Industrial Crystallization

Bridging the gap between theory and practice, this text provides the reader with a comprehensive overview of industrial crystallization.

Newcomers will learn all of the most important topics in industrial crystallization, from key concepts and basic theory, to industrial practices. Topics covered include the characterization of a crystalline product and the basic process design for continuous evaporative and cooling crystallization, as well as batch crystallization. Also included are measurement techniques, and details on precipitation, melt crystallization, and polymorphism, as well as the impact of additives and impurities on process and product features.

Each chapter begins with an introduction explaining the importance of the topic, and is supported by homework problems and worked examples. Real-world case studies are also provided, as well as new industry-relevant information, making this an ideal resource for industry practitioners, students, and researchers in the fields of industrial crystallization, separation processes, particle synthesis, and particle technology.

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Fundamentals and Applications

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Nomenclature

Main

Variable	Description	Unit	Chapter
a	activity	M	1
a	surface area of a solute entity in the cluster surface	m^2	4
A	crystal or particle surface area	m^2	2
A	Rate parameter of primary nucleation rate expression	$m^{-3}\ s^{-1}$	4
A_1, A_2	parameters for stress energy of a crystal, see eq. 5.45		5
A_{AZ}	cross sectional area of the annular zone of a DTB crystallizer	m^2	3
A_{hex}	circulation heat exchanger area	m^2	3
A_T	total crystal surface area per unit crystallizer volume	$m^2\ m^{-3}$	2
b, i, j, h, k	parameters in secondary nucleation rate expressions eq. 4.33 and eq. 4.34	–	4
B, B_0	secondary nucleation rate	# sm^{-3}	4, 5, 7, 8
$B(L, t), B(v, t)$	particle birth rate for nuclei of size L or volume v	# $m^{-3}\ m^{-1}\ s^{-1}$	7, 8
$b(\varepsilon, v)$	breakage function	m^{-1}	7
B	Burgers vector	m	5
C	attrition constant defined in eq. 4.46		4
C	empirical kernel of agglomeration		6
c	molar concentration	kmol m^{-3}	4
c	massic concentration	kg m^{-3}	5
c_b	impurity concentration in the bulk	kmol m^{-3}	2, 12
c_c	impurity concentration in the crystal	kmol m^{-3}	2, 12
$c_{c,G}$	impurity concentration in the crystal growing at a growth rate G	kmol m^{-3}	12
c_i	impurity concentration at the interface	kmol m^{-3}	12
$C(n)$	concentration of clusters of size n in solution	# molecules m^{-3}	4
C_o	concentration of nucleation sites in solution	# molecules m^{-3}	4, 5
$\overline{C_{A0}}$	initial reactant concentration	kmol m^{-3}	11

(*cont.*)

(*cont.*)

