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Crystallization of α -glycine by anti-solvent assisted by ultrasound

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Introduction

► Ultrasound and Crystallization by cooling:

Reduction of the induction time and a reduction of the size of the formed crystals with equivalent supersaturation.

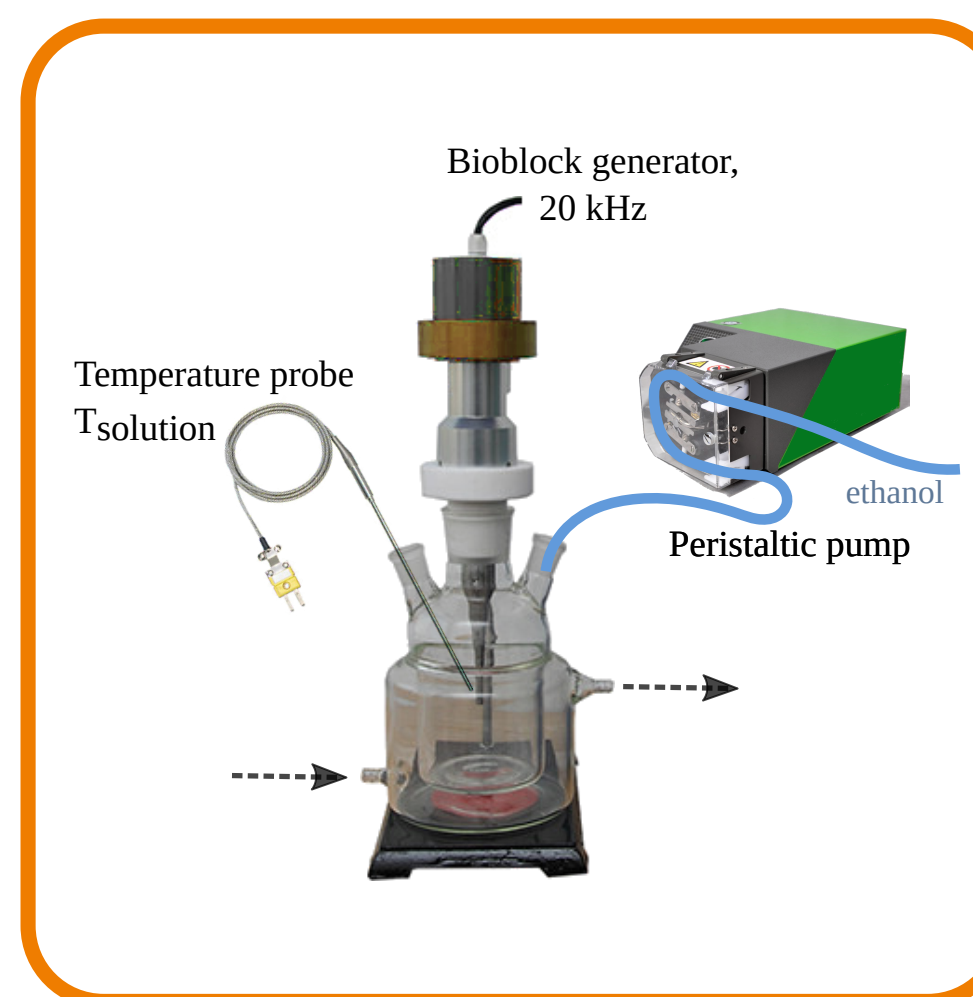
► Ultrasound and Crystallization by anti-solvent effect:

Do ultrasound have an influence on the crystallization of glycine by antisolvent effect in batch system (induction time, crystal size distribution, polymorphism)?

