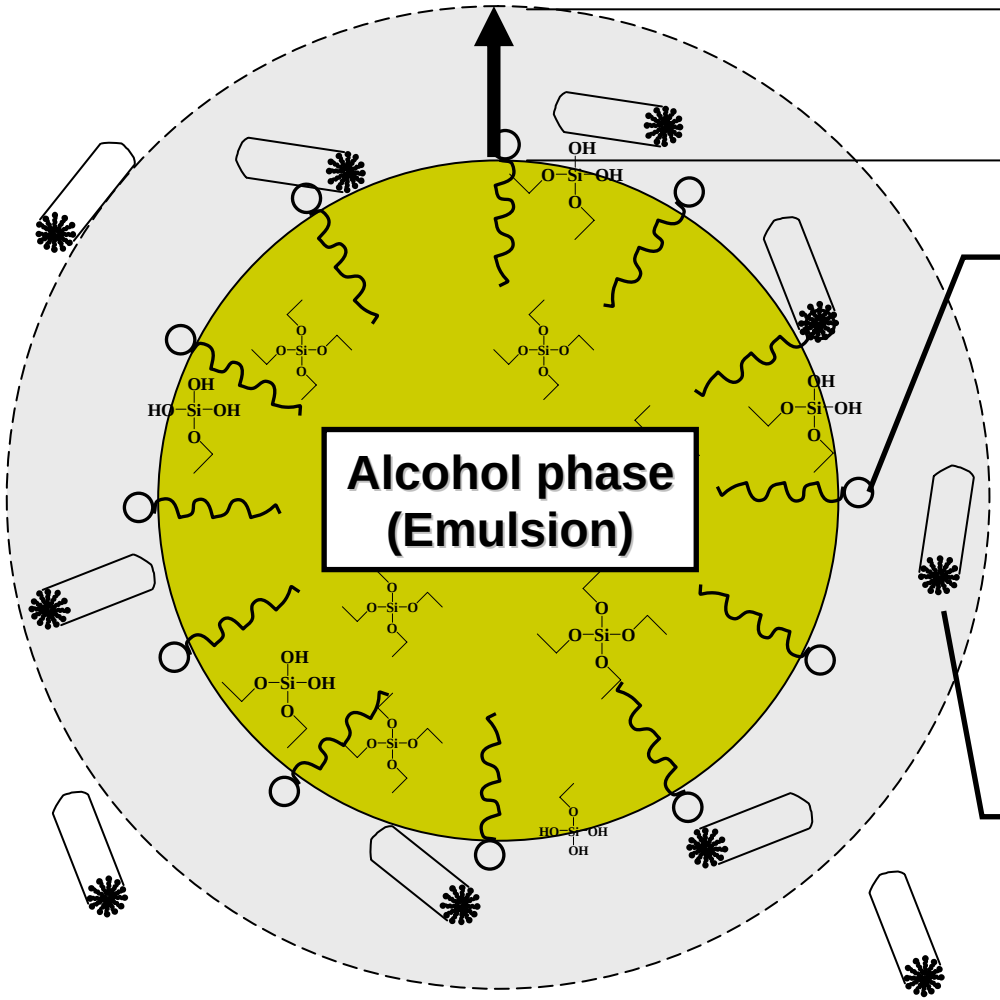
Synthesis and characterization of mesostructered sphere particles with core space

INHA University
Department of Chemical Engineering
Catalysis and Nano-materials Labortory

