



HAL
open science

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

Hector Uriel Rodriguez Vera, Fabien Baillon, Philippe Accart, Fabienne Espitalier, Olivier Louisnard

► **To cite this version:**

Hector Uriel Rodriguez Vera, Fabien Baillon, Philippe Accart, Fabienne Espitalier, Olivier Louisnard. Crystallization of α -glycine by anti-solvent assisted by ultrasound. 16th Meeting of European Society of Sonochemistry (ESS16), Apr 2018, Besançon, France. hal-01773871

HAL Id: hal-01773871

<https://hal.science/hal-01773871>

Submitted on 23 Apr 2018

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Introduction

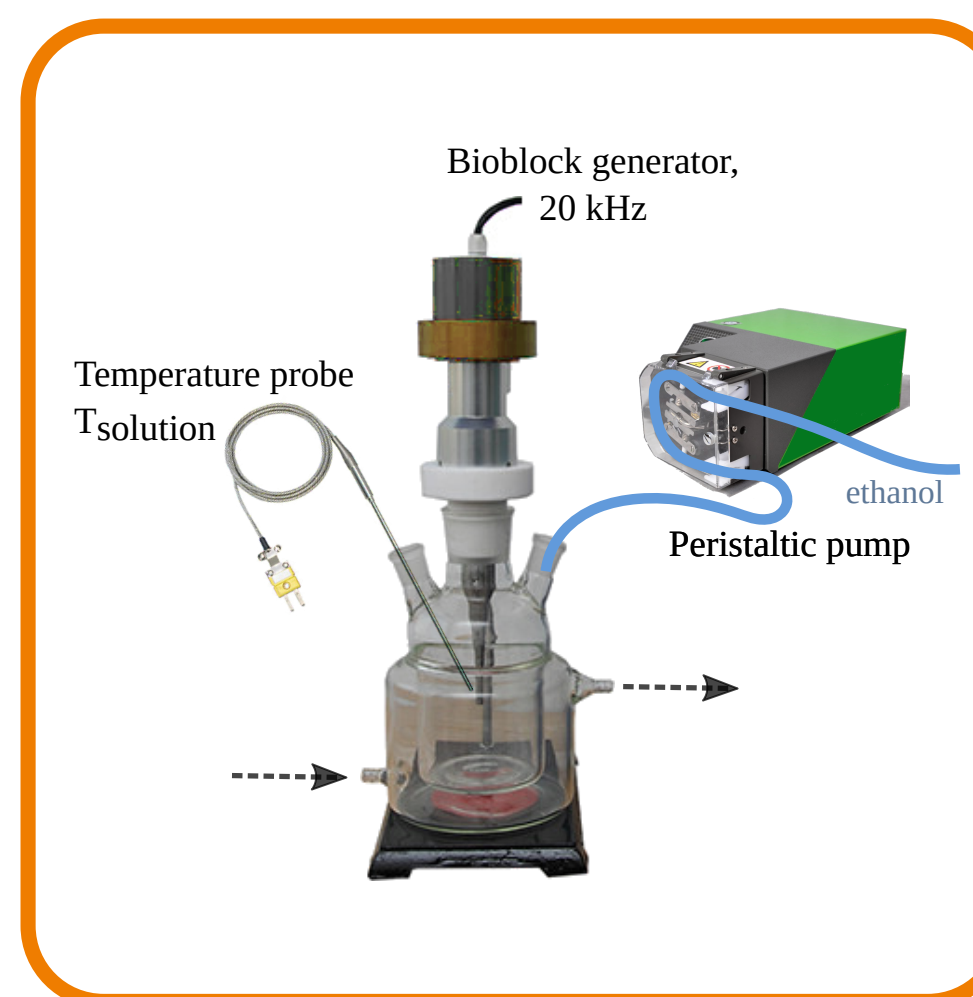
► **Ultrasound and Crystallization by cooling:**
Reduction of the induction time and a reduction of the size of the formed crystals with equivalent supersaturation.