Materials and methods

► Glycine

- 3 polymorphic: α , β and γ (stable)
- Initial solid: α (mass purity 99.9%)
- Soluble in water
- Insoluble in ethanol



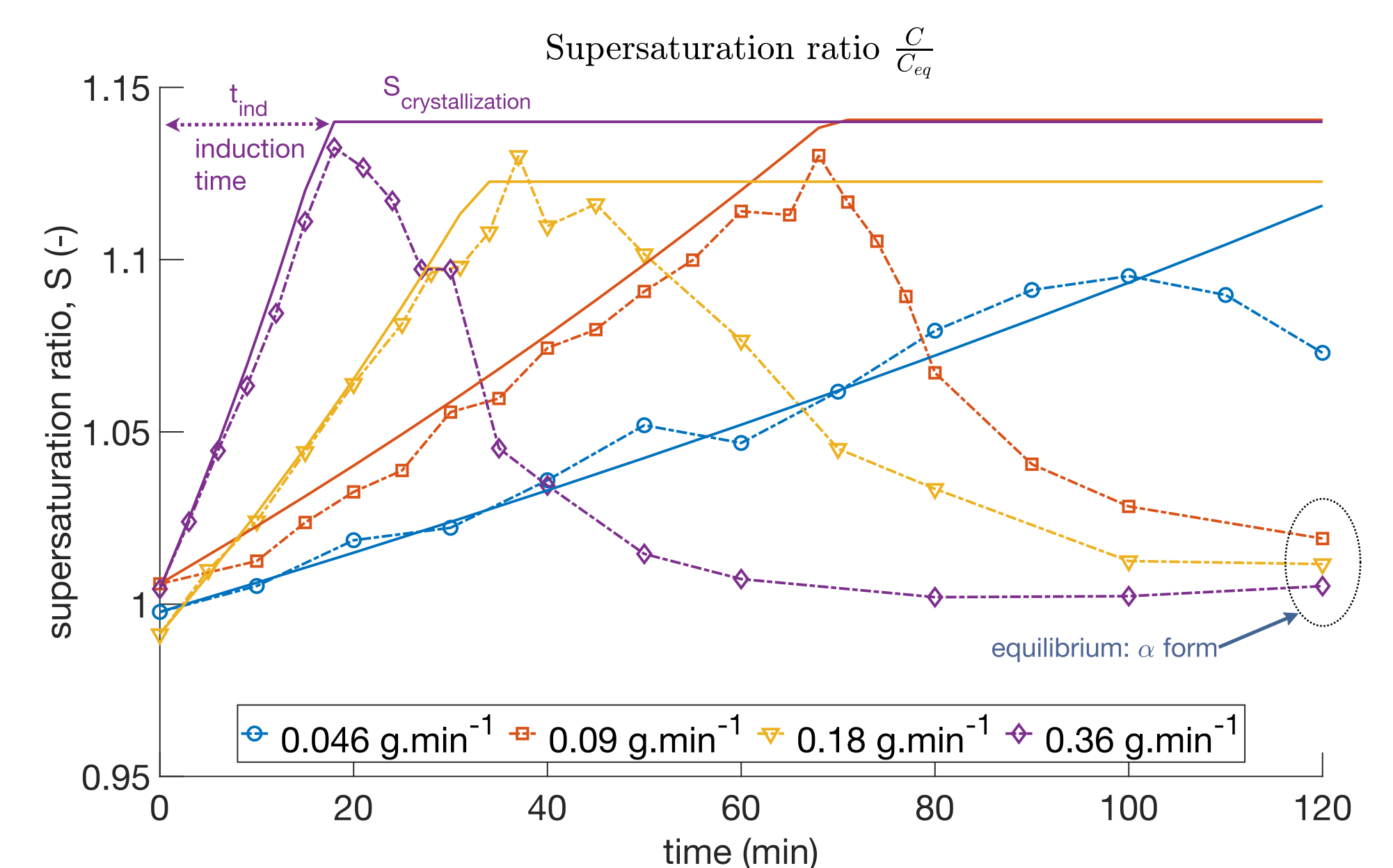
► Analyzes

- Refractive index
- MEB
- Laser granulometry
- Dissipated power measured in water

