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Mathematical modelling and analysis of an industrial scale evaporative crystallizer producing lactose monohydrate

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ABSTRACT

Lactose is industrially produced by a series of concentration steps to supersaturate the solution allowing crystallization to take place. This papers deals with the operation and analysis of a forced circulation evaporative crystallizer producing pharmaceutical lactose. A mathematical model of the operation of the crystallizer was developed. The model comprises of the mass balance, population balance, nucleation kinetics, growth kinetics and lactose solubility and mutarotation rate expressions. The steady state operation of the crystallizer was analysed from the classical mixed suspension mixed product removal (MSMPR) model perspective. It was found that the crystallizer behaviour deviated from ideal MSMPR behaviour with a steep upward curvature in the population density curve for crystal sizes below 40 μm. A size dependent growth (SDG) rate model was used to model the deviation from ideal behaviour. The parameters of the SDG model were estimated by fitting it to the measured population density curve from the crystallizer. The crystallization kinetic parameters like growth and nucleation rate constants were then estimated by fitting the simulated trends to the measured date. The model showed that the average crystal size follows a transient decaying dynamics before settling down to a constant value. This was reflected in the actual data collected from the crystallizer. The transient dynamics disappeared from the model results when a simple size independent growth model was used.

1. Introduction

Lactose is generally produced by concentration of whey permeate followed by a series of batch cooling crystallisers. Mathematical models have been developed for cooling batch lactose crystallization by numerous researchers (McLeod, 2007; Mimouni et al., 2009; Vu et al., 2006; Wong et al., 2010). During the permeate concentration step, the supersaturation must remain below that where spontaneous nucleation occurs. This limits the concentration that can be achieved and thus the yield (Paterson, 2009).Evaporative crystallisation does not have this limitation, as the continuous crystallization that occurs, exhausts the supersaturation created due to the water removal by evaporation. This allows the yield to be increased. However, evaporative crystallization is a process of continuous nucleation and growth that leads to a broader particle size distribution than that in the batch crystallization with a single nucleation event. Understanding the dynamics and crystallisation processes occurring in the evaporative crystallisers will enable better control of the crystal size distribution (CSD) coming out of the evaporative crystalliser. This work presents, to the best of authors' knowledge, the first detailed study of an industrial scale forced circulation evaporative crystallizer for lactose monohydrate.

2. Mathematical model development

In the development of this model the following assumptions have been made:

(i) The crystallizer operates under ideal mixed suspension mixed product removal (MSMPR) conditions. It is well mixed and the temperature, crystal suspension and supersaturation are uniform throughout the crystallizer. The product removal is isokinetic and no classification of the crystals occurs.
(ii) Lactose crystals are spherical.
(iii) No agglomeration or crystal breakage occurs. This assumption was supported by the microscopic images of the lactose crystals which showed intact, single crystals coming out of the crystallizer.

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Nomenclature

C_α	dissolved α-lactose concentration [kg (kg of solution)$^{-1}$]
C_β	dissolved β-lactose concentration [kg (kg of solution)$^{-1}$]
$C_{\alpha s}$	equilibrium solubility of α-lactose [kg (kg of water)$^{-1}$]
C_{LS}	equilibrium solubility of lactose [kg (kg of water)$^{-1}$]
C	total dissolved lactose concentration [kg (kg of solution)$^{-1}$]
C_c	crystal content of slurry [kg (kg of slurry)$^{-1}$]
B_s	secondary nucleation rate [# min^{-1} (kg of slurry)$^{-1}$]
B_p	primary nucleation rate [# min^{-1} (kg of slurry)$^{-1}$]
D[4,3]	volumetric weighted mean diameter (μm)
F	correction factor for α-LMH solubility
G	growth rate (μm min^{-1}))
G_e	effective size independent growth rate in the SDG model of Mydlarz (μm min^{-1})
K_m	mutarotation rate constant
L	bin size/crystal size (μm)
M	mass of the slurry present in the crystallizer (kg)
Q	mass flow rate (kg min^{-1})
T	crystallizer temperature (°C)
b	secondary nucleation rate order
g	growth rate order
j	exponential dependence of secondary nucleation on crystal content
k_{evap}	evaporation rate (kg min^{-1})
$k_{n,p}$	primary nucleation rate constant [# min^{-1} (kg of slurry)$^{-1}$((kg of α-LMH) (kg of water)$^{-1}$)$^{-b}$]
$k_{n,s}$	empirical secondary nucleation rate constant [# min^{-1} (kg of crystal)$^{-1}$((kg of α-LMH) (kg of water)$^{-1}$)$^{-b}$]
k_g	growth rate constant [μm min^{-1} ((kg of α-LMH)(kg of water)$^{-1}$)$^{-g}$]
m	mass of single crystal (kg)
m_0	0th momentof CSD [# (kg of slurry)$^{-1}$]
m_1	1st moment of CSD [m (kg of slurry)$^{-1}$]
m_2	2nd moment of CSD [m^2 (kg of slurry)$^{-1}$]
m_3	3rd moment of CSD [m^3 (kg of slurry)$^{-1}$]
m_4	4th moment of CSD [m^4 (kg of slurry)$^{-1}$]
$n(L)$	population density of crystal size L [# (kg of slurry)$^{-1}$ (μm)$^{-1}$]
n_p	primary nucleation rate order
t	time (min^{-1})