► **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



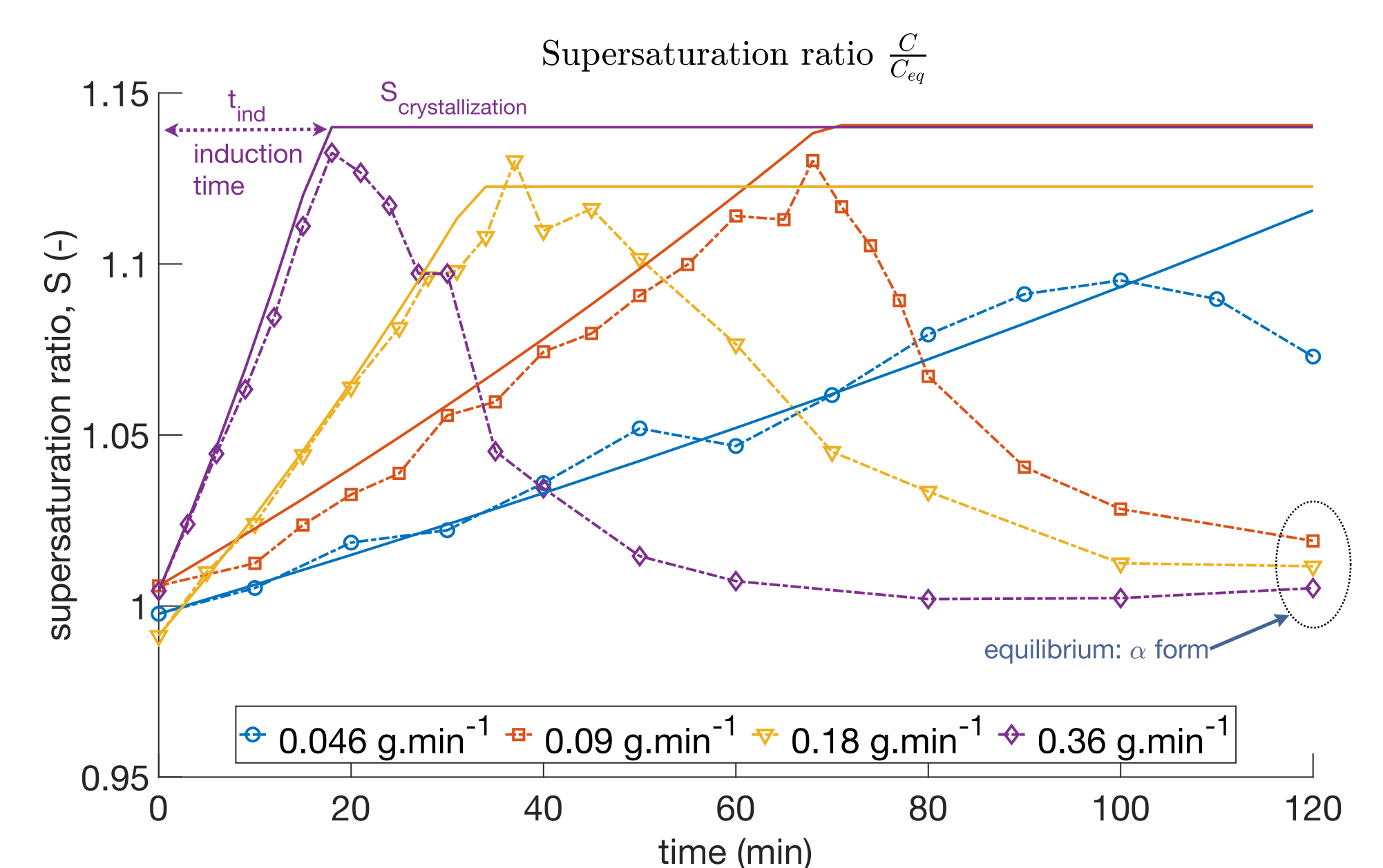
► Analyzes

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

