Batch crystallization of xylitol by cooling, evaporation and anti-solvent addition

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This contribution focusses on investigating various crystallization methods for xylitol to achieve crystal uniformity as well as a desired size and shape. The particle size distribution and morphology influence significantly the product suspensions' filterability, drying properties and other down-stream processes. Xylitol is a five-carbon sugar alcohol with applications in food, pharmaceutical, ontological and cosmetic industries, and many other fields. In this study, three different techniques for xylitol crystallization and their operation parameters were evaluated regarding their influence on the product crystal properties: cooling, evaporative, and anti-solvent crystallization.

Xylitol is highly soluble in water and saturated xylitol solutions have relatively high viscosities, as shown in Figure 1. The viscosity can have a key role in crystallization kinetics especially in cooling and evaporative crystallization. In anti-solvent crystallization, the saturated solutions have usually lower viscosity due to lower solubility levels and the presence of anti-solvent compared to cooling and evaporative crystallization.

In the case of batch cooling crystallization, the temperature of the saturated solution (T_{sat} = 40°C) was decreased from 40 to 25 °C at a constant cooling rate. When the temperature reached 37.5 °C, seed crystals were added, which is required in xylitol crystallization due to extremely slow nucleation. It was found that no crystallization occurs without seeding. The amount of added seeds was 1% of the theoretical crystal yield, which was calculated from the solubility difference between 40 °C and 25 °C. During this series of experiments, various batch times were applied – from 1 to 5 hours - to investigate the resulting, transient supersaturation.

The second method investigated was semi-batch evaporative crystallization at a constant temperature of 50 °C and atmospheric pressure. The used evaporation conditions in terms of evaporation time and evaporation rates were varied to change

the supersaturation degree. Seeding was carried out 15 minutes after the beginning of the experiment.

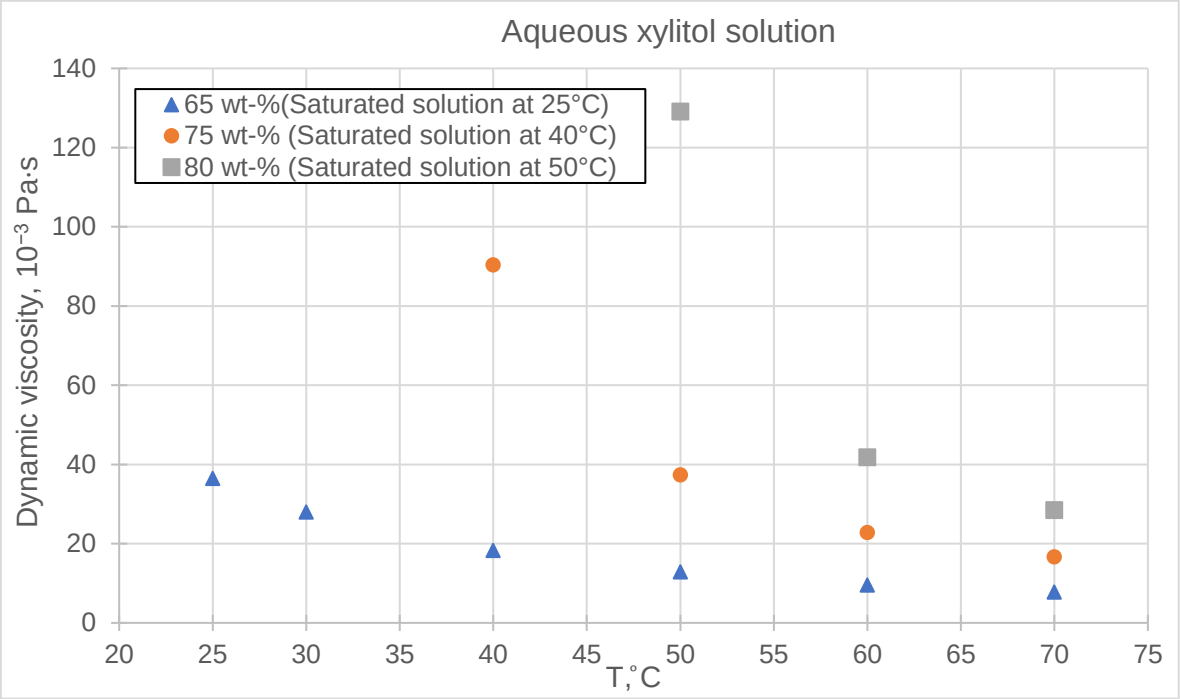

Figure 1– Measured dynamic viscosity of aqueous xylitol solutions at various concentrations

The third investigated technique was semi-batch anti-solvent crystallization utilizing ethanol for the aqueous xylitol solutions. Anti-solvent crystallization is a .method where supersaturation is achieved by using an anti-solvent reducing the solubility of the main compound. In this study, a certain amount of ethanol was continuously pumped to aqueous xylitol solution at a constant temperature of 40 °C. The addition time of ethanol was varied to change the driving force, i.e. supersaturation degree.

