

OPTIMISATION OF THE MULTICHAMBER CRYSTALLISER

Jaroslav NÝVLT* and Luděk PROVAZNÍK

Chemopetrol, Research Institute of Inorganic Chemistry, 400 60 Ústí nad Labem

Received November 8th, 1977

Mathematical model of the multichamber crystalliser is proposed and is solved for various combinations of temperature distributions among individual chambers. Results of a series of calculations have demonstrated that the most important factors are the temperatures in the first two chambers of the crystalliser while sufficient crystallisation surface area in other chambers suffices for reduction of supersaturation even at a large temperature difference. Optimisation of the five-chamber crystalliser with the mean size of product crystals chosen as the optimisation criteria is then performed for constants corresponding to crystallisation of Glauber salt.

Multichamber crystallisers can be considered to be the economically operated vacuum crystallisers. While a simple vacuum crystalliser operates at the final-low temperature which is from the point of view of the output disadvantageous, the vacuum in the multichamber crystalliser and thus the temperatures in individual chambers can be graded. In first chambers, evaporation takes place at the temperature only slightly smaller than that of the feed so that conditions for making use of the lowered pressure are more advantageous. This type of crystallisers is constructed as horizontal cylindrical vessel of larger size divided by partitions into three to eight chambers¹. Each chamber is connected usually to independent vacuum system with the suspension of crystals succeedingly overflowing from one chamber to the other. Individual chambers are more or less operated as adiabatic vacuum crystallisers so that a certain temperature corresponds to each chamber in steady state dependent among other on the fixed lowered pressure. Along the crystalliser thus exists a successively decreasing temperature profile.

In principle, it would be possible to describe the chamber crystalliser as a cascade of crystallisers with approximately constant volume of individual stages and with nucleation taking place in all stages. Mathematical models² have been proposed for such crystallisation cascade earlier which (for the sake of solubility of the describing relations) are based on some of the following assumptions: *a*) constant supersaturation in all stages of the cascade *b*) constant product $\bar{L}\bar{t}_i$ in all stages of the

* Present address: Czechoslovak Academy of Sciences, Institute of Inorganic Chemistry, Prague. The major part of the study has been presented at the National conference CHISA 77, in Bratislava 1977.

cascade. However these conditions are not fulfilled in the usual chamber crystalliser and thus it is necessary to look for computation to alternative solutions.

THEORETICAL

Let us assume that the chamber crystalliser is composed of individual stages which in general have the inlet of suspension, evaporation and outlet of suspension (Fig. 1). Parameters of the suspension entering the i -th stage are in general identical with those of the suspension leaving the $(i-1)$ st stage. Into the 1st stage enters only the fed solution so that the 1st stage can be expressed in the usual way as the stirred crystalliser with the mixed withdrawal of suspension (MSMPR) without seeding², the other stages are then MSMPR crystallisers with continuous seeding³. For the first stage holds approximately

$$\left(\frac{\bar{L}_1}{L_N}\right)^{1+3g/n} = 3B m_{c(1)}^{1-cg/n} \dot{m}_{c(1)}^{g/n-1} \quad (1)$$

while the mean size of crystals in the second and other stages is described by the relation

$$\begin{aligned} & \left(\frac{\bar{L}_i}{L_N}\right)^{1+3g/n} \left(1 - \frac{\dot{m}_{c(i-1)}}{\dot{m}_{c(i)}}\right) \left(1 - \frac{\dot{m}_{c(i-1)}}{\dot{m}_{c(i)}} \left(\frac{\bar{L}_i}{\bar{L}_{i-1}}\right)^3\right)^{-g/n} - \\ & - 4 \cdot 5 \cdot \left(\frac{\bar{L}_i}{L_N}\right)^{3g/n-2} \left(1 - \frac{\dot{m}_{c(i-1)}}{\dot{m}_{c(i)}} \left(\frac{\bar{L}_i}{\bar{L}_{i-1}}\right)^3\right)^{1-g/n} = \\ & = 3B m_{c(i)}^{1-cg/n} \dot{m}_{c(i)}^{g/n-1} \end{aligned} \quad (2)$$

