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Synthesis of gold nanoparticles: a new approach in using a nanoporous membrane in conjunction with ultrasonication

ABSTRACT

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Gold nanoparticles were synthesized by chemical reduction of HAuCl₄ inside the pores of a polycarbonate-based membrane followed by dissolving the membrane in dichloromethane and further sonication. Sonication time as the main affecting factor on the nanoparticle size was investigated. The characterization by transmission electron microscopy showed the formation of gold nanoparticles with diameters less than 20 nm and a uniform distribution of the nanoparticle size. This template method is particularly interesting, because it allows including metallic constituents inside the voids of nanoporous host materials resulting in monodisperse diameter nanomaterials.

Keywords: *Gold nanoparticles, Template synthesis, Polycarbonate membrane, TEM, Size distribution*

INTRODUCTION

The size and shape of materials govern many of the physical and chemical properties of nanoscale materials [1-3]. Nanomaterials show various physical properties such as magnetism, specific heat, conductivity, band gap energy, as well as luminescent and optical properties. Until now, some control over size and shape for several metal and semiconductor materials has been achieved and this has led to their size/shape-selective applications in various fields, e.g., SERS [4-6], catalysis [7-9], fluorescence/optical probes, biosensors, sensors and actuators [10-13]. It is necessary to have a homogenous distribution of gold nanoparticles with diameters between 5 nm and 20 nm for excellent catalytic activity. Several methods have been developed to achieve the controlled synthesis of dispersed gold nanoparticles with a narrow size range. Among those different strategies to synthesize nanoscopic materials reported in the literature, the template method is particularly the interesting one [14].

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This synthesis method consists of including metallic or organic constituents inside the voids of nanoporous host materials. In this context, polycarbonate particle track-etched membranes (PC-PTMs) show a significant advantage because they lead to the production of nanostructured materials with monodisperse diameters and lengths [15]. In fact, lots of different preparation routes have been reported for the synthesis of spherical gold colloidal particles. However, most of them involve the chemical reduction of Au ions, for example, reducing agents such as NaBH_4 , or white phosphorus produce small gold particles (diameter: <20 nm), while reductants such as ascorbic acid yield nanoparticles with diameters larger than 20 nm [16-19].

In this study, the possibility of reaching a method for the synthesis and separation of gold nanoparticles by using polycarbonate track-etched membranes was studied and its possibility was confirmed. Gold nanorods were synthesized within the pores of the membrane. Gold nanoparticles were obtained after dissolving membrane and nanorods sonication.

MATERIALS AND METHODS

Materials

Microporous and nanoporous PTM (Whatman) were used as template membranes for the chemical synthesis of gold nanorods. These membranes were available in a variety of pore diameters and densities. The typical template membrane used in this study had the following characteristics: pore diameter: 200 nm, pore density: 6.0×10^5 (pores/cm²), thickness: around 6 μm . Sodium boron hydride (NaBH_4) and dichloromethane (DC Chemical Co.) were used without any purification. Throughout the study, deionized water (Puris MR-RU 890) was used.

Methods

As shown in Figure 1, the polycarbonate membrane was used as a dividing wall in a two-compartment cell. In the first compartment, an aqueous solution of HAuCl_4 (0.5 M) was added and allowed to diffuse through the membrane during 5 minutes prior to the introduction of the sodium boron hydride (0.5M) solution in the second compartment. The gold ions and the reducing

reagent diffused toward each other through the pores of the membrane and reacted to yield gold nanorods. Ultrasonic bath (ultrasonic processor from Sonics and Materials, Inc., Model VC 750) was used for the sonication of solution of nanorods. For the study of the morphology of gold nanoparticles, the host membrane was dissolved in dichloromethane and the solution was sonicated in ultrasonic bath for different times. The formation of the gold nanorods and nanoparticles were investigated by utilizing TEM (JEOL, JEM-2100).

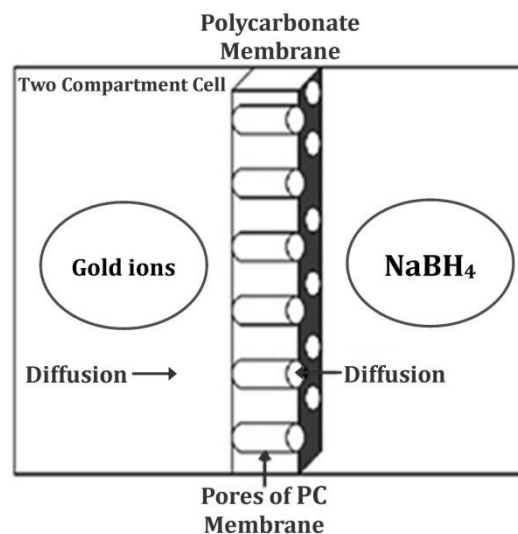


Fig.1. Schematic of the template synthesis of gold nanorods.

RESULTS AND DISCUSSION

Gold nanorods were chemically synthesized within the pores of commercial nanoporous polycarbonate membranes as a template. TEM micrographs clearly confirmed the formation of gold nanorods with a diameter size of about 150 nm (Figure 2). After the preparation of gold nanorods, the PTM membrane was dissolved in dichloromethane as a suitable solvent followed by the sonication of this solution for 20 and 30 minutes. Figure 3a shows the gold nanoparticles with diameters less than 20 nm. As it can be seen from Figure 3b, by increasing the time of sonication to about 30 min, the diameter of the prepared gold nanoparticles was increased to some extent. The gold ions were successfully loaded inside of the pores of the polycarbonate membrane because of the presence of anionic sites at the walls of the pores.

