2382
doi:10.1017/S1431927619012649
Microsc. Microanal. 25 (Suppl 2), 2019

Controlled Growth of Silicon Dioxide Nanospheres by Regulation in the Addition Rate of the Precursor

Isidoro López-Miranda[1], Eduardo Ortiz-Rascón[2*], Leonardo Baez-Castillo[1], Roberto C. Carrillo-Torres[1] and Mario E. Álvarez-Ramos[1].

[1.] Departamento de Física, Universidad de Sonora, Hermosillo, Sonora, México.
[2.] CONACYT–Departamento de Física, Universidad de Sonora, Hermosillo, Sonora, México.
* Corresponding author: eduardo.ortiz@fisica.uson.mx

A silicon dioxide nanospheres suspension can simulate the optical properties of biological tissues [1-3], allowing to compare the Mie solutions to Maxwell equations for dielectric spheres with the diffusion approximation for the radiative transport equation. Experimental results obtained from the measurement of light transmitted through these phantoms allow us to obtain the scattering and absorption coefficients, these coefficients can be also predicted theoretically by the Mie solutions when the morphological properties of the silica nanospheres are known, then the diameter of these nanospheres must be controlled with precision.

In this work a modified version of the Stöber method is used [4], where by using tetraethylorthosilicate as precursor (TEOS) different sizes of silica spheres [5] are obtained in the presence of a catalyst (ammonium hydroxide), by means of the controlled addition of the precursor by dripping it into the reaction [6], in this way a monodisperse suspension of silica nanospheres can be achieved. These nanospheres are analyzed by dynamic light scattering (DLS, Malvern Zetasizer Nano Range analyzer) and by scanning electron microscopy (SEM, FE-SEM JEOL JSM-7800F) where, from a statistical analysis, its average diameter is calculated [7].

In this modified version of the Stöber method, and by using the variation in the addition rate proposed by Lei et al [6], we supply only the TEOS precursor without the presence of any alcohol, in different addition rates, to the solution (alcohol, water and ammonium hydroxide), and then we analyze the effect on the growth of the silicon dioxide nanospheres. The synthesis was carried out at room temperature in 20 ml samples with a concentration of ethanol in water at 20% and magnetic stirring at 250 rpm for 8 hours.

Figure 1 shows the micrographs corresponding to each addition rate of the TEOS to the solution, where (a) corresponds to the slowest addition rate and, in descending order, (g) corresponds to the fastest addition rate. The averaged diameters of these micrographs are plotted in Figure 2 (measured with DLS and SEM); for each point of the graph, its corresponding micrograph is indicated. By analyzing Figure 2, we note that, in the faster addition rate, the nanospheres with the smallest diameter were obtained, while for the slowest addition rates the largest diameters were achieved, just as previously reported results suggested [6]. This effect on the diameter could be related to the low concentration of the precursor in the solution; which would in return favor the hydrolysis of the dripped precursor in the formation of the SiO_2 nanospheres [8].

References:

[1] E Ortiz-Rascón et al., AIP Conference Proceedings (2010) p. 130.
[2] CJ Maughan Jones and PRT Munro, Journal of Biomedical Optics **22(9)** (2017), p. 095004.

[3] L Baez-Castillo et al., Microscopy and Microanalysis **24(S1)** (2018), p. 1432.
[4] W Stöber, A Fink and E Bohn, Journal of Colloid and Interface Science **26(1)** (1968), p. 62.
[5] R Sato-Berrú et al, Journal of Materials Science and Engineering A **3(4)** (2013), p. 237.
[6] X Lei et al., Integrated Ferroelectrics **154:1** (2014), p. 142.
[7] E Ortiz-Rascón et al, Microscopy and Microanalysis **23(S1)** (2017), p. 1924.
[8] The authors acknowledge funding from the Consejo Nacional de Ciencia y Tecnología (CONACyT), through projects 573 and 255791-INFR 2015.