Previous work without US

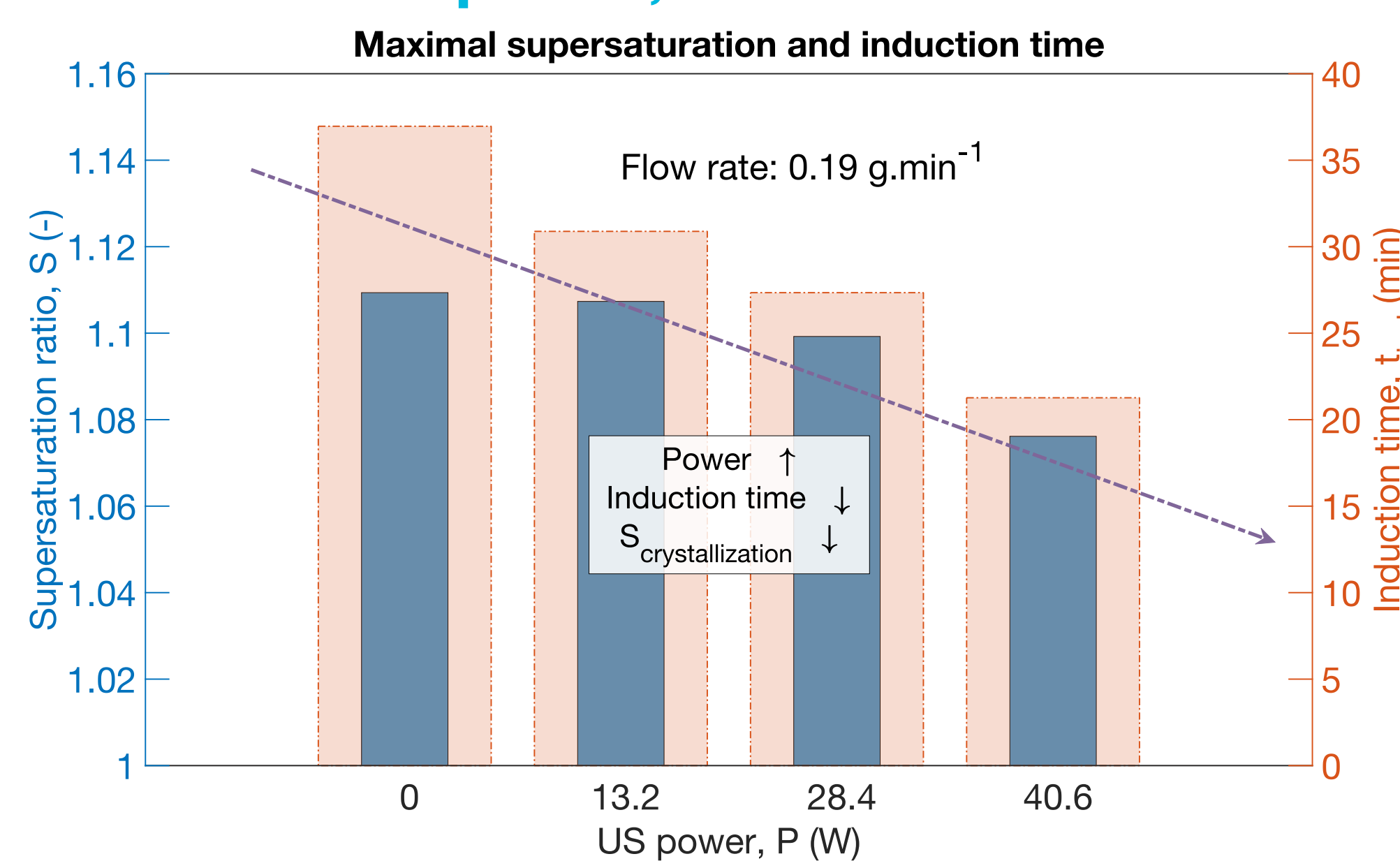
► Experiments without ultrasound (reference)