All crystallization experiments were accomplished in an EasyMax 402 stirred reactor station. Figure 1 shows the initial solution concentrations used at 40 °C (75 wt-%) and 50 °C (80 wt-%) and the solubility of xylitol at 25 °C (65 wt-%). In addition, a peristaltic pump, connected to a crystallization system allowed a continuous supply of anti-solvent. Moreover, a Mettler-Toledo Particle Track G400 based on the Focused Beam Reflectance Measurement (FBRM) was connected to the reactor. It should be mentioned here that, using the FBRM, it is possible to obtain some kinetic data

corresponding to nucleation and crystal growth rate. FBRM provides precise and highly sensitive chord length data collection to capture real-time changes.

Scanning electron microscopy was used to study the morphology of obtained xylitol samples. Typical results of scanning electron microscopy (SEM) are illustrated in the figure below (Figure 2).

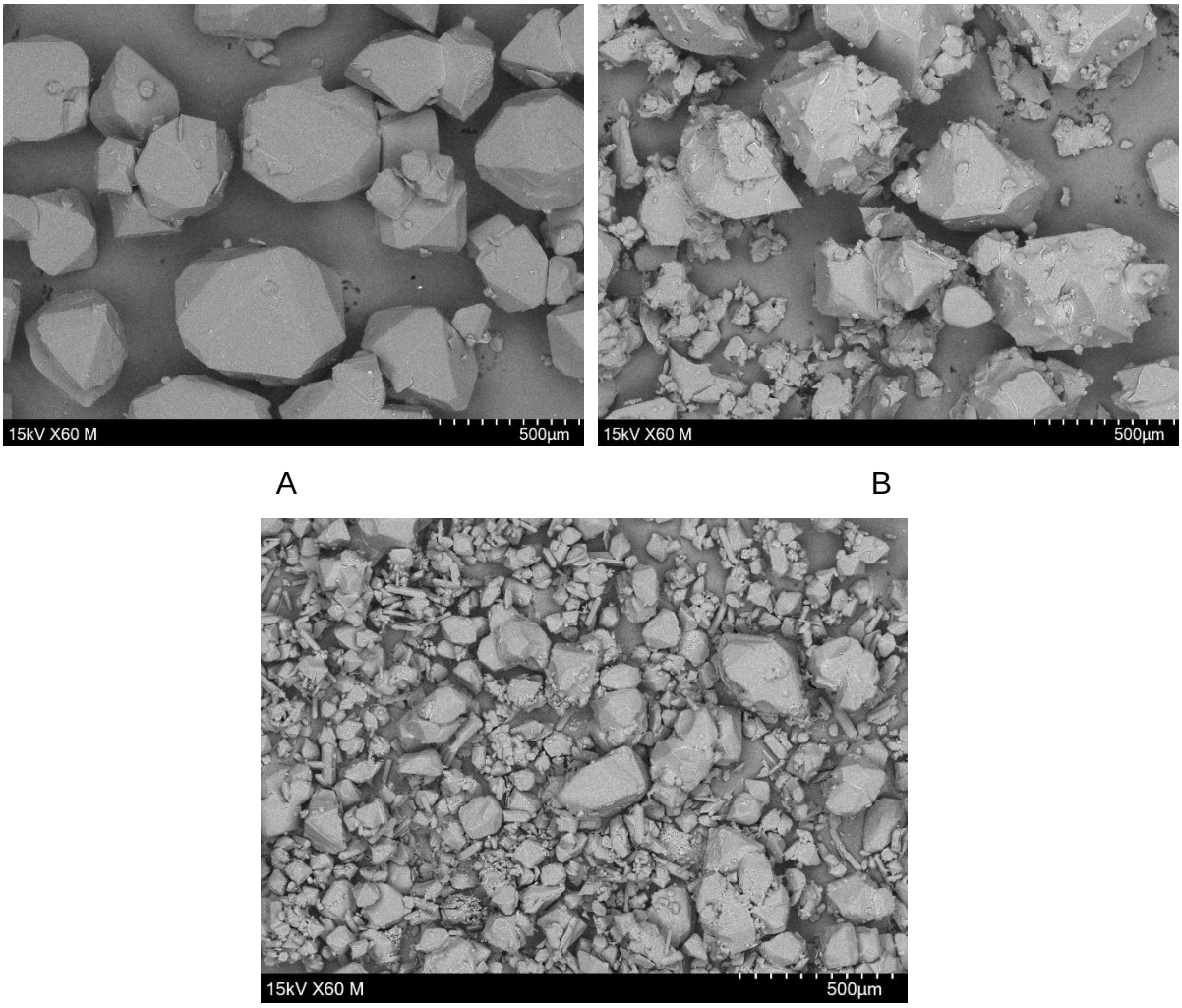

Figure 2 – Typical SEM micrographs of xylitol obtained by cooling (a), evaporative (b) and anti-solvent crystallization (c).

Micrographs of different xylitol materials are shown in Figure 2 using a 500 nm scale and 60 magnification. It should be mentioned here that anti-solvent seems to increase the supersaturation in such a way that very large nucleation rates of xylitol are induced. Thus, as the SEM pictures are indicating, if the nucleation rate is high,

smaller sizes are obtained. As expected, anti-solvent crystallization resulted in the smallest xylitol crystals, whereas the largest crystals were obtained by cooling crystallization applying the mentioned crystallization conditions. In the case of evaporative crystallization, obtained xylitol crystals were smaller and less uniform than the crystal product obtained by cooling crystallization.

Acknowledgments

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