Previous work without US

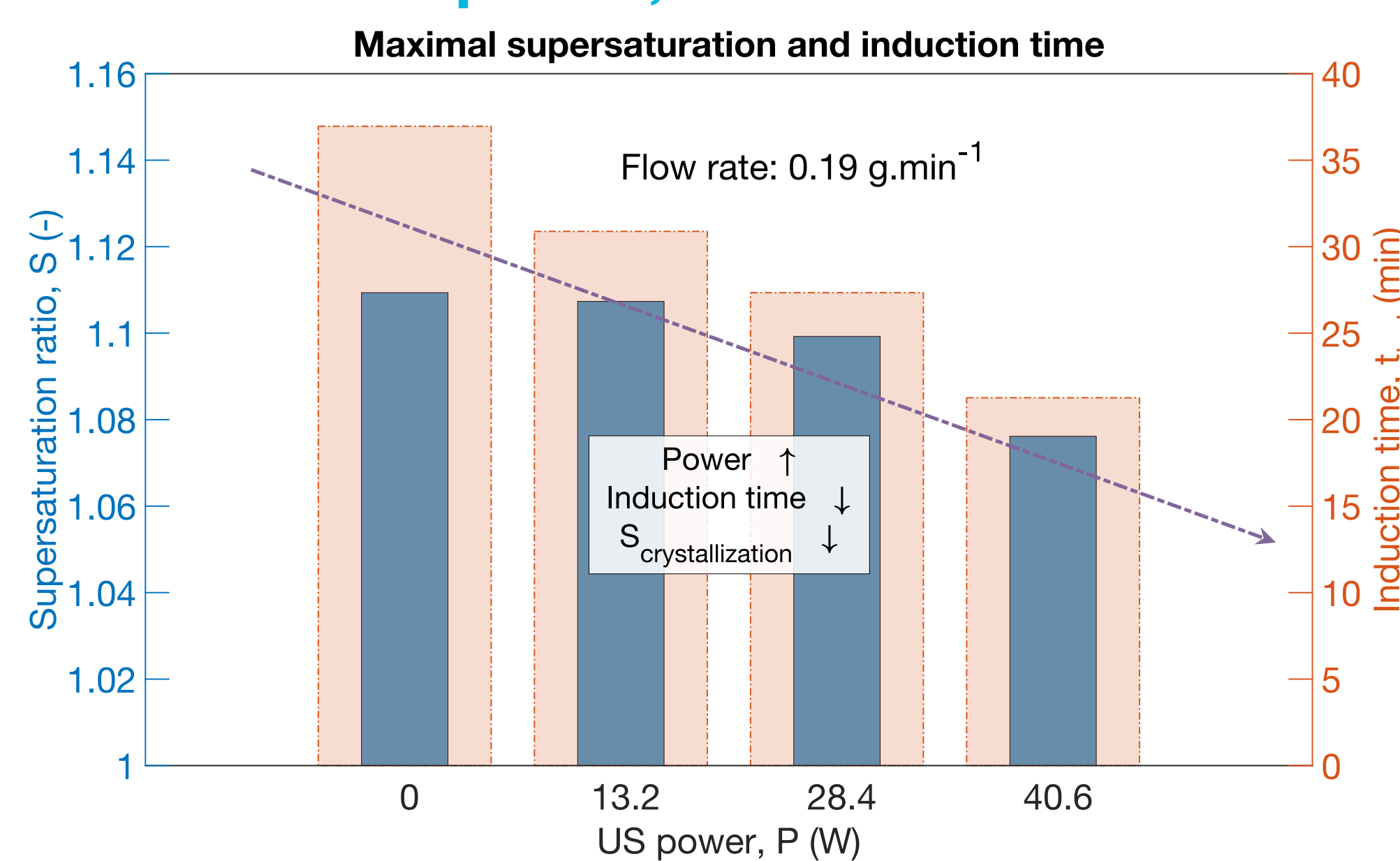
► Experiments without ultrasound (reference)

- 4 flow rates ($\text{g}\cdot\text{min}^{-1}$) of ethanol
- Supersaturation ratio $S = \frac{C}{C_{eq}(T,R)}$