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

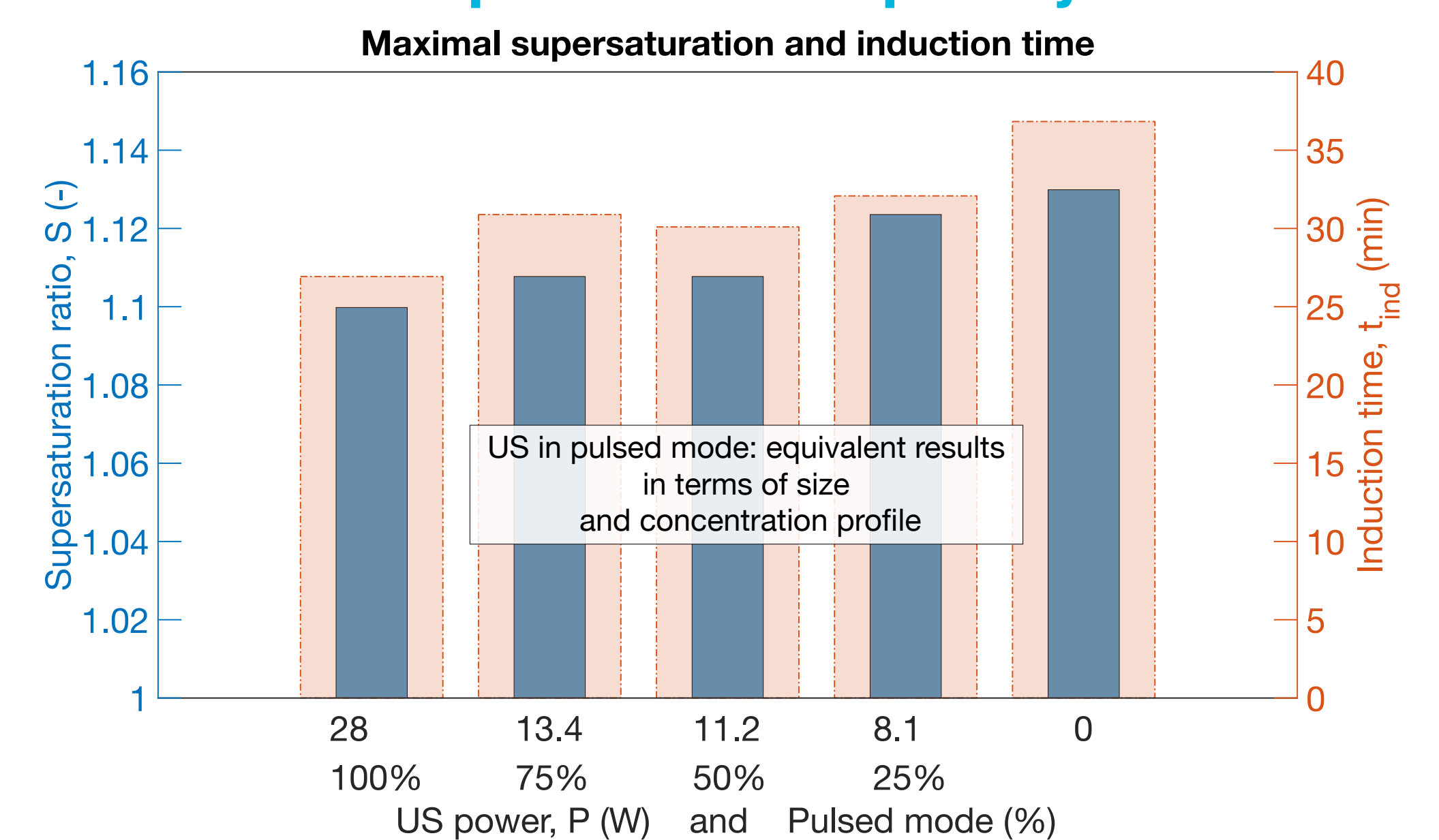