Variable	Description	Unit	Chapter
c_{eq}, $c_{eq,real}$	solubility; solubility of secondary nuclei with stress in the crystal lattice	kmol m^{-3}	4
c_p	specific heat	kJ kg^{-1}°C^{-1}	3
C_s, C_s*	seed loading ratio defined in eq. 8.5, critical seed loading ratio	–	8
C_v	parameter for eq. 3.8	m s^{-1}	3
CV	coefficient of variation of the CSD	–	2
d, D	tee branch and tee run diameters in T-mixer	m	11
D	molecular diffusivity	m^2 s^{-1}	4, 5
$D(L, t)$, $D(v, t)$	particle death rate	# m^{-3} m^{-1} s^{-1}, # m^{-3} m^{-3} s^{-1}	7, 8
$D_{crystallizer}$	crystallizer diameter	m	3
$D_{DTB\_crystallizer}$	DTB crystallizer diameter	m	3
D_i	turbulent diffusivity	m^2 s^{-1}	11
D_{imp}	impeller diameter	m	3, 4, 11
d_m	molecular diameter	m	4
D_{lin}	linear crystal dissolution rate	m s^{-1}	5
$D_{vapor\text{-}head}$	crystallizer diameter that avoids liquid entrainment in the vaporhead	m	3
D_τ	crystallizer diameter that provides the residence time τ	m	3
E_D,E_R	Arrhenius activation energy for the crystal growth rate parameter, defined in eq. 5.37 and eq. 5.38		5
F	force	J mol^{-1}	1
f	flux of growth units towards a crystal surface	# m^{-2} s^{-1}	5
$f(n)$	frequency of attachment of entities to a cluster of size n	# s^{-1}	4
$f_o(n^*)$	equilibrium frequency of attachment	# s^{-1}	4
$f_o(n^*_{2D})$	frequency of attachment of growth units at the edge of a critical nucleus with a surface area of $2\pi\ r^*_{2D}h$	s^{-1}	5
g	acceleration due to gravity	m s^{-2}	11
g	flux of growth units leaving a crystal surface	# m^{-2} s^{-1}	5
G	Gibbs free energy	J mol^{-1}	1
G	overall crystal growth rate	m s^{-1}	3, 4, 5
$g(n)$	frequency of detachment of entities to a cluster of size n	# s^{-1}	4
$G(L)$	size-dependent component of the overall crystal growth rate in eq. 5.45	–	5
$G(t)$	time-dependent component of the overall crystal growth rate with rate dispersion	m s^{-1}	5
$G(L, t)$, $G(\eta, L, t)$	overall crystal growth rate	m s^{-1}	5
G_{eff}, G_{kin}, G_{att}	effective, kinetic and attrition crystal growth rate, see eq. 4.37.	m s^{-1}	4
G_L	size-dependent overall crystal growth rate	m s^{-1}	5, 7, 8

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Variable	Description	Unit	Chapter
G_v	volumetric growth rate	$m^3\ s^{-1}$	5
h	step height of a growing crystal	m	5
H	enthalpy	$J\ mol^{-1}$	1
H	Vickers hardness		4
H	enthalpy of crystallizer contents	J	3
$h(L)$	classification function	–	7
$H(L)$	secondary nuclei size distribution function, eq. 4.41	–	4
J	nucleation rate	$\#\ m^3\ s^{-1}$	4, 7, 8
J_{2D}	nucleation rate on a crystal surface	$\#\ m^{-2}\ s^{-1}$	5
K	Newton number for impeller, eq. 4.36	–	4
k	Boltzmann constant, 1.3806503×10^{-3}	JK^{-1}	4, 6
k_2	rate constant for two-step nucleation eq. 4.32		4
k_a	particle surface shape factor	–	2,4
K_{att}	parameter for eq. 4.39		4
k_d	mass transfer coefficient of solute in a solution adjacent to a growing or a dissolving crystal, defined in eq. 5.28 and eq. 5.47	$m\ s^{-1}$	5
K_D	massic crystal growth rate coefficient considering diffusion and rough surface disintegration, defined in eq. 5.48	$m\ s^{-1}$	5
k_{d0}	parameter defined in eq. 5.38	$m\ s^{-1}$	5
$k_{distr,i}$	effective distribution coefficient for incorporation of impurity *i* in a particle, molar or massic base	–	2, 3, 12
K_E, $K_{c\text{-}c}$, $K_{c\text{-}i}$	parameters for eq. 4.36		4
k_g	linear growth rate-based crystal growth rate coefficient defined in eq. 5.35 and eq. 5.36	$m\ s^{-1}$	5
K_G	mass-based crystal growth rate coefficient considering diffusion and surface integration, defined in eq. 5.31	$m\ s^{-1}$	5
$k_{m\&h}$	mass-based crystal growth rate coefficient considering diffusion and heat transfer, defined in eq. 5.40	$m\ s^{-1}$	5
k_n	reaction rate constant	$m^3\ kg^{-1}\ s^{-1}$ for first-order reaction	11
k_N, k_N^1	secondary nucleation rate constant, defined in eqs. 4.33 and 4.34		4
k_r	parabolic growth rate constant (eq. 5.10), linear growth rate constant (eqs. 5.11 and 5.21), exponential growth rate constant (eqs. 5.16 and 5.17) or overall surface integration controlled growth rate constant (eq. 5.29)	$[m\ s^{-1}]$, $[m\ s^{-1}]$, $[m\ s^{-1}]$ or $[kg^{-1}\ m^4\ s^{-1}]$	5

