P022 Developing a method to determine the flux of material arriving and leaving a crystal at equilibrium

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It is often said that a crystal in a saturated solution neither grows nor dissolves, however, this is an equilibrium so there must be some flux between the crystal and the solution. The question is how could this be measured. The aim of this work was to develop a method to make this measurement, as a prelude to investigating the effect of ultrasound on the rate of molecular exchange between the surface layer of a growing crystal and the surrounding slightly supersaturated solution. The challenge of labelling the molecules in the crystal as distinct to those in the surrounding solution can be overcome by taking an isotopically labelled material where the atoms in the label are not labile and do not play a critical part in intermolecular hydrogen bonds thus 2H labelled samples were considered unsuitable. Therefore, a ^{15}N $^{13}C_2$ labelled paracetamol was selected as the model compound. The next challenge was to develop an experimental methodology in order to control both the saturation of the surrounding solution and the ultrasonic intensity, which the crystal is exposed. Starting from a paracetamol solution saturated at 20°C, slight temperature decreases in the order of 0.20C, were made to achieve different saturation values. The ultrasonic field was quantified using a needle hydrophone for intensity measurement and by varying the percentage of the power of the ultrasonic bath. In addition, the mass and morphological changes to the crystal before and after ultrasonic intervention were measured (Figure 1). The final component in the process was to establish a method to quantify the amount of labelled material leaving the crystal. The extent and rate of molecular transfer from the crystal into the solution as a function of relative supersaturation and ultrasonic field intensity were measured using complementary techniques NMR and LC-MS. This enables assessment of the range of conditions under which crystal growth is accompanied by loss from the surface by dissolution and estimation of the relative magnitude of the flux in each direction.

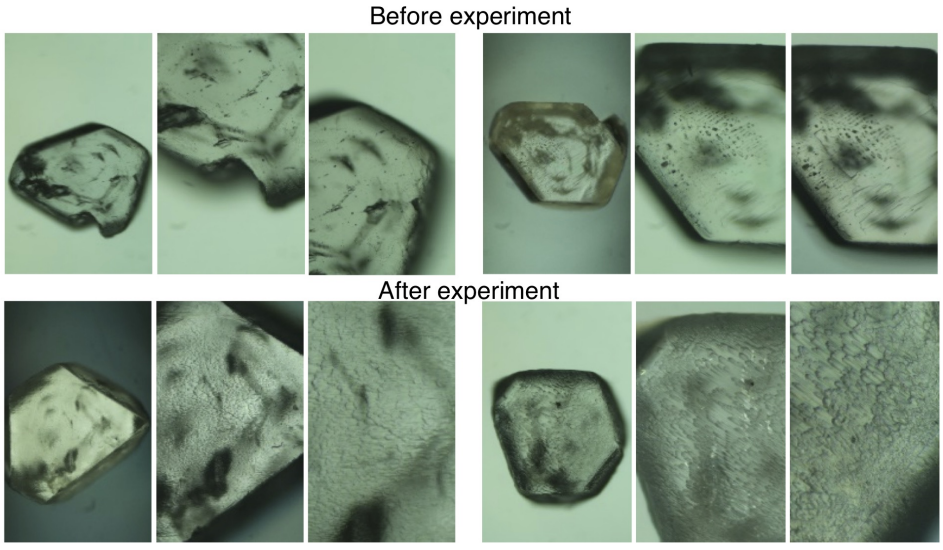

Figure 1: Two crystal faces of an isotopic paracetamol crystal before and after ultrasonic intervention (carried out at 50% power and 20°C) .

References

Thai T. H. Nguyen et al. Crystals 2017, 7, 294.