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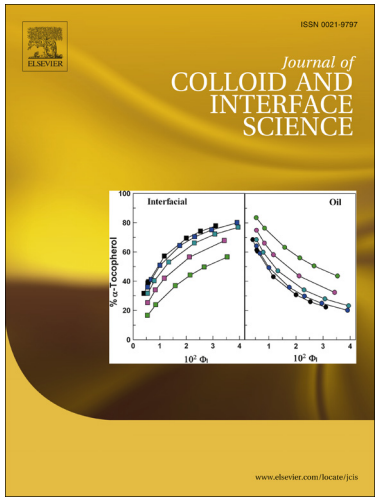

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Insights into the Synthesis of Layered Double Hydroxide (LDH) Nanoparticles: Part 1. Optimization and Controlled Synthesis of Chloride-Intercalated LDH

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ABSTRACT

Layered double hydroxide (LDH) nanoparticles have excellent anion-intercalating property, and their potential as theranostic nanovectors is high. However, understanding of the control of the mean particle size (MPS) and achievement of monodispersed particle size distribution (PSD) remains elusive. Herein, with the aid of statistical design of experiments on a model system of Cl^--intercalated (Zn, Al)-LDH, controlled synthesis of single crystalline nanoparticles using the coprecipitation method followed by hydrothermal treatment (HT) is achieved in three steps. First, a 2^{4-1} design enabled the identification of influential parameters for MPS (i.e., salt concentration, molar ratio of carbonate to aluminum, solution addition rate, and interaction between salt concentration and stirring rate) and PSD (i.e., salt concentration and stirring rate), as well as the optimum coprecipitation conditions that result in a monodispersed PSD (i.e., low salt concentration and high stirring rate). Second, a preliminary explanation of the HT was suggested and the optimum HT conditions for obtaining ideal Gaussian PSD with chi-squared (χ^2) < 3 were found to be 85 °C for 5 h. Third, using a central composite design, a quantitative MPS model, expressed in terms of the significant factors, was developed and experimentally verified to synthesize nearly monodispersed LDH nanoparticles with MPS ~200-500 nm.