Greek symbols

ρ	density (kg m^{-3})
φ	voidage [(kg of solution) (kg of slurry)$^{-1}$]
$1-\varphi$	crystal content of slurry [(kg of crystalline lactose) (kg of slurry)$^{-1}$]
τ	residence time (min)

Subscripts

f	inlet/feed stream
o	outlet/product stream
c	crystal
s	lactose solution
S	slurry
w	water
CSD	crystal size distribution
LMH	lactose monohydrate
MOL	method of lines
MSMPR	mixed suspension mixed product removal crystallizer
ODEs	ordinary differential equations
PDEs	partial differential equations
GRD	growth rate dispersion
SDG	size dependent growth
TS	total solids

(iv) The orders for growth and nucleation kinetics from the literature can be applied to the industrial crystallizer under study i.e. they are scale independent.
(v) The fluctuations and disturbances to flow rates and temperatures common during industrial operation are neglected.

2.1. Mass balance

A schematic representation of an evaporative crystallizer is shown in Fig. 1. Q(kg min^{-1}), φ[(kg of solution) (kg of slurry)$^{-1}$], C[(kg of dissolved lactose) (kg of crystal free slurry)$^{-1}$] represents the mass flow rate, voidage and dissolved lactose concentration respectively. Subscripts f and o denotes the feed and the discharge streams to and from the crystallizer. M(kg) is the mass of the slurry present in the crystallizer at any given time. k_{evap}(kg min^{-1}) is the evaporation rate.

The overall lactose solute balance in the crystallizer is given by:

$$\frac{d}{dt}[M\varphi C] + \frac{d}{dt}[0.95M(1-\varphi)] = Q_f\varphi_f C_f - Q_o\varphi_o C_o + 0.95Q_f(1-\varphi_f) - 0.95Q_o(1-\varphi_o) \quad (1)$$

where the terms on the left hand side represent the rate of accumulation of the dissolved and crystalline lactose in the crystallizer. The first two terms on the right hand side give the feed and discharge rate of dissolved lactose and the last two terms gives the feed and discharge rate of crystalline lactose. The crystalline lactose terms are multiplied by 0.95 to account for the water of crystallization.

The industrial crystallizer being modelled operates in two distinct modes. During the start-up, lactose solution is fed into the crystallizer vessel until a targeted volume is achieved. The steam flow to the calandria (heat exchanger) is then initiated, providing energy to concentrate the solution. The feed flow is controlled to maintain a constant volume in the crystallizer. Fresh lactose solution is continuously fed to the crystallizer to make up for the reduction in volume due to evaporation. The start-up phase can therefore be considered as a semi-batch or fed batch operation. This semi-batch concentration process continues till the desired slurry density is achieved. Then the discharge valve is opened and slurry is pumped forward to the next stage in the process. At this point the feed flow is set to maintain a constant level in the crystallizer, allowing for evaporation and the discharge flow. This marks the start of the continuous operation.

Eq. (1) for a semi-batch system under no discharge ($Q_o = 0$) and crystal free feed ($\varphi_f = 1$) conditions, becomes:

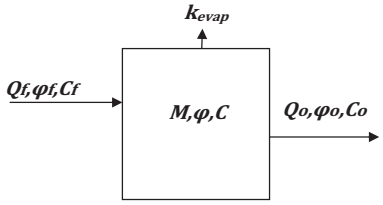

Fig. 1. A generalized schematic of an evaporative crystallizer.