International Dairy Journal 72 (2017) 14–22

Contents lists available at ScienceDirect

International Dairy Journal

journal homepage: www.elsevier.com/locate/idairyj

Laminar flow continuous settling crystalliser. Part 2. Modifications

Konrad R. Shaffer [a], Anthony H.J. Paterson [b,*], Clive E. Davies [b], Gerald Hebbink [c]

[a] Fonterra Cooperative Group Ltd, London Street, Auckland, New Zealand
[b] School of Engineering and Advanced Technology, Massey University, Private Bag, 11-222, Palmerston North, New Zealand
[c] DFE Pharma, Klever Strasse 187, 47568 Goch, Germany

ARTICLE INFO

Article history:
Received 29 November 2016
Received in revised form
2 March 2017
Accepted 16 March 2017
Available online 7 April 2017

ABSTRACT

This paper (Part 2 of 2) revises and expands on the original model for the laminar flow continuous settling crystalliser presented in Part 1 in an effort to understand why the original model over-predicted product particle d_{10} and d_{50} and under-predicted the span and d_{90}. Because agglomerates were observed experimentally, an agglomeration term was added to the initial theoretical model resulting in no significant particle size distribution prediction changes. On further analysing potential flow conditions a region of high flow and a subsequent area of low flow explained the d_{50} and span deviations found experimentally. It was concluded that the difference between the ideal predicted spans and the experimental spans was due to non-ideal laminar flow conditions existing in the column. Although not achieved experimentally, it is postulated that an ideal parabolic laminar column flow profile would result in a narrow particle size distribution.

1. Introduction

In Shaffer, Paterson, Davies, and Hebbink (2017), we presented a theoretical model and experimental results for a continuous settling crystalliser (CSC). The CSC experimental product results were found to have a significantly larger d_{90} and smaller d_{10} and d_{50} than the CSC theoretical model predictions. The experimental product span was approximately 3 times the theoretical predictions and greater than the objective span of 1. An investigation was carried out into the experimental effects that could be causing the span deviation from the ideal model conditions. The key model discrepancy, revealed from the investigation, found that crystal agglomeration that was occurring inside the column and potential flow deviations from ideal laminar flow could be the causes for the larger span. Initial explanations were based around the ability for agglomeration or increased flow to produce larger crystals, increasing the d_{90}. Likewise, agglomeration or a lower flow region has the possibility to settle out smaller slow growing crystals, decreasing the d_{10}. In this paper a range of agglomeration and flow conditions was theoretically modelled to test this hypothesis and compared with the experimental results.

* Corresponding author. Tel.: +64 6951 7116.
E-mail address: A.paterson@massey.ac.nz (A.H.J. Paterson).

http://dx.doi.org/10.1016/j.idairyj.2017.03.015

2. Continuous settling crystalliser theoretical model revisions

2.1. CSC agglomeration term

An agglomeration term representing the condition where all particle sizes have the ability to agglomerate after meeting a column radius and height interaction criteria was formulated. The column radius interaction criterion, Eq. (1), states that the particles column radius position must reside within 20% of the first crystal's diameter. Under normal model simulations, the individual particle column radius position does not change with time. The height interaction criterion (AGG1) was initially solved at each time step, Eq. (2), assessing if particles were touching based on particle height position and diameter; as depicted in Fig. 1:

$$r_1 - 0.2d_1 < r_2 < r_1 + 0.2d_1 \quad (1)$$

$$z_1 + 0.5d_1 \geq z_2 - 0.5d_2 \quad (2)$$

where r_1 and r_2 (m) are the radial positions of the two crystals in the column, d_1 and d_2 (m) are their diameters and z_1 and z_2 are their heights, (m). Using AGG1 height interaction criterion potentially meant crystals could come in and out of contact during the time step movement and not be counted, likewise crystals that were close together but not colliding may unnecessary agglomerate. The AGG1 height interaction criterion was then altered to

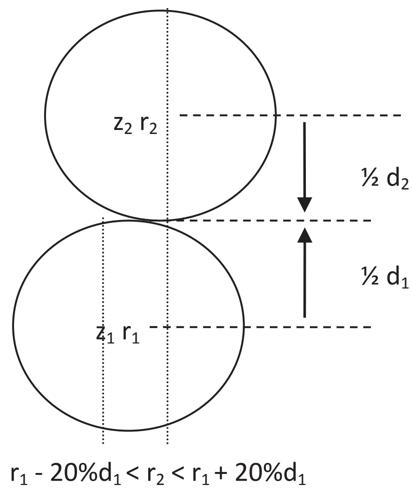

Fig. 1. Agglomeration 1 (AGG1) column height and radius criteria.

assess if contact occurred during the time step particle movement; agglomeration 2 (AGG2) used Eq. (3) instead of Eq. (2). The agglomeration predictions can be seen in Table 1 where Q_f is the feed rate (mL min^{-1}), C_T is the lactose concentration of feed (g lactose 100 g water^{-1}), t is time (s), Δt is the time step (s) and P_N is the number of unique particles entering per Δt (#).

$$\text{Using } z_{1,t1} \text{ and } z_{2,t1} : z_{1,t2} \geq z_{2,t2} \quad (3)$$

The agglomeration model particle size distribution (PSD) predictions did not differ significantly from normal model predictions. AGG1 criteria produced a larger number percent of product agglomerates than the AGG2 criteria. This difference is due to AGG2 strict collide criteria rather than AGG1 proximity criteria. After adding agglomeration conditions to the theoretical CSC model, larger particles can be produced, but agglomerates are removed from the column faster due to their faster settling rates resulting in no significant increase in the d_{90}.

