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Abstract

The paper deals with the design and operation of cooling and evaporation crystallizers and precipitators for reaction crystallization. It will be shown that the median crystal size L_{50} of a crystalline product and the Crystal Size Distribution (CSD) mainly depend on the crystallization kinetics, e.g. the rates of nucleation and crystal growth, and that this kinetics is controlled by supersaturation. Therefore, the main objective in crystallization and precipitation is to choose and to maintain the optimal supersaturation with respect to product quality at all times and all locations in the crystallizer or precipitator.

1. The role of supersaturation for the median crystal size

The selection of the apparatus or reactor and its operation condition depend on the chemico-physical properties of the system which can be described by the dimensionless solubility c^*/c_c (c^* is the solubility of the solute and c_c the density of the crystalline material) and the slope $d(\ln c^*)/d(\ln T)$ of the solubility curve $c^*=f(T)$ with T as the absolute temperature, see Fig. 1. Evaporative crystallization is necessary in the case of flat solubility curves $d(\ln c^*)/d(\ln T) < 1$.

Drowning out crystallization is carried out by the addition of an antisolvent, for instance alcohols or ketones. The maximum possible supersaturation in the absence of any formation of solid matter (e.g. a theoretical supersaturation not occurring in crystallizers) is given by

the ratio $\frac{\text{solution flow rate}}{\text{cooling rate}}$
in cooling crystallization or

the ratio $\frac{\text{solution flow rate}}{\text{evaporation rate}}$
in evaporative crystallization or

the ratio $\frac{\text{solution flow rate}}{\text{antisolvent flow rate}}$
in drowning out crystallization

However, we demonstrate that the actual supersaturation $\Delta c \equiv (c - c^*)$ or relative supersaturation $\sigma = \Delta c / c^*$ is the result of crystallization kinetics and the presence of crystals and foreign particles in the apparatus.

In reaction crystallization, two or more components

react chemically with the result that a new product is formed. If the initial concentration of the reactants is high and, therefore, the initial concentration $c_{\max}$ of the product is also very high but the solubility c^* is very low, the resulting high supersaturation $S_{\max} = 1 + \sigma_{\max} = c_{\max}/c^*$ or $\sigma_{\max} = \Delta c_{\max}/c^*$ leads to high rates of primary nucleation. Since foreign particles in the nanometer range are always present in the reactant

Fig. 1 Solubility of various solutes

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solutions, heterogeneous nucleation will take place. Homogeneous nucleation can be dominant at very high supersaturation ($\Delta c/c_c > 0.1$ for non-dissociating products).

Primary nucleation is an activated process which means that local or temporary peaks of supersaturation Δc are quickly reduced to the metastable supersaturation $\Delta c_{\text{met}} (\Delta c \rightarrow \Delta c_{\text{met}})$. The rate, B_{prim} , of primary nucleation is a function of the actual local supersaturation S . After this nucleation step, particles grow at the moderate supersaturation $\Delta c \leq \Delta c_{\text{met}}$ with the growth rate $G = f(\Delta c)$. If the residence time τ of nuclei in the precipitator or the batch time are small, the size increase $\Delta L = G\tau$ is small, too. When the crystal size exceeds approximately 100 μm , attrition fragments are formed which can act as secondary nuclei. The total nucleation rate is the sum $B = B_{\text{prim}} + B_{\text{sec}}$ of the contributions of primary and secondary nucleation. The median crystal size and the crystal size distribution (CSD) depend on the kinetic parameters B and G .

In the case of MSMPR (Mixed Suspension Mixed Product Removal) crystallizers or precipitators, the median crystal size L_{50} is given by

$$L_{50} = 3.67 \left(\frac{G m_T}{6 \alpha B \rho_c} \right)^{1/4}$$

with the suspension density m_T and the density ρ_c of crystals. With reaction crystallization, the suspension density results from a mass balance of the reactants in the case of complete conversion. According to equation (1), nanoparticles can be obtained by applying very high nucleation rates, B_{prim} , which require very strong supersaturations S_{max} . Such high supersaturations can be achieved by

- high concentrated reactants
- products with high concentrations c_{max} but low solubilities c^*
- rapid micromixing at the feed point of the reactants

for a fast chemical reaction. If, however, a coarse product is desired, the supersaturation must be kept low by

- low concentrated reactants (perhaps by dilution)
- good macromixing in the entire precipitator and
- seeding by high recycling rates of slurry in order to reduce local supersaturation peaks by crystal growth.

In systems with high solubilities ($c^*/c_c > 0.01$), the supersaturation is so small ($\sigma < 0.1$) that activated nucleation does not take place and new nuclei are only formed as attrition fragments.

