypochlorite solution was added respectively and kept for 2 h incubation at ambient condition. This IPB coloured solution was further diluted as required to detect UV-Vis spectra at the reasonable absorption maxima value. In fact, a total of 50-time dilution was made prior to detection of NH_3 through UV-Vis spectra. The UV-Vis spectrum of the solution was recorded by a single beam UV-Vis spectrophotometer and the absorbance value at 630 nm was determined. A series of known concentrations of NH_4Cl solution were used to calibrate the absorbance values.

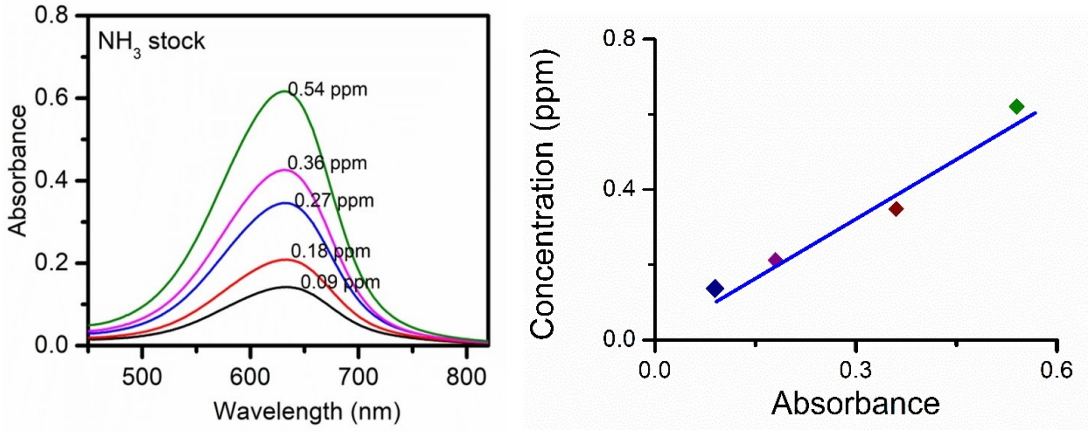

Fig. SI 1. (Left side) Indo-phenol complex formation at different concentration of NH_3 as shown in the graph. (Right side) Linear relationship of the Indo-phenol complex with NH_3 concentration at 630 nm passing through the origin.