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CRYSTALLIZATION AND CRYSTAL GROWTH RATE: THE EXAMPLE OF MONOHYDRATE CITRIC ACID CRYSTAL GROWTH

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Summary

Liquid fluidization techniques offer many advantages to crystallization processes. The crystals move uniformly and there are no shocks between them. More than ten thousand crystals of monohydrate citric acid were grown in a transparent fluidizing column of 94 mm diameter. Their characteristic sizes range from 1.12 mm to 4.62 mm. The temperature of each run was held at 25 °C.

All the results presented obey the following correlation

$$Sh = 0.21\ Re^{0.18}\ Ga^{0.31}\ Sc^{1/3}$$

and the growth rate was of an overall first order with respect to supersaturation.

Introduction

Crystallization can be defined as the process where by a solid phase is deposited from a liquid or gaseous solution, or from a pure vapor or melt. It must surely be considered the oldest unit operation in chemical engineering. Today many sections of the chemical industry use crystallization as a method of production, purification or recovery of solid material. Apart from the fact that the final product has an attractive appearance, crystallization is frequently the cheapest and sometimes the easiest way to produce a pure substance.

However, despite the large number of advances in technology in recent years, crystallization is often considered a part of empiricism. Nevertheless, there is a growing trend now to study crystallization problems using both empirical and theoretical approaches.

General Processes of Crystallization

The deposition of a solid crystalline phase can only occur if the solute concentration in the solution is greater than its own solubility in the solvent under the same condition of temperature. In this case the solution is said to be supersaturated[1,2]. The attainment of the supersaturated state is essential for any crystallization operation. Supersaturation can be achieved in two main ways: by thermal processes or by chemical processes[2,3]. In thermal processes, the supersaturation is achieved by cooling the solution, by solvent evaporation or by adiabatic vacuum cooling of the solution which consists of flashing off the solvent into a vessel maintained at low pressure. In chemical processes, a foreign substance, which is generally a liquid, is added into the solution to modify the equilibrium in a physico-chemical or purely chemical way.

However, the supersaturated state alone is not a sufficient condition for a system to begin to crystallize. If the system does not initially contain any crystals, a number of minute solid bodies has to be formed before the crystals can grow. This phenomenon is called nucleation, and it only occurs if some degree of supersaturation has been achieved. This critical degree of supersaturation is called supersolubility of the solute in the solvent. Supersolubility curve is not well defined. It depends on numerous parameters such as the nature of the solvent and solute, the cooling or evaporating rate to produce supersaturation, hydrodynamic conditions, presence of liquid or solid impurities, mechanical shocks, thermal shocks, pH, and more generally on the history of the solution[1,2]. Since the system is not in an equilibrium state, it is very important to note that supersolubility is not a physical property as, for example, solubility.

The formation of nuclei results from a very complicated process which is yet not well explained. However it is thought that molecular clusters can be formed in the solution by collisions of molecules with one another. Therefore short chains may be formed and eventually the lattice structure of the crystals is built up. The formation of nuclei requires sufficiently high chemical potential of the solute in the solution. This fact explains why nucleation cannot occur if supersaturation is not sufficiently high.

As soon as nuclei have been formed in a supersaturated system, they begin to grow into crystals. The nucleation step can be avoided by seeding the solution with small crystals. Many attempts have been made to explain the mechanism and the rate of crystal growth and there may be broadly classified under three general headings: surface energy, adsorption layer and diffusion theories[1].

According to the Burton–Cabrera–Frank model, the growth of the crystals involves linking of molecules to the edge of screw dislocations standing on the surface. The growth rate obeys the equation:

$$R_g = B_1\sigma^2 \tanh (B_2/\sigma) \qquad (1)$$

where R_g is the weight of crystals deposited per unit time per unit surface area. B_1 and B_2 are complex temperature dependent constants and σ is the degree of supersaturation.

