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Characterization of Cu nanoparticles via X-ray photoelectron spectroscopy in combination with SESSA calculations

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Copper nanoparticles (NP) have attracted great interest due to their unique properties which make them ideal candidates for applications in catalysis [1].

Within this study we have investigated the properties of nanoparticles produced in ionic liquid-aqueous micellar systems, which would provide a media for further coupling reactions.
Despite the protective stabilizing environment of the ionic liquid, several question like possible ox-ide/hydroxide-shell formation within the aqueous solution including parameters, e.g. coverage and thickness of these layers need to be addressed to understand NP behavior in possible applications.
Therefore, we have employed X-ray photoelectrons spectroscopy (XPS) in combination with theoretical calculations using the SESSA software package [2, 3].

Cu NP were formed via chemical reduction of $CuCl_2$ using sodium borohydride or hydrazine in ionic liquid (1-alkyl-3-methyl-imidazolium chloride)/aqueous solutions [4, 5].

Cu 2p and Cu LMM signals of the different NP were recorded and compared to signals from high-purity planar Cu and Cu-oxide reference samples.
As Cu 2p signals show no traces of Cu(II), we determined the Cu(0)/Cu(I) ratios from the Cu LMM signals.

Subsequently, different over-layer thicknesses have been calculated using SESSA. Assuming homogeneous coverage, the thickness of the oxide layer can be determined in the sub-nm range with accuracy better than one monolayer.

We could evidence the formation of few atomic layers of oxides, which exhibit varying thickness due to changes in the fabrication process. These variations can distinctly influence the NPs properties when they are used in catalytic reactions, thus proving that XPS is an essential tool for NP metrology.

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