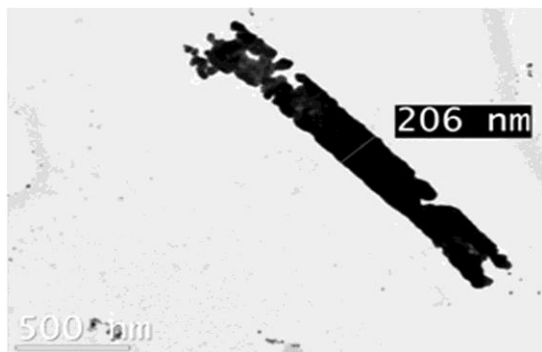


Fig.2. TEM image of a gold nanorod in the pore of the PC membrane

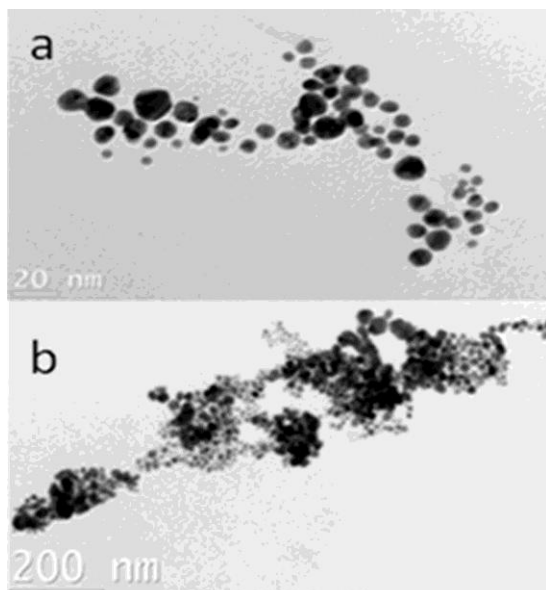


Fig.3. TEM images of gold nanoparticles after dissolving the membrane and sonication for (a) 20 and (b) 30 min.

Figure 4 shows the size distribution of gold nanoparticles after dissolving the membrane and sonication. As it is evident from Figure 4, sonication for 30 minutes leads to the formation of more uniform size distribution with a little more averaged particle size. As mentioned earlier, from a practical point of view, the size distribution has a more profound effect on the nanoparticles physico-chemical properties. Thus, we could conclude that, in general, by increasing the sonication time one can achieve a more uniform particle distribution. However, a further study reveals another view: a sonication time of more than 60 minutes, no gold nanoparticles could be found in the TEM images, but rather a dispersed gold phase was identified (Figure5). Thus, sonication time is also an important parameter to be considered and

optimized in order to obtain the well suited characteristics.

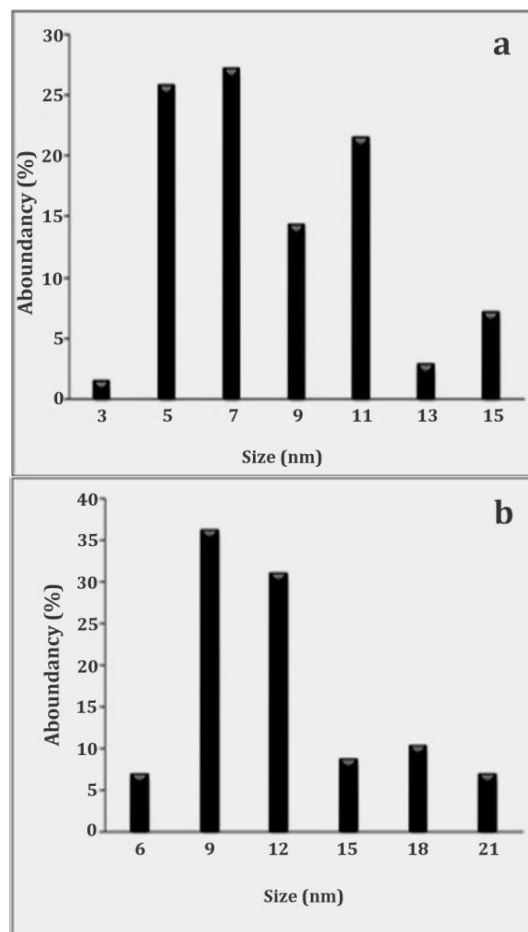


Fig.4. Size distribution of gold nanoparticles after dissolving of PC membrane and sonication for (a) 20 and (b) 30 min.

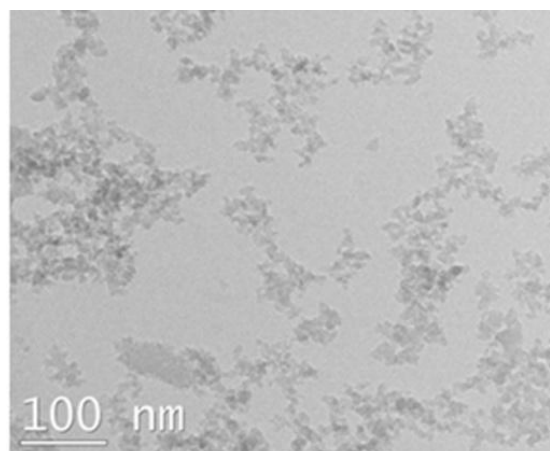


Fig.5. TEM image of dispersed gold phase after sonication for 60 min.

CONCLUSION

This work focused on the synthesis of gold nanoparticles by using a polycarbonate membrane as the template. The morphology of the gold nanoparticles has been analyzed using the transmission electron microscopy and the formation of gold nanoparticles was clearly confirmed. The results show the formation of gold nanoparticles with diameters of less than 20 nm. Also, the sonication time was found to be an effective factor on the average size and size distribution of the resulting nanoparticles. This method may offer reliable opportunities for the practical preparation of gold nanoparticles.

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