

Research Article

Electrochemical Method for Ag-PEG Nanoparticles Synthesis

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In this work we present an electrochemical method to successfully prepare silver nanoparticles using only polyethylene glycol as stabilizer and without any other reactive. Here we study the use of the polymeric stabilizer to allow the introduction of a potential tool to reinforce the control of the size and shape of the nanoparticles throughout the synthesis process. The evolution of the reactions was followed by UV-Vis spectroscopy. The electrode processes were characterized by cyclic voltammetric measurements and the final product was studied by Atomic Force Microscopy, Transmission Electron Microscopy, and X-Ray Diffraction. The influences of the current density, polymer length, and concentration media were analyzed.

1. Introduction

Noble metal nanoparticles have been intensely investigated due to their amazing properties such as optical, catalytic, and electric ones that can be controlled depending on the particles size, the size distribution, and shape [1]. Metallic silver particles in particular are technologically important because they show unique properties normally related to noble metals (excellent conductivity [2], chemical stability, nonlinear optical behavior [3, 4], etc.) besides other specific ones (catalytic activity [5], antibacterial action [6]). These characteristics make them suitable for a variety of potential applications on several devices [7–9].

Several methods have been reported for Ag nanoparticles synthesis, including Ag ions chemical reduction in aqueous solutions with or without stabilizing agents [10–13], thermal decomposition in organic solvents [14, 15], biochemical reduction [16, 17], chemical and photo reduction in reverse micelles [18–20], “nanosphere lithography” (NSL) [21], electrochemical reduction [22–27], irradiation reduction [28], microwave assisted [29–31] and chemical reduction in nonaqueous solvents with surface modifiers [32, 33]. Each method has typical advantages and disadvantages.

Particularly, the electrochemical techniques are quite interesting because they allow obtaining particles with a high purity using fast and simple procedures and controlling the

particle size easily by adjusting the current density [22, 23]. Through these techniques particles have been obtained with determined size and shape [24, 25] of several compositions [26, 27, 34–36]. Also, this method is eco-friendly because it avoids the use of reducer agents that usually are toxic.

Different stabilizers have been used in electrochemical techniques, which include organic monomers as electrostatic stabilizers [26] and polymeric compounds as steric stabilizers [37]. Ionic organic compounds can act as stabilizers and support electrolytes simultaneously avoiding the use of additional chemicals [26]. However, some authors showed that steric stabilizers allow obtaining more stable nanoparticles than electrostatic stabilizers [38]. Also, chemical reduction techniques assisted by polymers allow controlling the shape and size of nanoparticles by changing the polymer chain length [39, 40]. On the other hand, some authors showed that polyethylene glycol-stabilized silver nanoparticles are highly stable and sufficiently permeable to allow the diffusion of small molecules [41]. Thus, they are useful to several applications as Surface-enhanced Raman Scattering techniques and others. Then, we propose the use of the polymeric stabilizer polyethylene glycol to obtain stable nanoparticles and we study their ability to control the nanoparticles morphology.

In this work we present an electrochemical method to prepare silver nanoparticles using polyethylene glycol as stabilizer. We study the effect of the stabilizer concentration,

polymer chain length, and current density over the particle size and shape. These are common strategies for the control of the particle morphology, but they have dissimilar behavior at different systems, mainly the stabilizer concentration. Also, the use of a polymeric stabilizer allows the introduction of a potential tool to reinforce the control of the size and shape of the nanoparticles by using different polymer chains lengths and so we study this strategy too.

2. Experimental Section

2.1. Electrochemical Synthesis of Silver Nanoparticles. Silver nanoparticles were prepared by electrochemical reduction within a simple two-electrode type cell. The volume of the electrolysis cell was 50 ml. A platinum sheet (0.5×7 cm) was employed as cathode and a platinum rod (0.1×7 cm) as anode, the two being 3 cm apart. The cell was maintained into an ultrasonic bath. Silver nitrate (AgNO_3 , Merck) was used as silver ion precursor and polyethylene glycol (PEG, Aldrich) with different chains lengths $M_w = 400$ (PEG-400), 600 (PEG-600), 1450 (PEG-1450), 2000 (PEG-2000), and 6000 (PEG-6000) which was employed as stabilizing agent.

