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Materials Letters 59 (2005) 210–214

materials letters

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Formation of calcium phosphate nanoparticles in reverse microemulsions

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Received 14 May 2004; received in revised form 16 August 2004; accepted 22 August 2004
Available online 27 October 2004

Abstract

Nanosized calcium phosphate powder had been synthesized via an inverse microemulsion processing route, which used cyclohexane as the oil phases, octaethylene glycol monododecyl ether ($C_{12}EO_8$) as surfactant phase, and an aqueous $Ca(NO_3)_2$ and $(NH_4)_2HPO_4$ solution as the water phase. A wide variety of morphologies were encountered in synthesis which produced nanosphere (25–40 nm in diameter), sheet-like (3–16 nm in width and 50–300 nm in length), rod-like (10–17 nm in diameter and 24–50 nm in length) and needle-like (4–8 nm in diameter and 80–100 nm in length). The great structural diversity was resulted from the different value of the molar ratio of water to surfactant, W_0. Particles were visualized by transmission electron microscopy (TEM) and the identities of powder confirmed by FT-IR and X-ray diffraction (XRD).
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Keywords: Nanoparticles; Microemulsion

1. Introduction

Microemulsions, particular in W/O microemulsions, have received considerable attention as reaction media for the synthesis of nanomaterials. Such as oxides carbonate, silver chloride, sliver bromide, and high Tc $Yba_2Cu_3O_{7-x}$ [1–4]. Reverse micelles can solubilize relatively large amounts of water in organic solvents. It is this water pool that makes the reverse micelles particularly favorable for the synthesis of nanoparticles because the water pool is in the range of nanometer size which can be controlled by adjusting the water content. Solubilization of water in the reverse micelles can be expressed by W_0, molar ratio of water to surfactant (W_0=H_2O/Surfactant). W_0 is an important parameter in determining the size of the reverse micelles and the structure of water. The morphology of produces is often sensitive to the W_0. As a result, different shapes of nanosized particles could be synthesize at different value of W_0, such as nanowires [5,6], nanocones [7,8], nanoneedles [9], nanorods [10,11], and nanospheres [12,13].

Calcium phosphate has been used as artificial bone and tooth substitute. Besides its significance in biology, calcium phosphate is also a good candidate for applications in catalysts and ion exchangers due to its unique surface structure and substitutions. The preparation of calcium phosphate powders with given characteristics of morphology, stoichiometry, crystallinity, and crystal size distribution is important in biomedicine and material science. For example, to coat calcium phosphate onto silicone substrate with covalent linkage, calcium phosphate particles with flat and wide plane are essentially necessary [14]. Various processes have been employed to prepare calcium phosphate powders, including chemical coprecipitation [15], sol–gel process [16,17], spray–pyrolysis [18], hydrothermal synthesis [20,19], emulsion processing [21–23], mechano–chemical method [24], and autocombustion methods [25].

In this paper, we described the use of W/O microemulsion system for the formation of calcium phosphate nanoparticles. Various morphologies such as needle, sphere rod, and sheet-like were obtained because of different reaction conditions. The aim of this study was to further

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doi:10.1016/j.matlet.2004.08.037

our understanding of these processes by seeking some general features which could control the synthesis of calcium phosphate nanoparticles.

2. Experimental

2.1. Materials and methods

Analytical-grade cyclohexane, octaethylene glycol monododecyl ether ($C_{12}E_8$), $(NH_4)_2HPO_4$ (0.12 M aqueous solution), and $Ca(NO_3)_2$ (0.2 M aqueous solution) were used as reactants. pH was adjusted to 10 by $NH_3 \cdot H_2O$.

As a typical synthesis, two identical solution were prepared by dissolving $C_{12}E_8$ (9.415 g) in 50 ml of cyclohexane. The reverse micellar solution was transparent. Under drastical stirring, 0.12 M $(NH_4)_2HPO_4$ and 0.2 M $Ca(NO_3)_2$ aqueous solution were injected into the reverse micellar solution synchronously. The solution kept transparent until 4 days at room temperature. The value of W_0 was 5, 8, 10, and 5, respectively. After 4 days of reaction, a white product was collected by centrifugation, washed repeatedly with hot ethanol, then dried it to solid in 50 °C for 4 h.