(*cont.*)

(*cont.*)

Variable	Description	Unit	Chapter
k_{r0}	parameter defined in eq. 5.37	–	5
$k_{r,d}$	massic dissolution rate by rough surface disintegration	m s^{-1}	5
K_{sp}	solubility product	kmol m^{-3}	
k_v	particle volume shape factor	–	2, 4, 7
L	crystal size, particle size	m	2, 4, 5, 6
$L_{1,0}$	number weighted mean of the CSD	m	2
$L_{2,1}$	length weighted mean of the CSD	m	2
$L_{4,3}$	volume weighted mean of the CSD	m	2, 3
$L_{c\text{-}c}$, $L_{c\text{-}i}$	parameters for eq. 4.36	–	4
L_D	mode of the CSD	m	2
L_F	cut size for fines removal in an MSMPR crystallizer	m	7
L_g	parameter for size dependent growth rate expression, eq. 5.43	–	5
L_i, L_j	sizes of particles of size classes i, j	–	6
$\overline{L_k}$	average of upper and lower bound of the k size interval	m	6
L_M, L_{50}	median size of the CSD	m	2, 10
L_P	cut size for classified product removal in an MSMPR crystallizer	m	7
L_T	total crystal length per unit crystallizer volume	m m^{-3}	2
M	mass	kg	5, 8
m, n	number of streams entering and leaving a crystallizer	–	7
m	molecular mass	kg $kmol^{-1}$	4
$M_{stream,comp}$	mass of a component in a stream. See subscripts for the list of streams and components	kg	4
$m(L)$, $m(L, t)$	mass density distribution	kg m^{-3} m^{-1}	2, 7
$M(L)$	cumulative oversize mass distribution	kg m^{-3}	2
$m+$, $m-$	concentration of the cations, anions	mol L^{-1}	
$M_{B,y}$	yield in melt crystallization	(mol solids) (mol $feed)^{-1}$	12
m_j	jth moment of the crystal size distribution with length as internal coordinate	m^j m^{-3}	4, 6, 7
m_j	jth moment of the crystal size distribution with volume as internal coordinate	m^j m^{-3j}	7
M_T	crystal mass per unit suspension volume	kg m^{-3}susp	2, 3, 4
n	reaction order	–	11
n	parameter for size dependent growth rate expression, eq. 5.43	–	5
N	number of supercritical clusters	–	4
N	stirrer speed	s^{-1}	3,11
$n(L)$, $n(v)$ $n(L, t)$, $n(v, t)$	number density distribution based on particle size, on particle volume	# m^{-3} m^{-1}	2, 5, 6, 7, 8
$N(L)$	cumulative oversize number distribution	# m^{-3}	2

(*cont.*)