In a typical synthesis, the reaction medium was prepared to obtain an aqueous solution of silver nitrate 2.5 mM with 0.5 to 2% w/v of PEG. The solution was mixed and purged with N_2 during 20 minutes. Then, the electrolysis was carried out under ultrasonication at constant current and N_2 atmosphere during 30 minutes. The current chosen (7 mA, 10 mA, or 13 mA) was given by adjusting the applied potential.

2.2. Characterization of Silver Nanoparticles. In order to study the optical behavior of silver colloid, ultraviolet-visible (UV-Vis) absorption spectra of different samples were recorded by a Jasco V-530 UV-Vis spectrophotometer using as reference a corresponding blank sample.

The shapes and sizes of the nanoparticles were determined by Atomic Force Microscopy (AFM) and Transmission Electron Microscopy (TEM). AFM images were taken using NanoTec ELECTRONICA equipment in tapping mode configuration with a Si_3N_4 tip in air at room temperature. In order to prepare the sample for the AFM study, the particles were redissolved in ethanol and drop-cast onto a freshly peeled HOPG substrate. TEM images were taken with Philips EM 301 equipment. Samples were prepared by placing a drop over a TEM grid and leaving it to evaporate the solvent. To study the crystalline structure of the nanoparticles, X-ray Diffraction patterns were recorded by grazing incidence with a 2° incidence angle with an X'Pert Phillips PW 1700 diffractometer using CuK radiation (1.5405 \AA) and a graphite monochromator (the step size was of $2\theta = 0.2^\circ$; 10 s/step) for samples prepared over SiO_2 by drop-cast.

The cyclic voltammetric measurements (CV) were performed with an EG&G potentiostat/galvanostat in a conventional three-electrode cell at room temperature. A platinum rod embedded in epoxy resin was used as a working electrode and only its cross section was allowed to contact the electrolytes; a platinum microelectrode and a saturated

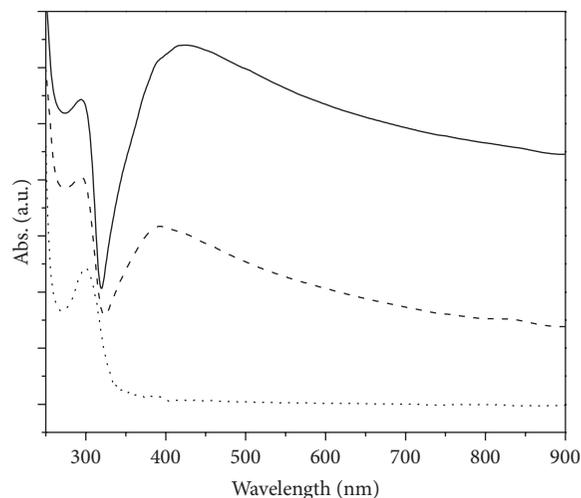


FIGURE 1: Absorption spectra of silver nanoparticles prepared using PEG-2000 at a concentration of 1% w/v with a current equal to 10 mA. Initial spectrum (dotted line) and spectra after 15 (dashed line) and 30 minutes of reaction (solid line).

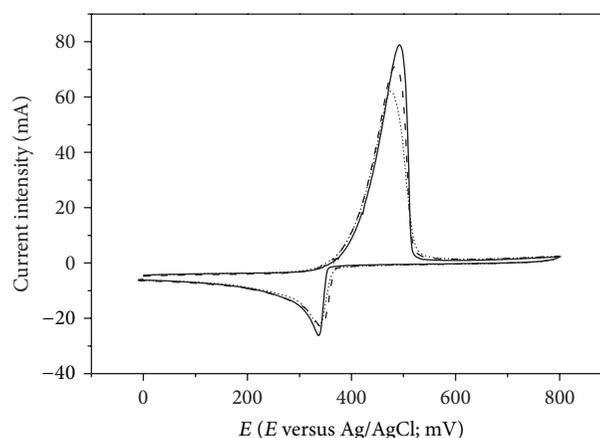


FIGURE 2: Cyclic voltammograms of 2.5 mM AgNO_3 aqueous solutions with 1% w/v (dashed line), 2% w/v (dotted line), and without PEG-2000 (solid line).

