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Cellophane and filter paper as cellulosic support for silver nanoparticles and its thermal decomposition catalysis

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ABSTRACT

Silver nanoparticles (AgNPs) have attracted great attention due to its optical, electrical and thermal properties. Cellulosic supports for these nanoparticles are of particular interest because of its availability, flexibility and biocompatibility. In this work, AgNPs were synthesized using two cellulosic materials, cellophane (CP) and filter paper (FP), as matrix support. Cellulosic materials were immersed in an aqueous solution of silver nitrate containing polyvinylpyrrolidone (PVP) and then reduced with hydroxylamine. The obtained nanocomposites (CP-AgNPs and FP-AgNPs) were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (DRX) and scanning electron microscopy (SEM). AgNPs of near 15 nm anchored onto cellulosic surfaces were detected. The thermal properties of these materials were investigated through thermogravimetry (TG). Their kinetic of thermal decomposition was studied by the Vyasovkin method of dynamic isoconvertion, which indicated a catalytic effect of AgNPs in the cellulose thermal decomposition reaction.

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1. Introduction

Metal nanoparticles have different properties from those of bulk metal because of their small sizes and very high surface area. Nanostructured materials have enormous applications potential such as in photoeletronic, magnetic, sensor and biomedical areas (Carrión et al., 2014; Guerrero-Martínez, Barbosa, & Pastoriza-Santos, 2011; Johnston, 2012, chap. 1; Ng, Mohammad, Leo, & Hilal, 2013; Prakash, Chakrabarty, & Singh, 2013; Rao, Gruenberg, & Geckeler, 2014). In particular, silver nanoparticles (AgNPs) present unique optical, thermal and catalytic properties (Cacciotti, Fortunati, Puglia, & Kenny, 2014; Meng, Tang, & Vongehr, 2010; Ravindran, Chandran, & Khan, 2013; Tolaymat et al., 2010; Seekell, Price, & Marinakos, 2012). Silver nanoparticles can be prepared by different methods, including formation of Ag nanoparticles attached to polymeric supports (Barud et al., 2008; El-Nour, Eftaiha, AL-Warthan, & Ammar, 2010; El-Rafie, El-Rafie, & Zahran, 2013; Qin et al., 2010; Roy, Gaur, Jain, Bhattacharya, & Rani, 2013). Cellulose is a polymer used in preparations of these nanoparticles due to their abundance in nature, and being an, biodegradable and biocompatible polymer. It is a linear homopolymer of D-glucopyranose units which are linked by β-1, 4- glycosidic bonds (Fengel & Wegener, 1989, chap. 4; Lavoine, Desloges, Dufresne, & Bras, 2012). The hydroxyl and ether groups available at the surface of cellulosic materials act as active sites for metal ions adsorption via ion-dipole interactions. This availability depends on the source and crystallinity of the cellulosic sample. The Ag^+ ions can then react forming supported silver nanoparticles when proper reagent and conditions are provided (Li, He, & Zhang, 2015).

Several sources of cellulose have been used as support in the preparation of AgNPs. Thang et al., 2015 obtained silver nanoparticles supported on bamboo pulp with excellent UV protection and

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antimicrobial activity. Bacterial cellulose has also been used to synthesize nanoparticles, and Wu et al. (2014) synthesized AgNPs with average size of 10–30 nm using this cellulosic material. There are studies that show AgNPs synthesis using cellulose nanofibrils and membranes (Barud et al., 2008; Dong, Snyder, Tran, & Leadore, 2013). In addition, these nanoparticles can be synthesized on the filter paper surface, where Sallum, Soares, Ardila obtained AgNPs with an average size of 180 nm. Khalilabad, Yazdanshenas, and Etemadifar (2013) synthesized AgNPs with an average size of 53 nm using cotton fabric as support matrix.

To characterize cellulose-AgNPs materials, important determinations are particle size/size distribution together with particle morphology, crystalline phases identification, thermal and optical properties evaluation. The thermal decomposition of cellulosic materials has been studied using TG techniques (Adel, El–Wahab, Ibrahim, & Al–Shemy, 2010). There are some studies on the thermal degradation of cellulosic materials treated with inorganic salts (Wu & Zavarin, 1986; Kahur, Jain, Gur, Bhatnagar, & Schulten, 1986), although we did not find studies for such materials containing adsorbed metal nanoparticles.

The investigation of the kinetic of the thermal decomposition of a material is a study of great interest in thermal analysis, because the reaction parameters such as rate and activation energy are very important in determining the mechanisms of the reactions of solid phase, especially in decomposition of composites. In this way, an appropriate method is the dynamic isoconversional method (Vyazovkin & Wight, 1999), to evaluate the apparent activation energy in any decomposition temperature. This method was developed because the apparent activation energy of decomposition can be evaluated both in simple reactions and also in complex reactions. It is considered that methods based in the use of three different heating rates give more reliable results than those based on data from a single heating rate, and the integral methods present fewer experimental errors (Fan-fei, Ming-xu, & Qing-ru, 2007).

