INFLUENCE OF PH, TEMPERATURE AND SAMPLE SIZE ON NATURAL AND ENFORCED SYNERESIS OF PRECIPITATED SILICA

Sebastian Wilhelm, Karlsruhe Institute of Technology (KIT), Department of Thermal Process Engineering, Germany
Sebastian.Wilhelm@kit.edu
Matthias Kind, Karlsruhe Institute of Technology (KIT), Department of Thermal Process Engineering, Germany

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Precipitated silica is an amorphous solid and has a wide range of industrial applications. It may be used not only as adsorbens in liquids and gases or as pigment in paintings, but it is also added to polymeric and pharmaceutical products as an inexpensive and inert filling material. In industrial production, monomeric silicic acid is produced by mixing an inorganic, silicate-based precursor and an acid in a stirred semi-batch process. During this process, the monomeric silicic acid polymerizes to amorphous silica particles. Finally, further polymerization and agglomeration of the particles lead to a colloidal gel network. This colloidal gel network shows syneresis behavior resulting in shrinkage and expulsion of liquid which was enclosed within the gel due to the continuing polymerization reaction. Thus, the product properties (e.g., agglomerate size, porosity or internal surface) are strongly influenced by syneresis. Understanding the dependency between the process parameters and the polymerization and syneresis respectively will give the opportunity to control the precipitation process of silica in order to produce a product with properties specially designed for any specific application purpose.

Syneresis of silica depends among other parameters on pH, temperature and sample size and can be a time-consuming process. Therefore, we developed the unique concept of “enforced” syneresis as the superposition of “natural” syneresis with an additional, external force in order to accelerate the syneresis of colloidal gels. Hereby, two open key aspects are of particular interest. On the one hand, the question arises whether natural and enforced syneresis are analogous processes with respect to their dependence on the process parameters. On the other hand, a model is to be developed that allows for predicting natural syneresis, based upon data determined by enforced syneresis.

The dependency of the maximum volume shrinkage $\Delta V/V_0|_{max}$ and the rate of natural syneresis on pH (one acidic and one basic reaction regime), on the temperature (20, 40 and 60°C) and different sample sizes are determined experimentally. The courses of shrinkage can be represented sufficiently well using an empirical model with two fitting parameters (the maximum volume decrease $\Delta V/V_0|_{max}$ and a characteristic time constant τ considering the rate of syneresis). Experimental syneresis processes of at maximum ~10 days are necessary to obtain these characterizing values. In the case of enforced syneresis, larger values for $\Delta V/V_0|_{max}$ and smaller values for τ respectively are obtained, but in a significantly shorter time (~5 h). Furthermore, the dependency of enforced syneresis behavior on varied process parameters will be presented. We can show that the pH-, temperature- and sample size-dependency of natural and enforced syneresis are indeed analogous. Finally, a correlative model will be derived and discussed that allows for a prediction of natural syneresis.

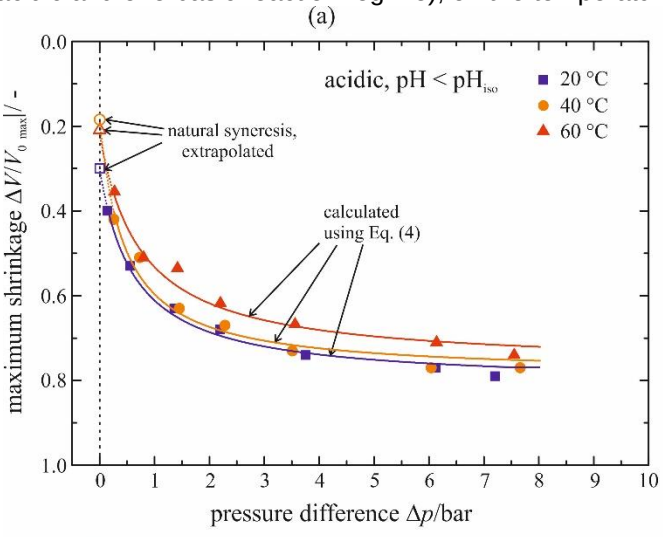

Figure 1 – $\Delta V/V_0|_{max}$ as a function of applied, external pressure difference