

The background of the cover is a microscopic image showing a dense field of blue, crystalline structures. These structures appear as intricate, branching, and somewhat fibrous patterns, characteristic of a crystallization process. The overall color is a vibrant blue with varying shades and textures.

# **Crystallization Process Systems**

**Alan G. Jones**



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# Crystallization Process Systems

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## Preface

Crystallization from solution is a core technology in major sectors of the chemical process and allied industries. Crystals are produced in varying sizes ranging from as small as a few tens of nanometers to several millimetres or more, both as discrete particles and as structured agglomerates. Well-established examples include bulk and fine chemicals and their intermediates, such as common salt, sodium carbonate, zeolite catalysts and absorbents, ceramic and polyester pre-cursors, detergents, fertilizers, foodstuffs, pharmaceuticals and pigments. Applications that are more recent include crystalline materials and substances for electronics devices, healthcare products, and a wide variety of speciality applications. Thus, the tonnage and variety of particulate crystal products worldwide is enormous, amounting to about half the output of the modern chemical industry. The economic value, social benefit and technical sophistication of crystal products and processes are ever increasing, particularly in the newer high added value sectors of global markets. This places yet greater demands on the knowledge, skill and ingenuity of the scientist and engineer to form novel materials of the required product characteristics and to devise viable process engineering schemes for their manufacture.

Particulate crystallization processes often require subsequent solid–liquid separation. Thus, the unit operation of crystallization is normally only part of a wider processing system. These systems should preferably be designed and optimized as a whole – problems detected in one part of the plant (poor filtration say) may in fact arise in another (inadequate crystallizer control). Attention to the latter rather than the former can result in a simpler, cheaper and more robust solution. Similarly, the scale of crystallizer operation can have a large effect on crystal product characteristics and hence their subsequent separation requirements. Previously a largely empirical art, the design of process systems for manufacturing particulate crystals has now begun to be put on a rational basis and the more complex precipitation processes whereby crystallization follows fast chemical reactions have been analysed more deeply. This progress has been aided by the growing power of the population balance and kinetic models, computational fluid dynamics, and mixing theory. This not only increases understanding of existing processes but also enhances the possibility of innovative product and process designs, and speedier times to market. Several large gaps in knowledge remain to be filled, however, thereby providing opportunities for further research. This perspective gives the reason for writing the book, and provides its theme.

*Crystallization Process Systems* brings together essential aspects of the concepts, information and techniques for the design, operation and scale up of particulate crystallization processes as integrated crystal formation and solid–liquid separation systems. The focus of the book, however, is on crystallization; only dealing with related unit operations as far as is necessary. It is therefore

not intended to be comprehensive but is designed to be complementary to existing texts on the unit operations of crystallization, solid–liquid separation and allied techniques, and important sources of further detailed information are given for the interested reader. The work is presented initially at an introductory level together with examples while later providing a window into the details of more advanced and research topics. Particular attention is paid both to the fundamental mechanisms and the formulation of computer aided mathematical methods, whilst emphasizing throughout the continuing need for careful yet efficient practical experimentation to collect basic data; the latter being essential in order to discriminate between competing theories, to inform and validate process models, and to discover the unexpected.

The book is mainly based on undergraduate BEng and MEng, and graduate MSc lecture courses that I have given at UCL since 1980 on Particulate Systems and Crystallization, respectively, together with those on a continuing professional development (CPD) course on Industrial Crystallization. To these is added the results of several research projects. Consequently, the book is aimed equally at students, researchers and practitioners in industry, particularly chemical engineers, process chemists and materials scientists.

