

Development of a Method for the In-situ Investigation of the CO₂ Induced Gelation of Biopolymer Solutions towards Aerogel Production

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CO₂ induced gelation is a promising method for the gelation of biopolymer solutions yielding homogenous and stable aerogels with high specific surface area after supercritical drying with CO₂. In this gelation method, pressurized CO₂ is applied to an aqueous biopolymer system with or without additional gelation agents. The ongoing gelation process after the pressurization is separated in different possible process steps: 1) dissolution of pressurized CO₂ into the aqueous biopolymer solution, 2) reaction of the CO₂ with water and formation of carbonic acid, 3) dissociation of the carbonic acid and drop of the pH value, 4) pH induced dissolution of a gelation agent, and induction of the gelation by 5.1) the drop of the pH value or by 5.2) the liberated gelation agent (see Figure 1).

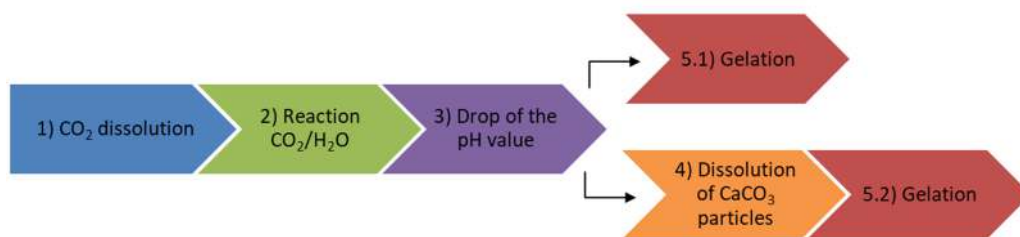


Figure 1: Scheme of the process steps of the CO₂ induced gelation of biopolymer solutions.

Characteristics of each process step depend strongly on the processed gelation system and gelation parameters like CO₂ pressure and temperature and, therefore, have an impact on the overall gelation process.

In this work different in-situ and ex-situ measurement methods for the investigation of the single process steps and the overall gelation process were developed in order to characterize the gelation mechanism and its kinetics.

Step 1: In-situ measurement of the dissolution kinetics of CO₂ into the aqueous biopolymer systems with a magnetic suspension balance in high pressure CO₂ environment

Step 3: In-situ measurement of the pH value inside the biopolymer solution via fluorescence pH sensor at increased pressure of CO₂

- Step 4: In-situ and ex-situ measurements of gelation agent particle dissolution via optical turbidity measurements
- Step 5: Ex-situ determination of the change of system's viscosity
- Overall process: In-situ visual observation of the gelation process by the use of a high pressure viewing cell

The evaluation of the developed measurement methods is performed with the aqueous gelation system composed of amidated pectin with calcium carbonate as additional gelation agent.