Diffusion theories are more practical[3,4]. It is generally accepted that crystal growth results from two steps. Firstly, a diffusion step whereby solute molecules are transported from the bulk solution to the solid surface. This step is defined by the following equation:

$$\frac{dm_c}{dt} = k_d\ a_c\ (C - C_i) \qquad (2)$$

Secondly, a surface reaction step in which molecules arrange themselves into the crystal lattice. This step is defined by the following equation,

$$\frac{dm_c}{dt} = k_r\ a_c\ (C_i - C)^r \qquad (3)$$

dm_c/dt	is the increase of the mass of the crystal per unit time
k_d and k_r	are respectively the mass transfer coefficient and the surface reaction constant
a_c	is the crystal surface area
C	is the solute concentration in the bulk solution
C_i	is the solute concentration at the crystals-solution interface
C^*	is the equilibrium saturation concentration
r	is the order of the surface reaction.

The equations 2 and 3 are not easy to apply in practice because they involve interfacial concentration C_i that cannot be measured. It is more convenient to consider an overall concentration driving force $C - C^*$ which is quite easily measured. A general equation for crystallization based on this overall driving force can be written as

$$\frac{dm_c}{dt} = k_g\ a_c\ (C - C^*)^g \qquad (4)$$

where k_g is the overall growth coefficient and g is the overall order of growth. It may be different from r. Two limiting cases can occur:

a) the surface reaction is extremely rapid so that the process is controlled only by the diffusional step. In this case $C_i \simeq C^*$ and

$$\frac{dm_c}{dt} = k_d\ a_c\ (C - C^*) = k_g\ a_c\ (C - C^*)^g \qquad (5)$$

Thus $g = 1$, and $k_d = k_g$

b) the diffusion step is very much faster than the reaction step so that the entire process is controlled by the surface reaction step. In this case $C_i \simeq C$

$$\frac{dm_c}{dt} = k_r\ a_c\ (C - C^*)^r = k_g\ a_c\ (C - C^*)^g \qquad (6)$$

Thus $g = r$, and $k_r = k_g$

In all other cases, k_g is a more or less complex function of k_d and k_r. The growth rate depends on the supersaturation of the solution, on its temperature and generally on the crystal-solution relative velocity and also on the crystal size. Moreover it may also be influenced by crystal defects and impurities in the solution. From theory, the value of the overall order g varies between 1 and 2.

Expression for and Measurements of Crystal Growth Rates

Crystal growth rates can be expressed in different manners:

a) as a mass deposition rate dm_c/dt

b) as a mass deposition rate per unit surface area

$$R_g = \frac{1}{a_c} \cdot \frac{dm_c}{dt}$$

and for N crystals, $R_g = (1/A)\ dM/dt$
where $M = Nm_c$ and $A = Na_c$

c) as a face displacement linear velocity in the perpendicular direction or as an overall linear growth rate, $G = dL/dt$ corresponding to the rate of increase of some characteristic length L of the crystal.

This characteristic length can be chosen as the equivalent diameter of the sphere having the same volume as the crystal, or having the same surface area as the crystal, or even as the sieve which just permits the particle to pass though.

It is then convenient to consider two shape factors:

1. the volume shape factor defined by

$$m_c = \alpha\, \rho_c\, L^3 \tag{7}$$

where ρ_c is the density of the crystal.

2. the surface shape factor β which is defined by

$$a_c = \beta\, L^2 \tag{8}$$

Thus, all these rates are linked by the following relations

$$R_g = \frac{1}{a_c} \cdot \frac{dm_c}{dt} = k_g\ \Delta\ C^g = 3\ \frac{\alpha \rho_c}{\beta}\ G \tag{9}$$

Many different techniques have been employed to facilitate crystal growth rate measurements[3,4]. They include face growth rate of a single crystal fixed in a moving solution or in an agitated vessel, or rotated in a stagnant solution, and overall growth rates measured in multiparticle systems in agitated vessels and fluidized bed.

Liquid fluidization exhibits many advantages for mass transfer operations which respect to mechanical agitation[4,5]. The flow pattern of the liquid is generally considered as the piston flow so that a higher mass transfer driving forces than that produced in agitated vessel can be maintained between the two phases. Moreover, attrition may be reduced because collisions between crystals with one another or with the walls in fluidized bed are less violent than shocks between crystals and the impellers of the stirrer.

