Hindawi Publishing Corporation
ISRN Nanotechnology
Volume 2013, Article ID 815071, 4 pages
http://dx.doi.org/10.1155/2013/815071

Research Article

Sol-Gel Synthesis and Structural Characterization of Nano-Thiamine Hydrochloride Structure

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Received 30 June 2013; Accepted 25 July 2013

Academic Editors: G. Alfieri and B. Coasne

The study presents the synthesis of nano-thiamine hydrochloride structure (NTH) using sol-gel method by hydrolysis of tetraethyl orthosilicate with ethanol and water mixture as silica source and nitric acid as catalyst support in which thiamine hydrochloride nanocrystals were dispersed in the silica glassy matrix. The synthesized nanocomposite was characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and differential thermal analysis (DTA). The morphological observation of the SEM results reveals that the nano-thiamine hydrochloride composites are in the range of 5–15 nm in size.

1. Introduction

Nanomaterials with an average grain size in the range from 1 to 20 nm have attracted research interest for more than a decade since their physical properties are greatly influenced by controlling the material at atomic scale [1]. In recent years much attention has been concentrated on metal nanocatalysts due to their novel characteristics and wide application in numerous reactions [2–4].

Many methods have been developed to control the size of nanoparticles such as Langmuir Blodgett films [5], vesicles [6], and reverse microemulsion [7]. The chemical and physical properties exhibited by these materials depend, among others, on both the composition and the degree of the homogeneity. Therefore different synthesis strategies have been developed [8, 9], such as coprecipitation [10], flame hydrolysis, microwave radiation, impregnation, and chemical vapor deposition. The sol-gel method has demonstrated the high potential to control the bulk and surface properties of the oxides [11–13].

Some of the advantages of the sol-gel method are its versatility and the possibility to obtain high purity materials, the provision of an easy way for the introduction of trace elements, allowance of the synthesis of special materials, and energy saving by using low processing temperature. Additionally, nonhydrolytic sol-gel methods have been also reported in the literature [14].

In this work, a novel sol-gel method to the synthesis of nano-thiamine hydrochloride composite (NTHC) is presented and the composite was analyzed by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and differential thermal analysis (DTA).

2. Experimental

2.1. Materials and Methods. Thiamine hydrochloride [$C_{12}H_{17}ClN_4OS{\cdot}HCl$ (VB1)] was purchased from "Novin Kavosh Mamatir Company in Arak, Iran". Tetraethyl orthosilicate (TEOS), nitric acid, and ethanol were obtained from Merck and were used as a silica source, acid catalyst, and homogenizing agent, respectively. All materials were used without further purification.

2.2. Characterization. The XRD measurements of synthesized samples were carried out using a Philips X-pert PRO powder diffractometer with Cu-Kα radiation (λ = 1.54060 Å) in the scan range 0–100°. The morphology of synthesized sample was studied using scanning electron microscopy

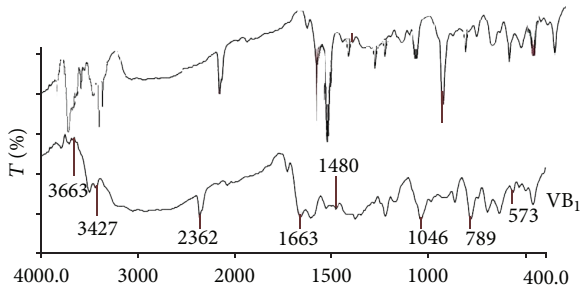

Figure 1: FTIR spectrum of the VB1 and the sol-gel.

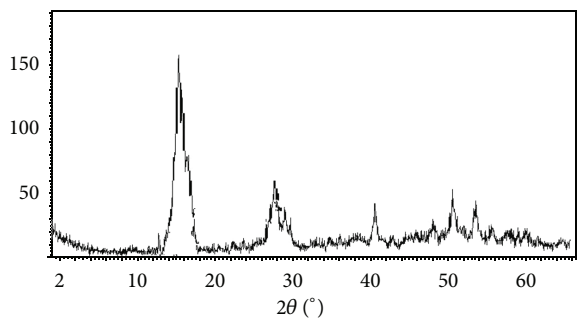

Figure 2: XRD pattern of thiamine·HCl nanostructure.

(Philips-XL30) by a sputtering technique with gold as covering contrast material. The FTIR spectra were recorded using Bruker spectrometer with KBr pellets in the range from 400 to 4000 cm^{-1}. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) profiles were performed with a Shimadzu-50 thermoanalyzer apparatus under air flow with a heating rate of 10°C/min.

2.3. Sol-Gel Process. The sol-gel composites were obtained through modification of the method reported by Oter et al. [15]. One of the advantages of the sol-gel technique is the possibility of using different precursors. In the current work, tetraethyl orthosilicate (TEOS) was used as the precursor. Two major sets of reactions take place during sol-gel processing: (i) hydrolysis of the precursor and (ii) polycondensation of the hydrolyzed products [16].

Nanostructure of thiamine hydrochloride was prepared through the sol-gel process. This sol was synthesized by hydrolyzing TEOS in a mixture of water, nitric acid, and ethanol. The molar ratio of components was 1 : 10 : 2 : 1, respectively. Briefly, under continuous stirring condition TEOS was dissolved in alcohol with later addition of a mixture of deionized water and acid drop by drop at 80°C. The ending solution was aged for 3 h under reflux at 80°C to obtain a clear silica sol. Following the formation of transparent and homogenous silica sol, the various amounts of thiamine hydrochloride were added to the sol. After 45 min, 120 μL of Triton X-114, as surfactants (the surfactant served to prevent fracturing of the gels when they were placed in solution; the amount of Triton X-114 is below its critical micelle concentration, 0.2 mM) was added into the sol, and the mixture was stirred for an additional 120 min at 100°C.

