An investigation into parameters affecting purity of crystals in OBC and STC

CMAC PhD student: Hannah McLachlan

Supervisor: Prof Xiong-Wei Ni, School of Engineering & Physical Science,

Heriot-Watt University, Edinburgh

Background

• As cooling rate in stirred tank crystallisers (STC) increases, the purity of crystals produced decreases [1].
• Crystals produced by cooling crystallization in Oscillatory Baffled Crystallizer (OBC) had higher purities than that in traditional STC under the same operational conditions, as well as at higher cooling rates than that at STC [2].

Objectives

Seek scientific explanations as to why higher purity of crystals were obtained in the OBC

Hypotheses

a) the mixing mechanisms present within the two systems may have contributed to this, as better heat transfer characteristics in OBC due to more uniform mixing may have led to better control of cooling rates;
b) the cooling rates achieved at the two systems may be different due to the different ways of generating mixing;
c) the combination of mixing and cooling rate may be responsible.

Plan

i. Carry out a selection of benchmark experiments in both the OBC and the STC to confirm the purity of crystals produced at fixed mixing, concentration and cooling rates in cooling crystallisation of pure urea (>99.5%) [3] in water;
ii. Analyse the collected samples using UV/Vis to calculate the purity of samples produced using the Lambert-Beer Law.
iii. Vary mixing intensity, cooling rate independently and collectively in the two systems;
iv. Add a known amount of biuret as impurity [4] into the solution to examine the rejection rate of impurities in the two systems.

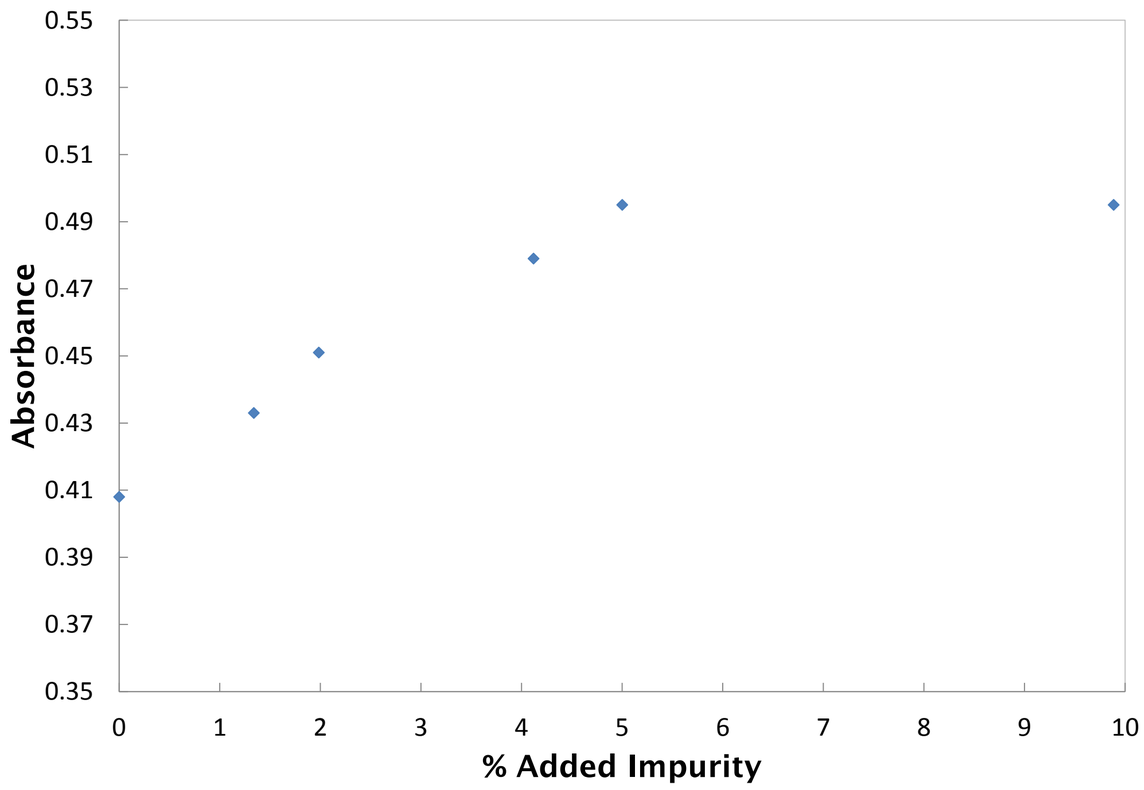

Figure 1 – Calibration Curve for Urea with Impurity

Analysis

• To use UV/Vis spectrophotometry to analyse the compound urea and impurity biuret, the samples must be derivitised with p-Dimethylaminobenzaldehyde to form a chromophore.
• Following derivitization the purity of the sample can be calculated using a calibration curve of known sample concentration. An example of the calibration curve is shown in Figure 1

Note - These experiments are to be carried out in a STC, a moving baffle OBC and a moving fluid OBC.

- The crystal habit of pure urea and urea with the added impurity is shown in Figure 2.

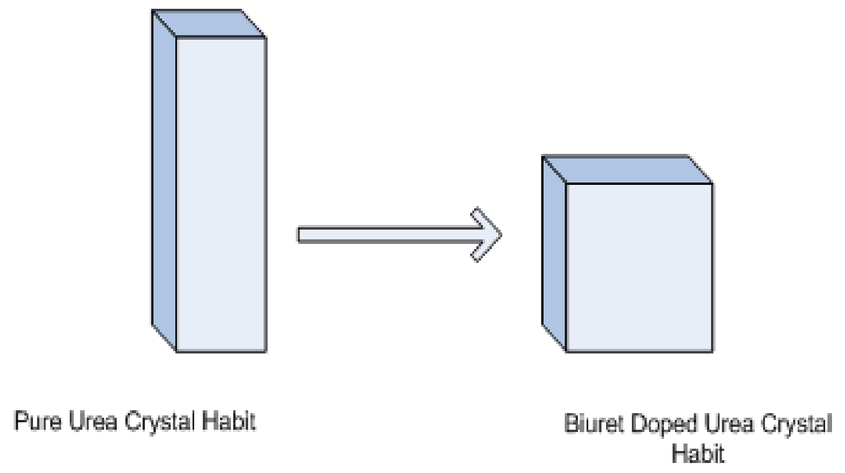

Figure 2 – Example of Crystal Habit of Pure Urea and Doped Urea

[1] Matthews, H. B. and J. B. Rawlings (1998). AICHE JOURNAL **44(5): 1119-1127.**
[2] Ni, X. and A. Liao (2008). Crystal Growth & Design **8(8): 2875 - 2881.**
[3] Davey, R., W. Fila, et al. (1986). Journal of Crystal Growth **79(1-3, Part 2): 607-613.**
[4] Scott, C. and S. Black (2005). ORGANIC PROCESS RESEARCH AND DEVELOPMENT **9(6): 890-893.**
[5] Saito, N., M. Yokota, et al. (2001). "Chemical Engineering Journal **84(3): 573-575.**

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