

Developing a crystallisation for a disaccharide



The challenge

CatSci was tasked with developing a crystallization for a disaccharide that was produced from an enzymatic reaction. The solid state chemistry was unknown in the literature and by analogy with another well known disaccharide, lactose, was likely to be complex.



How?

A solubility screen in mixed organic aqueous solvents identified solvent options that could give the right yield, volume efficiency, EHS characteristics and particle physical properties. The solubility data was used to design small scale crystallisations from aq. IPA and aq. Ethanol using purified material as input. In parallel, as samples were generated, these were analysed using PLM, DSC, XRD and VT-XRD to better understand the solid state chemistry. A single option, aq. Ethanol, was scaled to 1L using inputs that mimicked the anticipated impurity profile of the feedstock and finally, a trial was performed using real feedstock.



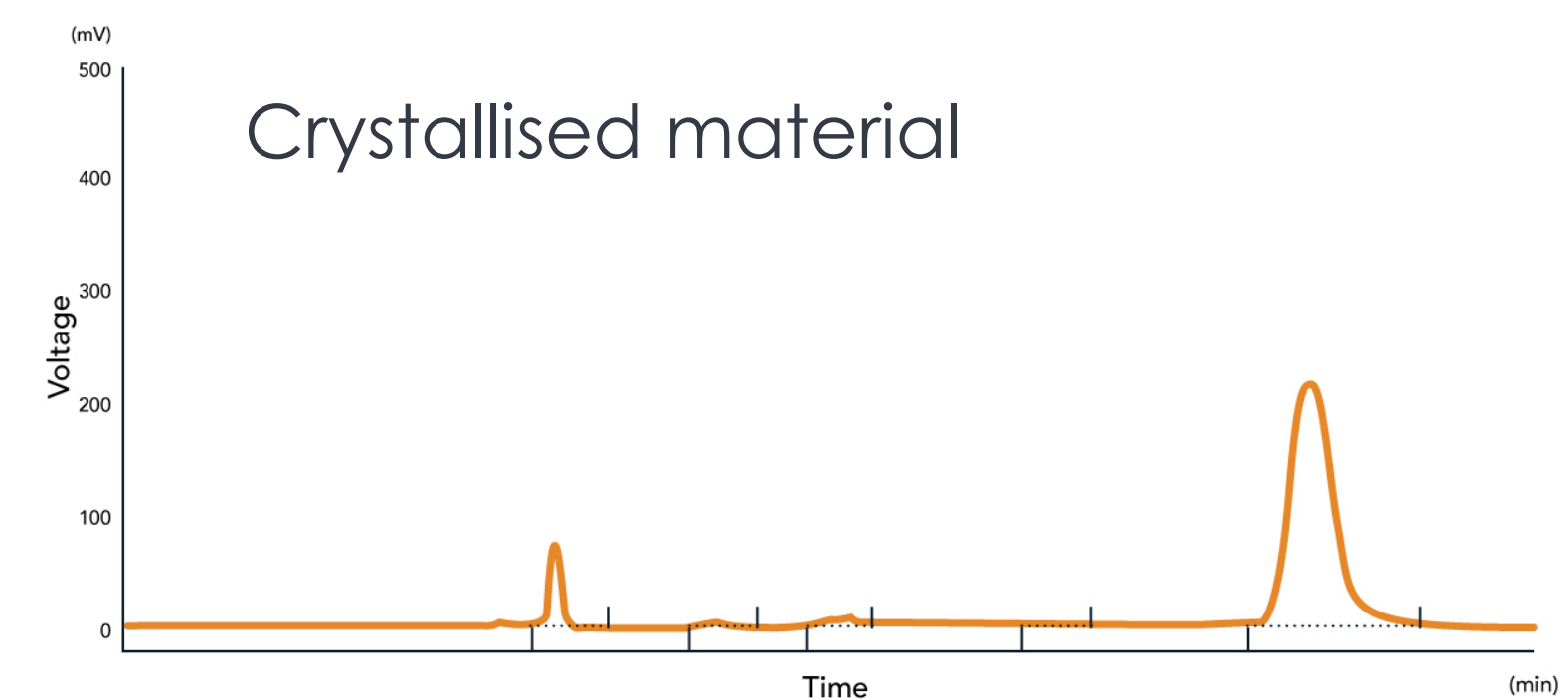
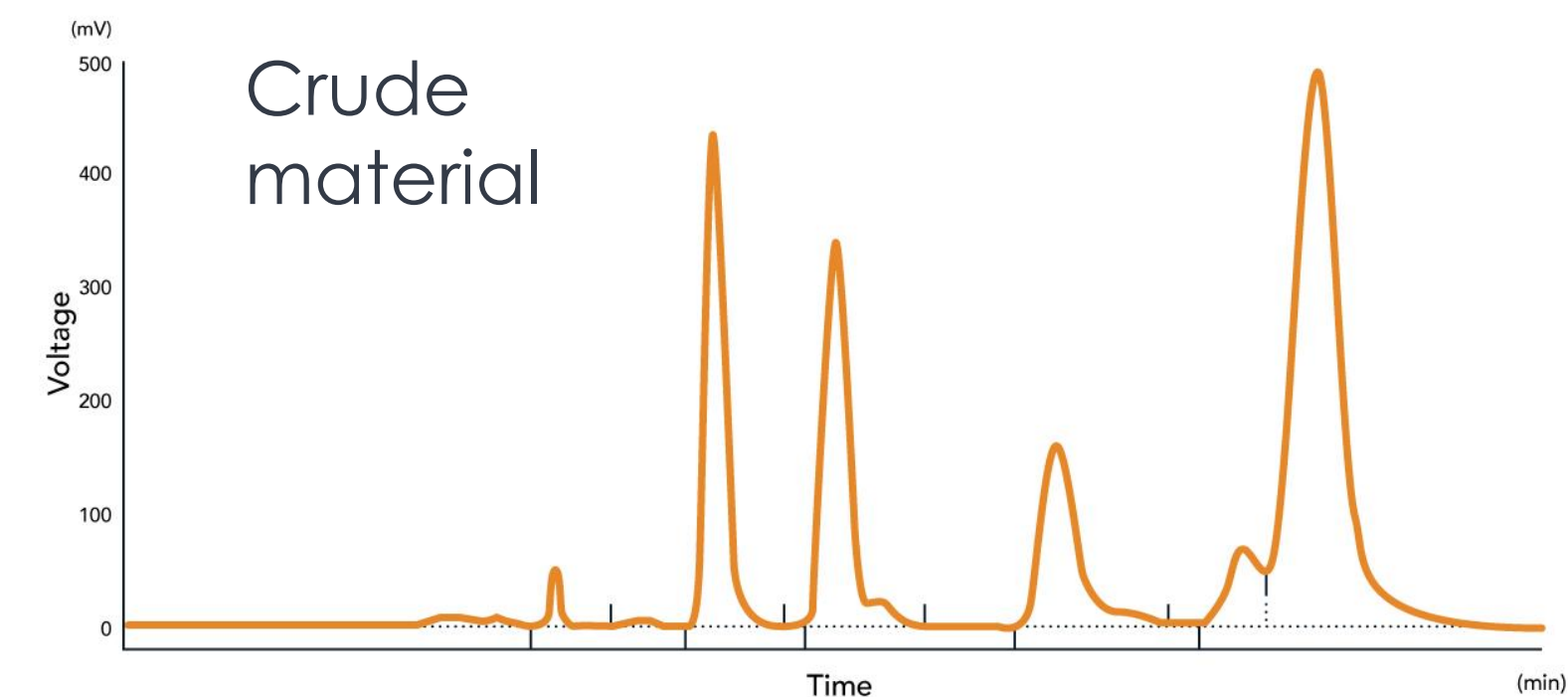
The achievement

The CatSci team successfully defined a crystallisation process that met the manufacturing, chemical purity and solid state targets:

- Went from 47.7% area purity in crude to 99.6% area purity.
- Obtained the desired disaccharide in 74% yield starting from a highly impure process feed.
- Significantly increased solid state understanding identifying that the product was actually a monohydrate and existed as anomers in solution.



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Polarised Light Microscopy
of crystallised material