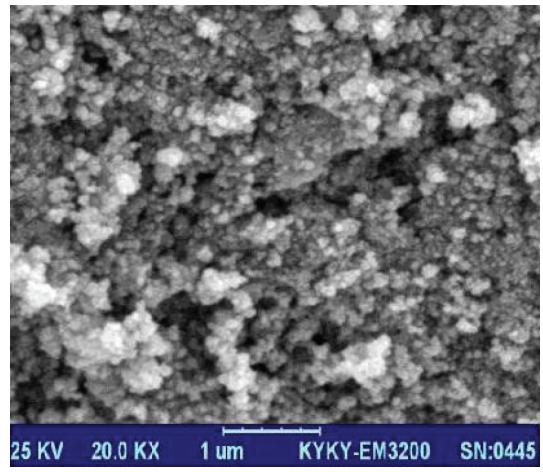

Figure 3: SEM image of thiamine·HCl nanostructure.

3. Results and Discussion

3.1. FTIR. FTIR spectra of the sol-gels are shown in Figure 1. The low frequency peak near 434 cm^{-1} is assigned to Si–O–Si out-of-plane bending. The bands at 789 and 1046 cm^{-1} are ascribed to Si–O–Si symmetric and anti-symmetric stretching vibrations, respectively. The peaks at 956 and 1035 cm^{-1} are related to Si–OH and Si–O–C, respectively. FTIR spectrum is narrower in thiamine hydrochloride nanostructure than the typical FTIR spectrum of the thiamine hydrochloride powder, and it is a reason that nano-thiamine hydrochloride was obtained.

3.2. X-Ray Diffraction. Figure 2 shows X-ray diffraction pattern of sol-gel nano-thiamine hydrochloride structure. The average crystallite size was determined by carrying slow scan of the powders in the range of 5–15 nm with the step of 0.01 o min^{-1} from the Scherer's equation. An estimate of the grain size (G) from the broadening of the main peak can be done by using the Scherer's formula bellow [10]:

$$G = \frac{0.9\lambda}{\Delta(2\theta)\cos\theta}, \quad (1)$$

where λ is the Cu-Kα radiation wavelength, $\Delta(2\theta)$ is peak width at half-height, and θ is the diffraction angle. The nanocrystallite sizes were found to be 5–15 nm.

3.3. Scanning Electron Microscopy (SEM). The particle morphologies of the prepared nano-thiamine hydrochloride structure were observed by SEM. Figure 3 shows the SEM images of the nano-thiamine hydrochloride structure at different magnifications. The SEM observation clearly illustrates that the nano-thiamine hydrochloride structure is formed by sol-gel method. Also it is to be noted that the nanostructure varied in size from 5 to 15 nm which is in good agreement with

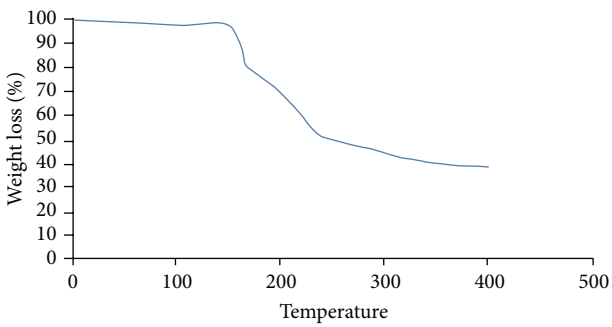

Figure 4: TG curve of thiamine hydrochloride nanostructure.

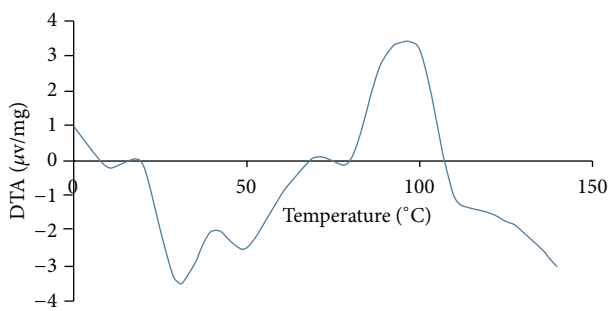

Figure 5: DTA curve of thiamine hydrochloride nanostructure.

that estimated by Debye-Scherrer formula from the XRD pattern.

3.4. Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA). Thermogravimetry is one of the most widely used techniques to monitor the composition and structural dependence on the thermal degradation of a composite. Figures 4 and 5 show the results of thermogravimetric analyses (TGA and DTA) of the thiamine hydrochloride nanostructure. The TGA curve shows an initial peak at 50°C which was related to moisture evaporation. After this peak, TGA shows major weight loss, in the range from 170 to 230°C, which shows the evaporation of some crystallized water molecules. The last sharp TG peak centered at about 210°C should arise from the oxidation decomposition of thiamine hydrochloride nanostructure in the air.

Figure 5 shows the DTA curve of nano-thiamine hydrochloride structure. Endothermic peaks at 35, 50, and 170°C may correspond to the loss of water molecules present in the dried gel capillaries; the strong exothermic peak at 100°C may be indicated by the formation of nano-thiamine hydrochloride structure through the sol-gel process.

4. Conclusion

Nano-thiamine hydrochloride structure was prepared by the sol-gel method in uniform diameters in the range of 5–15 nm at 100°C and was investigated by using powder X-ray diffraction, Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA), and differential thermal analysis (DTA). It is confirmed that nano-thiamine hydrochloride structure had high thermal stability. It is noteworthy that the sol-gel method is effective in obtaining pure phase nanomaterials with controllable size, uniform morphology and shape.

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