MECHANISM



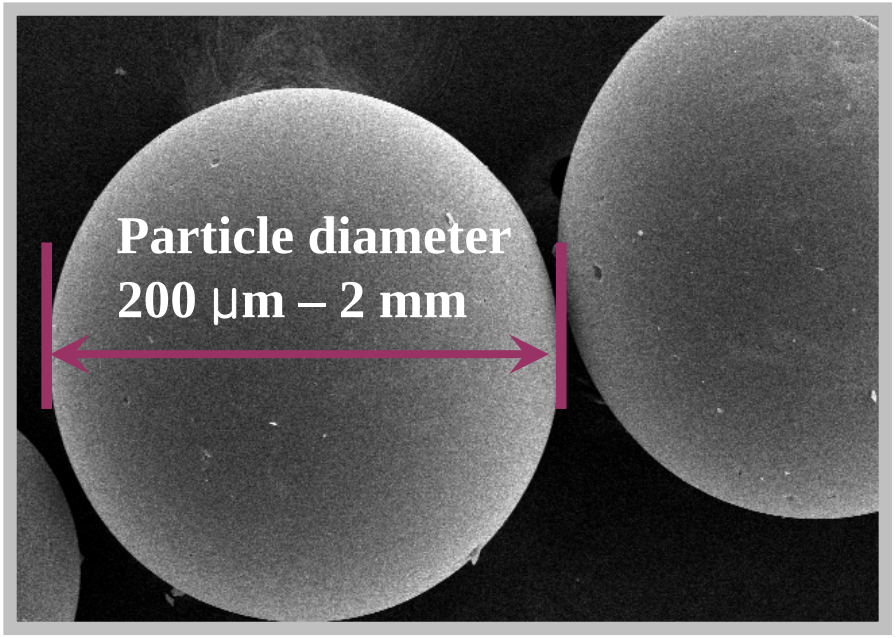
Direction of silica growth

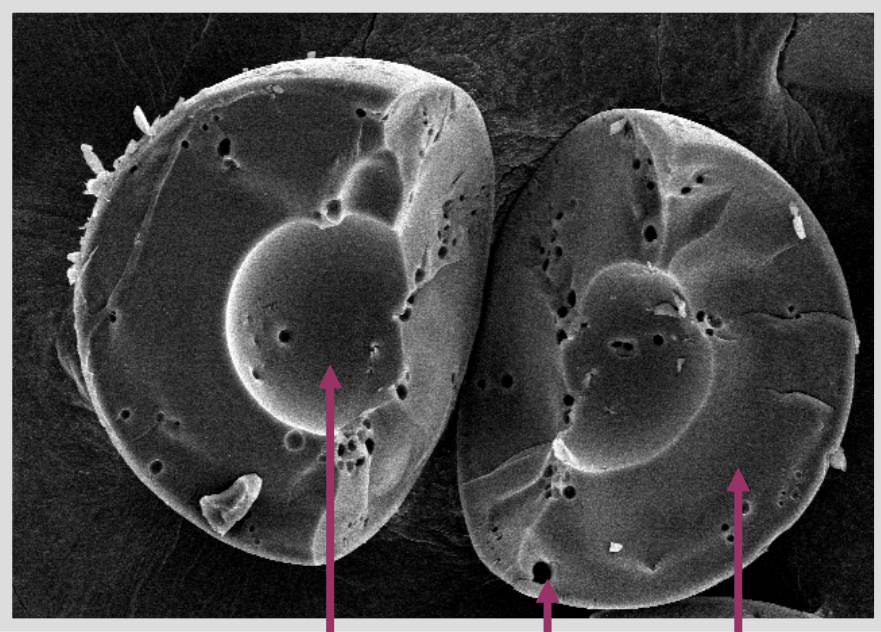
Surfactant as emulsion stabilizer

Surfactant as micell template with random orientation

Aqueous phase

PORE STRUCTURE SYSTEMS





Hollow space diameter : 100 – 200 μm

Macropore diameter : 1 - 5 μm

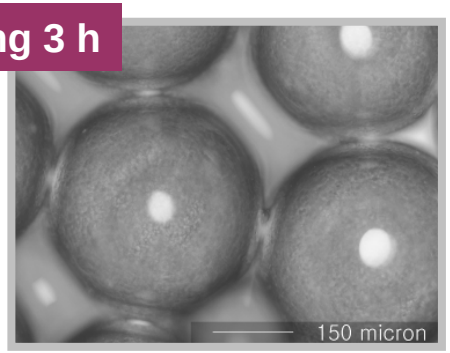
Mesopore diameter : 3 – 5 nm

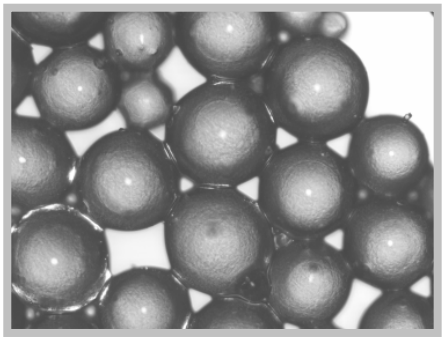
Morphology control factors :