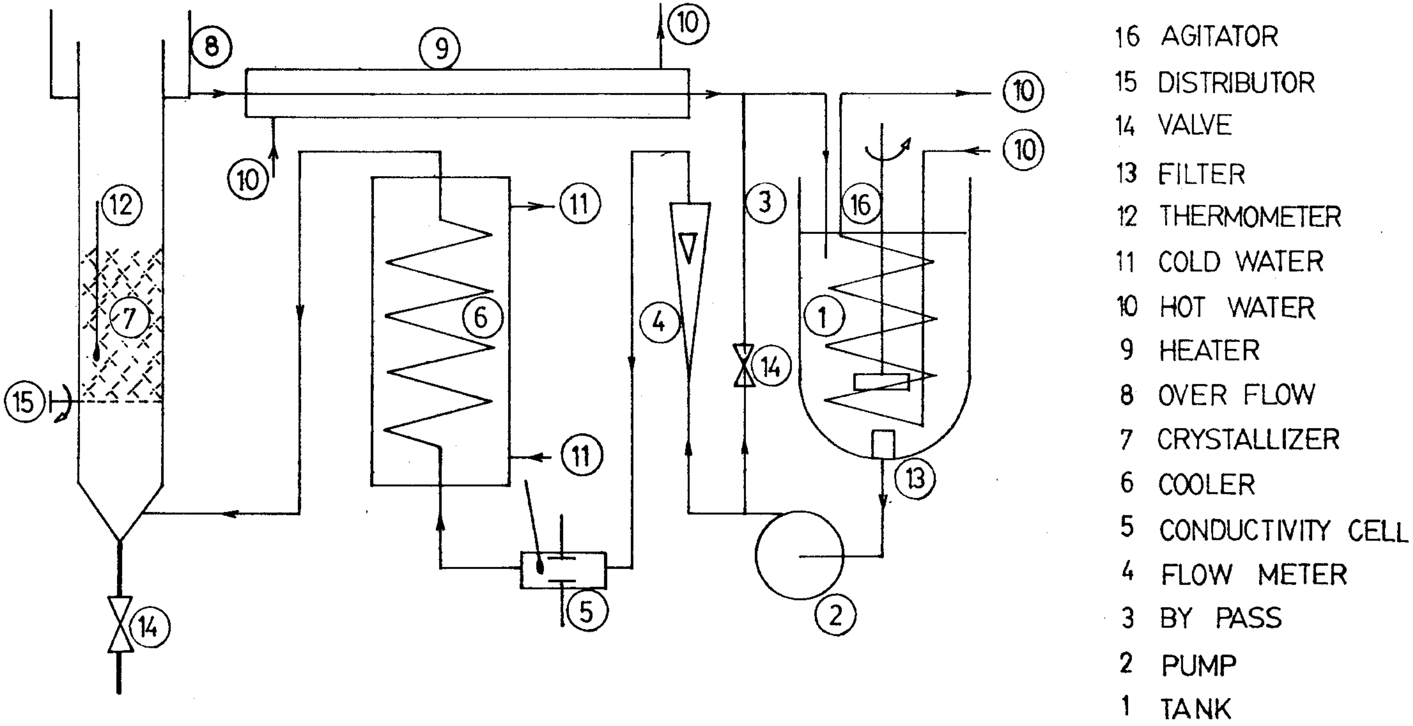

Fig. 1. Diagram of the crystallization apparatus.

