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Spectroscopic study of the experimental parameters controlling the structural properties of chitosan–Ag nanoparticles composite

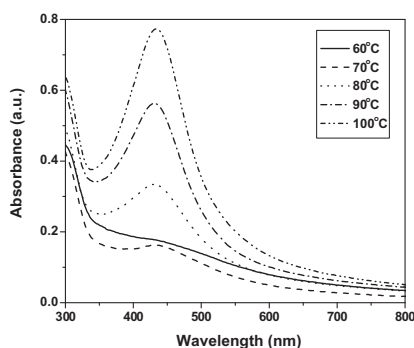
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HIGHLIGHTS

- Distinct spherical Ag nanoparticles were prepared by chemical reduction method.
- The reduction process and nucleation stage were enhanced by the parameters.
- Nanoparticles shape, size and distribution are influenced by the parameters.

GRAPHICAL ABSTRACT

UV–Vis absorption spectra of chitosan silver nanoparticles composites as function of reduction temperature for 6 h.



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ABSTRACT

Chitosan as reducing, stabilizing and capping agent was used to synthesize chitosan–silver nanoparticles composite under different experimental conditions of temperature or time. The UV–Vis spectra exhibited a single peak at 430 nm which provided strong evidence for the formation of surface plasmon resonance (SPR) band of Ag nanoparticles. The rate of the increase of this absorbance with temperature increases with increasing the time of reduction. It was found that the variation of the temperature from 60 °C to 100 °C and the time of reduction from 6 h to 16 h resulted in no significant changes in the intensities and positions of the FTIR absorption bands of the composite. The TEM micrographs showed distinct typical spherical silver nanoparticles separated from each other quite well at reduction temperature range (60–80 °C) and displayed some of accumulations at high temperature range (90–100 °C). The TEM micrographs investigation indicated various shapes with different reduction time. The SEM images of the prepared samples were discussed.

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Introduction

Chitosan is the N-deacetylated derivative of chitin. Chitosan is actually copolymers of 2-amino 2-deoxy D-glucopyranose and

2-acetamido-2-deoxy-D-glucopyranose [1]. Chitosan is primarily characterized by its molecular weight (MW) and the degree of acetylation (DA) [2]. Both chitin and chitosan are becoming increasingly important natural polymers because of their unique combination of properties like biodegradability, biocompatibility and bioactivity, in addition to attractive physical and mechanical properties [1]. For these reasons, chitosan is widely used in many

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different fields, including medicine, food and chemical engineering, pharmaceuticals, nutrition and agriculture [3].

At the present time nanochemistry becomes one of the main growing directions of nanoscience. Frequently, nanometer-size metallic particles show unique and considerable changed physical, chemical and biological properties owing to the dominance of quantum mechanics [4,5] compared to their macro scaled counterparts, due to their high surface to volume ratio. Thus, these nanoparticles have been the subject of substantial research in recent years. Metallic nanoparticles exhibit size and shape-dependent properties that are of interest for applications ranging from catalyst and sensing to optics, antibacterial activity and data storage [6]. Nanosilver, a particle of Ag element, is a new class of material with remarkably different physical, chemical and physicochemical characteristics such as increased optical, electromagnetic and catalytic properties from the bulk materials [7]. Recently different authors [8–10] used various chemical routes for production of nanostructure silver compounds as Ag_2CrO_4 , Ag_2CrO_7 and AgVO_3 which are important in many applications as visible-light sensitive and photocatalyst and electrochemical cells.

The most common method for producing silver nanoparticles is chemical reduction of a silver salt dissolved in water with a reducing compound such as NaBH_4 , citrate, glucose, hydrazine and ascorbate [11]. A reducing agent provides the free electrons need to reduce silver ions and to form silver nanoparticles [12]. Since reducing agents for silver nanoparticles synthesis are often considered toxic or hazardous, the use of green synthesis methods is becoming a priority [13,11]. A stabilizing agent (also well known as capping agent) is used in the synthesis process to prevent nanoparticles from aggregation and to control the size of final product, with agglomeration mainly caused by excess surface energy and high thermodynamic instability of the nanoparticles surface [13,11].

The present work aims to study the structural properties of chitosan–Ag nanoparticles composite prepared by using chemical reduction method under different experimental conditions. The molecular and morphological properties of the prepared samples will be investigated by using the most common spectroscopic technique.

