

MSc graduation assignment

## Polymorphism of *trans*-cinnamic acid

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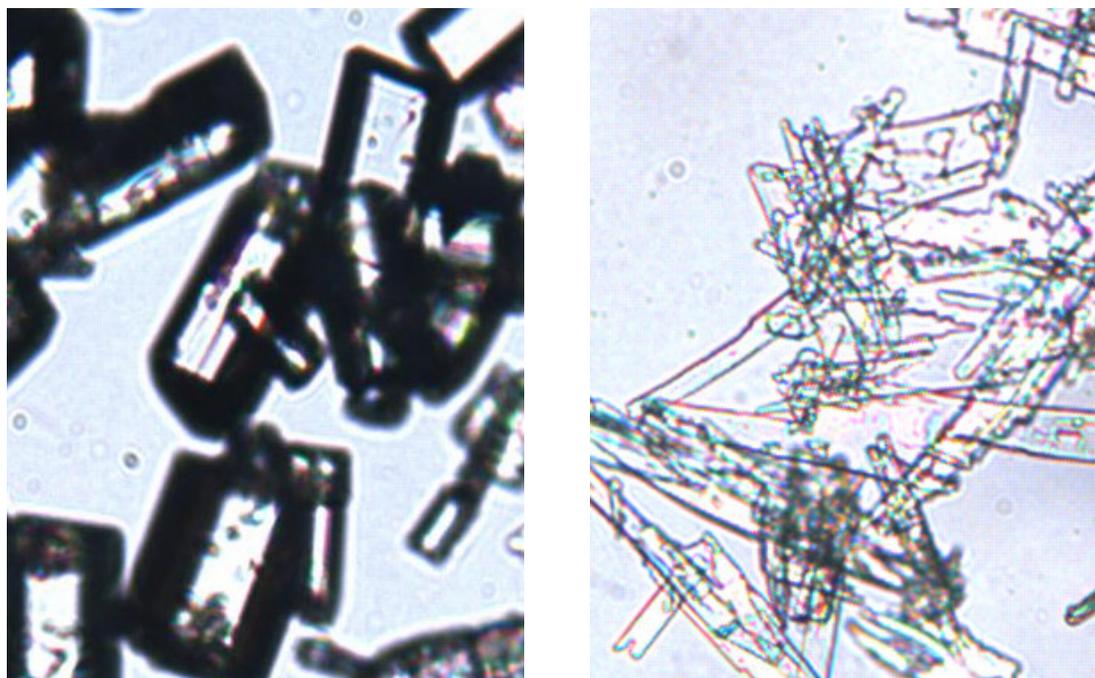
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### **Introduction:**

TNO & TUD are developing *In-Situ* Product Recovery (ISPR) techniques to improve the yield and productivity of fermentation processes. ISPR can in principle be achieved using Template Induced Crystallization (TIC). With TIC, templates are added to the crystallization solution as a specific surface upon which the solute may build its crystal lattice. *Trans*-cinnamic acid is used as the model compound to study TIC, because it can already be produced by *Pseudomonas putida* S12 at TNO.

Polymorphism is the phenomenon that a certain compound has more than one specific crystal structure. Different polymorphs have different chemical and physical properties, such as solubility and crystal morphology. These differences may affect the way these crystals nucleate and grow. Therefore polymorphism is one of the critical parameters that influence template induced crystallization.

Studies concerning polymorphism of crystals of *trans*-cinnamic acid are thus far limited to the most stable  $\alpha$ -polymorph<sup>1-3</sup>. The less stable  $\beta$ -polymorph however is formed in precipitation reactions. The percentage of the  $\alpha$ -polymorph (depicted in fig 1a) will gradually increase upon slow conversion in the presence of a liquid phase due to a solvent mediated transformation of the metastable  $\beta$ -polymorph (depicted in fig 1b) to the stable  $\alpha$ -polymorph.



**Figure 1: Crystals of cinnamic acid. (a)  $\alpha$ -polymorph (b)  $\beta$ -polymorph**

In order to study the precipitation of *trans*-cinnamic acid onto foreign surfaces properly, more insight in the morphology and characteristics of the  $\beta$ -polymorph is required. Results of this research will attribute to better knowledge about the crystallization behaviour of *trans*-cinnamic acid, which will in turn lead to greater understanding of the principles of template induced crystallization. Eventually this information will be used to develop a suited integrated process of fermentation and crystallization.

The goal of the research is the characterization and controlled production of the polymorphs of *trans*-cinnamic acid. For this purpose growth of single crystals of the different polymorphs must be realized. The polymorphs can be differentiated using spectrophotometer techniques. The solubility of the polymorphs has to be determined for several solvent(s) (mixtures). Once the different crystals are formed, the morphology and structure have to be characterized using microscopy and XRD techniques. Results from the XRD can later on be used as input for the molecular modelling of the crystal morphology (which can be checked with SEM pictures).

### **Goal of the research:**

The characterization and controlled production of the polymorphs of *trans*-cinnamic acid.

### **Tasks:**

- Crystallization of single crystals of different ( $\alpha$ ,  $\beta$  &  $\gamma$ ) polymorphs
- Determination of solubility of polymorphs in several solvent(s) (mixtures).
- Determination of type of polymorph (using e.g. Raman / IR spectrometer / XRPD)
- Characterization of crystal morphology (using Microscope / SEM / DSC)
- Determination of crystal structure (XRD)
- Molecular modelling of crystal morphology

### **Literature**

- (1) Atkinson S.D.M., Almond M.J., Hollins P., Jenkins S.L. *Spectrochimica Acta Part A* **2003** 59 629-635.
- (2) Schmidt G.M.J. *Journal of the chemical society* **1964** 2014-2021
- (3) Abdelmoty, I. et al. *Crystal growth & design* **2005** 5 2210-2217