# EFFECT OF SEED MASS ON SONOCRYSTALLIZATION KINETICS OF POTASSIUM SULFATE

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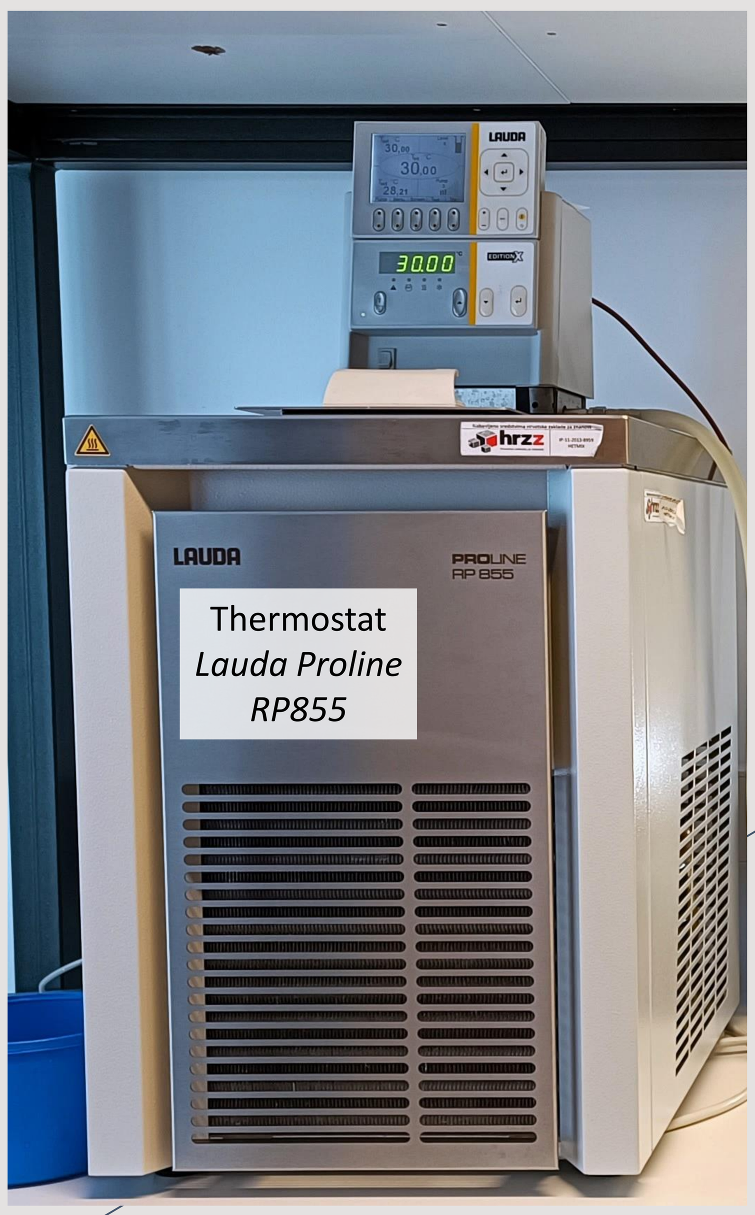
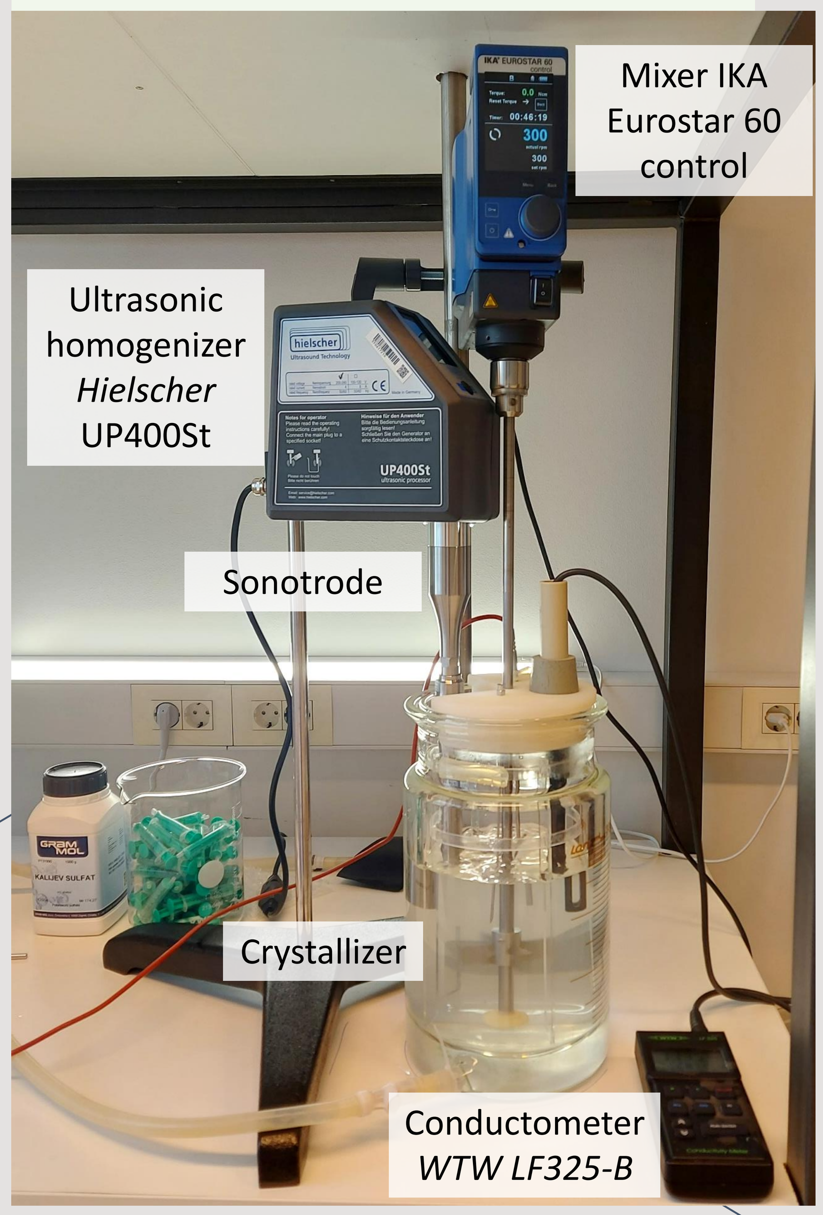
## INTRODUCTION