The book is divided into 9 chapters. For the guidance of lecturers and students, Chapters 1–4 largely comprise the earlier undergraduate taught topics together with some enhanced material; with Chapters 4–8 mainly covering the more advanced and research topics. Chapter 9 should be of particular help to those undertaking crystallization process design projects. Thus, Chapter 1 provides the definition of the basic characteristics that are common to all particulate crystals, notably their molecular structure, particle size, size distribution and shape. This is followed in Chapter 2 by the relevant transport processes of chemical engineering as applied to crystallization, namely the hydrodynamic factors affecting crystallizers and solid–liquid separation equipment, viz. the motion of single particles, slurries and suspensions in both agitated and quiescent vessels, and flow through porous media, respectively. Then follows a more advanced description of mixing models, the unified population balance approach to the analysis and prediction of particle size distributions and its coupling with fluid flow. Having provided the theoretical basis, these chapters naturally lead on to an introduction to the fundamentals and techniques of crystallization and related precipitation processes *per se* in Chapter 3. As mentioned above, the next process stage is normally solid–liquid separation and drying. Thus, simple procedures for the design and operation of gravity settlers, thickeners, filters, centrifuges and driers are considered relatively briefly in Chapter 4, this time in the light of the underlying solid–liquid transport theory covered in Chapter 2. Particle formation processes occurring within crystallizers, viz. nucleation, crystal growth, agglomeration and breakage, together with methods for their determination, are then considered in greater depth in Chapters 5 and 6. Then in Chapter 7, the design and performance of well-mixed batch and continuous crystallizers is considered with the population balance from Chapter 2 underlying their theoretical analysis. Chapter 8 extends these considerations to crystallization systems where imperfect fluid–particle-mixing is an important factor, and is illustrated in detail in

respect of the scale up of precipitation processes. Finally, in Chapter 9 the design of crystallization process systems is considered as a whole, bringing together the essential features of the analysis of particle formation and solid–liquid separation processes described earlier with the logic of process synthesis, optimization and control, again illustrated by some examples.

I am indebted to John Mullin for his encouragement during the writing of this book. Many other academic and industrial colleagues are also worthy of thanks, together with visitors and research students in the UCL crystallization group over the last two decades. Too numerous to mention individually, they each contributed much for which I am most grateful. I hope that each will see some of their many contributions reflected with due acknowledgement in the text. Thanks are also due to present and former students for their patience in working through the examples and giving feedback on courses. Finally, but most importantly, my special thanks are due to Judith, Robert and Stephen for their continual support during the preparation of the manuscript.

Alan Jones  
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*October 2001*

## Nomenclature and abbreviations

$a_i$	coefficients
$A$	area, $m^2$
$a_L$	specific surface area based on liquid volume, $m^{-1}$
$a_R$	specific surface area based on reactor volume, $m^{-1}$
$B^0$	nucleation rate, $\#/m^3 s$
$B_a$	birth rate of particles due to aggregation, $\#/m^3 s$
$B_d$	birth rate of particles due to disruption, $\#/m^3 s$
$B_I$	birth rate in region I, $\#/m^3 s$
$B_{II}$	birth rate in region II, $\#/m^3 s$
$B_L$	source function, $\#/m^3 s$
$B_n$	birth rate due to nucleation, $\#/m^3 s$
$c$	solution concentration, kg anhydrous solute/kg solvent
$c^*$	equilibrium saturation concentration, kg anhydrous substance/kg solvent
$C$	cooling rate ( $d\theta/dt$ ), $K s^{-1}$
$C_p$	specific heat of solution, $kJ kg^{-1}$
$Cr$	contraction factor at the inlet of riser
$\Delta c$	concentration driving force ( $c - c^*$ ), kg anhydrous substance/kg solvent $^{-1}$
$d$	diameter, m
$D_a$	death rate of particles due to aggregation, $\#/m^3 s$
$d_B$	bubble diameter, m
$D_d$	death rate of particles due to disruption, $\#/m^3 s$
$D_d$	rate of particle death due to disruption, $\#/m^3 s$
$d_L$	droplet diameter, m
$D_M$	molecular diffusivity, $m^2 s^{-1}$
$D_p$	particle diffusivity, $m^2 s^{-1}$
$D_a$	death rate of particles due to aggregation, $\#/m^3 s^{-1}$
$D_d$	death rate of particles due to disruption, $\#/m^3 s^{-1}$
$E$	energy of contact, kJ
$E_L$	longitudinal dispersion coefficient of liquid, $m^2 s^{-1}$
$\dot{E}_t$	rate of energy transfer to crystals by collision, $kJ s^{-1}$
$f$	mixture fraction for the non-reacting fluid = $c_A^0/c_{A0}$
$f(\alpha)$	fraction of crystals exiting CFR at point $\alpha$
$f_{ij}$	fraction of crystals exiting discretized CFR at point $\alpha_{ij}$
$f_s$	surface shape factor (area = $f_s L^2$ )
$f_v$	volume shape factor (volume = $f_v L^3$ )
$f$	collision frequency, exchange rate between zones I and II
$F$	crystal bed permeability, $m^{-2}$
$F$	flow rate of gas, $m^3 s^{-1}$
$F$	fraction of solvent evaporated, kg solvent evaporated/kg original solvent
$G$	linear growth rate, $m s^{-1}$
$G'$	mean velocity gradient, fluid shear rate, $s^{-1}$



