

Synthesis and Characterization of Silica Nanoparticles Obtained by Additions of Amino Acids into the Stöber Reaction

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Nanosized colloids have found a wide range of applications in the industry and medicine due to their tunable physiochemical properties [1]. It is well known that the properties of colloidal materials depend on their nature, size and distribution [1,2]. In particular, silica nanoparticles (SiO₂ NP's) are one of the most versatile colloidal materials due to their ease preparation and availability of precursors [2]. The Stöber process is widely used to obtain colloids of SiO₂ [3]. However, the method produces SiO₂ NP's with relatively large polydispersity especially for small size of NPs [3]. For this reason, the Stöber process has been modified to obtain better monodispersity by replacing ammonia with amino acids as base catalysts [2,4]. Herein we report the characterization by electron microscopy of SiO₂ NP's synthesized with additions of two different amino acids, L-glutamic acid (L-Glu) and L-arginine (L-Arg), with negative and positive side chains, respectively, into the Stöber reaction. It is important to say that in the present work ammonia has not been replaced by the amino acids, instead we are analysing the effects of both compounds (ammonia and amino acids) in the final characteristics of the SiO₂ NP's.

SiO₂ NP's have been synthesized using a 50/50 volume ratio of ethanol/water. The experimental procedure was as follows: 1 mmol of the amino acid, (0.147 g of L-Glu, Sigma, ≥99% or 0.174 g of L-Arg Sigma, 98.5%), was completely dissolved in a solution of 25.0 mL of ethanol and 25.0 mL of deionized water, then 3.0 mL of ammonium hydroxide (FagaLab, 72%) and 1.5 mL of TEOS (Sigma, ≥99%) were added under constant agitation. The solution was kept in agitation for one hour at room temperature. SEM and EDS analyses were carried out with a FESEM JEOL JSM-7800F, 15 KeV under secondary electron mode and an EDS Bruker QUANTAX XFlash 6|60 detector. For the analysis, the samples were placed on an aluminium alloy holder.

Both experiments produced spherical NP's with some degree of polydispersity (see Figure 1). However, the L-Glu SiO₂ NP's are bigger (290.86±17.65 nm) than L-Arg SiO₂ NP's (113.92±14.59 nm). EDS maps showed that there is carbon over the NP's (see Figures 2 and 3). Further, it was observed that there is material connecting the NP's (see Figures 4 and 5). Also, EDS line scan analysis revealed that this material contains carbon. Besides, in both analysis, the amount of carbon in L-Arg SiO₂ NP's is greater than in L-Glu SiO₂ NP's, which would suggest that L-Arg bound easily on the NP's surface due to its positive charge.

References:

- [1] I. Ojea-Jiménez *et al*, ACS Appl. Mater. Interfaces **8** (2016), p. 4838.
- [2] K. D. Hartlen, A. P. T. Athanasopoulos, and V. Kitaev, Langmuir **24** (2008), p. 1714.
- [3] W. Stöber, A. Fink, and E. Bohn, J. Colloid Interface Sci. **26** (1968), p. 62.
- [4] T. Yokoi *et al*, J. Am. Chem. Soc. **128** (2006), p. 13664.

[5] The authors acknowledge funding from CONACyT (255791-INFR-2015) to acquire the FESEM JEOL JSM-7800F microscope. F. J. Carrillo-Pesqueira gratefully acknowledges the scholarship provided by CONACyT (México).

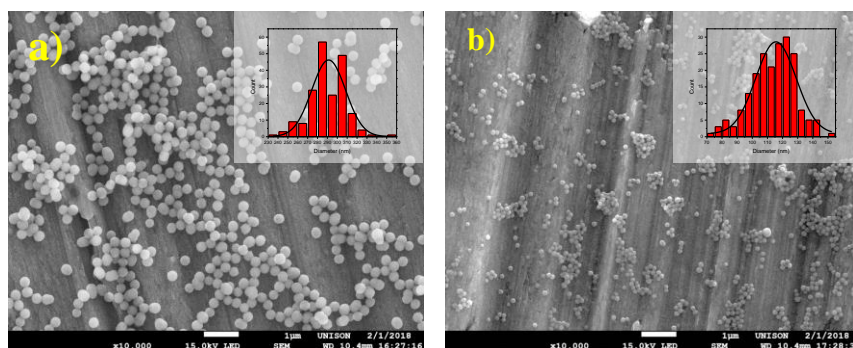


Figure 1. SEM images and size distribution of: a) L-Glu-SiO₂ NP's and b) L-Arg-SiO₂ NP's.