The use of ultrasound in crystallization has gained significant attention in recent years. Compared to conventional crystallization, sonocrystallization offers increased repeatability, one of its key advantages. Sonocrystallization may also provide an increased crystal growth rate, reduced agglomeration, and narrower CDS. Seeded crystallization is also used to control the CSD. Seed is introduced into a supersaturated solution to act as nucleation sites. The main focus of this pilot study was to **analyze the effect of sonication on the process of seeded batch crystallization of potassium sulfate**. Crystallization was conducted in a 2,65 dm$^3$ vessel in which mechanical mixing and ultrasonic irradiation were applied. Potassium sulfate served here as a model salt to study the kinetics of sonocrystallization processes.

*Keywords: sonocrystallization, mechanical mixing, seeding, pulsed sonication.*




## EXPERIMENTAL SETUP

Crystallizer volume: 2,65 dm$^3$
Saturation temperature: 30 °C
Cooling rate: 6 °C/h

**Temperature**: Pt100 probe
**Solution concentration**: determined based on measured solution conductivity
**Crystal size distribution (CSD)**: laser diffraction (*Horiba LA300*)
**Crystal morphology**: FEG-SEM (*JEOL JSM 7610F Plus* )

**Mechanical mixing:**
Straight blade turbine (SBT)
$N$ = 300 rpm
$D/d_T$ = 0,53
$C/H$ = 0,33

**Sonication**:
Sonotrode *H22L2D*
$A$ = 20 % (11 μm)
$P$ = 20 %

## RESULTS

To determine the seeding conditions, unseeded crystallization was conducted to determine the seeding temperature and seed load. It was found that seeding should be performed at 29 °C (in the first half of the metastable zone width). To determine the seed load and size, empirical expressions were used (Mullin, 2001; Somarriba et al., 2004.):

$$\frac{m_T}{m_s} = \left(\frac{L_{s,fin}}{L_s}\right)^3 \qquad \frac{L_{s,fin}}{L_s} = \frac{1}{5} \qquad \frac{m_T}{m_s} = 2 - 6\%$$

Potassium sulfate solution was saturated at 30 °C and was cooled down at 6 °C/h. Solution temperature and concentration were monitored contiuously (*in-situ*), while crystal size distribution was determined *off-line*.