Variable	Description	Unit	Chapter
n^*	number of entities in a critical radius cluster	–	4
n_0	number density of nuclei	$\# \ m^{-3}$	4, 5
N_A	Avogadro's number, 6.023×10^{26}	$\# \ k \ mol^{-1}$	4
N_i	number of particles in size class i	$\# \ m^{-3}$	6, 8
N_p	power number of an impeller, also called Newton number, defined by eq. 11.6	–	11
N_q	flow number of an impeller	–	11
N_T	total crystal number per unit crystallizer volume	$\# \ m^{-3}$	2
N_c, N_0	number of crystals and droplets	–	4
Nu	Nusselt number, $Nu = \alpha \ L/\lambda$	–	5
P	pressure	bar	1
P	probability of nucleation	–	4
P	production rate of a crystallizer	(kg crystals) s^{-1}, (kg crystals) $batch^{-1}$	3
P_0	power input of stirrer	W or W (kg $suspension)^{-1}$	4, 7, 11
P_{susp}	minimum power needed to suspend particles in a mixed vessel	W(kg $suspension)^{-1}$	3
q_c	pumping capacity of an impeller	$m^3 \ s^{-1}$	11
q_L	number density of secondary nuclei	$m^{-3} \ m^{-1}$	
Q_{heat}	heat duty to a crystallizer	$J \ s^{-1}$	3
r	ratio between upper and lower bounds of a size interval	–	
r, r^*, r^*_{2D}	cluster radius, critical radius, 2D critical nucleus radius	m	4
r, s	parameters for disruption rate given in eq. 6.17	–	6
R	ideal gas constant	$J \ mol^{-1} \ K^{-1}$	1
$R - 1$	fines-to-product flow ratio in an MSMPR crystallizer	–	7
$r(L_1, L_2)$	rate of collisions or rate of agglomeration	$\# \ m^{-3} \ s^{-1}$	6, 7
$r(v_1, v_2)$	rate of collisions or rate of agglomeration	$\# \ m^{-3} \ s^{-1}$	6, 7
r_0	radius of the molecule	m	
r_{agg}	agglomeration rate	$\# \ m^{-3} \ s^{-1}$	6
R_A	mass-based crystal growth rate	$kg \ m^{-2} \ s^{-1}$	5
R_{HD}	crystallizer height-to-diameter ratio	–	3
$r_{i,j}$	collision rate between particles from interval i and j	$\# \ m^{-3} \ s^{-1}$	6
R_{lin}	average linear crystal growth rate	$m \ s^{-1}$	5
$\mathrm{r_c}$	length scale below which linear elasticity theory breaks down	[m]	5
S	entropy	$J \ mol^{-1} \ K^{-1}$	1
S	supersaturation ratio	–	4, 6
$S(v)$	selection function for particle breakage	s^{-1}	7
Sh	Sherwood number, $Sh = k_d L D^{-1}$	–	

(*cont.*)

(*cont.*)

Variable	Description	Unit	Chapter
T	temperature	°C or K	1, 5, 6
$T - T_{eq}$	undercooling	K	1, 4, 9, 12
T_R	roughening temperature	K	5
t, t_i	time or residence time, induction time	s	3, 4, 5
u	fluid velocity	$m\ s^{-1}$	11
v, V	side stream and main stream velocity of a T-mixer	$m\ s^{-1}$	
V	particle volume	m^3	2
$V, V(t)$	suspension volume	m^3	4, 7, 8, 11
$v(L)$	volume density distribution	$m^3\ m^{-3}\ m^{-1}$	7
V_A, V_R, V_{TOT}	interaction potential between particles: attractive, repulsive and total	J	6
$\dot{V}_{att}, V_{att,ij}$	volumetric attrition of all crystals and of crystals of size i with impeller section j	$m^3\ m^{-3}\ s^{-1}$	4
$v_{coll,ij}$	collision velocity of a crystal with an impeller	$m\ s^{-1}$	4
V_{cryst}	crystallizer volume, eq. 4.43	m^3	4
V_M	molecular volume	m^3 molecule^{-1}	4, 5
V_{macr}	detectable macroscopic volume	m^3	4
v_{max}	allowable vapor velocity in crystallizer head	$m\ s^{-1}$	3
v_{ss}	settling velocity of a particle in a suspension	$m\ s^{-1}$	3
v_{step}	step velocity of a growing crystal	$m\ s^{-1}$	5
v_{sup}	superficial velocity of a fluid in a pipe	$m\ s^{-1}$	3
V_T	crystal volume per unit suspension volume	$m^3\ m^{-3}$	2, 7
v_{tip}	tip speed of stirrer	$m\ s^{-1}$	3
w	solute mass fraction	kg (kg solution)$^{-1}$	1
W^*	nucleation work	J	4
w_{eq}	solubility in mass fraction	kg (kg solution)$^{-1}$	2, 3
W_i	stress energy of one crystal	J	5
$W_{p,ij} W_{p,min}$	energy of the impact of a crystal with an impeller, eq. 4.44. minimum impact energy, eq. 4.47	J	4
$w_{phase,comp}$	mass fraction of component in a given phase of the crystallizer product stream	(kg i) (kg solid)$^{-1}$	3
W_{stir}, W_{pump}	electrical energy costs for stirring and recirculating in a continuous crystallizer	W	3
$w_{stream,phase,comp}$	mass fraction of component in a given phase of a stream. The stream is usually a suspension	(kg component) (kg phase)$^{-1}$	3
$w_{stream,comp}$	mass fraction of component or solvent in the liquid phase of the stream	(kg solvent) (kg solution)$^{-1}$	3
x	impurity mol fraction in liquid phase	(mol impurity) (mol solution)$^{-1}$	12
y	impurity mol fraction in crystalline phase	(mol impurity) (mol crystal)$^{-1}$	12