Concentration of suspension in the crystalliser is determined by the material balance of an adiabatic crystalliser⁴

$$m_{c(i)} = \dot{m}_0 \bar{t}_i \left[h_{1g} \frac{w_0 - w_i}{w_0 + 1} + c_{p0}(T_0 - T_i) w_i \right] / (h_{1g} - h_{1s} w_i) \quad (3)$$

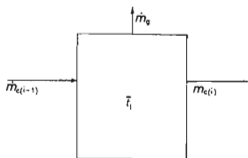


FIG. 1
Schematic Drawing of the i -th Chamber of the Multi-Chamber Crystalliser

$$\dot{m}_g = [\dot{m}_{c(i)} h_{1s} + \dot{m}_0 c_{p0}(T_0 - T_i)]/h_{1s}. \quad (4)$$

The dependence of solubility on temperature (*i.e.* the dependence of w_i on T_i) is determined by the equation⁵

$$\log x_i = A_0 + A_1/(T) + A_2 \log T. \quad (5)$$

The relation between the concentration of suspension and the crystalliser output is given by $m_{c(i)} = \dot{m}_{c(i)} \bar{t}_i$.

RESULTS AND DISCUSSION

In view of the complexity of solution (Eq. (2) has been solved by the trial and error method), the whole solution of the chamber crystalliser has been performed on the computer TESLA 200 with values of parameters close to those at crystallisation of Glauber salt. This is because the Glauber salt is one of the most frequent products for the crystallisation of which is applied the chamber crystalliser. The basical assumption has been the inlet of the solution saturated at $T_0 = 30^\circ\text{C}$, which was in all cases cooled to the final temperature $T_f = 0^\circ\text{C}$. However distribution of temperatures to individual chambers has been different: Temperature of the 1st stage has changed by 1 K, temperature of the 2nd stage by 2 K and temperature of the 3rd and 4th stages by 5 K in such a way that no heating of the solution took place. Other parameters used for calculation have had the values $m_0 = 3.5 \text{ kg s}^{-1}$; $w_0 = 1.9394 \text{ kg/kg}_0$; $A_0 = -150.93607$; $A_1 = 4229.3746$; $A_2 = 54.672663$; $h_{1g} = 2457.65 \text{ kJ/kg}$; $h_{1s} = 232.78 \text{ kJ/kg}$; $M_{anh} = 142.037$; $M_{byd}/M_{anh} = 2.268$; $B = 0.1 \text{ kg}^{-g/n} \text{ kg}_0^{g/n} \cdot \text{s}^{g/n-1}$; $g = 1.0$; $n = 2.55$; $c = 0$; $\bar{t}_1 = 1060 \text{ s}$; $L_N = 1.5 \cdot 10^{-4} \text{ m}$.

In all calculation has been performed for 1600 distributions of temperatures: Some of the most interesting results are summarized in Table I, for the optimum are the resulting data given in Table II.

From the data given in Table I results that the difference in temperature among individual chambers $\Delta T_i = T_{i-1} - T_i$ should successively increase proportionally to the increase of the crystallisation area with the passage through the whole crystalliser. The curve 1 in Fig. 2 corresponds to the optimum distribution of temperatures and the corresponding mean size of crystals has reached 0.83 mm. The curve 2 represents more or less the usual temperature distribution in the five chamber crystalliser and the corresponding mean size of crystals was 0.76 mm. Though the difference in mean sizes of crystals does not seem to be too significant, it is necessary to realize that the simple mixed suspension crystalliser (curve 3) under analogical conditions with the effective volume identical with the effective volume of one chamber, would supply product with the mean size of crystals 0.72 mm so that the differences of the required sizes of crystals in hundredths mm would be significantly reflected in investment costs.