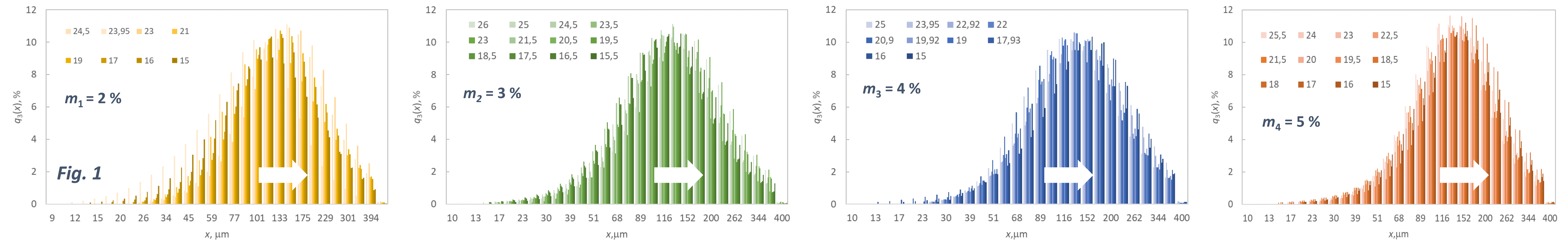



Fig. 1

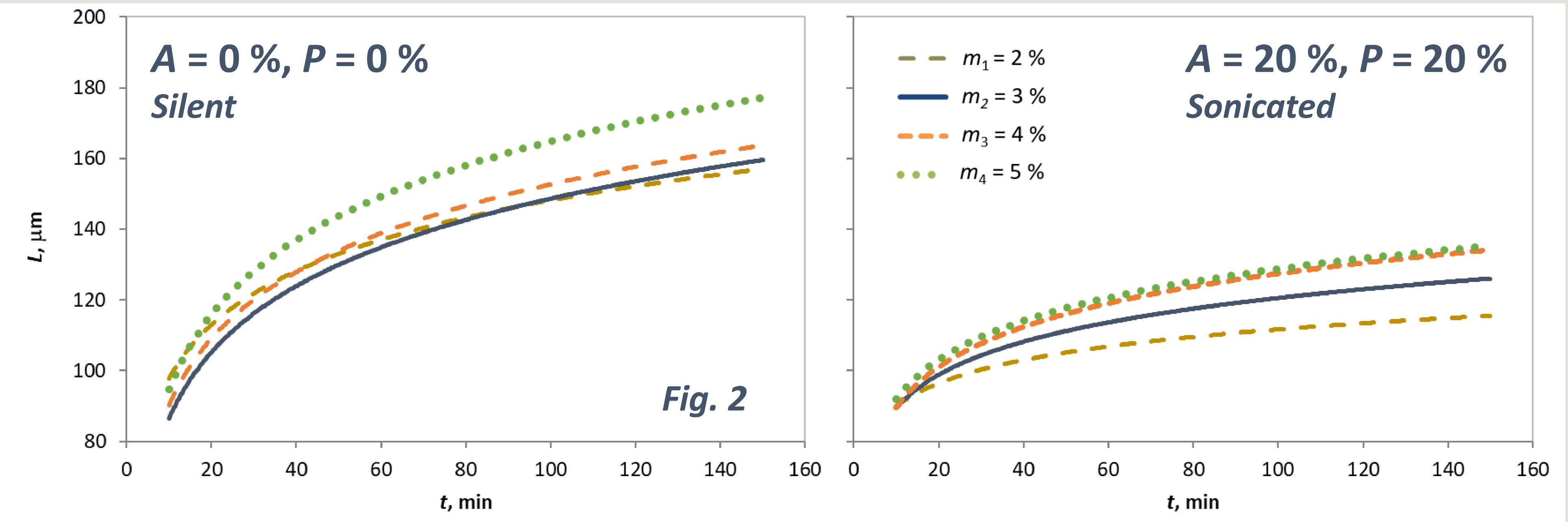



Fig. 2

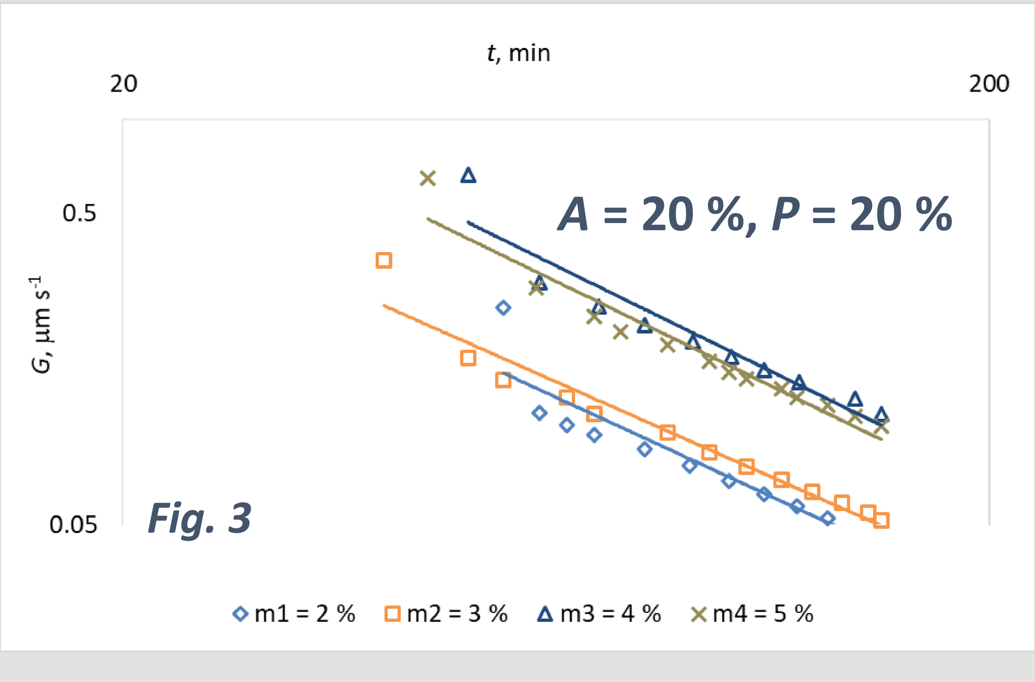



Fig. 3

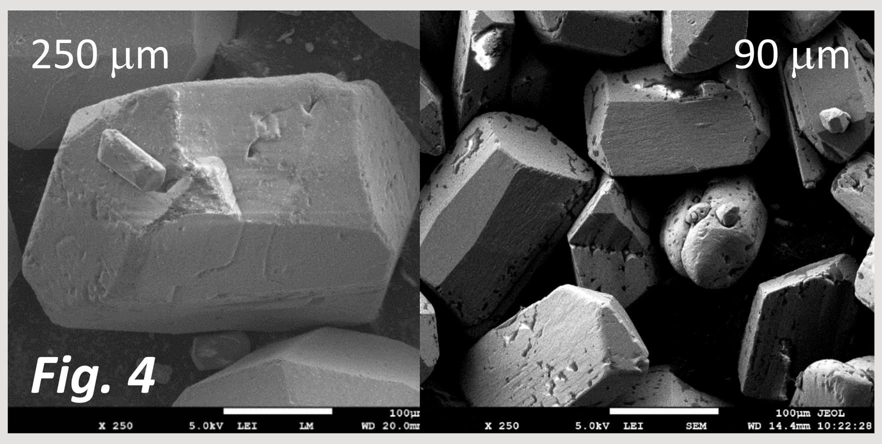



Fig. 4

## CONCLUSION

CSD (Fig. 1) showed that the crystal size distribution function shifts to the right as temperature decreases, meaning that crystal size increases over process time. Crystals were significantly larger in silent system (A = 0 %, P = 0 %) than in the sonicated one (A = 20 %, P = 20 %) indicating that ultrasound limits the size to which crystals can grow (Fig. 2). Thus, in the sonicated system, crystals achieve their final size quite early in process time. Growth rate shows that the higher the seed load, the higher the growth rate in the sonicated system (Fig. 3). Similar was observed in silent system (not shown here for brevity). However, this was much more pronounced in the sonicated system where crystals achieved final mean volume diameter earlier in process time. this indicates that they could be prone to attrition due to mechanical forces caused by the impeller but even more by bubbles implosion in the sonicated systems. SEM was used to inspect the crystal morphology of samples of average size = 250 μm and 90 μm. It was found that they were regularly shaped indicating that sonication ensures regular growth as opposed to agglomeration which is often encountered in mechanically agitated silent systems.

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