2.2. Characterization

Morphology of powders as-synthesized were dispersed in ethanol and characterized by transmission electron microscopy (TEM; PHILIPS CM300), using an accelerating voltage of 300 kV. Using IR spectra which were obtained with a Perkin Elmer System 2000 FT-IR Spectrophotometer, the samples were dispersed in KBr. The phase composition and crystallinity of the calcined powders were analyzed by X-ray diffraction (XRD) using a Brüker AXS, D8 Advance Diffractometer with CuKa radiation at 40 kV and 40 mA.

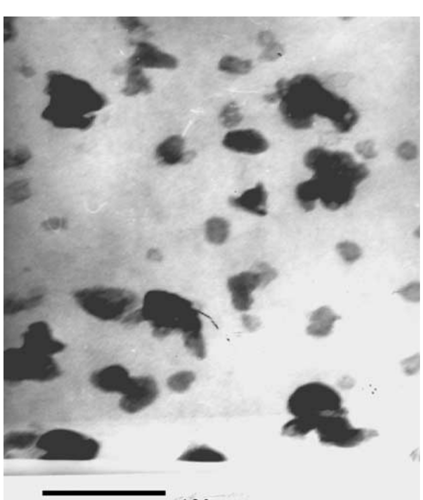

Fig. 1. TEM micrographs of irregular spherical calcium phosphate nanoparticles formed from W_0=5, 0.35 M $C_{12}E_8$ in cyclohexane microemulsion (scale bar=130 nm).

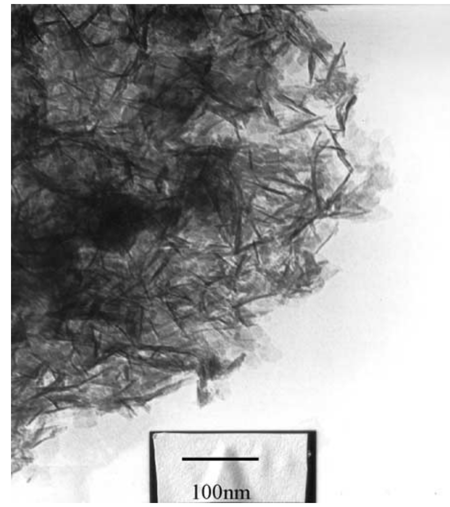

Fig. 2. TEM micrographs of needle-like calcium phosphate nanoparticles formed from W_0=8, 0.35 M $C_{12}E_8$ in cyclohexane microemulsion (scale bar=100 nm).

3. Results and discussion

Fig. 1 showed the morphology of calcium phosphate particles produced in microemulsion at W_0=5. After 4 days, the inorganic material was in the form of an irregular spherical particle (Fig. 1), showing the individual nanospheres in the region of 25–40 nm in diameter with curved and round edges. As mentioned above, reverse micelle could form different morphology in microemulsion depending on the value of W_0. When W_0 was low as in our experiment W_0=5, the morphology of reverse micelle were

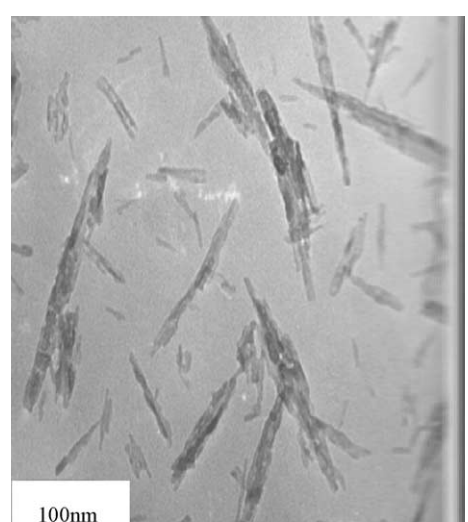

Fig. 3. TEM micrographs of long sheet-like calcium phosphate nanoparticles formed from W_0=10, 0.35 M $C_{12}E_8$ in cyclohexane microemulsion (scale bar=100 nm).

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