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Binder-free Setup to Assess Magnetic Nanocatalysts in a Study of Novel CuxOy-IONP-clusters for Electrochemical Nitrate Reduction

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Electrochemical reduction enhanced by nanocatalysts has big potential for applications in sensing or conversion reactions (e.g., CO2) due to high catalytic activity, selectivity and large surface area 1. To function properly, these catalysts have to be electrically connected to a working electrode (WE) in an electrochemical setup to apply a respective electrical potential. As conductive polymer binders like poly-3,4-ethylendioxythiophen (PEDOT) [2] are expensive and raise environmental concerns, this work aimed to magnetically attach the catalysts. As model system electrochemical reduction of NO3- on Cu-based catalysts was chosen as promising approach for environmental applications or synthesis of NH3 [2].

In a first step, ferrimagnetic large single domain iron oxide nanoparticles (IONP; prepared at 35 °C following [3]) were functionalized by electroless Cu deposition (adapted from 4), induced by intensive light irradiation for 1h (white light LED, 60 W). The resulting CuxOy-IONP-clusters exhibited a Cu-content of βCu ~ 17 –27 wt-% (atomic absorption spectroscopy), a spherical morphology with fuzzy surface (d ~ 500 nm, transmission electron microscopy, fig. 1 a) consisting of a mixture of CuO, Cu2O and Cu(OH)2 (X-ray photoelectron spectroscopy).

To assess their catalytic activity for NO3–reduction, the clusters were applied to a masked glassy carbon holder with an exposed circular surface of A ~ 0,049 cm². The clusters were held in place by a NdFeB permanent magnet on the back. The holder served as binder-free WE in an electrochemical setup (counter electrode: Pt; reference electrode: Ag/AgCl (sat.); fig. 1 b, c). Cyclic voltammetry (EWE = 0.5 to 1.2 V; dE/dt = 20 mV/s) and chronoamperometry (EA = 1,2 V) were performed. In Ar-purged 0.5 M K2SO4 solution current densities in the µA/cm² range were obtained. Adding KNO3 (0.01 M) resulted in current densities in the mA/cm² range, proving sensitivity for NO3-. Faradaic efficiencies for the NH4+ production of up to 94% were measured photometrically with NH4+-test kits, proving good catalytic performance.

The binder-free electrochemical setup can be used to investigate other magnetic nanocatalysts (e.g., Au-, Pt-, Ag-coated IONP, Co-ferrites) and catalytic reactions like CO2-reduction.

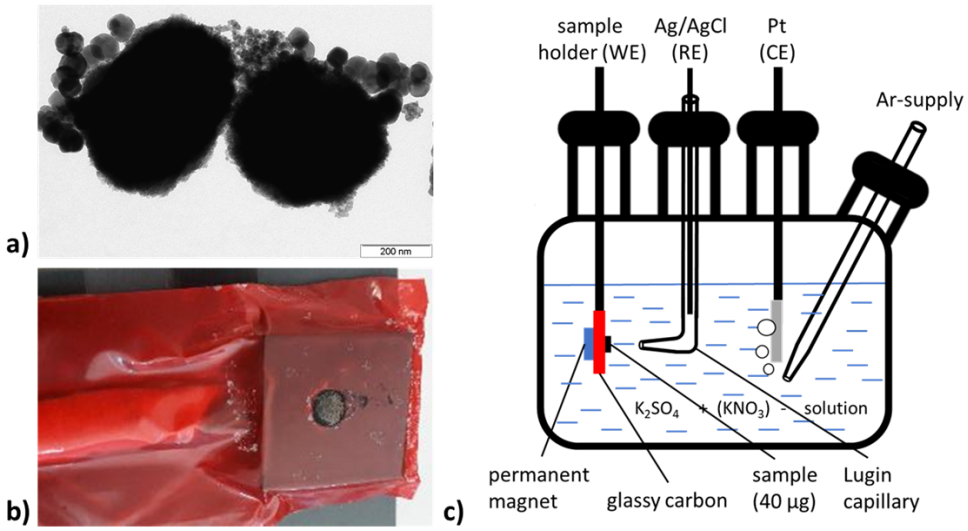

Fig. 1) a) TEM-image of Cu_xO_y-IONP-clusters **b)** loaded sample holder **c)** Electrochemical setup with loaded sample holder as WE

Figure 10: Photographs of (a) millimeter-scale UCST hydrogels containing MNPs, (b) ground UCST hydrogels containing MNPs, and (c) DLS results.

References:
1 Gawande et al., 2015;
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