

Stabilization of Copper Oxide Nanoparticles in (w/o) Microemulsions: Experimental and Modeling Results

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1. Introduction

Formation of well-dispersed colloidal nanoparticle catalyst is crucial for heterogeneous catalysis in organic media. However, maintaining stable dispersion of the particles is a challenging task since these particles tend to agglomerate with time in order to reduce the high surface energy. One approach of minimizing particle agglomeration is to prepare the fresh catalyst *in-situ*. Very high reaction rates were attained when *in-situ* prepared ultradispersed catalysts were employed for hydrocracking, coprocessing, and coal liquefaction. Water-in-oil (w/o) microemulsions, or reverse micelles, are very good candidates for the *in-situ* catalyst preparation, since they provide control over the particle size, and produce stable ultradispersed nanoparticles. It was found that microemulsion-prepared nanoparticle catalysts exhibited smaller particle size and showed higher catalytic activity compared with the ones prepared by the conventional techniques.

2. Objectives

An *in-situ* approach of copper oxide nanoparticle preparation, in single AOT/water/isooctane microemulsions is demonstrated. The effect of different microemulsion and operating variables, including surfactant concentration, water content and mixing time, on the colloidal concentration (nanoparticle uptake), size and stability of copper oxide nanoparticle in AOT/water/isooctane microemulsions is evaluated. In addition, a mathematical model describing the effect of the aforementioned variables on the nanoparticle uptake and stability is provided.

3. Methodology

The colloidal nanoparticles are prepared *in-situ* by exposing AOT/water/isooctane microemulsions to a soluble copper precursor followed by addition of NaOH at a later stage. Upon addition of NaOH, copper oxide nanoparticles stabilized in the water pools form in addition to a bulk precipitate at the bottom. The concentration of the colloidal copper oxide and the particle size are then measured at different times using inductive coupled plasma (ICP), UV-Vis spectroscopy and transmission electron microscope (TEM). Monitoring the concentration of colloidal metal oxide particles and their sizes with respect to time gives indication on their stability.

4. Results:

The experimental results have shown that particle size increased with increasing the surfactant concentration, concentration of the precursor salt, and water to surfactant mole ratio. The nanoparticle uptake increased linearly with the surfactant concentration, and displayed an optimum with R and a power function with the concentration of the precursor salt. The model, which was based on water uptake by Winsor type II microemulsion, described well the effect of the variables considered in the study on copper oxide nanoparticle uptake. Below are some samples of our finding. Figures 1 to 3 show a maximum copper oxide nanoparticle uptake coupled with an increase in the particle size, and broader size distribution as the value of R increased. Figure 1 shows that the maximum uptake occurs at $R = 5.0$. The increase and decrease in the nanoparticle uptake reported in Figure 1 is captured by the increase and the decrease in the size of the UV-absorption peaks of Figure 2. The TEM images of Figures 3a,b show an increase in the nanoparticle size and size distribution.

References:

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Figures:

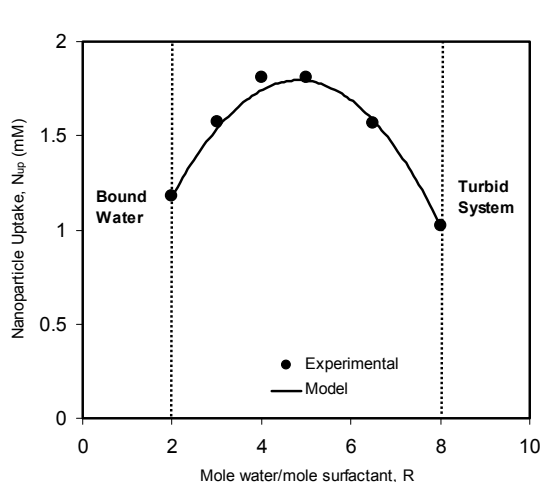


Figure 1. Nanoparticle uptake as a function of water to surfactant mole ratio, R , obtained when mixing a stoichiometric amount of CuCl_2 -containing microemulsions $\{[\text{CuCl}_2] = 3.0 \text{ mM}, [\text{AOT}] = 200 \text{ mM}, R = 3.0\}$ with NaOH for 3 h at 300 rpm.

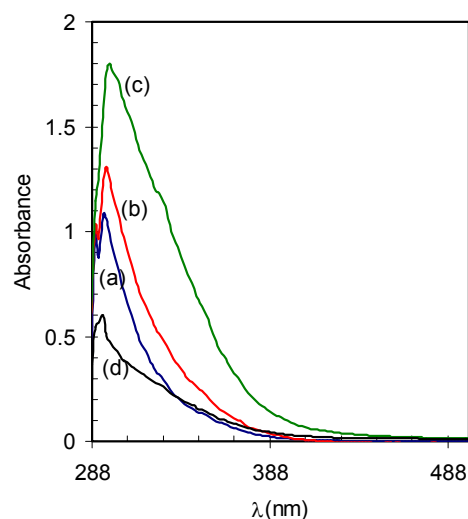


Figure 2. UV-absorption spectra of colloidal copper oxide nanoparticles obtained when mixing a stoichiometric amount of CuCl_2 -containing microemulsions $\{[\text{CuCl}_2] = 3.0 \text{ mM}, [\text{AOT}] = 200 \text{ mM}, (a) R = 2.0; (b) R = 3.0; (c) R = 5.0; (d) R = 8.0\}$ with NaOH for 3 h at 300 rpm.

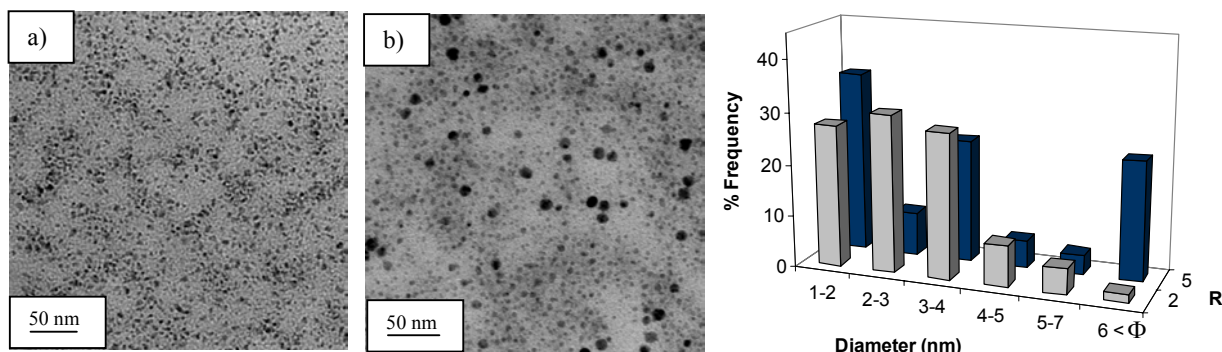


Figure 3. TEM photographs and the corresponding particle size distribution histograms obtained when mixing a stoichiometric amount of CuCl_2 -containing microemulsions $\{[\text{CuCl}_2] = 3.0 \text{ mM}, [\text{AOT}] = 200 \text{ mM}, (a) R = 2.0; (b) R = 5.0\}$ with NaOH for 3 h at 300 rpm.