In the present work, CP and FP were chosen because of their different crystallinity and cellulose type. Silver nanoparticles were synthesized on these cellulosic samples using a chemical reduction method. FTIR was used to evaluate the cellulose crystallinity of the samples before and after the formation of the adsorbed AgNPs. X-ray diffraction was used to investigate the AgNPs formation as well as to determine their average crystallite size and cellulose crystallinity. The thermal behavior of CP, CP-AgNPs, FP and FP-AgNPs was studied using the TG technique. The thermal decomposition was carried out at four different heating rates and the Vyazovkinís method of dynamic isoconvertion (Vyazovkin & Wight, 1999) was used to evaluate apparent activation energies (Eα) of the cellulose pyrolysis. Previously published reports do not present kinetic data of the pyrolysis of cellulose-AgNPs composites.

2. Experimental

2.1. Materials

Cellophane (CP), filter paper (FP), silver nitrate, polyvinylpyrrolidone (PVP) molecular weight of 1.300.000, sodium hydroxide and hydroxylamine, all of P.A. degree, were commercially purchased and used as received.

2.2. Cellulose-AgNPs composites preparation

AgNPs adsorbed onto cellulosic materials were prepared by a methodology proposed in the literature (Santa Maria et al., 2009) with modifications. Three aqueous solutions were prepared: 0.06 mol/L $AgNO_3$ and 0.2% wt/v PVP (solution I), 0.2% wt/v PVP with sufficient NaOH to attain pH in the 13–14 range (solution II), and 0.2% wt/v PVP with 0.002 mol/L hydroxylamine (solution III). The CP or FP (0.8 g) was left in contact with 100 mL of solution I for 24 h. Thereafter, the material was immersed in 50 mL of solution II and solution III was then added dropwise. The materials were dried at 40 °C.

2.3. Characterization

The samples were analyzed by Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR), using a Bomem spectrometer, model MB-100, with zinc selenide ATR attachment, in the range 4000–600 cm^{-1}. Lateral order index (LOI) of cellulose was estimated with the aid of the FTIR graphs using the ratio between the areas of the absorption bands at 1418 cm^{-1} and 894 cm^{-1} (OíConnor, Dupré, & Mitcham, 1958; Siroký, Blackburn, Bechtold, Taylor, & White, 2010). X-ray diffraction patterns (XRD) were obtained in a Shimadzu D6000 X-ray diffractometer, using a Co Kα 1.74 Å irradiation. The experimental conditions used were: $2\theta = 10–80°$, voltage 40 kV and 30 mA. The average crystallite sizes of AgNPs were estimated by the Scherrer's equation in the characteristic peak at $2\theta = 38.1°$ as shown in equation 1 (Langford & Wilson, 1978; Aldersley, Joshi, Price, & Ferris, 2011):

$$d = \frac{0.9\lambda}{C\cos\theta_B} \quad (1)$$

In which d is the average size of the crystallites, 0.9 is a constant which is related to the spherical shape of the nanoparticle, λ is the wavelength of the cobalt irradiation, B is the width at half peak height and θ_B is the Bragg angle accordingly.

The percentage cellulose crystallinity of the samples was calculated using the following equation (Segal, Creely, Martin, & Conrad, 1959; Besbes, Alila, & Boufi, 2010):

$$Cr(\%) = \left(\frac{I_{[002]} - I_{amorphous}}{I_{[002]}}\right) \quad (2)$$

In which Cr represents the percent of cellulose crystallinity. For the type I cellulose, the minimum intensity (relative to the amorphous halo, $I_{amorphous}$) lies between the peaks in the region of $2\theta = 18–19°$, and the maximum intensity (due to the crystalline region, $I_{[002]}$) corresponds to the diffraction peaks in the region of $2\theta = 22–23°$. For the cellulose II, the minimum intensity lies at $2\theta = 13–15°$, and the maximum intensity at $2\theta = 18–22°$ (Roncero, Colom, & Vidal, 2003). Scanning electron microscopy micrographs (SEM) were conducted using a Shimadzu SSX-550 microscope. The samples were prepared according to a standard procedure, fixed with conductive glue and coated with a thin layer of gold. Examination was performed at a magnification of 3000 and 4000 times. Thermogravimetric analyses (TG) of the samples were carried out using a Shimadzu TGA 50 thermobalance and analysis for activation energy computation were repeated in a PerkinElmer STA6000 instrument. Approximately 6 mg of the cellulosic material were placed in a platinum sample pan, subjected to heating from room temperature to 800 °C with heating rate of 2.5, 5.0, 10.0 and 20.0 °C/min. TG analyses were carried out in inert atmosphere (N_2) flow of 10 mL/min. Decomposition onset temperature (T_{onset}), maximum rate of decomposition temperature (T_{max}), and decomposition end temperature (T_{end}) were obtained from the first derivative of the thermogravimetric curve.

2.4. Kinetic procedure

Apparent activation energy of the thermal decompositionreaction of the materials was computed by the Vyazovkin method of dynamic isoconversion (Vyazovkin & Wight, 1999). In this method, Vyazovkin propose that TG data ofthe solid sample must be available for at least three different heating rates. It considers that

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