Structural Characterization of Monodisperse SiO₂ Spherical Nanoparticles Grown by Controlled Method to Develop Optical Phantoms

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Among the diverse applications of silica nanoparticles, the suitability of these to reproduce scattering and absorption properties in tissue simulating phantoms for diffuse optical imaging, through the use of Mie theory, has been recently proposed [1]. An important limitation to adjust the Mie theory to the pattern of scattered light by scattering spheres is the relative ratio between the wavelength of the incident light and the size of the scattering sphere, in this way, it is important to achieve monodisperse and controlled growth of the spheres diameter, so this ratio will be close to 1, allowing the Mie theory to work according to its own limitations.

The development of diagnostic imaging systems has required the use of tissue simulating materials to mimic the optical properties of human or animal tissues [2,3]. The properties of these phantoms usually are inconsistent and change over time, making comparison of different imaging systems a serious problem when also different animal-derived phantoms are being used [4]. The use of a standard phantom suitable to be reproduced in any laboratory with constant and confident optical properties is the main goal of using silica nanoparticles to develop this standard.

In this work a modified version of the Stöber Method [5,6] is used to obtain spherical silica particles by varying the proportion of water and ethanol contained in a solution while maintaining a constant proportion in the rest of the reagents: tetraethyl orthosilicate and hydroxide of ammonium. To analyze the structure of each sample the hydrodynamic diameters of SiO₂ spheres were measured by dynamic light scattering (DLS) using a Malvern Zetasizer Nano Range analyzer. Then the nanoparticle diameters were measured by scanning electron microscopy (SEM) observing spherical and monodisperse particles; SEM analyses were carried on with a FE-SEM JEOL JSM-7800F.

There are several methods for the synthesis of SiO_2 , we use a modified version of the Stöber Method. This method consists of controlled hydrolysis, catalyzed on alcoholic medium. The following precursors were used: tetraethylorthosilicate (TEOS, $Si(OC_2H_5)_4$) as precursor of silica, ethanol and deionized water as solvents and ammonia hydroxide (NH₄OH) as a catalyst; the synthesis took place at room temperature with magnetic stirring for 24 hours.

Figure 1 shows the hydrodynamic diameter distribution of the particles as a function of the percentage of water diluted in ethanol, it is observed that in the range of 4 to 8% of water volume the diameter of the particle increases rapidly to around 750 nm, while outside this range the diameter of the nanoparticle decreases; these results are in relative agreement with others reported in the literature [6]. This controlled growth will allow to vary the wavelength used in accordance with de particle diameter. In Figure 2 the scattering silica nanoparticles SEM images are shown.

By using this method, it was possible to control the size of the SiO₂ particles by varying only the water vs ethanol ratio contained in the solution, and by keeping the amounts of the TEOS and NH₄OH reactants constant, obtaining monodisperse particles suitable to reproduce properly the theoretical scattering patterns predicted by Mie scattering theory. These results allow us to have a consistent and confident method to develop the precursors for the tissue simulating optical phantoms, pursuing to make a standard for diffuse infrared and optical imaging systems.

References:

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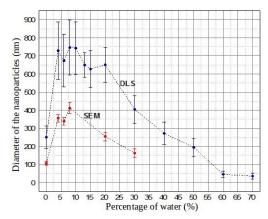


Figure 1. Comparison of diameters of the silica nanoparticles measured with DLS and SEM.

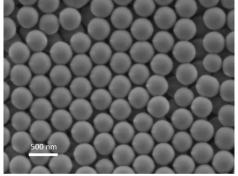


Figure 2. Morphology of SiO₂ spheres with average diameter 411 nm, 8% water, imaged by SEM; the scale bar shows 500 nm.