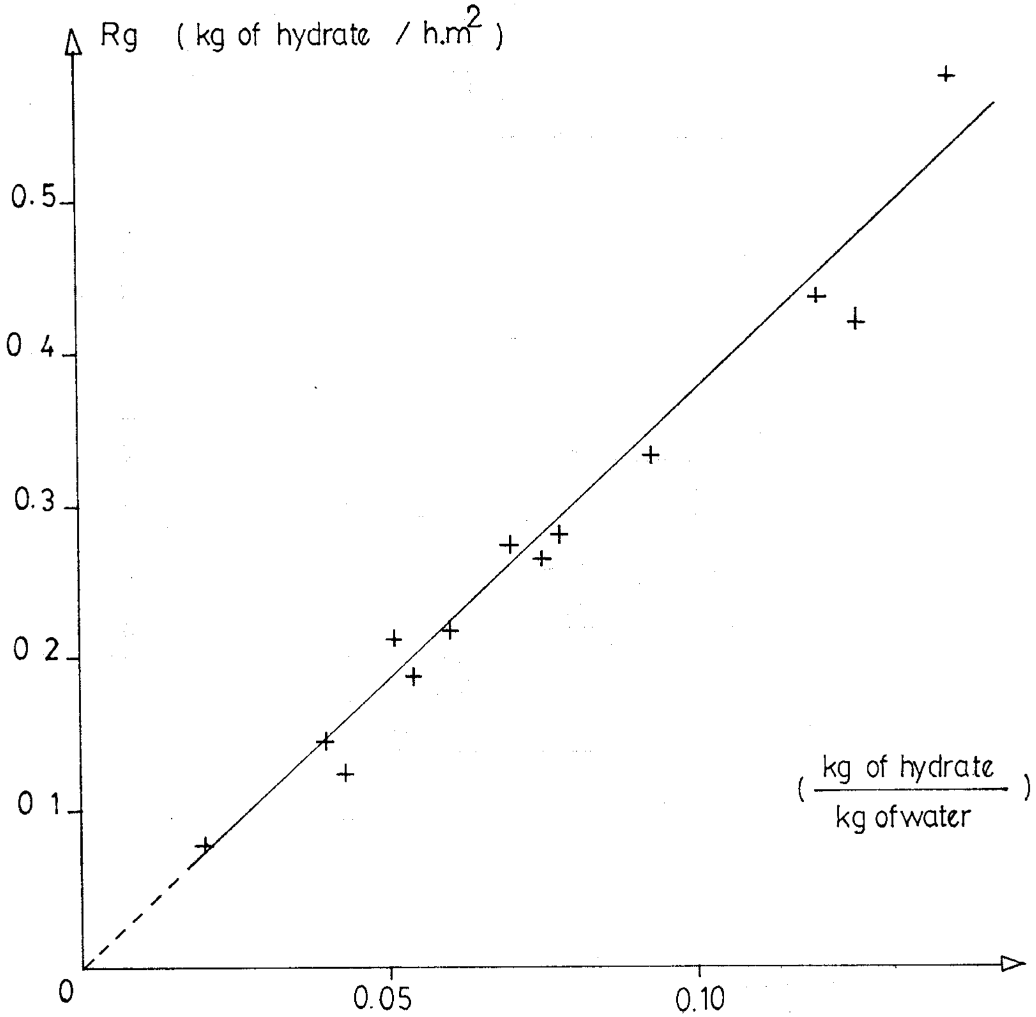

Fig. 2. Relationship between mass of crystals deposited per hour per unit surface area (R_g) and supersaturation of the solution.

Apparatus and Method

The apparatus is shown schematically in Fig. 1. The main part is the crystallizer. It consists of a perspex column of 94 mm in diameter with a conic section for its lower part and a symmetrical overflow on its upper part. The distributor is a 15 mm thick perforated plate which can move about its diameter horizontal axis so that magma can be removed through the column basis. The solution was circulated by means of a small stainless steel centrifugal pump. The flow rate could be adjusted by means of a bypass and was measured by a flow meter. A stirred tank of 35 litres was maintained at 30 °C by a coil in which hot water was circulated and this provided a nearly constant concentration of the column inlet solution during the experiment.

Supersaturation is achieved by cooling the solution in a coiled heat exchanger. The bed temperature is indicated on a thermometer graduated to 0.1 °C and the cooling water flow is regulated to maintain a temperature of about 25 °C. A solution of desired concentration was prepared by adding either citric acid or water into the stirred tank. Crystals were sieved, weighed and numbered by taking samples of three hundred to five hundred crystals. They were then introduced at the top of the column. The inlet concentration was continuously measured by means of a conductimeter while the outlet concentration was determined by titration with caustic soda. At the end of each experiment, the crystals were filtered, washed with ethyl ether, air dried, weighed and numbered.

Results

Effect of supersaturation

A set of 13 experiments were carried out for the same solution velocity ($U = 2.46$ mm/s), and for approximate characteristic sizes, but with varying supersaturation of the solution between 0.02 and 0.14 kg monohydrate acid per kg water. It was assumed that the size variations during each experiment did not strongly affect the growth rate. The results are presented as the mass of crystals deposited per hour per unit surface area (R_g) versus the supersaturation ΔC and are plotted in Fig. 2. It can be noted that all the data can be correlated by a straight line, the equation of which is given by

$$R_g = 3.75\ \Delta C \qquad (10)$$

Effect of solution velocity and characteristic size

The effect of solution velocity and characteristic size can be shown by the following equation

$$R_g = kU^m L^n\ (C - C^*) \qquad (11)$$

where k is a temperature dependent constant,

U is the solution velocity,

L is the characteristic size assumed to be the diameter of the same volume sphere.