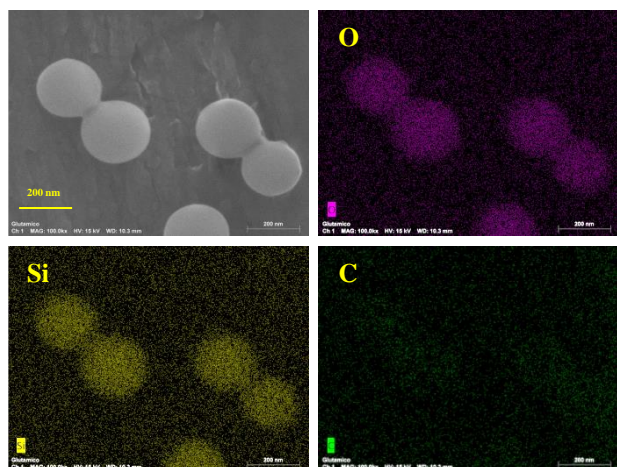


Figure 2. EDS map analysis of the L-Glu-SiO₂ NP's. The scale bar is 200 nm.

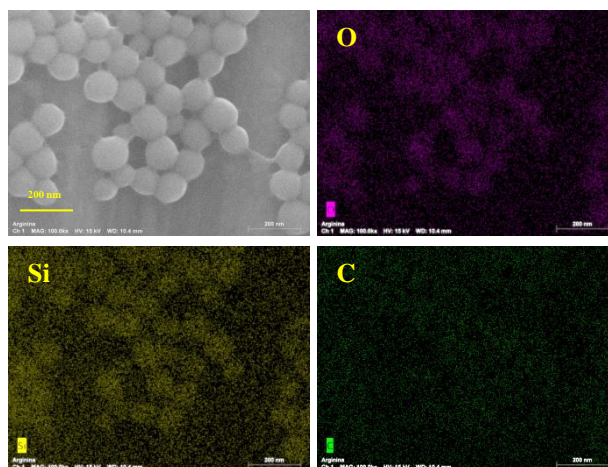


Figure 3. EDS map analysis of the L-Arg-SiO₂ NP's. The scale bar is 200 nm.

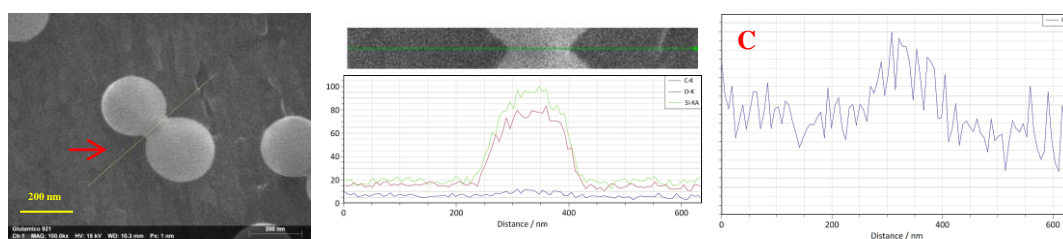


Figure 4. EDS line scan analysis of the L-Glu-SiO₂ NP's. The scale bar is 200 nm.

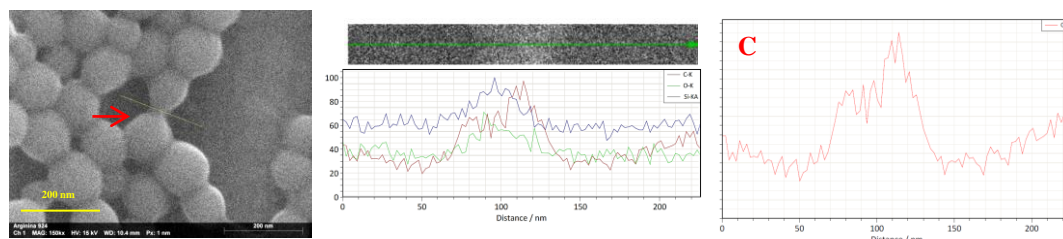


Figure 5. EDS line scan analysis of the L-Arg-SiO₂ NP's. The scale bar is 200 nm.