1. ***Alcohol concentration***
2. ***Silica precursor concentration***
3. ***Stirring speed***

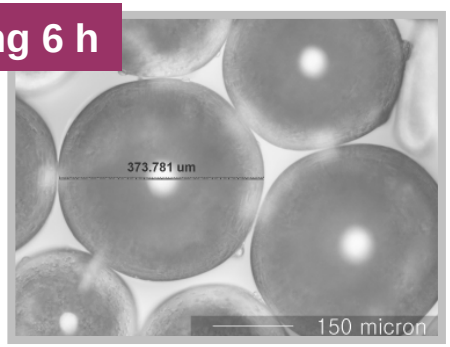
GROWTH OF SILICA SPHERES

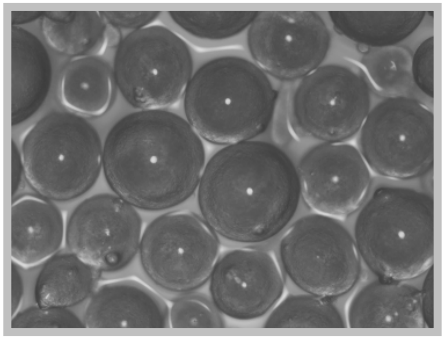
Aging 3 h



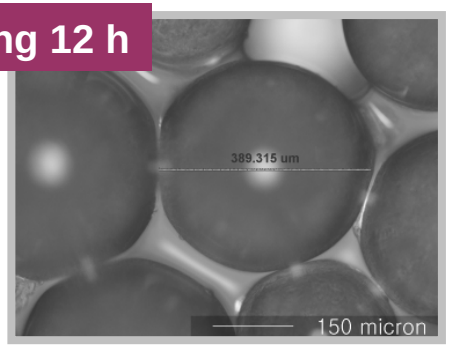


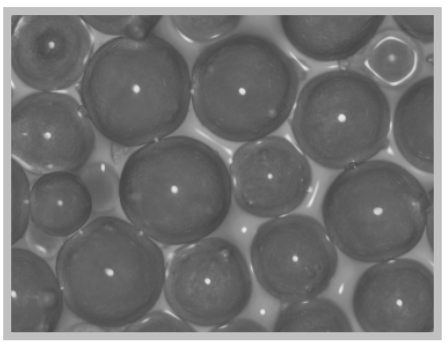
Aging 6 h





Aging 12 h





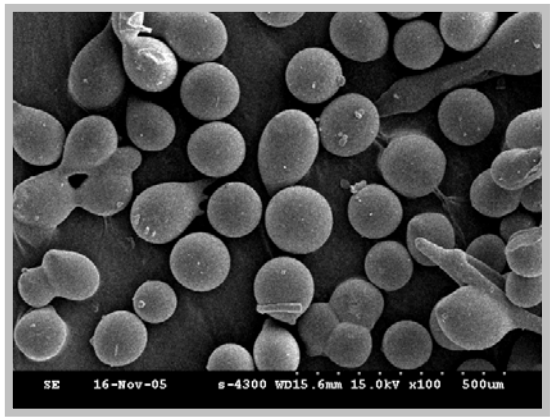
P123 + H_2O + 2M HCl

TEOS and alcohol dropwise addition

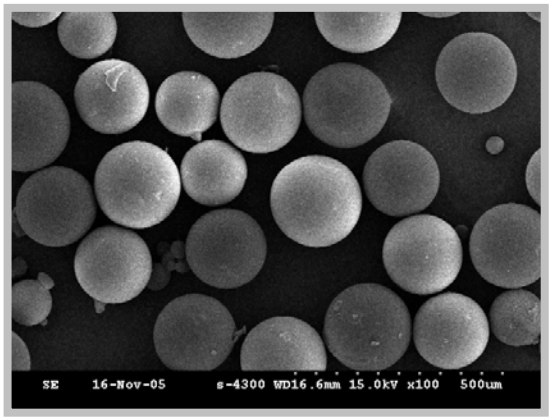
stirring at 35℃ for 1 day (300 rpm)