Ag/AgCl electrode were used as the auxiliary electrode and the reference electrode, respectively.

3. Results and Discussion

3.1. Characterization of the Reaction Progress. The reaction mixture was characterized in different stages of the reaction progress by UV-Vis spectroscopy. Figure 1 shows the evolution of the optical properties for silver nanoparticles prepared by electrochemical reduction using PEG-2000 as stabilizer at a concentration of 1% w/v and applying a current equal to 10 mA. At the initial time, the absorption spectrum exhibits a single shoulder at a wavelength near to 300 nm, which was attributed to ions NO_3^- in aqueous solution [42] with a lower contribution of Ag^+ [43]. After 15 minutes of reaction, a broad band appeared at 392 nm and its intensity increased until an

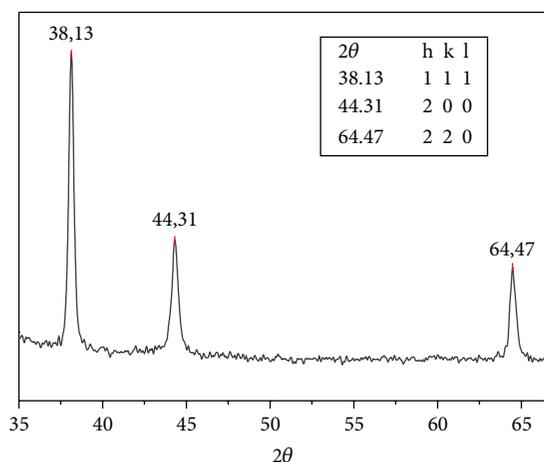


FIGURE 3: X-Ray Diffraction pattern of silver nanoparticles. The principal position peaks are shown in the figure.

absorption maximum centered at 425 nm which was obtained after 30 minutes of reaction. This latter absorption band corresponds to the surface plasmon resonance of spherical silver nanoparticles [44]. An absorption minimum appears at 320 nm, which corresponds to a minimum in the imaginary part of the refractive index for bulk silver [45]. In a control experiment a similar solution was placed in the ultrasonic bath with nitrogen atmosphere, but no one current was applied. After 50 minutes no spectral change was observed (not shown here); then reducing action by PEG-2000 was ruled out in these conditions.

In the electrochemical synthesis of silver nanoparticles, there is a competition between two different cathode surface processes: the silver particle formation and the silver deposition on cathode [46]. The key to electrochemical synthesis of silver nanoparticles in aqueous medium is to avoid formation of silver plating and to force reduced particles to leave the cathode surface. In order to test the PEG influence over these processes, CV experiments were carried out [37]. Figure 2 shows the CV in silver nitrate solutions with different amounts of PEG. All CV experiments present one reduction peak near 337.4 mV and one oxidation peak near 492.0 mV approximately. The reduction peak is attributed to silver ions reduction and their electrodeposition on the platinum surface; the oxidation peak is related to oxidation of the silver electrodeposition on the electrode [42]. CV show that the reduction peak intensity is the same for all the cases. The oxidation peak intensity decreases when the PEG amount increases and this is attributed to the PEG influence over the electrode processes. In the PEG-free solution the reduced silver was almost completely deposited on the surface of working electrode and then, when the scan was swept in the oxidation sense, almost all the reduced silver was oxidized. On the other hand, in the PEG containing solution, the silver ions reduced were divided: a part was deposited on the electrode surface whereas another part was reduced to nanoparticles and stabilized by PEG. Thus, when the scan was swept in the oxidation sense, only the deposited silver was oxidized again. So, CV results show that PEG

effectively enhances the nanoparticles formation and reduces the electrodeposition on the electrode surface.