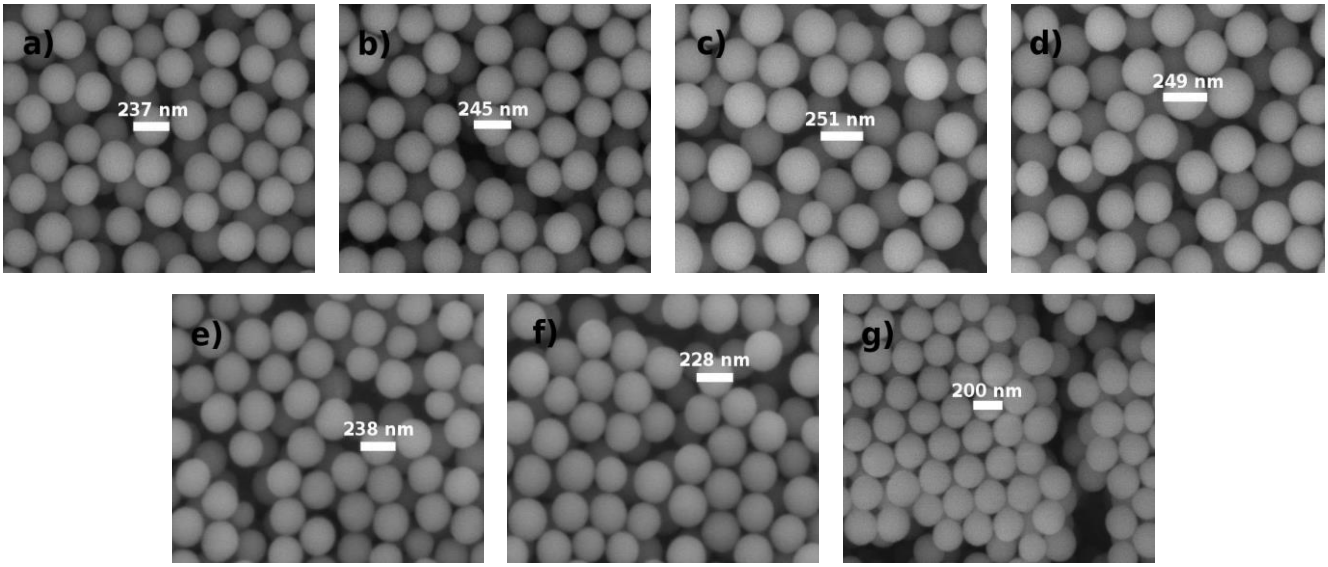

Figure 1. SEM micrographs of the SiO_2 nanospheres. The addition rates of the TEOS for each sample were: a) 5.6 µL/min, b) 8.3 µL/min, c) 16.7 µL/min, d) 50.0 µL/min, e) 62.5 µL/min, f) 111.1 µL/min, and g) 250 µL/min.

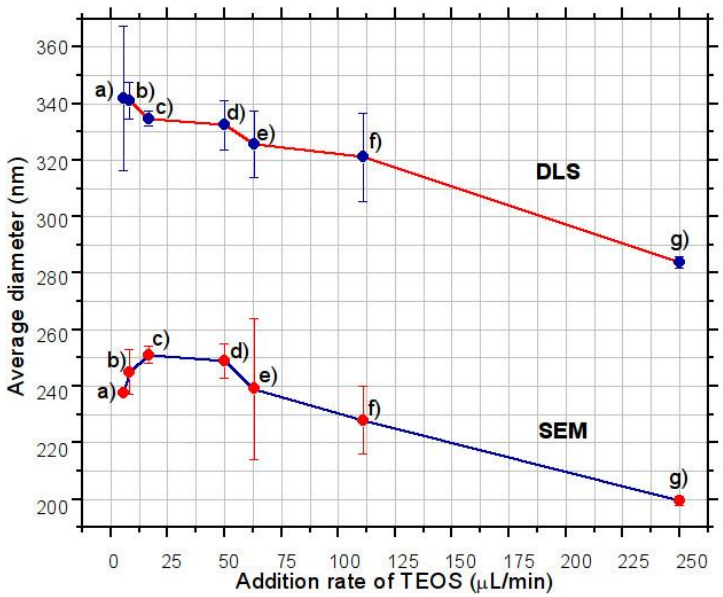

Figure 2. DLS measurements (upper curve in red) and SEM measurements (lower curve in blue). Each point corresponds to a different addition rate of the precursor (TEOS), as indicated in the micrographs in Figure 1.

https://doi.org/10.1017/S1431927619012649 Published online by Cambridge University Press