(*cont.*)

Variable	Description	Unit	Chapter
z	Zeldovitch factor	–	4, 5
z-1	ratio between the volumetric flow rate of the classifier recycle to the classifier product	–	7
$Z(n, t)$	number concentration of clusters with n solute entities	–	4
$Z_{i,j}$	number of crystals of size class i that collide with the impeller segment j	# m^{-3} s^{-1}	4

Greek letters

Variable	Description	Unit	Chapter
α	recycle ratio, liquid recycle flow per unit liquid flow out of the crystallizer	–	3
α	heat transfer coefficient	W m^{-2} K^{-1}	5
β	parameter in correlation for interfacial tension in eq. 4.10. Value 0.414	–	4
β	collision frequency kernel for agglomeration	m^3 $\#^{-1}$ s^{-1}	6
β_0	Size independent part of agglomeration kernel	–	6
β_1	Size dependent part of agglomeration kernel	m^3 $\#^{-1}$ s^{-1}	6
β_{agg}	agglomeration rate constant or agglomeration kernel	m^3 s^{-1}	6, 7
β_{disr}	disruption kernel	m^3 s^{-1}	6
δ, δ_h	boundary layer thickness for mass transfer and for heat transfer around a growing crystal	M	5
γ	activity coefficient	–	1
γ	particle surface energy	J m^{-2}	2
γ, γ_{eff}	interfacial free energy for homogeneous and heterogeneous primary nucleation	J m^{-2}	4
γ_{edge}	edge free energy of a growing crystal	J	4
λ	thermal conductivity	W m^{-1} K^{-1}	5
λ_0	step distance of a growing crystal	M	5
Λ	macroscale of turbulence	M	11
Γ	shear rate	s^{-1}	6
Γ / K_r	fracture resistance	–	4
ΔG	Gibb's free energy of phase change	J $molecule^{-1}$	2, 4
ΔG^*, $\Delta G_2{}^*$	energy barrier for nucleation from solution and for nucleation inside a cluster	J $molecule^{-1}$	4
ΔH_{evap}	enthalpy of evaporation	kJ kg^{-1}	3
ΔH_{cr}	enthalpy of crystallization	kJ kg^{-1}	5

(*cont.*)

(*cont.*)

