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Study of organic molecule crystallization assisted by ultrasound preliminary study with SONOCRISTAL apparatus

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Introduction

Crystallization operations play a key role in the process steps of solid transformation. They concern different industrial sectors such as pharmacy, fine chemistry, and food and minerals industries. They allow the production of high value-added products but also to recover waste and to contribute to the remediation of some media.

The use of ultrasound, during a crystallization operation, appears to be promising because it allows nucleation control, as well as the design of the crystalline size, facies and possibly even structure of crystals. These effects have been observed in aqueous phase with different organic and mineral solids [1].

The technological challenges are to define how ultrasound needs to be applied at the industrial scale, to develop pulsed ultrasound for energy optimization and to conceive experimental micro-probes in order to speed knowledge transfer to industry.

This work proposes a new ultrasound device for application in continuous mode.

Materials and Methods

An organic component (solA) purchased by a French dairy industrial is used for the experiments. The experimental set-up consists of two 10 L stirred vessels for the dissolution of solA, one 20 L stirred vessel for the cooling of the solution and one tubular vessel of 1.3 L for the crystallization. All vessels are controlled in temperature thanks to a jacket. The tubular vessel, named, SONONOCRISTAL is equipped with a cylindrical sonotrode in titanium connected to a generator (maximal electrical power of 800 W and frequency 20.6 kHz). This device has been designed and conceived together with SYNETUDE SAS (Grenoble, France) and RAPSODEE (Albi, France). The transparent material used for the tubular vessel (polycarbonate) permits to visualise the flow and the formation of the solid.

In order to follow the crystallisation, the size distributions of solA solid (MALVERN, Master Sizer 3000) and the absorbance of the suspension at 560 nm (HORIBA, CAPA 700) are measured at the exit of the tubular vessel. Experiments have been conducted with an entrance mass fraction of 0.381 ± 0.004.

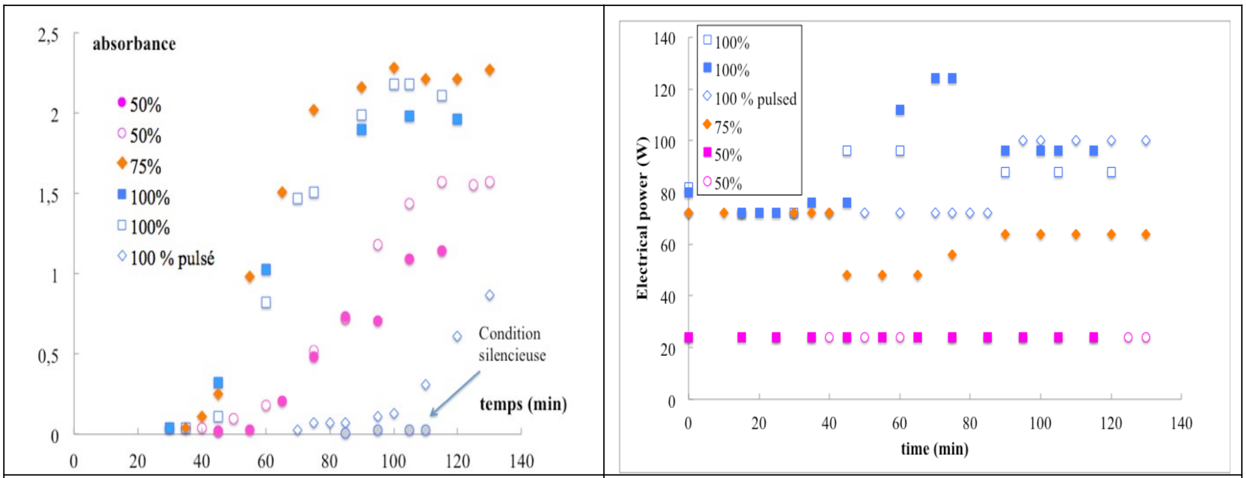
Results and conclusion

Experiments have been made with four vibrations amplitude (0, 50, 75 and 100%).

All experiments are repeatable in terms of diameters and solid formation.

In Figure 1(a), the absorbance at the exit of the tubular vessel is presented for different vibration amplitudes. An increase of the absorbance corresponds to a solid appearance. More the amplitude increase, sooner the crystallization begins. The quantile diameters (d10/d50/d90) measured at the steady state are very close for amplitude ranged between 50 and 75 % and 100 % pulsed (Table 1). These particles diameters seem to be larger for a vibration amplitude of 100 %.

The Figure 1(b) shows that the consumed electrical power is function of presence of solid for amplitude higher than 50 %. A threshold concentration seems to exist above which the generator must increase its electrical power to maintain a vibration amplitude constant.



(a) (b)

Figure 1. Absorbance and electrical power as function of time at the exit of the tubular vessel with a flowrate of 115 ml/min and 140 ml/min for a vibration amplitude of 100%

Vibration amplitude (%)	100	100	75	50	50	0	0	100 % pulsed 5s on/5s off
d10/d50/d90 (µm)	4/28/50	6/24/39	1/7/17	1/5/22	1/6/14	no crystal	no crystal	1/4/12

Table 1. Quantile diameters of crystals at the exit of the SONOCRISTAL at the steady state.

The experiment conducted with a vibration amplitude of 75% leads to attractive results: a significant decrease of the concentration, in dissolved solA (about 3 %) and crystals with a size lower than 20 µm. However, longer experimental time will need to be tested in order to confirm these results.

Thereafter, different frequencies of pulsation will be studied and as well as the material used for the tubular vessel.

References

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