washing, filtration, drying, and calcination

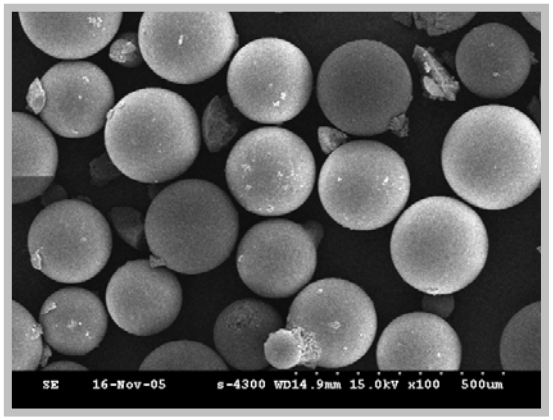
ALCOHOL CONCENTRATION (I)



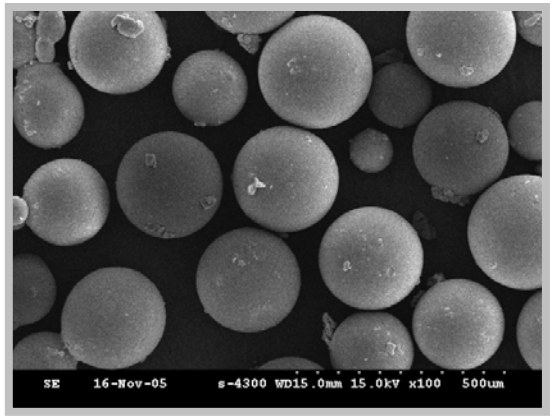
B181R6



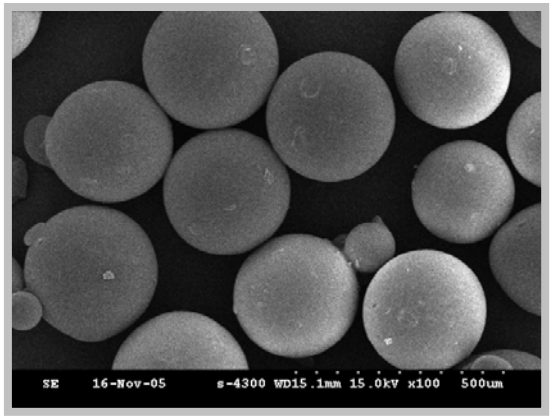
B217R6



B254R6