3.2. Characterization of Silver Nanoparticles. The nanoparticles were characterized by XRD, AFM microscopy, and UV-Vis spectroscopy. In Figure 3 the XRD pattern of nanoparticles, is shown where three distinct diffraction peaks were observed at 2θ values of 38.13° , 44.31° , and 64.47° , corresponding to the (111), (200), and (220) crystalline planes of cubic Ag, respectively. The broad nature of the XRD peaks is attributed to the nanosize of the particles [47].

In order to determine the size and shape of the particles, AFM images were registered. Figure 4 shows a typical AFM image. The particles are uniformly spherical in shape with an average diameter of 30.36 nm. The average diameter was determined by statistical analysis from histograms of several AFM images of the nanoparticles. This average diameter is near to the value obtained from the XRD spectrum. An average nanoparticle size of 45.85 nm was calculated from the breadth of (111) diffraction line at its half-intensity (Figure 3), with an instrumental broadening of 0.25° , using the Scherrer equation [48]. If we compare the mean sizes of the Ag nanoparticles estimated by microscopic technique and XRD, it seems that the Scherrer calculus overestimated the radius. This fact is due to the fact that the sample is polydisperse and the XRD is particularly sensitive to the largest particles or crystallites [49]; then, this result seems to be reasonable.

TEM images (Figure 5) show mainly spherical particles between 10 and 30 nm of size and a few nanospheres bigger, with diameter of 200 nm approximately. These last ones would be responsible for the dispersion effect that is observed as a mild decline of the signal towards longer wavelengths in the UV-Vis extinction spectra.

Furthermore, the morphological characteristics of the particles during different experiments stages were followed by the study of the UV-Vis spectra taking into account the knowing relations between the number and position of the absorption bands and the size and shape of the particles [50]. In silver nanospheres with diameter shorter than 100 nm only one peak in the UV-Vis spectra is observed between 400 and 500 nm [51].

In metallic nanoparticles, the size dependence of the surface plasmon absorption is not easily explained as in the case of semiconductor nanoparticles, where a blue shift or a red shift of the absorption onset undoubtedly results from decreasing size or increasing size, respectively.

The peak position of surface resonance is not well suited for discussion of size effect within the intrinsic size region in metallic nanoparticles [52]. However, the size dependence of the optical spectra of large nanoparticles ($2R > 25$ nm) is an extrinsic particle-size effect governed only by dimension of the particle with respect to the wavelength of the light, and therefore a red shift of the peak position of surface resonance undoubtedly results from increasing size [43]. This relationship is used to obtain qualitative information about relative media size between different samples.

3.3. Influence of Synthesis Conditions. In order to determine the influence of the current density, PEG concentration and

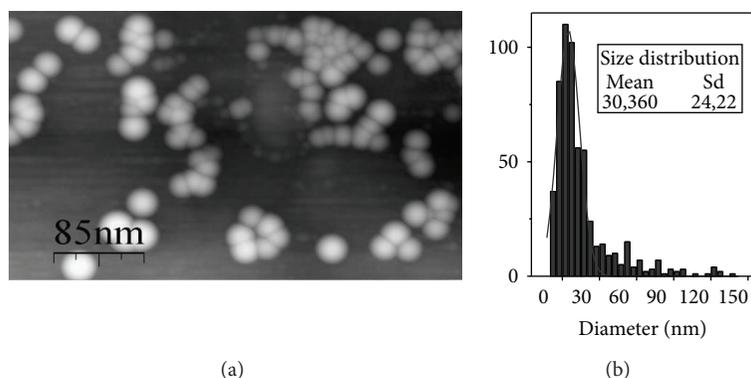


FIGURE 4: AFM image of PEG protected silver nanoparticles (a) and the corresponding size distribution analysis (b).