TABLE I
Selected Data on Mean Size of Crystals in Individual Chambers

ΔT_1	ΔT_2	ΔT_3	ΔT_4	ΔT_5	$\bar{L}_1 \cdot 10^3$	$\bar{L}_2 \cdot 10^3$	$\bar{L}_3 \cdot 10^3$	$\bar{L}_4 \cdot 10^3$	$\bar{L}_5 \cdot 10^3$
1	2	5	10	12	0.54	0.65	0.74	0.80	0.82
1	8	15	5	1	0.54	0.69	0.75	0.76	0.76
2	2	3.5	7.5	15	0.60	0.68	0.74	0.80	0.83
2	2	5	15	6	0.60	0.68	0.75	0.81	0.82
2	8	10	10	0	0.60	0.72	0.77	0.78	0.78
3	2	5	15	5	0.63	0.69	0.75	0.81	0.81
3	3	4	7.5	12.5	0.63	0.71	0.76	0.80	0.82
3	4	6.5	5	12.5	0.63	0.72	0.77	0.80	0.82
3	4	15	4	3	0.63	0.72	0.80	0.80	0.81
4	4	20	2	0	0.65	0.72	0.80	0.80	0.80
5	4	15	6	0	0.66	0.72	0.78	0.79	0.79
6	8	16	0	0	0.66	0.73	0.77	0.77	0.77
7	2	21	0	0	0.68	0.70	0.77	0.77	0.77
7	6	17	0	0	0.68	0.73	0.77	0.77	0.77
7	12	11	0	0	0.68	0.75	0.77	0.77	0.77
8	20	2	0	0	0.68	0.75	0.75	0.75	0.75
9	14	7	0	0	0.69	0.75	0.76	0.76	0.76
10	18	2	0	0	0.69	0.75	0.75	0.75	0.75
11	19	0	0	0	0.59	0.74	0.74	0.74	0.74
12	18	0	0	0	0.69	0.73	0.73	0.73	0.73
13	17	0	0	0	0.71	0.74	0.74	0.74	0.74
14	16	0	0	0	0.71	0.74	0.74	0.74	0.74
15	15	0	0	0	0.71	0.73	0.73	0.73	0.73
20	10	0	0	0	0.72	0.73	0.73	0.73	0.73
25	5	0	0	0	0.72	0.73	0.73	0.73	0.73
30	0	0	0	0	0.72	0.72	0.72	0.72	0.72

TABLE II
Optimum Temperature Distribution

i	T	ΔT_1	\dot{m}_g	$m_{c(i)}$	w_i	$\bar{L}_1 \cdot 10^3$
1	28.0	2.0	0.073	0.705	1.435	0.60
2	26.0	2.0	0.120	1.13	1.103	0.68
3	22.5	3.5	0.176	1.56	0.738	0.74
4	15.0	7.5	0.256	1.97	0.358	0.80
5	0.0	15.0	0.374	2.22	0.108	0.83

For the given ΔT_1 and ΔT_2 the dependence of the mean size of crystals on the value ΔT_3 has passed through the maximum which at low values of ΔT_1 and ΔT_2 is very significant and is situated at lower values ΔT_3 (e.g. at $\Delta T_1 = 1$ is for $\Delta T_2 = 6$ with the value $\Delta T_3 = 5$ K, for $\Delta T_2 = 4$ already at $\Delta T_3 = 10$ K and for $\Delta T_2 = 2$ at values $\Delta T_3 = 15$ K, for $\Delta T_1 = 3$ and $\Delta T_2 = 2$ at $\Delta T_3 = 5$ K, for $\Delta T_2 = 4$ already at $\Delta T_3 = 15$ K and finally for $\Delta T_1 = 6$ independently of ΔT_2 is situated at values $\Delta T_3 = 15$ K or also according to possibilities greater, for $\Delta T_1 \geq 8$ the maximum is already very inexpressive. This property (i.e. the fact that the resulting mean size of crystals depends first of all on values ΔT_1 and ΔT_2), has enabled construction of the optimisation graph (Fig. 3) in which the dependence of mean size of crystals on values ΔT_1 and ΔT_2 is plotted regardless of the distribution of temperatures to next individual chambers under the assumption that the above given condition is at least approximately satisfied for suitable values ΔT_3 . It is obvious from Fig. 3 that for the given values of parameters we obtain the largest crystals for $\Delta T_1 = \Delta T_2 = 2$ K. With the further increasing values ΔT_1 and ΔT_2 the size of product crystals is successively decreasing while it is less and less dependent on the value ΔT_2 .