In this case large median sizes L_{50} can be obtained if

- the attrition rate $B_{\text{att}} \sim B_{\text{sec}}$ is low,

- the crystal growth rate G has the maximum allowable value with respect to crystal purity and
- the mean residence time of the slurry is optimum.

In the case of batch crystallizers, the median crystal size L_{50} as well as the CSD are dependent on the actual supersaturation which varies with time and also with the location, if a voluminous apparatus is not ideally mixed.

The kinetic crystallization parameters, i.e. the nucleation rate, B , and the growth rate, G , are necessary for the evaluation of the median crystal size.

2. Nucleation rate

The rate of primary nucleation depends on the diffusivity D_{AB} , the solubility c^* , the density c_c of the crystals and the supersaturation S , and can be calculated according to the classical theory of nucleation [6]. However, with respect to a very high particle or nuclei density N in the form of particles/ m^3 , a very rapid perikinetic agglomeration takes place with the result that observed nucleation rates, B_{prim} , of nuclei in the micrometre range are several orders of magnitude smaller than predicted by theory [9]. In addition, foreign particles are present in every solution leading to heterogeneous nucleation. According to experiments carried out with foreign particles added to the reactants, the nucleation work is reduced to approximately 10% of the value for homogeneous primary nucleation [1, 8]. Furthermore, the rate of heterogeneous primary nucleation is proportional to the specific surface area of foreign particles present in the solution [1, 8]. These results and further considerations allow evaluation of the metastable zone widths for homogeneous and heterogeneous nucleation. Below a certain supersaturation $\sigma_{\text{met,sec}}$ or $\Delta c_{\text{met,sec}}/c^*$, the nucleation rate induced by activated nucleation is so small that new nuclei can only originate from attrition fragments [6].

In **Figure 2**, the nucleation rate is plotted against the relative supersaturation S for activated (surface) nucleation (theoretical lines) and for attrition-induced nucleation (shaded area). Further theoretical considerations and experimental results lead to **Figure 3** where the dimensionless metastable zone width, $\Delta c_{\text{met}}/c_c$, (valid for $B \gg 10^{12} \text{ m}^{-3} \text{ s}^{-1}$), is plotted against the dimensionless solubility, c^*/c_c , with c_c as the density of the solid crystals (valid for non-dissociating systems). In such a diagram, lines of constant relative supersaturation S can be drawn according to $\Delta c/c_c = (\Delta c/c^*)(c^*/c_c) = \sigma \cdot c^*/c_c$.

Fig. 2 Volumetric nucleation rate against the dimensionless supersaturation for activated and attrition-controlled nucleation

Fig. 3 Metastable zone widths $\Delta c_{met}/c_c$ against the dimensionless solubility c^*/c_c for homogeneous, heterogeneous and attrition-controlled nucleation

3. Growth rates

Experimental results and theoretical considerations have shown that the mean growth rate, G , of a crystal collective depends mainly on the mass transfer coefficient k_d , the dimensionless solubility, c^*/c_c , and the relative supersaturation $\sigma = \Delta c/c^*$ [4]. In **Figure 4**, the ratio v/k_d with $G=2v$ is plotted against σ with c^*/c_c as the parameter. As can be seen from the shaded area in this figure, the growth rate G is often in the range

$$10^{-9} \text{ m/s} < G < 10^{-7} \text{ m/s}$$

and can be calculated for growth controlled by diffusion for $\sigma > 1$ from

$$G = \frac{\beta}{3\alpha} k_d \frac{\Delta c}{c_c}$$

α and β are the volume or surface shape factors, resp.

Since the maximum supersaturation is $\Delta c_{met,act}$ after activated nucleation, the supersaturation Δc with $\Delta c_{met,hel} > \Delta c > 0$ is decisive for the growth of nuclei. With batch crystallizers, the growth rate depends on the time-averaged supersaturation

$$\bar{\Delta c} = \frac{1}{\tau} \int_0^{\tau} \Delta c \, dt$$

Here, τ is the residence time of the crystals in the crystallizer. The curves in **Figure 4** show maximum possible growth rates in the absence of impurities or additives which retard or even block crystal growth. Only agglomeration of submicron particles of high particle density N may enhance the increase of crystal size per unit time. With needle-like crystals or platelets, the definition and prediction of crystal growth rates are difficult.

If the crystallizer is not ideally mixed, the local distribution of supersaturation has to be taken into account. This can be done by dividing the total active volume into sections with approximately constant supersaturation.

