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Exploitation of the nanostructure of electrocrystallized ramified branches for an alternative synthesis of colloidal metallic nanoparticles

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We explore an alternative synthesis of colloidal metal nanoparticles (cMnP) by electrocrystallization. The principle is to make grow long metallic ramified branches by galvanostatic electrolysis of a stagnant metal salt aqueous solution inside a Hele-Shaw (thin gap) cell. In the absence of supporting electrolyte, the constraint of electroneutrality forces the deposit to grow as ramified branches. These branches grow by successive nucleation / growth events that leads to a fine branches structure made up of metal crystals whose size can be lower than 100 nm (1). The objectives of this work are to study the formation mechanism of such metal nanocrystals assemblies and elaborate a device able to dissociate and release them to propose an alternative synthesis of cMnP.

Concerning the formation mechanism, the growth of copper (from CuSO₄) and iron (from FeCl₂) branches have been studied varying systematically the two main parameters, the metal salt concentration C (0.01 - 0.5 M) and the applied current density J (1 - 500 mA/cm²) in a cell of 50 μm in depth maintained horizontally. For each experiment, the branches pattern (optical visualizations) and the branches structure (SEM) are collected. These data are analyzed with the support of theoretical modeling at both macro and micro scales. The main new result is the observed dependence of both the size and the form of the crystals, constituting the branches, on J . Indeed, for a given C , the SEM observations reveal that, at high J , for Cu and Fe, the branches are composed of dendrites ($\sim 10\ \mu\text{m}$ maximum length) on which small non-dendritic crystals ($\sim 100\ \text{nm}$) have nucleated and grown. The amount and the size of these crystals decrease when J increases. Conversely, for Cu, and for the lower J values investigated, no dendritic structure is observed, the branches are only composed of piles of non-dendritic crystals with rather wide sizes distribution ($\sim 100\ \text{nm}$ - $1\ \mu\text{m}$). This observed competition between out of equilibrium crystalline growth (dendrites growth) and nucleation is discussed considering the growth velocities for both non-dendritic crystals (Faraday law) and dendritic crystals (from the tip velocity provided by the dendrites growth theory) and also the required equality between anions migration velocity and the average growth velocity (1).

The dissociation and the release of Fe nanocrystals have been attempted in a specific microfluidic cell on which a vibrating element (PZT disc) has been integrated. The branches fragmentation into fine particles ($< 20\ \mu\text{m}$), involving the oscillations of co-formed H₂ bubbles, is observed whatever J and C used. From Fe branches composed mainly of dendrites (80 mA/cm², 0.1 M), submicrometric Fe dendrites (length $\sim 500\ \text{nm}$ arms diameter $\sim 50\ \text{nm}$, TEM observation) have been produced (2). These particles have a specific surface area $\sim 10^8\ \text{m}^{-1}$ comparable to spherical nP of 30 nm in diameter. Their chemical activity has been evaluated for the dechlorination of trichloroethylene and the results suggest that the whole surface is active. One important advantage of this method is the possibility of flushing the branches (and so the cMnP) before their fragmentation to avoid subsequent purification step as usually required for the widely used colloidal synthesis.

- (1) Brady, R.; Ball, R. *Nature* **1984**, 309, 225; Fleury, V. *Nature* **1997**, 390, 145.
- (2) Iranzo, A.; Chauvet, F.; Tzedakis, T. *Electrochim. Acta* **2017**, 250, 348.