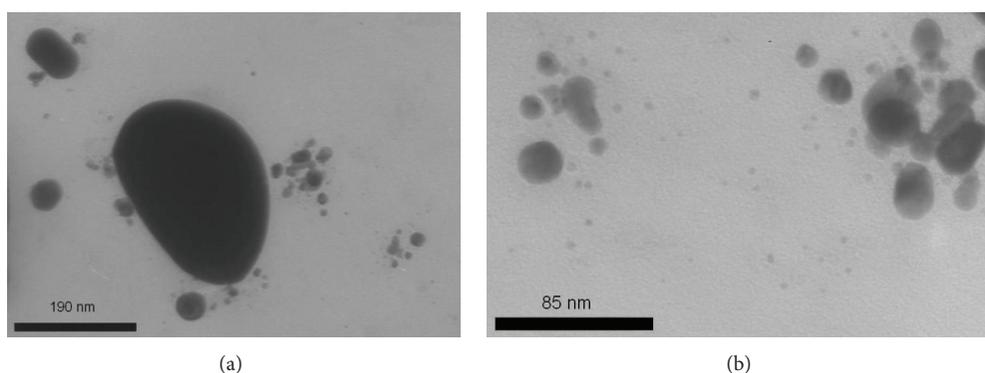


FIGURE 5: TEM images of PEG protected silver nanoparticles.

polymer chain length on the size and shape of the Ag nanoparticles several experiments were performed to study one by one each parameter of the experimental conditions. After each experiment, a UV-Vis spectrum was taken to analyze the number and position of the absorption bands and then to infer the nanoparticles shape and relative size.

3.3.1. Current Density. Reetz and Helbig determined that the particle size of Pd clusters obtained by electrochemical reduction can be controlled by variation of the current density [53]. In order to determine if the current density has some effect on silver particle size, the synthesis was repeated using currents of 7, 10, and 13 mA, respectively, whereas the other conditions were the same for these samples: 2.5 mM AgNO₃ and 1% w/v PEG-2000. Figure 6 shows the UV-Vis spectra of the obtained colloids. A blue shift in the plasmon peak can be observed as the current is increased, from 454 nm for the sample prepared at 7 mA to 405 nm for the sample prepared at 13 mA. This infers that for our samples the use of higher current density during the synthesis produces small particles. This behavior is similar to other reports by several authors for different systems with other metallic ions or stabilizers [45, 53].

3.3.2. PEG Concentration. The influence of PEG-2000 concentration on the silver particle size was studied through UV-Vis spectroscopy. The synthesis was repeated using PEG-2000 concentration of 0.5, 1, 1.5, and 2% w/v. Figure 7 shows the

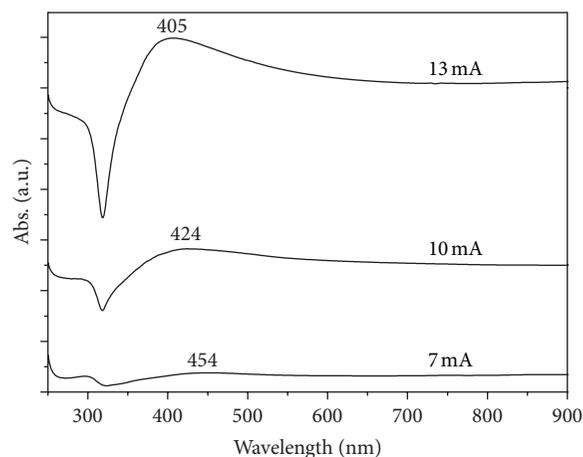


FIGURE 6: UV-Vis spectra of Ag nanoparticles obtained by using different current density during the synthesis process. From the bottom to the top: 7 mA, 10 mA and 13 mA. In the figure, the plasmon position is indicated for each spectrum.

UV-Vis spectra for the colloids obtained with different PEG-2000 amounts.