TABLE I: OPERATIVE CONDITIONS AND GROWTH CONSTANT k_g (ΔC_{lm} IN KG OF CITRIC ACID MONOHYDRATE/KG OF WATER AND k_g IN KG OF DEPOSITED ACID/H.M^2. ΔC)

Run	U (cm/s)	L_1 (cm)	L_2 (cm)	T (°C)	ΔC_{lm}	t (min)	k_g
1	0.25	0.181	0.191	25.0	0.021	40	3.77
2	0.25	0.139	0.152	24.9	0.030	30	4.92
3	0.25	0.165	0.180	24.9	0.033	40	3.77
4	0.25	0.151	0.170	24.6	0.051	30	4.15
5	0.25	0.139	0.156	25.0	0.055	30	3.42
6	0.24	0.151	0.177	25.2	0.060	40	3.59
7	0.25	0.138	0.162	24.8	0.071	30	3.85
8	0.25	0.139	0.161	25.0	0.075	30	3.41
9	0.26	0.141	0.166	25.0	0.078	30	3.61
10	0.25	0.140	0.180	24.9	0.120	30	3.64
11	0.25	0.142	0.179	24.6	0.126	30	3.25
12	0.25	0.136	0.180	24.8	0.141	25	4.10
13	0.26	0.113	0.144	24.6	0.093	30	3.69
14	0.18	0.179	0.219	25.3	0.094	40	3.52
15	0.30	0.162	0.180	25.3	0.042	40	3.61
16	0.32	0.162	0.179	25.1	0.040	40	3.62
17	0.38	0.146	0.173	25.0	0.063	40	3.54
18	0.44	0.142	0.164	25.3	0.049	40	3.76
19	0.21	0.138	0.158	25.1	0.048	40	3.45
20	0.16	0.144	0.167	25.0	0.066	40	3.01
21	0.25	0.192	0.218	25.1	0.063	40	3.57
22	0.25	0.198	0.221	25.3	0.053	40	3.58
23	0.29	0.196	0.216	25.0	0.046	40	3.78
24	0.34	0.200	0.219	25.1	0.038	40	4.11
25	0.37	0.198	0.225	25.3	0.054	40	4.08
26	0.44	0.207	0.232	25.0	0.049	40	4.19
27	0.18	0.188	0.214	24.7	0.057	40	3.72
28	0.31	0.165	0.183	24.7	0.040	40	3.73
29	0.36	0.164	0.179	24.9	0.037	40	3.48
30	0.41	0.168	0.182	25.4	0.031	40	3.80
31	0.46	0.169	0.187	25.2	0.040	40	3.74
32	0.20	0.167	0.190	25.0	0.056	40	3.53
33	0.17	0.172	0.195	25.0	0.057	40	3.35
34	0.30	0.188	0.198	25.0	0.025	40	3.42
35	0.35	0.190	0.202	25.0	0.029	40	3.52
36	0.39	0.186	0.201	24.8	0.033	40	3.87
37	0.44	0.182	0.199	25.0	0.044	40	3.26
38	0.20	0.181	0.200	25.0	0.049	40	3.20
39	0.16	0.181	0.198	25.1	0.052	40	2.85
40	0.23	0.220	0.244	25.3	0.059	40	3.49
41	0.30	0.218	0.238	25.5	0.047	40	3.62
42	0.34	0.215	0.253	25.4	0.084	40	3.88
43	0.39	0.217	0.248	25.4	0.067	40	3.98
44	0.44	0.225	0.250	25.2	0.054	40	3.92

