



Crystallization of phase-pure anhydrous calcium carbonate polymorphs with a modular crystallization reactor

H. Nebel, M. Epple

Institute of Inorganic Chemistry, University of Duisburg-Essen, Universitätsstrasse 5-7, 45117 Essen, Germany

Calcium carbonate can crystallize in three different anhydrous polymorphs, namely, aragonite (orthorhombic), calcite (rhombohedral) and vaterite (hexagonal). The controlled crystallization of the different anhydrous calcium carbonate polymorphs is difficult to accomplish because of marginal differences in the energy of crystallization.^[1–4] Furthermore the effect of the different parameters during crystallization is not yet completely understood. We have developed a modular crystallization reactor where the anhydrous calcium carbonate polymorphs can be synthesized in a phase-pure form by precipitation from aqueous solutions without any additives. All parts of the reactor are temperature-controlled by a thermostat. For the synthesis of aragonite and vaterite, a solution of CaCl_2 and a solution of Na_2CO_3 are pumped with two peristaltic pumps into two coil distillate condensers. After the coil distillate condensers, the solutions are led into a double-walled two-neck adapter. To ensure, that the solutions are well mixed, the reactive solution is passed through a Vigreux column which leads to a highly turbulent current. The mixed solution then flows through a coil distillate condenser again and after that into a 500 ml double-walled three-neck round bottom flask which acts as continuous stirring reactor. The dispersion is dispensed onto a temperature-controlled Büchner funnel where the calcium carbonate is filtered off. For the synthesis of calcite, the Vigreux column and the post connected coil distillate condenser are exchanged by a 1000 ml double-walled three neck round bottom flask, which acts as an additional stirring reactor. Therefore, the initially precipitated vaterite is fully converted into phase-pure calcite.

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