A displacement of the maximum of absorption was observed towards the blue with the increase of the concentration of PEG-2000 in the range between 0.5 and 2% w/v. Then, increasing the PEG concentration in the reaction medium,

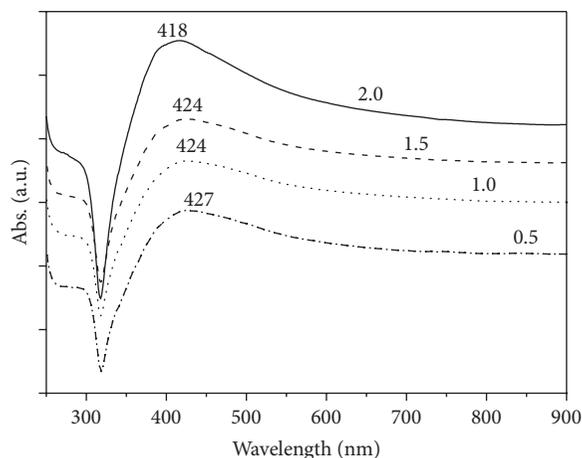


FIGURE 7: UV-Vis spectra of Ag nanoparticles obtained by using different polymer concentrations of PEG-2000 during the synthesis process. From the bottom to the top: 0.5, 1.0, 1.5, and 2.0% w/v. In the figure, the plasmon position is indicated for each spectrum.

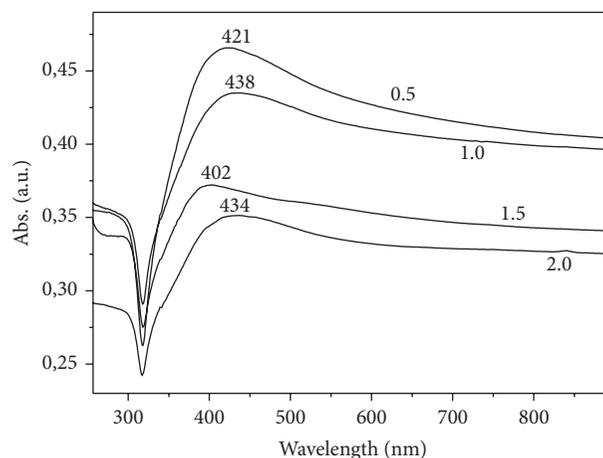


FIGURE 9: UV-Vis spectra of Ag nanoparticles obtained by using different polymer concentration of PEG-600 during the synthesis. From the bottom to the top: 0.5, 1.0, 1.5, and 2.0% w/v. In the figure, the plasmon position is indicated for each spectrum.

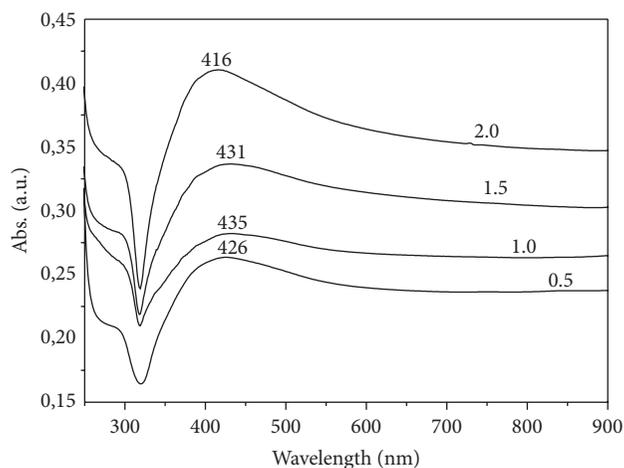


FIGURE 8: UV-Vis spectra of Ag nanoparticles obtained by using different polymer concentration of PEG-6000 during the synthesis process. From the bottom to the top: 0.5, 1.0, 1.5, and 2.0% w/v. In the figure, the plasmon position is indicated for each spectrum.