Results: effects of ultrasound

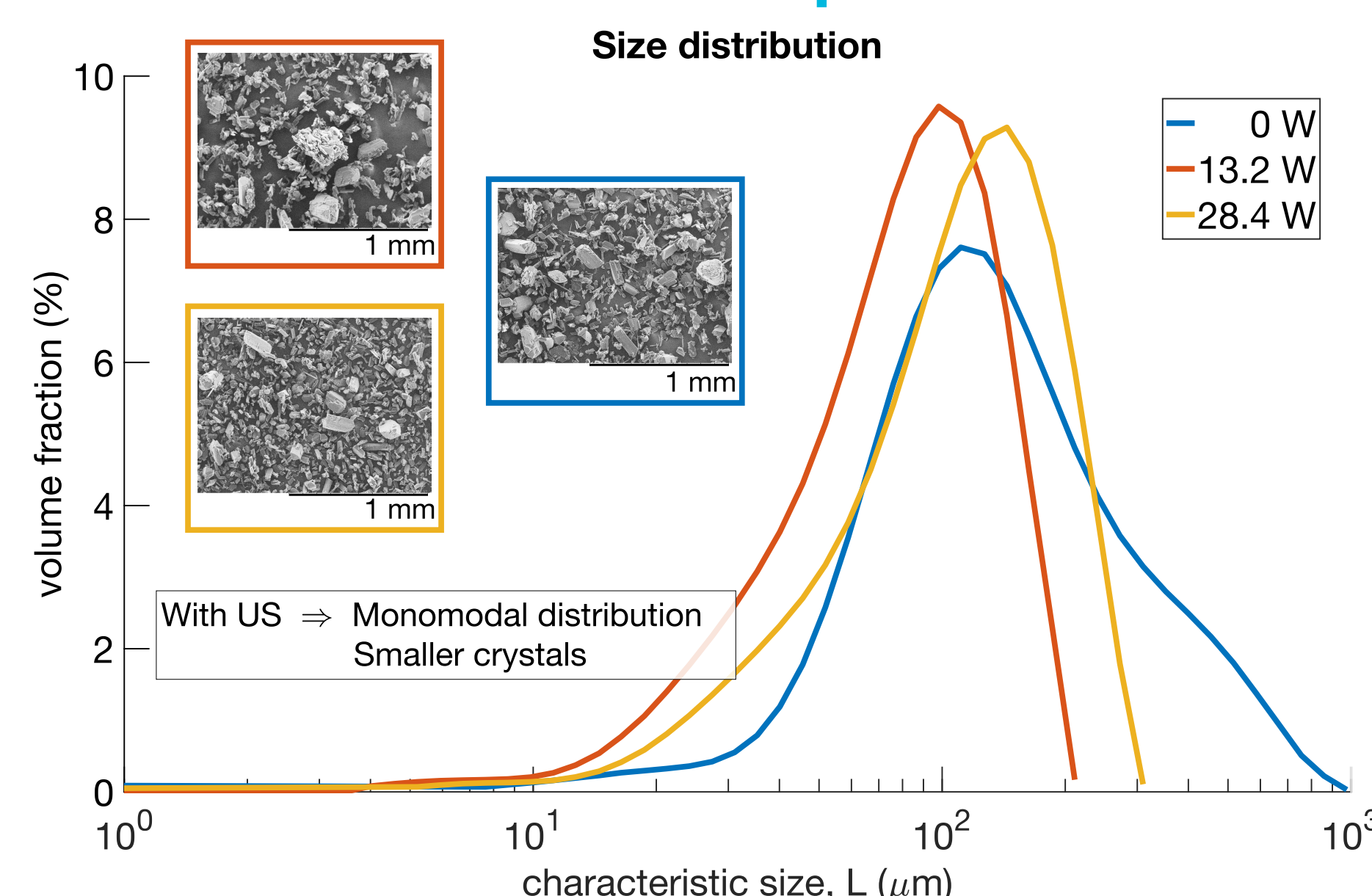
► Influence of US power, for constant flow rate