Fig. 4 Crystal growth rate against the relative supersaturation with the ratio c^*/c_c as the parameter

4. Median crystal size

In the case of activated nucleation, the rate B_{act} increases very rapidly with the supersaturation S ($B_{act} \sim S^{10}$ up to $B_{act} \sim S^{50}$ in the absence of agglomeration depending on c^*/c_c , see **Fig. 2**). In reality, however, strong agglomeration takes place for nuclei smaller than one micrometre [9]. The higher the initial supersaturation the smaller is the median crystal size L_{50} in the absence of agglomeration. In **Figure 5**, the median crystal size is plotted against the mean relative supersaturation σ (or the maximum supersaturation $\sigma_{max} = S_{max} - 1$ for reaction crystallization) for inorganic and organic systems with different solubilities c^* or ratios c^*/c_c (the density of crystals is in the range $5 \text{ mol/dm}^3 < c_c < 40 \text{ mol/dm}^3$). Additionally, the diagram shows experimental results of the aqueous systems of BaSO_4 , BaCO_3 , CaCO_3 , Mg(OH)_2 and $\text{MgC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. As can be seen from this figure, the median crystal size drops with increasing relative supersaturation. In reaction crystallization, the relative supersaturation which is decisive for the median crystal size depends on

- the rate constant of the chemical reaction,
- the number and size of foreign particles present in the reactants,
- the effectiveness of macro and micromixing, and
- the maximum product concentration.

Experiments carried out with continuously operated precipitators have shown that the mean crystal size is much larger in comparison to products obtained with batch precipitators in which large differences of supersaturation occur with respect to time and space.

Systems with high solubilities ($c^*/c_c > 0.01$) lead to median crystal sizes $L_{50} > 100 \text{ }\mu\text{m}$. The larger the crystal and the higher the collision velocity w_{col} of

Fig. 5 Median crystal size against the initial (maximum possible) supersaturation; shaded are: results of inorganic and organic systems [5]

Fig. 6 Relative attrition volume against the collision velocity (crystal resistance) for KNO_3 according to different models

crystals against a resistance (rotor, wall, other crystals), the stronger are the attrition effects which lead to attrition fragments in the size range $1 \text{ }\mu\text{m} < L_{att} < 150 \text{ }\mu\text{m}$ [11]. In **Figure 6**, the relative abraded crystal volume $\Delta V_c/V_c$ based on the volume V_c of a single crystal is plotted against the collision velocity w_{col} for 0.1 and 1 mm KNO_3 crystals according to a model of Gahn [11]. The diagram also shows lines according to a model developed earlier [12, 13, 14, 15]. In all cases, a huge number of attrition fragments are produced in industrial crystallizers with crystals larger than $100 \text{ }\mu\text{m}$. Therefore, it is necessary to restrict the circumferential velocity of rotors (stirrer, pumps) to $< 10 \text{ m/s}$ and the mean specific power input in stirred vessels to $\bar{\epsilon} < 0.5 \text{ W/kg}$ when a coarse product is desired. The growth rate, G , of small attrition fragments is reduced due to crystal deformation and an increased chemical potential [16].

This reduction can be described by

$$G = k_g \left[\frac{c - c^* \exp\left(\frac{\Gamma_s}{R T L_{att}}\right)}{c^*} \right]^g$$

with k_g as the growth coefficient and g as the growth exponent usually in the range $1 < g < 2$. The higher the fracture resistance Γ_s and the smaller the size L_{att} of the attrition fragment, the more the growth rate is reduced, see **Figure 7** valid for KNO_3 attrition fragments. With respect to individual differences in defor-

Fig. 7 Growth rate of 356 KNO₃ attrition fragments against their initial size

Fig. 8 Optimal supersaturation for cooling crystallization against the dimensionless solubility for different cooling rates or slopes of the solubility curve

mation, the growth rate differs from fragment to fragment (Growth Rate Dispersion, GRD). As a result of these considerations, large median sizes can only be obtained under the following conditions:

- the supersaturation has to be optimum (see Fig. 8), i.e.
- the residence time (continuously operated crystallizer) or the batch time has to be optimum (see below)
- the circumferential velocity u_{tip} of a rotor should be low and
- the specific power input should be low, too.

However, with respect to the suspension of coarse crystals in stirred vessels, a minimum value of $[u_{tip}(D/T)^{3/2}]$ of the stirrer is necessary for a given suspension [17]:

$$\left[u_{tip} \left(\frac{D}{T} \right)^{3/2} \right]_{min} = \frac{1}{0.088 Ne^{7/18}} \left[3c_w \cdot L_p \cdot g \frac{\Delta \rho}{\rho_L} \cdot w_s^2 \phi_v \right]^{1/4}$$

with D/T as the diameter ratio (stirrer D and tank T) and w_s as the settling velocity of the largest crystal with the drag coefficient c_w and the density difference $\Delta \rho = \rho_c - \rho_L$ between the crystal (c) and the liquid (L), ϕ_v is the volumetric crystal hold-up and Ne the Newton number of the stirrer. As can be seen from this equation, crystals with a large settling velocity w_s are mostly endangered by attrition.