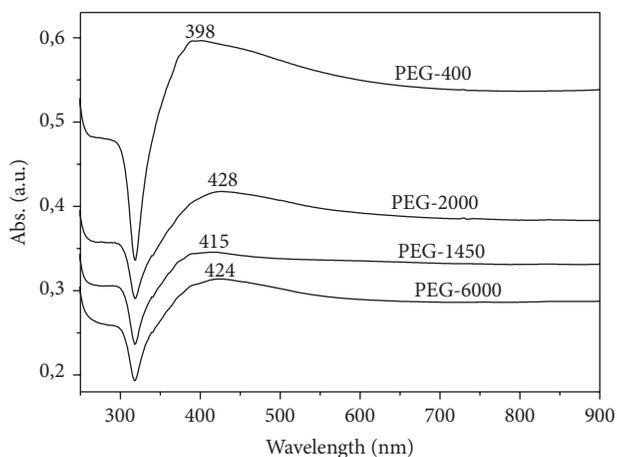


FIGURE 10: UV-Vis spectra of Ag nanoparticles obtained by using different polymer length during the synthesis process. From the bottom to the top: PEG-6000, PEG-1450, PEG-2000, and PEG-400. In the figure, the plasmon position is indicated for each spectrum.

the particle size obtained decreases. Then, it is concluded that it is possible to reinforce the control over the particle size by adjusting the PEG-2000 concentration. This behavior was similar when the same experiment was repeated using PEG-6000 in the range between 1 and 2% w/v (Figure 8). Meanwhile with PEG-600 an unsystematic behavior of the position of the maximum of absorption in function of the wavelength was found (Figure 9), and the same to PEG-400 (not shown here). Then, it is concluded that it is possible to control the particle size by adjusting the PEG-2000 and PEG-6000 concentration between 1 and 2% w/v.

3.3.3. PEG Chain Length. In some cases, when polymeric stabilizers are used, just by changing the chain length of the stabilizer, variations in the form of particles can be produced

[39]. The effect of PEG chain length on the particle size was also investigated by UV-Vis spectroscopy. Polymers with several molecular weights were used: $M_w = 400, 1450, 2000,$ and 6000 . Nevertheless, in all our experiments only one maximum of absorption between 400 and 430 nm appears to be assigned to the spherical appearance of nanoparticles (Figure 10). Then, it is concluded that the M_w did not influence the form of particles and nanospheres are obtained for all the cases.

With all the studies shown during these investigations we obtained stable nanoparticles using PEG as stabilizer and we found the correct experimental parameters that control the morphology. These nanoparticles have the possibility to be used for several purposes, such as bactericide, because they could be manipulated as a powder.

4. Conclusions

Spherical silver nanoparticles were synthesized by an electrochemical method with PEG as stabilizer. This method is a rapid and eco-friendly technique to obtain silver nanoparticles. PEG is an efficient stabilizer that favours the particle formation over silver deposition on the cathode. The influence of synthesis conditions was studied. The current density is an adequate parameter to control particle size. The PEG chain length does not show effects on the shape of the nanoparticles obtained, which are spherical for all the cases. Nevertheless, with the longer polymer chain length it is possible to control the particle size through the PEG concentration. Thus, the use of polymeric stabilizer of long chain adds an additional tool to control the particle size with respect to other electrochemistry techniques.

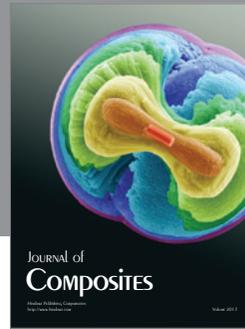
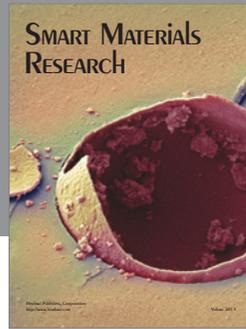
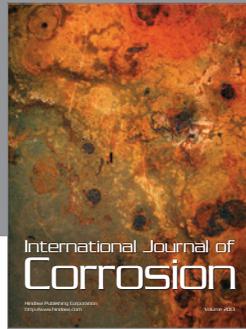
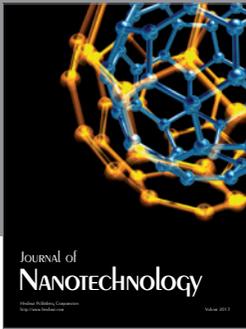
Acknowledgments

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