TABLE I: *(continued)*

Run	U (cm/s)	L_1 (cm)	L_2 (cm)	T (°C)	ΔC_{lm}	t (min)	k_g
45	0.15	0.236	0.260	25.1	0.061	40	3.32
46	0.43	0.246	0.270	25.1	0.049	40	4.30
47	0.18	0.231	0.257	24.8	0.060	40	3.67
48	0.14	0.214	0.231	25.3	0.040	40	3.40
49	0.19	0.254	0.275	25.2	0.058	40	3.02
50	0.23	0.246	0.266	24.9	0.054	40	3.17
51	0.29	0.248	0.258	25.3	0.022	40	3.88
52	0.34	0.255	0.269	25.2	0.034	40	3.51
53	0.39	0.230	0.250	25.0	0.044	40	3.83
54	0.46	0.126	0.140	25.4	0.029	40	4.14
55	0.40	0.126	0.138	25.4	0.029	40	3.69
56	0.34	0.128	0.148	24.9	0.049	40	3.47
57	0.92	0.128	0.142	25.3	0.036	40	3.19
58	0.23	0.127	0.141	25.2	0.038	40	3.16
59	0.19	0.124	0.141	25.2	0.050	40	2.90
60	0.14	0.124	0.139	25.2	0.047	40	2.82
61	0.32	0.147	0.175	25.1	0.063	40	3.67
62	0.33	0.149	0.176	25.2	0.061	40	3.75
63	0.33	0.261	0.287	25.5	0.059	40	3.61
64	0.33	0.190	0.217	25.3	0.071	40	3.28
65	0.33	0.192	0.216	25.4	0.048	45	3.69
66	0.33	0.288	0.317	25.1	0.068	40	3.58
67	0.33	0.215	0.247	24.8	0.080	40	3.35
68	0.33	0.322	0.353	24.8	0.071	40	3.73
69	0.33	0.176	0.201	25.0	0.057	40	3.68
70	0.36	0.180	0.200	25.4	0.044	40	3.85
71	0.45	0.236	0.264	24.6	0.083	30	3.76
72	0.45	0.354	0.378	24.7	0.055	30	4.73
73	0.39	0.249	0.282	25.0	0.085	30	4.27
74	0.45	0.375	0.390	24.9	0.038	30	4.57
75	0.45	0.287	0.306	24.5	0.051	30	4.28
76	0.45	0.392	0.408	24.6	0.043	30	4.34
77	0.45	0.412	0.437	24.8	0.065	30	4.38
78	0.45	0.300	0.323	24.8	0.066	30	3.93
79	0.45	0.322	0.349	24.8	0.068	30	4.51
80	0.45	0.436	0.462	24.9	0.067	30	4.31
81	0.44	0.113	0.139	25.2	0.063	30	4.65
82	0.40	0.113	0.147	24.7	0.105	30	3.57
83	0.35	0.113	0.143	24.8	0.089	30	3.68
84	0.31	0.113	0.145	24.5	0.094	30	3.77
85	0.22	0.113	0.160	25.5	0.136	30	3.77
86	0.17	0.114	0.155	25.4	0.125	30	3.61

TABLE II: OPERATIVE CONDITIONS OF DISSOLUTION AND CONSTANT k_d (ΔC_{lm} IN KG OF CITRIC MONOHYDRATE/KG OF WATER AND k_d IN KG OF DISSOLVED ACID/H.M^2. ΔC)

Run n°	U (cm/s)	L_1 (cm)	L_2 (cm)	T (°C)	ΔC_{lm}	t (min)	k_d
1	0.30	0.204	0.177	25.0	0.045	60	3.39
2	0.41	0.182	0.148	25.0	0.045	60	4.22
3	0.40	0.200	0.173	25.1	0.042	60	3.61
4	0.44	0.200	0.145	25.0	0.070	60	4.33
5	0.25	0.177	0.151	25.1	0.064	40	3.48
6	0.31	0.253	0.242	25.3	0.022	40	4.16
7	0.43	0.257	0.225	24.9	0.054	50	4.02
8	0.24	0.250	0.219	24.8	0.054	50	3.88
9	0.34	0.222	0.181	24.8	0.067	50	4.03
10	0.31	0.248	0.205	24.8	0.070	50	4.06
11	0.19	0.157	0.141	24.8	0.032	45	3.57
12	0.15	0.157	0.150	24.9	0.019	40	3.15
13	0.24	0.156	0.151	25.0	0.016	27	3.46
14	0.38	0.248	0.225	25.2	0.060	30	4.23
15	0.45	0.304	0.283	25.4	0.061	30	3.76
16	0.24	0.199	0.185	25.3	0.042	30	3.63
17	0.31	0.198	0.185	25.6	0.042	30	3.39
18	0.37	0.198	0.186	25.6	0.034	30	3.91
19	0.47	0.201	0.196	25.1	0.012	40	3.75
20	0.17	0.197	0.192	24.6	0.017	30	3.31
21	0.17	0.207	0.197	24.9	0.034	30	3.35
22	0.44	0.201	0.197	24.9	0.009	30	4.34
23	0.37	0.272	0.268	24.8	0.011	30	3.71
24	0.37	0.248	0.245	24.8	0.009	30	3.92
25	0.37	0.139	0.132	25.0	0.020	30	4.02
26	0.37	0.157	0.138	25.0	0.060	30	3.47
27	0.37	0.182	0.168	24.9	0.045	30	3.58