B290R6



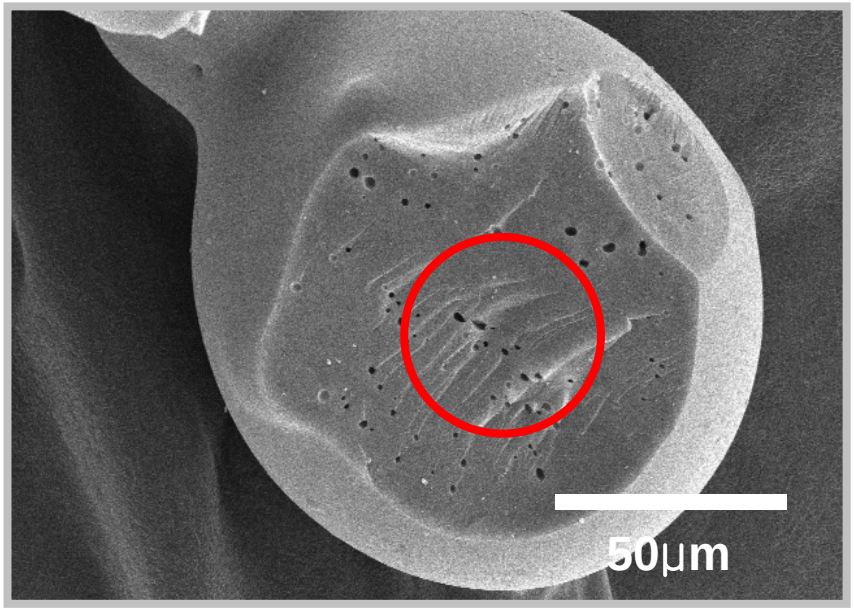
B326R6

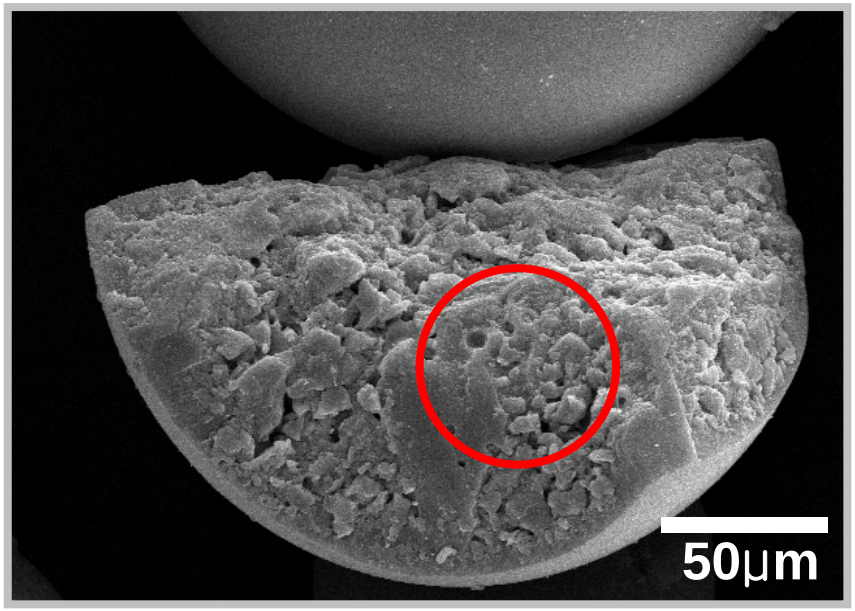
Sphere size increases with alcohol concentration.

B:alcohol conc.

R:stirring speed (600RPM)

ALCOHOL CONCENTRATION (II)

