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Synthesis of a New Polyoxometalate Loaded Stearic Acid Nanoparticles

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Abstract: The stearic acid nanoparticles loaded polyoxometalate $K_6[\gamma\text{-}(CpTi)_2SiW_{10}O_{38}]$ $[(CpTi)_2SiW_{10}]$ have been prepared and structurally characterized by elemental analysis, IR spectra. The particle size was estimated by transition electron microscope and zatesizer instrument. The result showed that the polyoxometalate retained the parent structure after encapsulation by stearic acid nanoparticles.

Keywords: Polyoxometalate, stearic acid nanoparticles, preparation.

Recently, many methods have been used to prepare polyoxometalates (POMs) in nanosize[1,2], but these methods were still limited in application of POMs used as drug deliveries.

Drug carrier material system has attracted more and more interest in controlled release applications. Our group has prepared and characterized the system of liposome encapsulated polyoxotungstate[3] and the system of polyoxometalate/starch nanomaterial[4]. Since the beginning of the nineties more and more attention has focused on a new drug carrier material system --- the solid lipid nanoparticles (SLN)[5-6]. SLN has many advantages such as improving the stability than the liposome, the possibility of controlling drug release and drug targeting. Stearic acid is a main composition of fat, and has better biocompatibility and low toxicity, and it is solid at room temperature. Stearic acid is available for pharmaceutical use and it is easy to prepare the nanoparticles.

Recently we use stearic acid as the matrix and combine the drug carrier system with POM to prepare new POM complex materials, which has more hydrolytic stability at physiological pH. Here we report the preparation of $(CpTi)_2SiW_{10}$[7] loaded stearic acid nanoparticles.

Stearic acid nanoparticles were prepared by emulisification and solvent evaporation method. Definite amount of stearic acid and lecithin were dissolved in 10 mL of $CHCl_3$ and 10 mL of CH_3COCH_3, and melted together at 70°C. Under stirring a water solution of $(CpTi)_2SiW_{10}$ and polyoxyethylene 40 stearate were added to obtain an optically transparent system. Continuing heating until the volume of the dispersion is about 5 mL, then the dispersion was immediately dispersed in cold water (2-3°C) at 1:10 ratio under

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mechanical stirring for 2 h, obtaining stearic acid nanoparticles. The untrapped $(CpTi)_2SiW_{10}$ was separated by dialysis at 4°C for 24 h. The dispersion was freeze-dried for 72 h with sucrose as cryoprotectant. The dry powder was sealed and stored in a refrigerator at 4°C. The contents of $(CpTi)_2SiW_{10}$ were analyzed by the photometric method.

The elemental analytic results of the POM loaded stearic acid nanoparticles indicated W, 7.80; Si, 0.23; Ti, 0.81; K, 0.9% respectively. It can be seen that when the POM was encapsulated by stearic acid nanoparticles, the ratio of W:Ti:Si is still 10:2:1, indicating that the structure of $(CpTi)_2SiW_{10}$ did not change during the process. The encapsulate efficiency = weight of encapsulated drug/added total weight of drug×100%, where the weight of encapsulated drug = added total weight of drug – the weight of uncapsulated drug. The weight of the uncapsulated drug was determined by the photometric method. From the experimental data, the standard curve of $(CpTi)_2SiW_{10}$ at 263 nm was a staight line. The standard curve equation was $A = -0.04513 + 0.00645C$, $R = 0.99611$ ($n = 6$). (A is absorbancy, C is concentration, R is linear relatively coefficient, n is number) Therefore the encapsulation efficiency of $(CpTi)_2SiW_{10}$ loaded stearic acid nanoparticles is 71.3%, and the contents of $(CpTi)_2SiW_{10}$ in stearic acid nanoparticles are 3%. The IR spectrum of the polyoxometalate loaded stearic acid nanoparticles shows the absorption peaks at 980, 950, 900, 870, 810, 750 cm^{-1}, which are charateristic peaks of heteropolyalions with Keggin structure of W-Od, W-Ob-W, W-Oc-W and Si-Oa. The peak at 1468 cm^{-1} shows a sharp absorption, which is charateristic of the C-C stretch for a η^5-C_5H_5 ligand bonded to Ti[8]. It confirms that the POMs did not change during encapsulation.

The transmission electron micrographs (TEM) (**Figure 1**) show that the stearic acid nanoparticles form relatively uniform spherical particles and the particles are not agglomerated. The size distribution (**Figure 2**) of stearic acid nanoparticles estimated by Zatesizer indicated that the size of the particles is distributed in two uncontinuous limits, in which 80% of the stearic acid nanoparticles distributed in the range of 245-308 nm, the average diameter is 260.3 nm; and 20% of the stearic acid nanoparticles distributed in range of 616-775 nm.

Figure 1 The TEM of the POMs loaded stearic acid nanoparticles

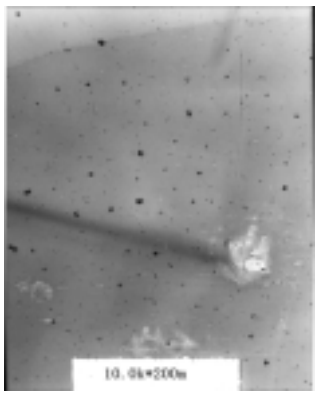
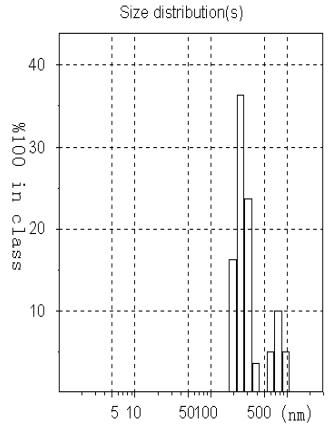


Figure 2 The size distribution of the POMs loaded stearic acid nanoparticles



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References

1. E. Coronado, C. J. Gomez-Gracia, *J. Comments Inorg. Chem.,* **1995**, *17*, 255.
2. E. Coronado, P. Delhaes, J.R. Galan Mascaros, *et al., Synth. Met.,* **1997**, *85*, 1467.
3. Y. Yang, J. H. He, X. H. Wang, B. Lin, J. F. Liu, *Trans. Met. Chem.,* in press.
4. X. H. Wang, J. F. Liu, M. T. Pope, *Dalton Trans.,* **2003**, 957.
5. R. H. Muller, K. Mader, S. Gohla, *Eur. J. Pharm. Biopharm.,* **2000**, *50*, 161.
6. W. Methnert, K. Mader, *Adv. Drug. Del. Rev.,* **2001**, *47*, 165.
7. X. H. Wang, J. T. Liu, J. X. Li, J. F. Liu, *Inorg. Chem. Comm.,* **2001**, *4*, 372.
8. W. H. Knoth, P. J. Domaille, R. D. Farlee, *Organometallics,* **1985**, *4*, 62.

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