For experiments carried out in fluidized bed with a batch of N uniform size crystals, assumption of perfect mixing of the crystals and of piston flow of the liquid leads to the following equation

$$R_g = kU^m L^n (C - C^*)_{lm} \tag{12}$$

where $(C - C^*)_{lm} = \dfrac{(C_1 - C^*) - (C_2 - C^*)}{\ln \dfrac{(C_1 - C^*)}{(C_2 - C^*)}}$.

and the rate of mass deposition

$$R_g = \frac{3\alpha\rho_c}{\beta} \cdot G = \frac{3\alpha\rho_c}{\beta} \cdot \frac{dL}{dt} \tag{13}$$

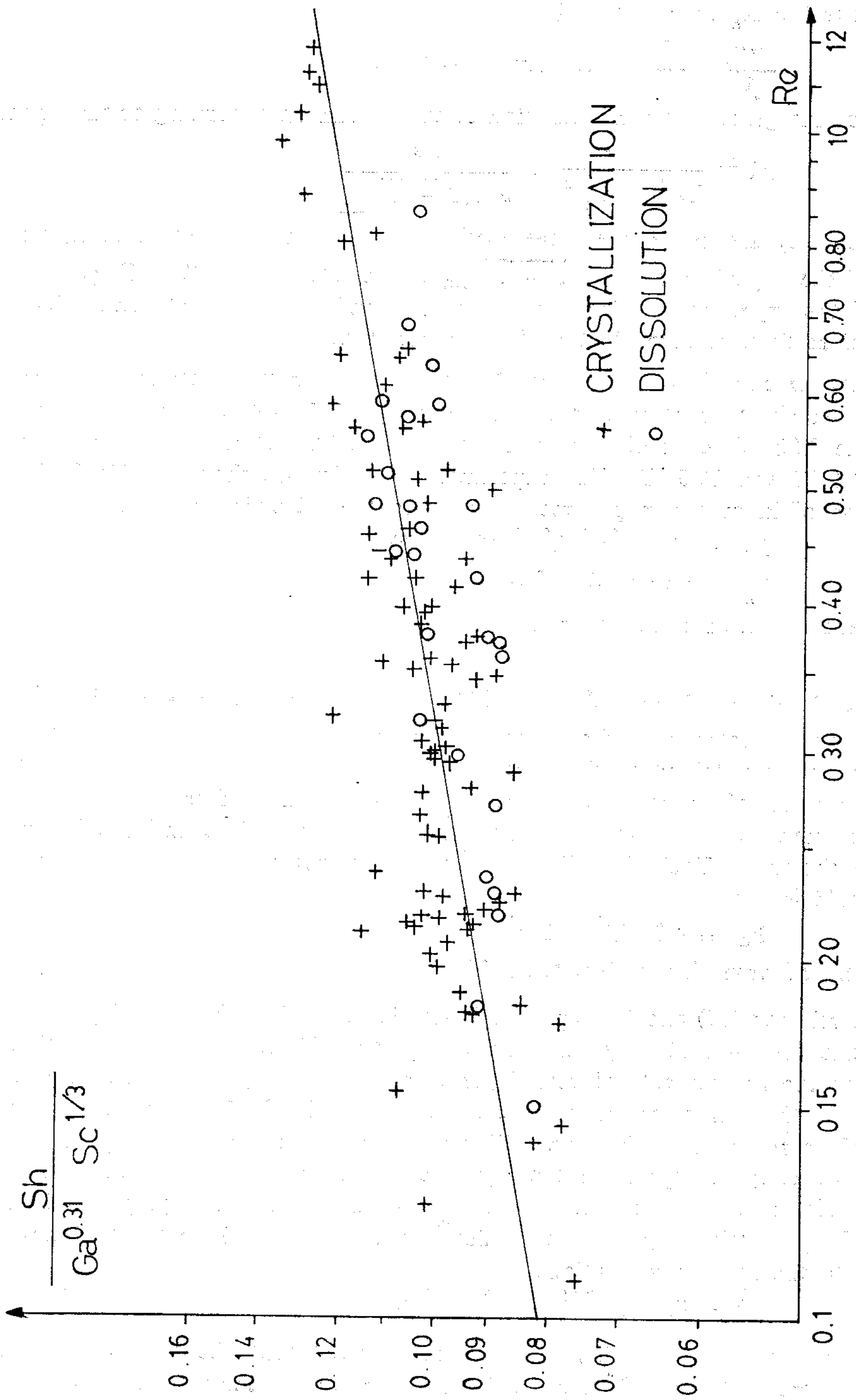

Fig. 3. The plot of log $(Sh/Ga^{0.31}\ Sc^{1/3})$ versus log Re.

so by substituting R_g in equation (12)

$$\frac{3\alpha\mu_c}{\beta} \cdot \frac{dL}{dt} = kU^m L^n (C - C^*)_{lm} \qquad (14)$$

which can be integrated over the duration of an experiment according to the equation

$$kU^m \frac{1 - n}{L_2^{1-n} - L_1^{1-n}} = \frac{3\alpha\rho_c}{\beta\, t\overline{(C - C^*)}_{lm}} \qquad (15)$$

where L_1 and L_2 are respectively the initial and final size of the crystals, t is the duration of the experiment, and $\overline{(C - C^*)}_{lm}$ is the average of $(C - C^*)_{lm}$ over the duration of the experiment. The parameter k, m and n can be determined by using a numerical method of identification from experiments.

Eighty-six runs have been carried out for solution velocities in the range 1.4 mm/s – 4.6 mm/s, characteristic sizes in the range 1.12 – 4.62 mm, supersaturation in the range 0.02 – 0.14 kg of monohydrate acid per kg water, and for temperatures between 24.5 °C and 25.5 °C. The experimental results are shown in Table I. The identification of k, m and n parameters leads to the following relation

