

Synthesis of silica nanoparticles in a hybrid microwave-plasma hot-wall reactor under well-controlled conditions

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Silica nanoparticles have a large range of practical applications. They are widely used as fillers in plastics and coatings to improve material properties such as hardness and thermal stability. Especially applications with a demand for high transparency require silica particles with a specific size, morphology, and surface coating. Therefore, the synthesis of particles with highly-defined particle-size distributions is desired. Most of the silica powder worldwide is produced by gas-phase processes such as flame reactors. However, these methods require exact knowledge of the kinetics of precursor decomposition, particle formation and particle growth. Tetraethoxysilane (TEOS) as a halide-free and inexpensive precursor material is subject to growing interest for particle formation from the gas-phase.

A microwave-induced plasma reactor combined with a hot-wall furnace has been constructed for gas-phase synthesis of high-purity silica nanoparticles. The microwave heats the injected gas mixture including TEOS vapor within few microseconds. This initiates the chemical reactions of the precursor decomposition which is then followed by particle formation. The hot-wall furnace allows to achieve longer and variable residence times at high temperature which allows further particle growth such as coalescence, agglomeration as well as sintering of particle agglomerates. The combined use of a plasma and hot-wall reactor allows studying particle growth in detail. Information about the growth kinetics can then be used for the reactor design. The plasma reactor can be initiated and the plasma can be maintained using precursor gaseous TEOS in the presence of O₂ and Ar or N₂. In order to obtain reproducible synthesis conditions the current apparatus has been designed to allow the control of gas flow rates, gas composition, pressure and temperature of the hot-wall furnace.

Particle sizes, shapes and morphologies have been characterized by transmission electron microscopy (TEM). For TEM measurements, the sample was collected through thermophoretic deposition of nanoparticles inside the reaction chamber. TEM results reveal that particles are spherical in shape with a high degree of agglomeration. The size of the primary silica nanoparticles was modified by varying the process parameters, such as TEOS concentration,

reactor pressure, residence time and the furnace temperature. With TEOS concentration of 1.2% by mass, reactor pressure of 40 mbar and the furnace temperature of 800°C, the particles with mean diameter of 9.8 nm were synthesized. The mean particle size was found to increase linearly with increasing the reactor pressure (fig. 1). The degree of agglomeration depends very strongly on the furnace temperature and the resident time. The maximum degree of agglomeration was observed at 400°C while varying the furnace temperature from 200°C to 1000°C. An additional ex-situ analysis was carried out by means of isothermal Brunauer-Emmett-Teller (BET) adsorption. The primary average BET particle diameter was calculated from the determined powder surface area.

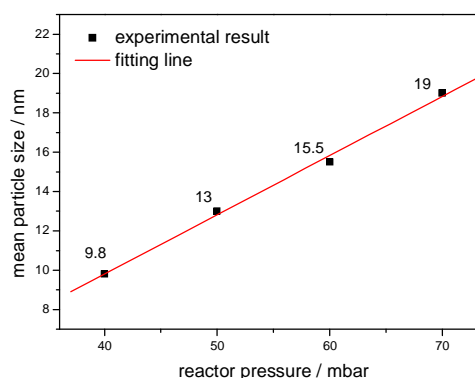


Figure 1. Influence of reactor pressure on the particle size at 800°C furnace temperature

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