Materials and methods

Materials

High molecular weight chitosan powder with practical grade >75% deacetylation of molecular weight range from 31,000 to 37,000 and the viscosity range from 800 to 2000 mPa s, purchased from Sigma–Aldrich Chemical company (Ireland), was used for preparation of chitosan–silver nanoparticles composite. Silver nitrate salt and acetic acid were of analytical grade and supplied by SRL Company, Mumbai, India.

Method

Chitosan–silver nanoparticles composites were prepared by using chemical reduction method according to the procedure first reported by Wei et al. [3]. Stock of chitosan solution was prepared by dissolving 6.92 g of chitosan in 1 liter of acetic acid. Stock of silver nitrate solution was prepared by dissolving 8.84 g of silver nitrate salt in 1 liter of de-ionized water. Each solution was stirred overnight at 25 °C till homogenous solution was reached. A mixture of two stocks was prepared from chitosan and AgNO_3 solutions using AgNO_3 : Chitosan ratio 1:2 (v/v) producing AgNO_3 final concentration 2.947 mg/ml. The mixture was stirred overnight at 25 °C then it was kept at 80 °C for 16 h for the reduction

process. Chitosan–Ag nanoparticles composite was reduced at different reduction temperatures namely; 60, 70, 80, 90 and 100 °C and for different times of reduction viz; 6, 8, 10, 12, 14 and 16 h at constant pH 4.5.

Characterization

UV–Vis spectra were performed with a UV–Vis spectrophotometer Model V-570 UV/VIS/NIR, operating in the absorption mode in the range of 200–1000 nm. FTIR spectrum was obtained using a Fourier-transform infrared instrument (JASCO FTIR-400 Japan) in the spectral range of 4000–400 cm^{-1} at room temperature. Transmission electron microscope (TEM) images were obtained by using JEOL JEM 2010 transmission electron microscope. The samples were prepared by drying a drop of composite on the carbon-coated copper grid. The particle sizes were measured using revolution (4pi-Analysis-v1.6.0b195) program. The surface morphology was examined by scanning electron microscope (SEM) (Philips XL 30).

Results and discussion

The effect of variation of concentration of silver nitrate in the composite relative to chitosan was previously studied by the authors [15]. The obtained results proved that the UV–Vis absorption spectra exhibited a single peak at 430 nm which provided strong evidence for the formation of surface plasmon resonance (SPR) peak of Ag nanoparticles. The FTIR spectra suggested that the shift at amides I and II bands to lower frequencies with increasing AgNO_3 : Chitosan ratios is due to the attachment of silver to nitrogen atoms. The TEM images showed that Ag nanoparticles have spherical shape and its mean particle size ranges from 4.58 to 29.01 nm depending on the AgNO_3 : Chitosan ratios.

Examples of the UV–Vis spectra of samples prepared at different temperatures for different times are shown in Figs. 1 and 2. Visual examination of the recorded spectra revealed that the absorbance of SPR peak is temperature and time dependent. In order to evaluate the changes of the absorbance with both temperature and time on quantitative basis the absorbance of the SPR peak for each sample was determined. The relationship between the determined absorbance and temperature for any given time is illustrated graphically in Fig. 3. It is clear from this figure that the change in absorbance over the range (60–80 °C) for any given time is slow which means that the rate of silver reduction under these experimental parameters is very slow. Elevating the temperature above 80 °C causes rapid increase in absorbance providing that high temperature accelerates the nucleation stage and reduction process. Both the UV–Vis spectra illustrated in Figs. 1 and 2 and the relationship shown in Fig. 3 give strong evidence that the appropriate temperature of the complete reduction is above 80 °C. It could be concluded that the reduction process and nucleation stage are enhanced by increasing both temperature and time. This result is in consistence with the data reported in literature [16,17].

FTIR spectroscopy was applied to follow up the alternation in structural properties of chitosan–Ag nanoparticles composite brought about by changing the reduction temperature and time during the reaction between chitosan and silver ions. The IR absorption spectra of all samples were recorded. Examples of IR absorption spectra of the chitosan–Ag nanoparticles composites under the effect of different reduction temperatures and time are shown in Fig. 4(a and b). The qualitative and quantitative analysis of the recorded spectra revealed that the variation of temperature from 60 °C to 100 °C and the time of reaction from 6 h to 16 h resulted in no significant changes in the intensities or positions of the absorption bands.

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