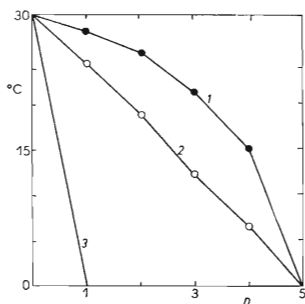


FIG. 2
Temperature Distribution Between Individual Chambers (Individual curves are denoted in the main part of this study)

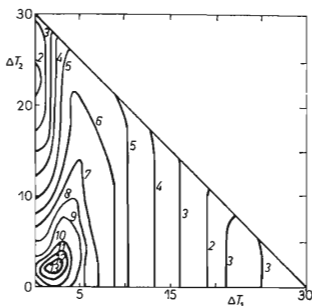


FIG. 3
Mean Size of Product Crystals from the Multi-Chamber Crystalliser, \bar{L} , in Dependence on Values ΔT_1 and ΔT_2
Values $\bar{L} \cdot 10^3$ (m): 2 0-72, 3 0-73, 4 0-74, 5 0-75, 6 0-76, 7 0-77, 8 0-78, 9 0-79, 10 0-80, 11 0-81, 12 0-82, 13 0-83.

From the data given in Table I is also obvious that the greater is the number of chambers of the crystalliser the smaller ΔT_1 should be chosen. While the optimum effect in the five-chamber crystalliser is reached at $\Delta T_1 = 2$ K the effective distribution of temperatures at $\Delta T_1 = 4$ K already corresponds utmost to the four chamber crystalliser, for $\Delta T_1 = 6$ K only to the three-chamber crystalliser, for $\Delta T_1 = 10$ K there suffice already only two crystallisers in the series, while at greater ΔT_1 the resulting effect is quite negligible in comparison with the simple crystalliser (as concerns the mean size of crystals but not the distribution of crystal sizes).

CONCLUSIONS

For the final size of product crystals from the chamber crystalliser, the distribution of temperatures first of all in the first two chambers is decisive. At the optimum temperature distribution the temperature differences between the adjoining chambers in the direction of suspension flow are steadily increasing. The choice of temperature difference of the saturated feed and temperature in the first chamber significantly affects the number of chambers in the given crystallisation device to be effectively used.

LIST OF SYMBOLS

A_0, A_1, A_2	constants in Eq. (5)
B	system constant ($\text{kg}^{-s/n} \text{kg}_0^{s/n} \text{s}^{s/n-1}$)
c	exponent of secondary nucleation
c_{p0}	specific heat of feed solution ($\text{kJ kg}^{-1} \text{K}^{-1}$)
g	order of crystal growth
h_{ig}	heat of evaporation (kJ kg^{-1})
h_{is}	crystallisation heat (kJ kg^{-1})
\bar{L}_i	mean size of crystals in the i -th chamber (m)
L_N	size of initial crystals (m)
M_{anh}	relative molecular weight of water-free compound (kg mol^{-1})
M_{hyd}	relative molecular weight of hydrate (kg mol^{-1})
\dot{m}_g	evaporation rate (kg s^{-1})
\dot{m}_0	feed rate (kg s^{-1})
$m_{c(i)}$	concentration of suspension in the i -th chamber (kg kg_0^{-1})
$\dot{m}_{c(i)}$	specific output of the i -th chamber ($\text{kg kg}_0^{-1} \text{s}^{-1}$)
n	nucleation order
T	temperature (K)
T_0	temperature of feed ($^{\circ}\text{C}$)
T_i	temperature in the i -th chamber ($^{\circ}\text{C}$)
$\Delta T_i = T_{i-1} - T_i$	

- \bar{t}_1 mean residence time of solution in the one chamber (s)
 w_0 feed concentration (kg kg_0^{-1})
 w_i concentration of solution in the i -th chamber (kg kg_0^{-1})
 x_i mole fraction of crystallising compound in the saturated solution at temperature T_i

REFERENCES

1. Bamforth A. W.: *Industrial Crystallization*. Hill, London 1956.
2. Nývlt J., Moudrý F., Veverka V.: This Journal 38, 1815 (1973).
3. Nývlt J.: This Journal 43, 209 (1978).
4. Nývlt J.; Kočová H.: Chem. Prům. 26, 567 (1976).
5. Broul M., Nývlt J., Söhnel O.: *Tabulky rozpustnosti anorganických solí ve vodě*. Academia, Prague 1979.

Translated by M. Rylek.