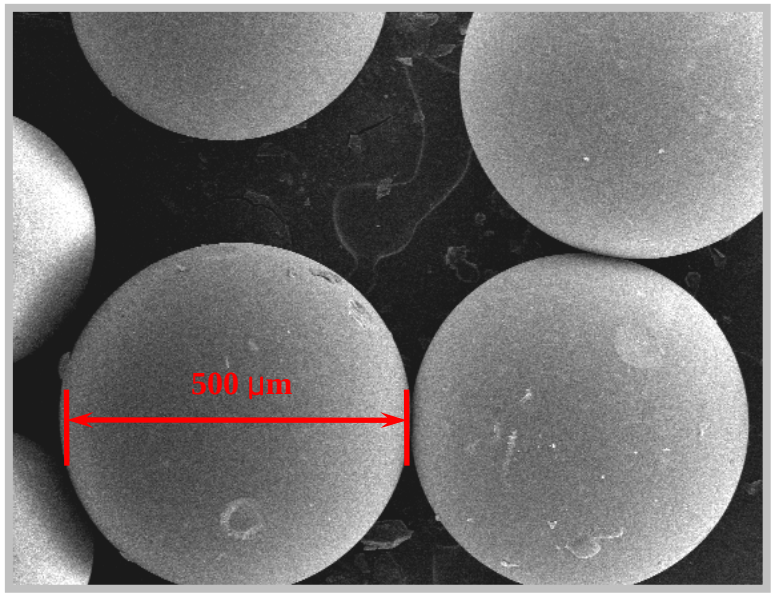




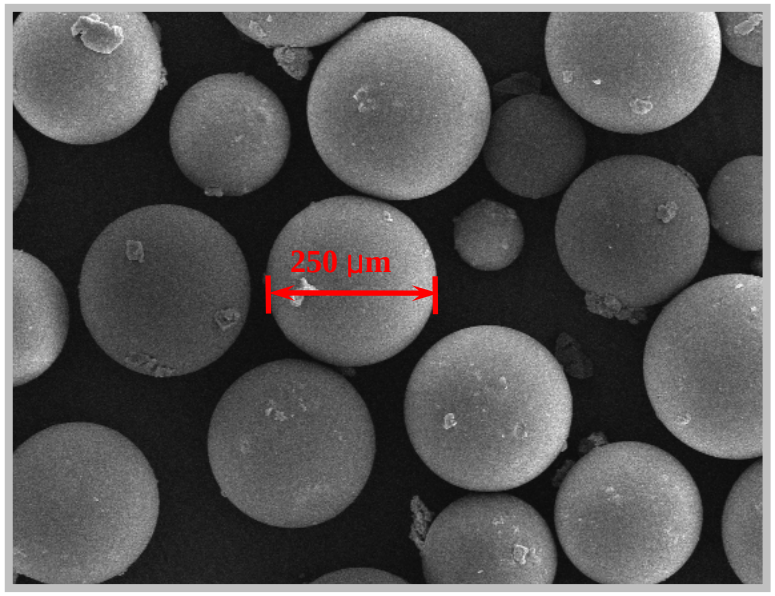
B181R6 **B254R6**

As the amount of alcohol increases, macropore structure developed.

STIRRING SPEED



B290R3

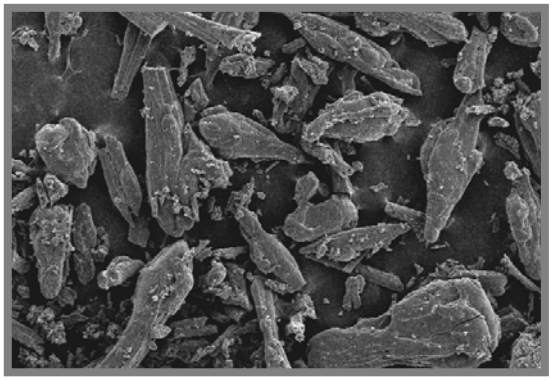


B290R6

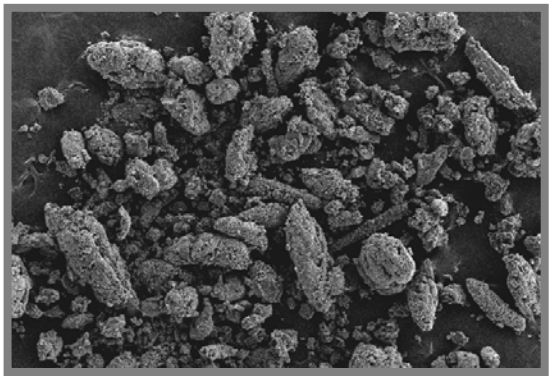
- *Sphere size decreased with increasing stirring speed.*
- *Spherical particles can be made over a wider region of stirring speed when the amount of alcohol in the mixture increases.*

