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Effects of Flow Rate and Cosolvent on the Preparation of Silica Microspheres in Supercritical CO₂

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The silica microparticles synthesized in supercritical media has been widely and intensively utilized at last decade for using many application areas like catalysis, controlled release, and separation science due to their superior properties such as high surface areas, porosity, and regular morphologies [1]. Besides silica microparticles which have sphere morphology has been also employed for using many areas like drug delivery, column packing, and insulating materials. Although there are several techniques for preparing silica microspheres, sol-gel technique using supercritical fluids is an efficient and easy technique. CO₂ is often used as a supercritical fluids for this technique due to its low critical temperature parameters, inertness, nontoxicity, nonflammability and low cost. The solubility effect of CO₂ can be changed by adding a small amounts of organic solvents. The most important advantage of the technique is that particle sizes can be controlled by adjusting parameters such as temperature, pressure, flow rate of supercritical CO₂ and cosolvent type.

In this study, the effects of flow rate of CO₂ and cosolvent type on silica microspheres prepared by using one step sol-gel technique in supercritical CO₂ were investigated. The CO₂ was flowed through the reactor at a rate of 1-10 mL min⁻¹ which was detected at the syringe pump. The cosolvent was added in the reactor with approximately 2% (v/v). Prepared silica microparticles at 80°C and 200 bar were characterized by scanning electron microscope (SEM), which was used to determine the size and morphology of particles. Functional group analysis were also performed by using Fourier Transform Infrared Spectroscopy (FT-IR) techniques.

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References

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