The potential ways in which column particles can interact to form agglomerates can be seen in Fig. 2. The large crystals (high flow region) do not interact with each other to produce the larger crystal sizes measured in practice. Slow growing small crystals in the low flow rate zones can agglomerate and settle out; these would otherwise be lost to the waste stream. Overall, the CSC theoretical model with agglomeration does not show a significant PSD effect compared with the no agglomeration model conditions, but the agglomeration term was retained in the model because agglomeration does occur in reality. Further CSC theoretical model analysis is required to explain the decrease in d_{10} and d_{50}, and increase in d_{90}, giving rise to the increased CSC product span.

2.2. CSC theoretical model factor analysis

Solutions for longer time, decreased time steps and increased individual particle input were carried out to assess any sensitivity issues that may be compromising predictions; these are summarised in Table 2.

CSC theoretical model factor variations did not give any significant changes to the product PSD with all conditions studied. However, it is important to note that having no nuclei input diameter, $d_i = 0$ m, better matched the experimental waste stream predictions and increases the product by waste (PW) crystal volume ratio. The theoretical model input nuclei diameter was based on Malvern MasterSizer volume predictions, converted to a number distribution for the experimental nuclei feed stream at 0 h of growth. The probable reason for the improved prediction by the $d_i = 0$ is because there was a significant amount of nuclei present that were too small to measure as the nuclei had yet to grow to a measurable size. This can be seen in Fig. 3 when the crystal numbers for the feed at 0 h of growth and the 4 and 6 h controls are compared and when the number of crystals in the product are compared with the number in the feed. Growth conditions were such that additional nucleation was limited.

The theoretical model waste crystal PSD increases by removing the input nuclei diameter despite the PW crystal volume ratio increasing because the product by waste crystal *number* ratio decreases from $8.1 \pm 0.6 \times 10^{-3}$ to $7.0 \pm 0.1 \times 10^{-3}$. Without the starting diameter more small crystals enter the waste stream, as they have not grown large enough to oppose the column flow. The average waste crystal size decreased from 5.70 ± 0.2 μm to 3.57 ± 01 μm. Smaller crystals have a lower PSD effect on a volume basis which means the d_{10} and d_{50} effectively increase.

2.3. CSC theoretical model flow modification

With the addition of an agglomeration term not satisfactorily explaining the difference in predictions and results, an investigation was carried out into what ***internal flow conditions*** occurring in the column could be causing both smaller and larger particles. A significant number percent of small crystals is required to lower the d_{10}, whereas only a small number percent of large crystals is required to increase the d_{90}. Alterations were made to the core CSC theoretical model to test the different flow condition hypotheses, and the most plausible theory investigated is presented below.

2.3.1. Channelling column model

Model predictions carried out with faster flow rates achieved significantly larger particle sizes; increases in the product d_{50} and d_{90} occurred. It is noted that the CSC is height and growth limited and at faster flow rates more crystals are lost from the column into the waste stream. Model predictions carried out with slower flow rates achieved significantly smaller particle sizes; decreases in the product d_{10}, d_{50} and d_{90} occurred. A combination of fast and slow

Table 1
Theoretical continuous settling crystalliser agglomeration results.[a]

Condition	d_{10} (μm)	d_{50} (μm)	d_{90} (μm)	Span	AGG%	PW
Product, AGG0	51.2 ± 0.4	73.2 ± 0.9	85.9 ± 0.9	0.47 ± 0.01	0	3.1 ± 0.2
Waste, AGG0	6.7 ± 0.0	22.5 ± 0.1	36.7 ± 0.3	1.33 ± 0.02	0	
Product, AGG1	48.2 ± 1.1	70.1 ± 1.6	85.4 ± 1.1	0.53 ± 0.04	55.7 ± 0.1	2.0 ± 0.1
Waste, AGG1	6.8 ± 0.1	22.7 ± 0.3	36.9 ± 0.5	1.32 ± 0.02	2.2 ± 0.1	
Product, AGG2	50.8 ± 1.6	73.3 ± 1.4	85.5 ± 0.9	0.47 ± 0.03	38.8 ± 2.7	2.3 ± 0.2
Waste, AGG2	6.8 ± 0.0	23.0 ± 0.4	37.4 ± 0.3	1.33 ± 0.03	2.4 ± 0.1	

[a] Abbreviations are: AGG0, no agglomeration; AGG1, agglomeration solved using Eq. (2); AGG2, agglomeration solved using Eq. (3); PW (–), the product to waste crystal volume ratio. $Q_f = 28$ mL min^{-1}, $C_T = 42$ g 100 g^{-1}, t = 16 h, Δt = 5 s, $P_N = 10$.