

## Study for ultrasonic stimulation effect on promotion of crystallization of triacylglycerols

Satoru UENO\*, Jun-ichi SAKATA, Morio TAKEUCHI and Kiyotaka SATO  
Hiroshima Univ., Higashi-Hiroshima 739-8528, Japan

### Introduction

There has been an increasing interest in application of ultrasonic waves to crystallisation from different phases. In particular, this applies to the nucleation and crystallisation of biological soft materials, which are often synthesised in a crystalline state. The crystallisation of these materials<sup>1</sup> is one of the areas in which application of ultrasonic irradiation (sono-crystallization) may play a key role, as an external factor, by controlling the crystal structure (polymorphism), shape, and rate of crystallisation. The close links between these physical properties and the functional properties such as melting and solidification, dissolution, aggregation and dispersion, crystal network formation, suspension of foreign substances, and stability of crystal containing systems, increase the importance of this study.

In the previous study [1, 2], we reported the following results of the effect of the ultrasound stimulation for the crystallization of tripalmitin(PPP) and trilaurin(LLL), the typical triacylglycerols (fats); (i) a marked decrease of induction times for both (PPP) and (LLL), (ii) an increased nucleation rate, and (iii) the crystallization of the most stable polymorph,  $\beta$  form, for both triacylglycerols were remarkably promoted. In this study, we will show the results of the promotion effect of ultrasound for the other kinds of triacylglycerols.

### Experimental

Tricaprin (CCC), trimyrustin (MMM), palm-mid-fraction (PMF) and cocoa butter (CB) were used as samples. CCC and MMM were purchased from Sigma Chem. Co. (St. Louis, MO, USA). Both of them had a purity of more than 99 % and were used without further purification. PMF and CB were supplied by Fuji Oil Co. (Tokyo, Japan).

Experiments were carried out using in-situ synchrotron radiation time-resolved small- and wide-angle X-ray scattering (SAXS/WAXS) simultaneous measurement combined with a sono-crystallization system at BL-9C and 15A. All of the set-up of this experiment were the same as the previous study [1, 2].

### Results and Discussion

Fig.1 shows the isothermal crystallization of CCC at 11.5 °C with ultrasound for 2 sec. In this case, the most stable form,  $\beta$  form, directly crystallized at 80 sec after the temperature reached at 11.5 °C cooling from melting state at 55 °C. In the case of the same sample and same experimental condition without ultrasound, the crystallization of the  $\beta$  form appeared at 1000 sec. This

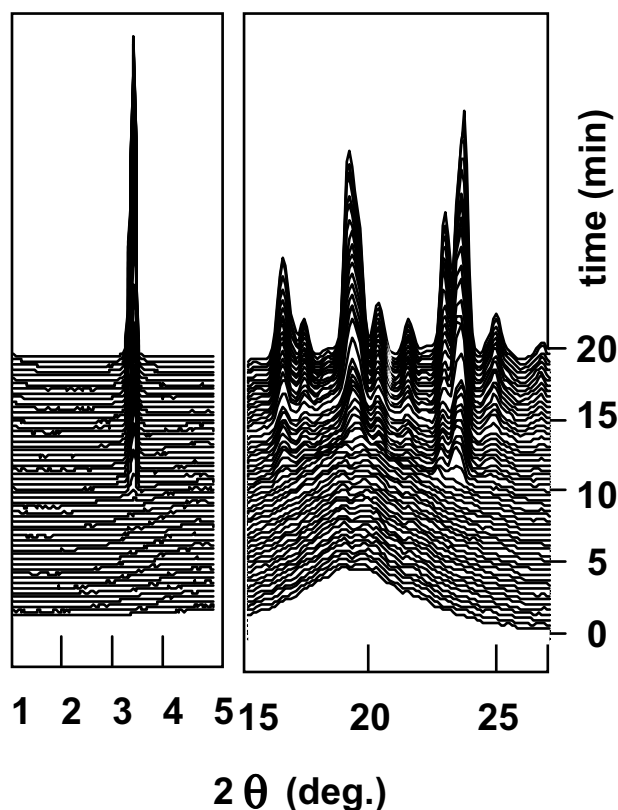


Fig.1 The time-resolved X-ray diffraction data for crystallization of (CCC) with ultrasound at 11.5 °C. 10 sec measurement were repeated. The diffraction peak shows the crystallization of the most stable form,  $\beta$  form.

shows the same result observed in the previous study; the promotion of the crystallization, especially the most stable form  $\beta$ . The same kinds of results were observed all temperature conditions of all samples, except for PMF, supporting the conclusion of the previous study.

The promotion effect of ultrasound mentioned above is able to be explained by the theory of the cavitation effect for crystallization from liquid [2,3]. The remarkable effect for the  $\beta$  form crystallization and the result of PMF is still open to question.

### References

- [1] S.Ueno et al., PF Activity Report 2001,19, B95 (2002).
- [2] S. Ueno et al., J. Phys. Chem. B 107, in press (2003).
- [3] C. Lee et al., J. Appl. Phys. 71, 5721 (1992)

\*sueno@hiroshima-u.ac.jp