TEOS CONCENTRATION

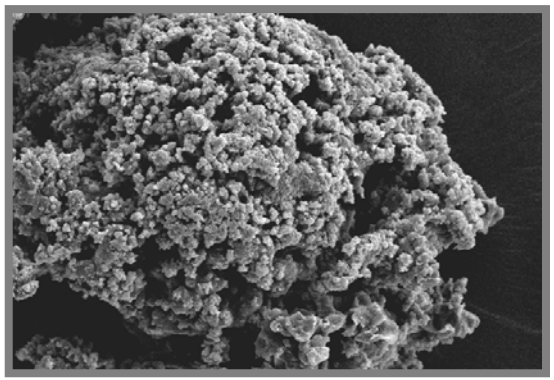
T102R3



T089R3

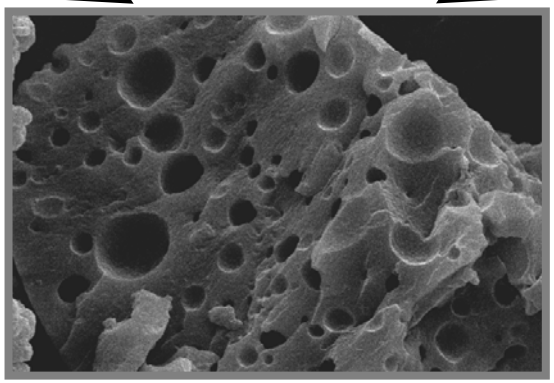


T076R3



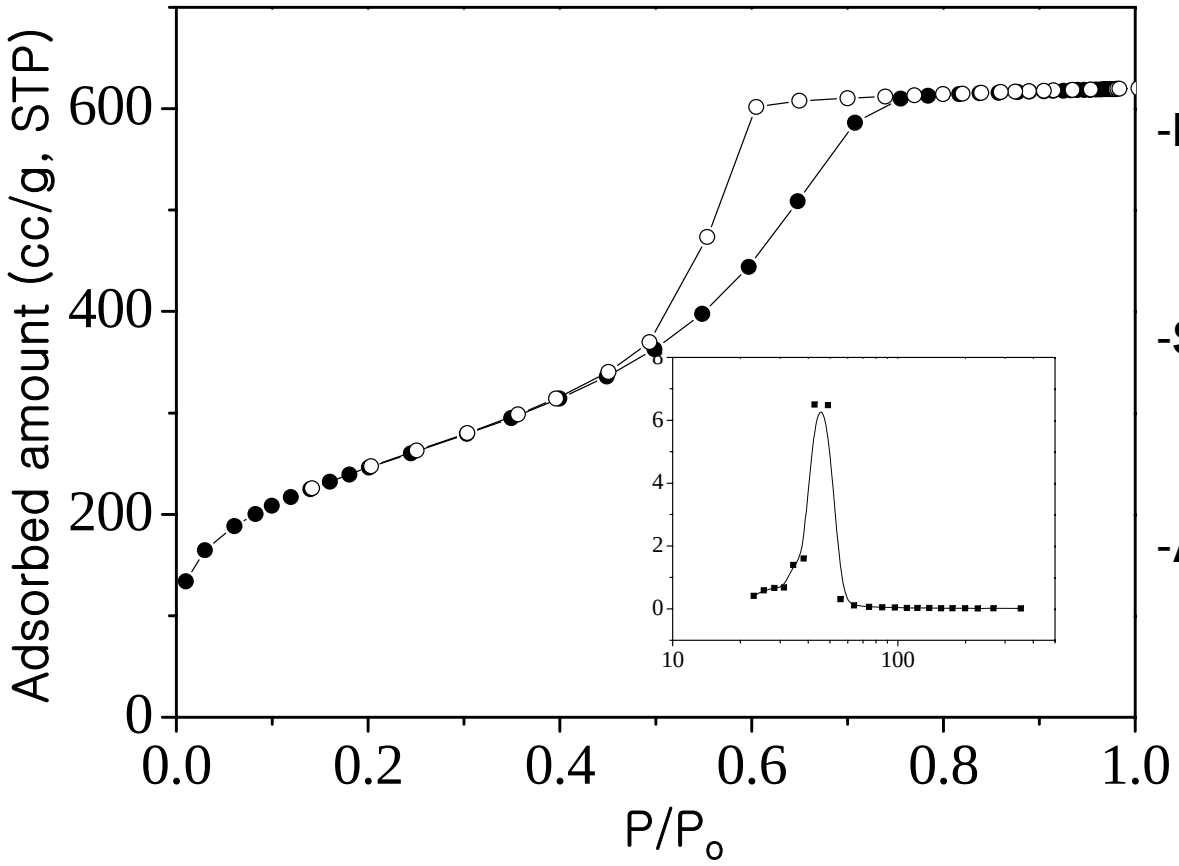
Random morphology

Below critical conc. of TEOS, nonspherical particles with sponge-like morphology were formed.



