Size, roughness and porosity control in mesoporous Stöber-type particles

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Abstract

Mesoporous silica particles are the inorganic bedrocks from which, through functionalization processes, it is possible to design inorganic and hybrid nanomaterials with multiple emerging applications. Thus, very complex systems that include sophisticated functionalities have been designed, but in many cases, underestimating the role played by the inorganic silica support. In this work we synthesized mesoporous Stöber particles combining the atrane route (based on the use of triethanolamine Si-based complexes as Si source) [1] and the Stöber method (using hydro-alcoholic reaction media) [2].

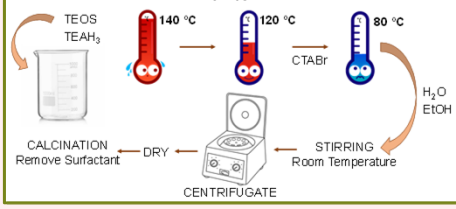
Objectives

- Control the particle size by varying the aging time (**Kinetic Control**)
- Study the effect of aging under hydrothermal conditions (**Hydrothermal Conditions**)
- Induce a massive and controlled degradation in silica mesoporous particles to increase their porosity using post-treatments in different medias (**Degradation**).

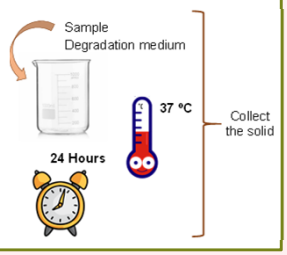
Experimental Procedure

Synthesis

2 TEOS (tetraethyl orthosilicate): 7 $TEAH_3$ (triethanolamine): 0.5 CTABr (cetyl trimethyl ammonium bromide): 1000 H_2O: 170 EtOH



Degradation



Kinetic Control

The particle size increases at the same time we increase the aging time

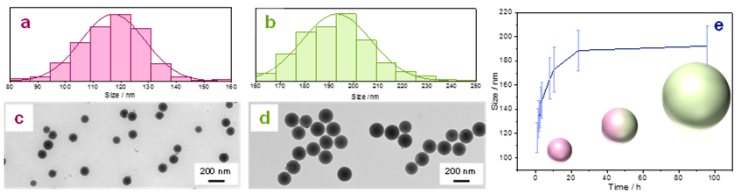


Figure 1. Size distribution of the samples at (a) 1 hour, (b) 96 hours. TEM images of the samples at (c) 1 hour, (d) 96 hours. (e) Size vs time

Hydrothermal Conditions

Modulating the particle surface

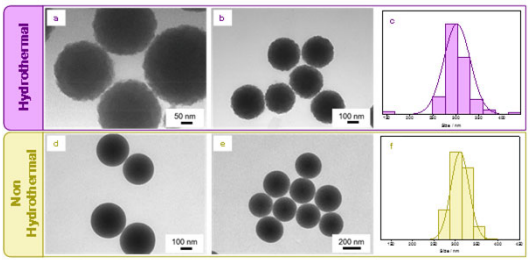


Figure 2. Hydrothermal sample: (a-b) TEM images, (c) Size distribution. Non-Hydrothermal sample: (d-e) TEM images, (f) Size distribution

Degradation

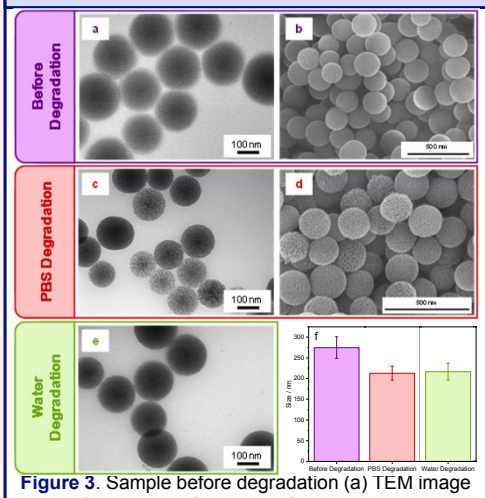


Figure 3. Sample before degradation (a) TEM image and (b) SEM image. Sample PBS degradation (c) TEM image and (d) SEM image. (e) TEM image of the sample in water degradation. (f) Comparison of the particle size of the different samples

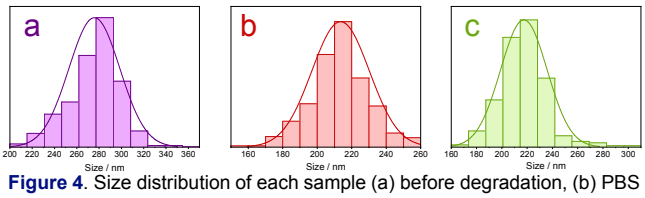


Figure 4. Size distribution of each sample (a) before degradation, (b) PBS degradation, (c) water degradation

The best of the degradation: Increase the porosity

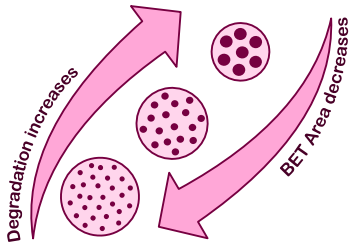


Table 1. BET area for each sample

	BET Area / m^2/g
Before Degradation	1132.4
PBS Degradation	707.5
Water Degradation	1111.1

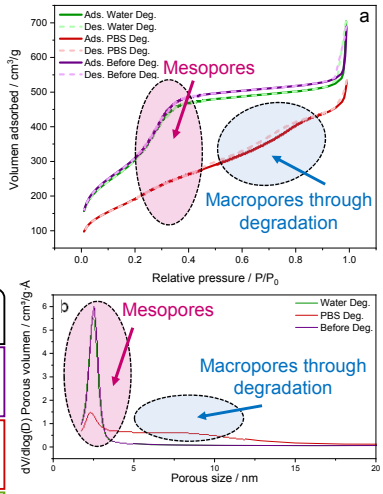


Figure 5. (a) Nitrogen adsorption-desorption isotherms and (b) Porous size distribution of each sample

Conclusion

- Varying the aging time: obtain particles with a controllable size (100-200 nm).
- Modulate the particle surface: change the roughness of the surface.
- Degradation: increase the porosity of the mesoporous particles using different medium.
- The particles can be used in catalysis or biomedical applications due to their small size.

[1] Nanoparticulated silicas with bimodal porosity: chemical control of the pore sizes. J. El Haskouri et al., Inorg. Chem., 2008, 47, 8267; doi.org/10.1021/ic800893a

[2] Controlled growth of monodisperse silica spheres in the micron size range. W. Stöber et al., J. Colloid Interface Sci., 1968, 26, 62; doi.org/10.1016/0021-9797(68)90272-5