$g$	gravitational acceleration, $\text{m s}^{-2}$
$H$	column height, m
Ha	Hatta number
$h_c$	enthalpy of crystallization, $\text{kJ kg}^{-1}$
$h_g$	enthalpy of vaporization, $\text{kJ kg}^{-1}$
$J_n$	primary nucleation rate, $\#/m^3 \text{ s}$
$J_s$	secondary nucleation rate, $\#/m^3 \text{ s}$
$k$	Boltzmann constant, $1.38054 \times 10^{-22} \text{ J K}^{-1}$
$K$	Kozeny coefficient
$k$	reaction rate constant, $\text{m}^3 \text{ mol}^{-1} \text{ s}^{-1}$
$K_a$	aggregation constant
$k_b$	number nucleation rate coefficient
$K_d$	disruption constant
$k_g$	crystal growth rate constant
$k_i$	kinetic rate coefficients
$k_L$	mass transfer coefficient, $\text{m s}^{-1}$
$k_n$	nucleation rate constant
$k_{sp}$	solubility product
$k'_g$	mass growth rate coefficient
$k_m$	mass nucleation rate coefficient
$K_N$	rate coefficient
$K_R$	rate coefficient
$L$	characteristic dimension, size co-ordinate, crystal size, m
$L_0$	size of nuclei, m
$L_{so}$	initial size of seed crystals, m
$L_p$	size of product crystals, m
$M_c$	mass of crystals, kg
$M_l$	mass of liquor in crystallizer, kg
$M_g$	mass of solvent evaporated, kg
$M_h$	mass solvent in crystallizer, kg
$M_{anh}$	molecular mass of anhydrous substance, $\text{kg kmol}^{-1}$
$M_{hyd}$	molecular mass of hydrated substance, $\text{kg kmol}^{-1}$
$M_p$	product crystal mass, kg
$M_{so}$	initial mass of seed crystals, kg
$M_T$	suspension, or magma, density, $\text{kg m}^{-3}$
$n(L, t)$	population density distribution function, $\#/m^4$
$n^0$	nuclei population density ( $\text{m}^{-1} \text{m}^{-3}$ )
$N$	total number of particles, $\#/m^{-3}$
$N_0$	initial total number of particles, $\#/m^{-3}$
$N_a$	agitator speed, Hz
$N_{so}$	initial number of seed crystals, $\#/kg \text{ solvent}$
$n_C$	number of crystals, $\#/m^{-3}$
$n_I$	number density of crystals in region I, $\#/m^4$
$n_{II}$	number density of crystals in region II, $\#/m^4$
$P$	crystal production rate, $\text{kg s}^{-1}$
$P$	degree of agglomeration
$p$	population density of particles, $\#/m^4$