Sponge-like macropores

TEXTUAL PROPERTIES



-**BET surface area**

802 - 891 m^2/g

-**Single point pore volume**

0.76 - 0.98 cm^3/g

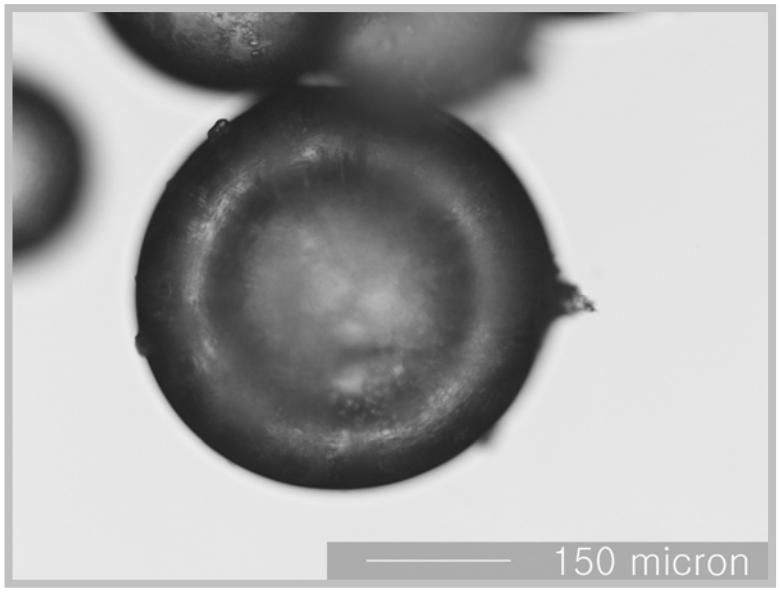
-**Average pore diameter**

3.2 - 4.9 nm

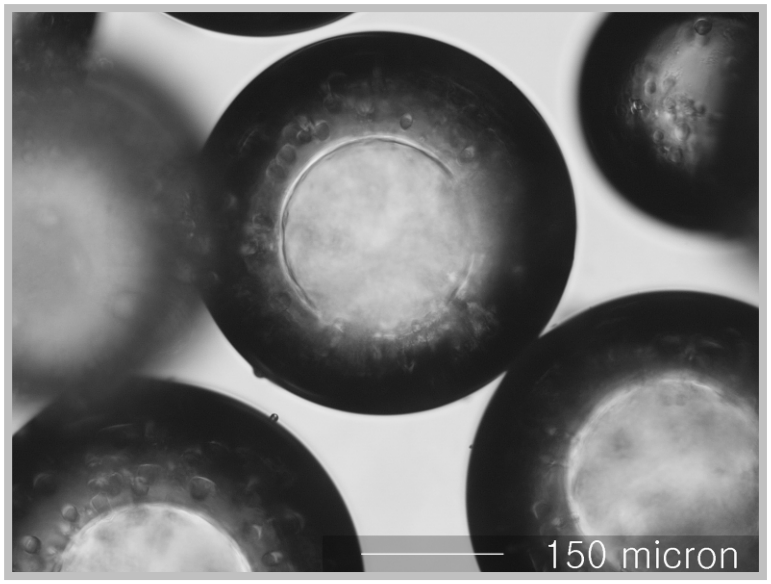
Type IV isotherm

Hysteresis roop was H2 type which was indicated cage-like mesopore

SILICA SOURCE EFFECT



TEOS 300 rpm



TPOS 300 rpm

-Other silica (TMOS, TBOS) sources can also be used to make spherical particles, but it is difficult to optimize the synthesis conditions due to different hydroloysis and condensation rates of the silica precursors.