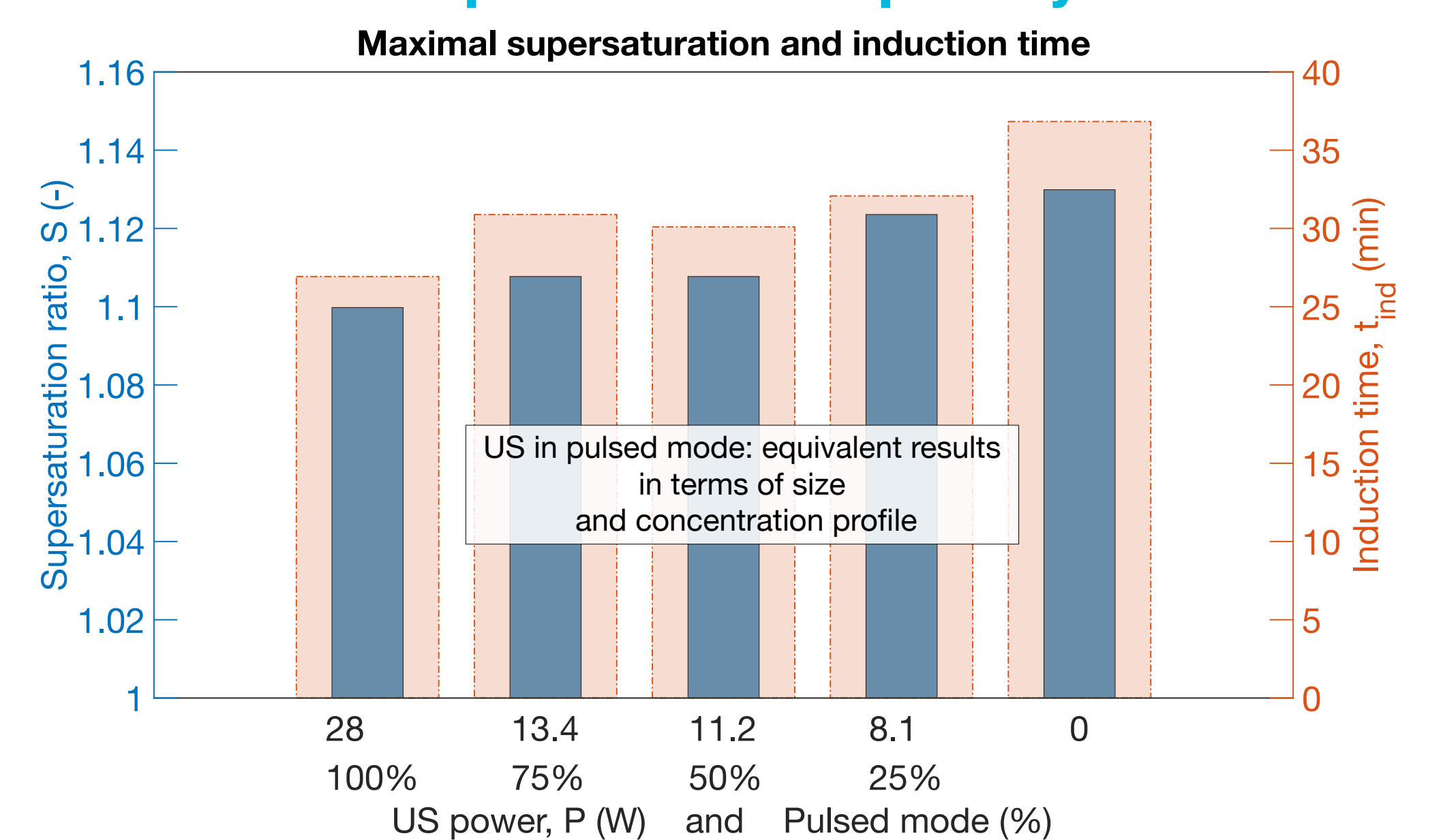


Results: effects of ultrasound

