Synthesis of Porous Silica from Water Glass by Dual Templating System

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Abstract

In this work, a method to synthesize mesoporous silica from water glass using dual templating system is proposed. Gelatin is used to control the silica porosity, whereas polyethylene glycol is to control the particle morphology.

The silica precursor was prepared by diluting water glass (SiO\(_2\):Na\(_2\)O = 3.3, concentration 26.84 wt. %) with the ratio 1:10. The diluted water glass was then passed through ion exchange resin H\(^+\) to produce silicic acid (pH ~ 2). Gelatin and PEG with various concentrations from 0.005 -0.02 g/cm\(^3\) was diluted into 3% aqueous formic acid and water respectively. Then, it was added into the previously prepared silicic acid. This solution was mixed with NH\(_4\)OH to form gel, and aged for about 18 hours. Then, it was dried in the oven at a temperature of 80 °C. The dried sample was then extracted in a Soxhlet using 5% aqueous formic and 5% aqueous DMSO. This process was done for about 48 hours until the yellow color of liquid in the Soxhlet disappeared. Some of the silica was dried in the oven at a temperature of 100 °C for 12 hours, and the other was calcined at a temperature of 550 °C for about 4 hours.

The pore volume of porous silica increases with the increase of template concentration. PEG can be removed from porous silica using Solvothermal extraction using DMSO. It can be seen from the IR-spectrum, which is similar to IR-spectrum of silica without template. On the other hand, gelatin can not be removed well using the same method, because it is more difficult to crack silica-gelatin hybrid. It is confirmed by IR-spectrum which has combination peak between silica peak and gelatin. Therefore, calcination was used to overcome this lack. Combination template removal (combine solvothermal extraction with calcination) can carbonize the gelatin and crack silica-gelatin hybrid. It can be seen from the IR spectrum that there are no hybrid detected. It is found that carbonized sample has higher surface area than original sample. For example: sample prepared using 0.005 g/cm\(^3\) gelatin template and 0.015 g/cm\(^3\) PEG template obtain surface area about 160.69 m\(^2\)/g, when carbonization was occurred, surface area increase to 281.44 m\(^2\)/g.

Keywords: porous silica, dual templating system, gelatin, PEG