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Monitoring crystallization processes using the Mixture Composition Probe (MCP)

One common method of industrial crystallization is to dissolve a solute material in a solvent and then slowly evaporate the solvent. Evaporative crystallization is used for many industrial processes including sucrose and sodium chloride production. Often, crystallization is used to purify or modify the properties of the solute material.

To demonstrate the capabilities of the MCP probe for monitoring crystallization processes, we performed a test re-crystallization of the chemical oxybenzophenone (2-Hydroxy-4-methoxybenzophenone) in an acetone solvent. This material was chosen because it is highly soluble in acetone and results in a high solids concentration during re-crystallization. High crystal concentration is typical in many industrial processes, and we wanted to test realistic conditions. In addition, this material could be mixed with a water "anti-solvent" to make slurry to add to the acetone solvent. Again, slurry transport of the solute is common to some industrial crystallization processes.

To start the process, the MCP probe tip was immersed in acetone held in a small glass vessel. The acetone was continuously stirred and slowly heated to 50 degrees C. Slurry of 66 weight percent oxybenzophenone in water was prepared to add to the 75 grams of acetone.

The figure below shows the measurements of the MCP probe during the entire dissolution and recrystallization process. Measurements of mean crystal size (blue), crystal concentration (green), water/acetone ratio (yellow), and temperature (red) are plotted against the minutes after slurry addition. Crystal size is plotted on the left y-axis; all other measurements on the right y-axis. The processing steps are indicated by the numbers in the figure. These steps include:

1. The crystal/water slurry is slowly added to the acetone and this cools the mixture. The crystals dissolve at first then begin to grow (0 to 4 minutes).
2. The acetone is heated and starts to evaporate and boil. This increases the water (anti-solvent) to acetone ratio so some crystals form with low concentration and uniform size (4-25 minutes).
3. With continued acetone evaporation, the water/acetone ratio reaches 30%. At this saturation, crystal concentration and mean size increase rapidly (25-35 minutes).
4. In the final stage, water/acetone ratio and crystal concentration slowly increase as more acetone is evaporated. Crystal size remains fairly steady and the mixture is very cloudy. Near the end of the tests so much acetone has evaporated that the probe gap is not always full of liquid. This results in transients in the water/acetone ratio(35-70 minutes)

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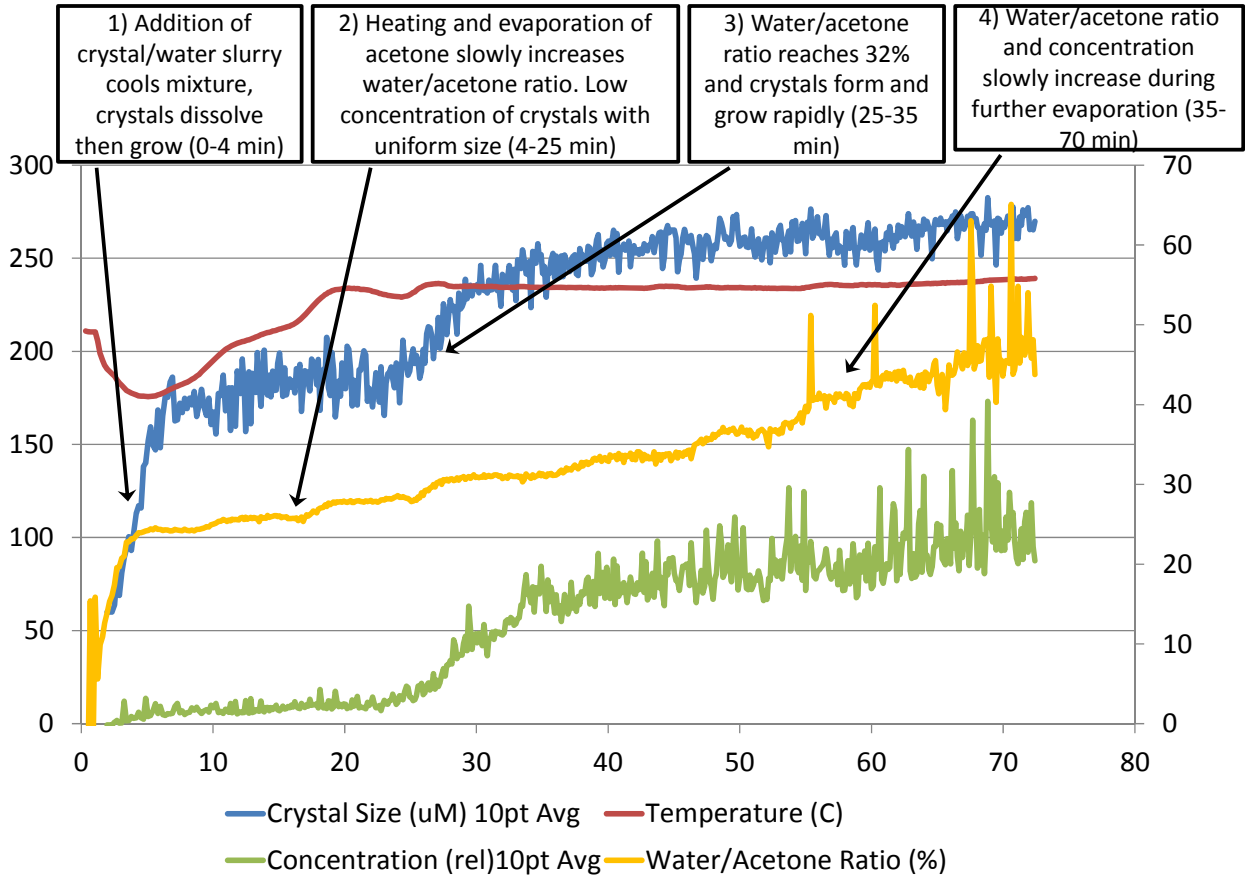

This experiment demonstrates that the MCP probe provides key measurements of crystallization processes. Monitoring these key parameters can lead to a better understanding of the process, as well as better control of final particle size and concentration.