Variable	Description	Unit	Chapter
Δm	mass of crystals formed in a batch	kg	8
ΔP	pressure drop around the circulation pump of a continuous crystallizer	Pa	3
$\Delta T_{hex,max}$	allowable temperature elevation of the suspension as it passes through the circulation heater of a crystallizer	°C	3
$\Delta\mu$	supersaturation	J mol^{-1} or J molecule^{-1}	4, 5, 9
ε	specific power input from impeller, energy dissipation rate	Wkg^{-1}	4, 6, 11
ε	volume fraction of liquid in a suspension	(m^3 liquid) (m^3 suspension)$^{-1}$	3, 7
η	mosaic spread associated with distortion of the crystal lattice	rad	5
η	Kolmogorov microscale of turbulence, defined by eq. 6.15	m	6
$\eta_{survival}$	secondary nuclei survival efficiency, eq. 4.42	–	4
$\eta_{geometric,j}$, $\eta_{target,ij}$	geometric and target efficiency parameters for eq. 4.42	–	4
$\eta(C_1, T)$	viscosity inside the clusters in two-step nucleation	–	4
θ	contact angle between a nucleus and a foreign substrate	°	4
μ	chemical potential	J mol^{-1}	1, 4, 9
μ	dynamic viscosity	Pa.s	3, 6
μ	shear modulus		4, 5
ν	kinematic viscosity	m^2 s^{-1}	6, 11
ν_0	volume occupied by a molecule in the cluster	m^3	
ρ	density	kg m^{-3}	2, 3, 4, 5, 11
$\rho_{feed,liquid}$	liquid density in the feed stream	(kg liquid) (m^3 liquid)$^{-1}$	3
$\rho_{crystal}$, ρ_{liquid}	density of the crystalline and the liquid phase respectively	kg m^{-3}	3, 5
σ	standard deviation		2
σ	relative supersaturation	–	3
τ	residence time	s	3, 5, 7
τ_P, τ_L	residence time of product crystals and liquid in an MSMPR crystallizer	s	7
τ_{macro}, τ_{meso}, τ_{micro}	macromixing, mesomixing and micromixing time	s	11
τ_c, τ_d	circulation time, time for turbulent dispersion	s	11
τ_e, τ_R	time constant for engulfment and for chemical reaction	s	11
υ	Poisson ratio	–	5

(*cont.*)

Variable	Description	Unit	Chapter
$\varphi_{crystal}$	volume fraction of crystals	–	4
φ_2	cluster volume fraction	–	4
$\varphi_{H,stream}$	enthalpy of a stream	J s^{-1}	3
$\varphi_{v,prod}$	volumetric production rate of a crystallizer	m^3 suspension s^{-1}	3
$\varphi_{v,stream}$	volumetric flow rate of a stream	m^3 s^{-1}	3
$\varphi_{hex,max}$	allowable heat flux in circulation heat exchanger of evaporative crystallizer	J m^{-2}	3
Ψ	overall agglomeration efficiency of collisions between particles	–	6
ψ_{profit}	profit of a crystallization unit	€ $year^{-1}$	3
$\psi_{capital}$	capital costs	€ $year^{-1}$	3
ψ_{mb}	economic material balance costs and operation costs	€ $year^{-1}$	3
ψ_{oper}	economic material balance		
ξ	particle volume in population balance for agglomeration	m^3	7

Subscripts

0,e	start and end of a batch	3
b	bulk	2
circ	circulation stream between crystallizer body and heater in an evaporative crystallizer	3
cla	clear liquor advance stream in an MSMPR	7
crystal	crystal phase	2, 3, 7
eq	equilibrium	1, 2
feed	feed stream to a crystallizer	3
final	final condition in a batch	3, 8
hetero	heterogenenous primary nucleation	4
homo	homogeneous primary nucleation	4
i	interface	5
i	a component	3, 7
in	inflow stream	7
init	initial condition in a batch	3, 8
liquid	liquid phase	3
out	outflow stream	7
product	product stream	3
solvent	a component	3
seed	seeds to batch crystallizer	8
solid	solid phase (for multi-component crystals)	7
susp	suspension (slurry)	1, 3
tank	mixing vessel	6
vapor	vapor stream leaving a crystallizer	3

Streams: crystallizer feed, crystallizer product flow. Phases: liquid and crystal. Components: *i*, solvent.

Abbreviations

CFD	computational fluid dynamics
CSD	crystal size distribution
DT	draft tube crystallizer
DTB	draft tube baffle crystallizer
FC	forced circulation crystallizer
MSMPR	mixed suspension mixed product removal
ODE	ordinary differential equation
PF	plug flow reactor
PSD	particle size distribution