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In-situ observation of particle formation in freezing aqueous solutions by using synchrotron X-ray techniques

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Freezing of water is prevalent in surface environments. To date, geochemical processes in ice have rarely been considered primarily due to the Arrhenius effect on chemical reactions as well as the fact that it is a solid phase. During the last decade, however, it has been demonstrated that various chemical reactions, including redox, photochemical, and (reductive) dissolution reactions, were abnormally accelerated in freezing solutions primarily by the freeze concentration effect. We are currently working on the freeze-induced reactions in solutions containing dissolved Mn(II) and carbonates under varying solution conditions.

Upon freezing at –5 or –20 °C, rhodochrosite ($MnCO_3$) formed in the frozen solutions, which did not occur at room temperature under otherwise the identical conditions. Yet, this observation was made after thawing the frozen solutions, which may alter the extent and/or products of the intrinsic reactions. In order to avoid any potential artefacts of thawing, we are now trying to develop *in-situ* X-ray absorption fine structure (XAFS) and transmission X-ray microscopy (TXM) analyses with custom-built cold stages to examine the $MnCO_3$ formation in frozen solutions without thawing. The *in-situ* XAFS analysis successfully demonstrated the progressive formation of $MnCO_3$ during the solution freezing. In addition, the *in-situ* TXM images displayed the distribution and size of $MnCO_3$ particles formed in ice. These *in-situ* techniques could further provide prerequisite information to unravel the freeze-induced reactions.