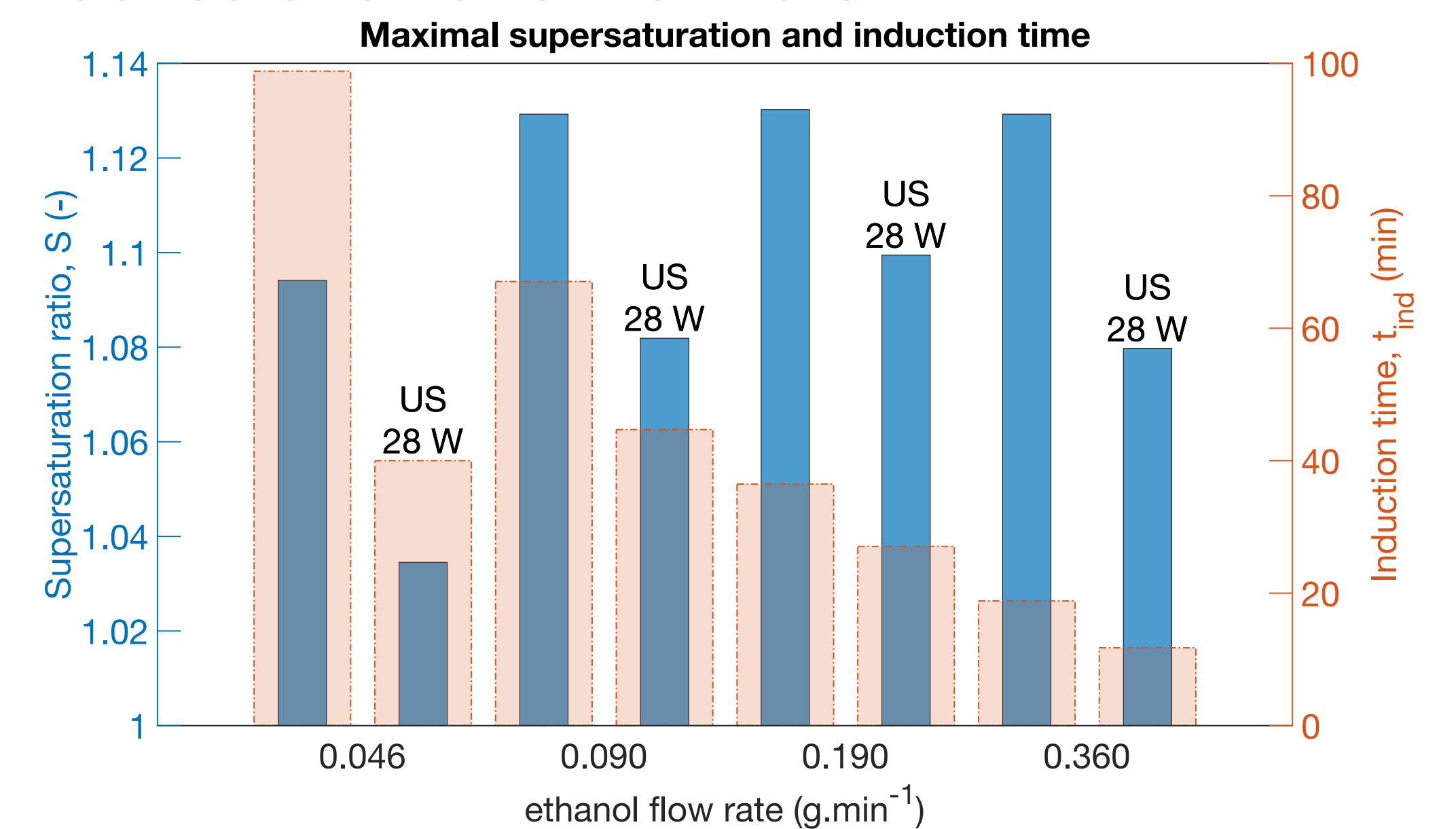
► Influence of the pulsation frequency



► 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)



Université de
Toulouse,
IMT Mines Albi,
Centre
RAPSODEE,
UMR CNRS 5302
Campus Jarlard,
81013 Albi France

References

Yang, X., Ching, C. B., Wang, X. J. & Lu, J., 2006, AIChE annual meeting, San Francisco, CA.

El Bazi, W., 2011, thèse du Conservatoire national des Arts et Métiers.

Louhikultanen, M., Karjalainen, M., Rantanen, J., Huhtanen, M. & Kallas, 2006, J. International Journal of Pharmaceutics, 320, 23–29.

Baillon, F., Espitalier F., Cogné C., Peczkalski R., Louisnard O., 2014. Power ultrasonics: Applications of high intensity ultrasound, 845-874, Eds: Juan A. Gallego-Juárez and Karl F. Graff. Woodhead Publishing.

Contacts

Fabienne.Espitalier@mines-albi.fr

Fabien.Baillon@mines-albi.fr