5. Supersaturation in crystallizers

The actual local supersaturation in a crystallizer under isothermal condition is the result of the following parameters and processes:

<ul style="list-style-type: none"> • Concentration of the reactants • Velocity of the chemical reaction • Intensity of macro and micromixing • Dilution of solution • Agglomeration 	important for sparingly soluble systems
<ul style="list-style-type: none"> • Crystallization kinetics (nucleation, growth) • Creation of crystal surface by attrition. 	important for highly soluble systems

Let us assume that precipitation is initiated by a very fast chemical reaction with the result that the progress of this reaction is controlled by macro and micromixing. This will be explained in more detail.

5.1 Effect of macro and micromixing

In Figure 9, the supersaturation S is plotted against a dimensionless time t^* which is defined by

$$t^* = \frac{1}{\tau_{macro}} \left(\frac{V}{\dot{V}_A + \dot{V}_B} \right) = \frac{\tau}{\tau_{macro}}$$

for MSMPR crystallizers and

$$t^* = t \cdot \left(\frac{\dot{V}_A + \dot{V}_B}{V} \right)$$

for batch crystallizers. With continuously operated crystallizers, the actual supersaturation depends on the ratio of the mean residence time $\tau = [V / (\dot{V}_A + \dot{V}_B)]$ and the macromixing time τ_{macro} according to

$$\tau_{macro} \approx 5 \frac{T^{2/3}}{\bar{\epsilon}^{1/3}} \approx 5 \left(\frac{\pi}{4 Ne} \right)^{1/3} \left(\frac{T}{D} \right)^{5/3} \cdot \frac{1}{N}$$

Here, $\bar{\epsilon}$ is the mean specific power input equivalent to $\bar{\epsilon} = (1/V) \int \epsilon dV$, T is the tank diameter and N is the speed of the stirrer. In batch crystallizers, the ratio $V / (\dot{V}_A + \dot{V}_B)$ is the time of the addition of the two reactants A and B or the solution, respectively. Let us assume that the addition time of a reaction crystal-

lizer is very short compared to the total batch time. If the micromixing time of the liquid mixture with the viscosity ν according to

$$\tau_{\text{micro}} \approx 50 \ln Sc \frac{\nu^{1/2}}{\epsilon_{\text{loc}}^{1/2}}$$

is very short ($\tau_{\text{micro}} \ll \tau_{\text{macro}}$ or $\tau_{\text{macro}}/\tau_{\text{micro}} \rightarrow \infty$), the maximum possible supersaturation S_{max} is obtained after a short time (batch crystallizers) or a short residence time (continuously operated crystallizers), see point A. The supersaturation decreases with increasing t^* . The longer the micromixing time τ_{micro} in comparison to the macromixing time τ_{macro} , the smaller is the supersaturation for a given t^* and, consequently, the nucleation rate.

5.2 Effect of dilution

Let us consider a T-mixer and a stirred vessel, see **Figure 10**. In the case of a stirred vessel, the flow rates $\dot{V}_A$ and $\dot{V}_B$ of the reactants are diluted in the vessel and the degree of dilution increases with the vessel volume V . Therefore, the actual concentration and supersaturation depend on the ratio $(\dot{V}_A + \dot{V}_B)/V$. The actual local supersaturation can be influenced by the processes of

- dilution ($\sigma \sim 1/V$), see point B in **Figure 9**
- macromixing ($\sigma \sim 1/N$) and
- micromixing ($\sigma \sim \epsilon_{\text{loc}}^{-1/2}$).

Therefore, the prediction of the median crystal size requires the prediction of the mixing phenomena and the calculation of local concentrations and supersaturation profiles.

5.3 Effect of attrition

In the case of strong attrition (e.g. high settling velocity w_s of crystals), the maximum and the median crystal size are reduced with the result that the coefficient of variation CV decreases. The CSD can be con-

Fig. 9 Supersaturation S against the dimensionless time with τ_{micro} as the parameter

Fig. 10 Operation of a T-mixer and a stirred vessel

Fig. 11 Median crystal size of KNO_3 crystals against the mean residence time

trolled by attrition, see **Figure 11**. In this diagram, the median crystal size is plotted against the residence time τ of a continuously operated crystallizer. Since KNO_3 (as well as potash alum and citric acid) are very prone to attrition, the optimum residence time τ_{opt} is only one hour. With attrition-resistant materials such as KCl , NaCl , $(\text{NH}_4)_2\text{SO}_4$ and tartaric acid, the residence time τ_{opt} can be two or three hours. Therefore, it is very important to know the attrition behaviour which depends mainly on the fracture resistance of the crystalline matter [11].

