Visual Measurements of Water Solubility in Methane and Hydrate Equilibrium at Cryogenic Conditions with Implications for LNG Production

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The formation and deposition of solids during the cryogenic processing of liquefied natural gas (LNG) is a perennial risk for plant operators. Lack of relevant experimental solid−liquid equilibrium data at LNG conditions limits the accuracy of model predictions and simulations. This can lead to: (1) unscheduled LNG plant shutdowns due to the blockages caused by the freeze-out of impurities, and (2) equipment design which is over-engineered to prevent blockages. Formation and deposition of impurities, especially ice or gas hydrates in LNG production are not very well understood. Currently, the LNG industry uses an estimate between 0.1 and 1 ppmv as an operational moisture limit. However, no experimental data for ice or hydrate formation and melting are available at such low concentrations, preventing verification of predicted phase boundaries for water solids and limiting our understanding of solidification kinetics in such systems. We present here outcomes from experiments using a specialized CryoSolids apparatus, consisting of a high-pressure sapphire cell (pressures up to 25 MPa) housed in a cryogenic environmental chamber which enables visual measurements at temperatures down to 87 K. The cell is equipped with a Peltier-cooled copper post, which can be sub-cooled relative to the bulk fluid mixture, allowing a controlled location for solid formation within the cell. In this work, the CryoSolids apparatus was used to produce the first solid-fluid-equilibrium data for water solids forming from a 200 ppm water in methane mixture at pressures to 20 MPa. This data, and ongoing experimental campaigns, will enable model verification and tuning of the binary interaction parameters for higher solute concentrations.