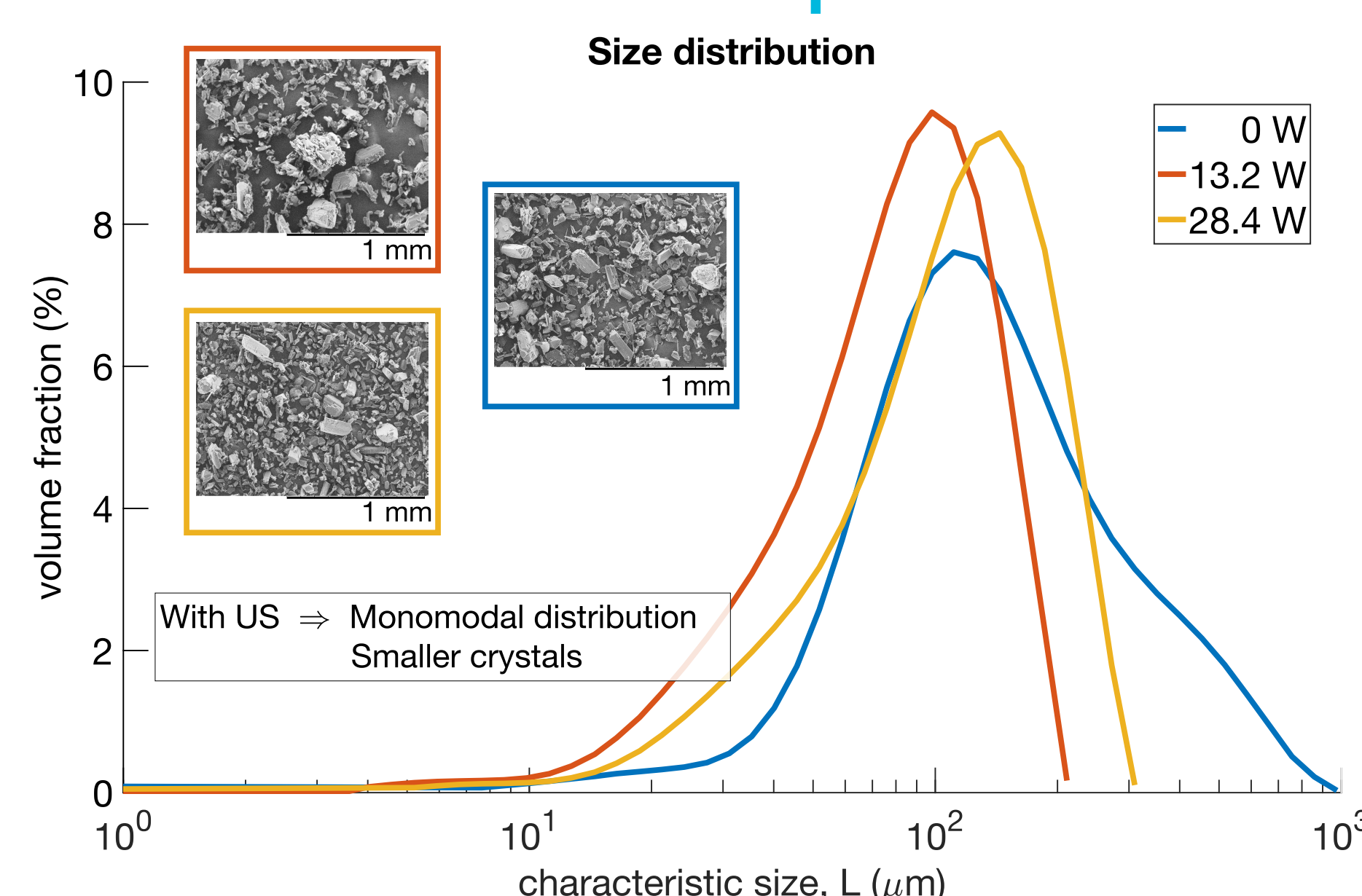
► Influence of US power, for constant flow rate