6. How to influence the median crystal size

The median crystal size L_{50} depends on the crystallization kinetics via $B(S)$ and $G(S)$ and on the growth time (batch) or $t = V/(\dot{V}_A + \dot{V}_B)$ with $L_{50} = 3.67 \times G\tau$ for MSMR crystallizers.

6.1 Continuously operated crystallizers

Very small particles (nanoparticles) can be produced by

- highly concentrated reactants and high $\Delta c/c_c$ ($\Delta c/c_c > 0.1$)
- avoidance of agglomeration (addition of surfactants and/or change in pH)
- rapid quenching or diluting in order to stop growth
- fast local micromixing of the reactants but poor macromixing
- no dilution (T-mixer) in order to produce many nuclei

The contrary is true for obtaining large particles. The reduction of attrition is the prerequisite for producing coarse crystals. As a rule, particles in the micrometre range are agglomerates formed from nanoparticles [8, 9].

6.2 Batch crystallizers

In order to produce very fine particles, the remarks given under 6.1 are valid. The addition tubes of the two reactants should be very close together at a point of a very high specific power input (discharge region of the impeller, preferably a Rushton turbine). Another possibility is to premix the reactants in a T-mixer where the fast chemical reaction takes place, see **Figure 10**. The suspension is then introduced into the vessel where a very weak (or zero) supersaturation leads to no further activated nucleation (or growth).

Large particles can be produced if

- the supersaturation is optimum (only very few nuclei but sufficient growth) and
- the residence time of the crystals is long ($L_{50} \sim G\tau$) [10].

Nomenclature

B	: nucleation rate	[m ⁻³ s ⁻¹]
c	: concentration	[kmolm ⁻³]
c _w	: drag coefficient	[–]
D	: stirrer diameter	[m]
D _{AB}	: diffusion coefficient	[m ² s ⁻¹]
f	: factor	[–]
G	: growth rate	[ms ⁻¹]
k _d	: mass transfer coefficient	[ms ⁻¹]
k _g	: growth coefficient	[ms ⁻¹]
L	: crystal size	[m]
m _T	: suspension density	[kgm ⁻³]
N	: stirrer speed	[s ⁻¹]
R	: gas constant	[Jmol ⁻¹ K ⁻¹]
S	: supersaturation	[–]
T	: tank diameter	[m]
T	: temperature	[K]

t	: time	[s]
V	: vessel volume	[m ³]
$\dot{V}$	: volumetric flow rate	[m ³ s ⁻¹]
w _s	: settling velocity	[ms ⁻¹]
α, β	: shape factors (volume, surface)	[–]
ϵ	: specific power input	[Wkg ⁻¹]
ν	: kinematic viscosity	[m ² s ⁻¹]
ρ	: density	[kgm ⁻³]
τ	: residence or mixing time	[s]
Γ_s	: fracture resistance	[Jmol ⁻¹ m]
ϕ_v	: volumetric hold-up	[–]
Ne	: Newton number of the stirrer	[–]
Sc $\equiv \nu/D_{AB}$	: Schmidt number	
t*	: dimensionless time	[–]

Subscripts and superscripts

A, B	: component A, B
act	: activated
att	: attrition
c	: crystalline
het	: heterogeneous
hom	: homogeneous
L	: liquid
max	: maximum
p	: parent
prim	: primary
sec	: secondary
tip	: tip speed
*	: equilibrium
50	: 50 percent

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Author's short biography

A. Mersmann

1931	Alfons Mersmann was born in Oer-Erkenschwick, Germany
1952-1957	he studied Mechanical Engineering at the Technical University of Hannover
1957-1961	worked at the Max-Planck Institute in Goettingen, Germany in the field of heat and refrigeration technology
1961	wrote his doctorate on: Pressure drop and foam thickness of liquid layers in a gas penetrated sieve tray
1961-1969	worked at Dynamit Nobel, Germany
since 1969	is dean of the Institute of Mechanical Engineering at the Technical University Munich, Germany
1983-1985	was dean of the department of Mechanical Engineering
1994	received the Emil-Kirschbaum-Medal for his continued research in the field of Thermal Process Technology
1996	received the Ernest-Solvay Prize

Prof. Mersmann prepared many international conferences, published more than 280 articles. He was and still is member of various special research projects.