$P_0$	power number
$p_b$	pressure at bottom, Pa
$P_1$	power input, W
$P_N$	power number
$p_t$	pressure at top, Pa
$q(\alpha)$	fraction of feed entering the system at point $\alpha$
$q_{ij}$	fraction of feed entering discretized CFR at point $\alpha_{ij}$
$Q$	volumetric throughput, $\text{m}^3 \text{s}^{-1}$
$Q$	heat input, kJ
$Q_0$	pumping (flow) number
$Q_m$	mol flow rate, $\text{mol s}^{-1}$
$r$	rate of chemical reaction, $\text{mol/m}^3/\text{s}$
$R$	universal gas constant, $8.315 \text{ J mol}^{-1} \text{ K}^{-1}$
$R$	ratio of molecular masses ( $M_{\text{hyd}}/M_{\text{anh}}$ )
Re	Reynolds number
$S$	relative supersaturation
$S$	co-ordinate of crystal number
Sc	Schmidt number
Sh	Sherwood number
$S_p$	bed specific surface area, $\text{m}^{-1}$
$t$	time, s
$T$	temperature, K
$u, v$	particle volume, $\text{m}^3$
$u_{\text{pi}}$	local instantaneous value of particle velocity
$V$	volume, $\text{m}^3$
$x$	distance from gas–liquid interface, m
$X$	solids moisture content
$Y$	dimensionless crystal size, $(L_p - L_{\text{so}})/L_{\text{so}}$
$Y$	gas humidity
$Y$	Young's modulus of elasticity, $\text{N m}^{-2}$
$Z$	dimensionless time, $t/\tau$

### Superscripts

$b$	'order' of nucleation
$c$	shear rate dependence of maximum agglomerate size
$g$	'order' of growth
$i$	relative kinetic order ( $=b/g$ )
$j$	magma density dependence of nucleation rate
*	equilibrium value

### Subscripts

$c$	crystal
$f$	final, at time $\tau$
$g$	growth
$I$	instantaneous
$i$	summation index
$j$	summation index

l	liquor (solution)
mm	mass mean
n	nuclei
0	initial, or zeroth
s	speed
t	time, $t$
wm	weight mean

**Greek**

$\alpha$	time along the length of a network
$\beta_a$	aggregation kernel, $\text{m}^3 \text{s}^{-1}$
$\beta_d$	disruption kernel, $\text{m}^3 \text{s}^{-1}$
$\delta(L - L)$	Dirac delta function of crystal size, m
$\varepsilon$	voidage fraction
$\varepsilon$	specific power input, $\text{W m}^{-3}$
$\dot{\gamma}$	shear rate, #/s
$\mu$	fluid viscosity, Pa s
$\mu_j$	$j$ th moment of size distribution
$\phi(f)$	probability density function for the statistics of fluid elements
$\nu$	kinematic viscosity, $\text{m}^2 \text{s}^{-1}$
$\rho$	fluid density, $\text{kg m}^{-3}$
$\rho_c$	crystal density, $\text{kg m}^{-3}$
$\rho_L$	liquid density, $\text{kg m}^{-3}$
$\rho_s$	particle density, $\text{kg m}^{-3}$
$\sigma$	Poisson's ratio, absolute or relative supersaturation
$\tau$	mean residence time in vessel, s
$\phi$	coefficient
$\theta$	solution temperature, K
$\tau$	batch time, mean residence time in vessel, s
$\psi$	coefficient
$\eta_T$	impeller target efficiency

**Others**

$\langle \rangle$	ensemble average
—	average or mean value
#	number of particles or crystals

**Abbreviations**

ATR	Attenuated Total Reflection
CSD	Crystal Size Distribution
CV	Coefficient of Variation
FTIR	Fourier Transform Infra Red
HSE	Health, safety and environment
MFB	Micro Force Balance
MSMPR	Mixed Suspension, Mixed Product Removal
PDF	Probability Density Function
PSD	Particle Size Distribution