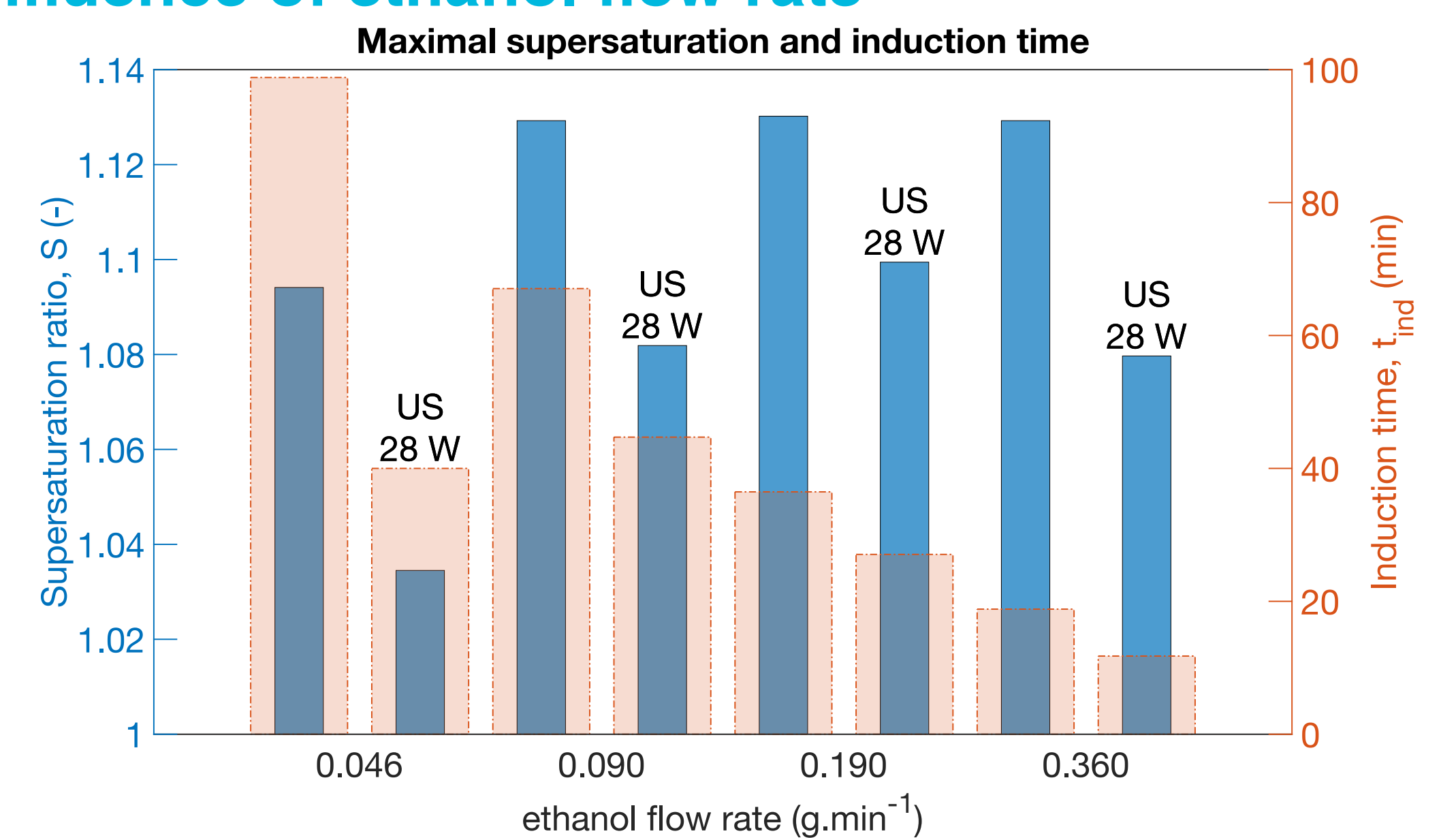
► Influence of the pulsation frequency



► Influence of the ultrasonic power



► Influence of ethanol flow rate



Conclusions and perspectives

► Trends with US vs. without US:

- Smaller crystals, monomodal size distributions
- Decrease of induction time
- The α -form is obtained with and without ultrasound
- Equilibrium concentration more quickly reached ($S \rightarrow 1$)

► Work in pulsed mode (> 25%):

identical results in continuous mode

► Transpose in continuous mode:

SONOCRISTAL® (prototype developed in collaboration with SYNETUDE SAS)



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