$$R_g = 5.37\ U^{0.18}\ L^{0.10}\ (C - C^*) \qquad (16)$$

or

$$k_g = 5.37\ U^{0.18}\ L^{0.10} \qquad (17)$$

The standard deviation is less than 0.1.

Comparison with results of dissolution of citric acid in fluidized bed

In order to determine whether the diffusional phenomenon is the limiting step or not, the results obtained on the crystallization were compared with those obtained by the dissolution of monohydrate citric acid crystals in fluidized bed. The dissolution experiment was achieved on temperature between 24.6 °C and 25.6 °C and the results are shown in Table II. The regression analysis gave a correlation of mass transfer coefficient as

$$k_d = 4.79\ U^{0.21}\ L^{0.01} \qquad (18)$$

with a standard deviation of less than 0.1.

The relation (17) and (18) appears fairly different, but the ratio k_d/k_g remains in the range 0.95 – 1.08. By referring to equation (5) it can be shown that this implies that the growth rate of monohydrate citric acid crystals in fluidized bed is controlled by the diffusional mass transfer step. It should be noted that this fact is extremely rare in crystallization. This result seems to be a consequence of the very high viscosity of supersaturated aqueous solutions of monohydrate citric acid. All the results had been presented according to the correlation proposed by Damronglerd[6, 7] by plotting log $(Sh/Ga^{0.31}\ Sc^{1/3})$ versus log (Re) (Fig. 3). They were correlated to the following relation:

$$Sh = 0.21\ Re^{0.18}\ Ga^{0.31}\ Sc^{0.33} \qquad (19)$$

The effect of Ga number on Sh number was about the same as determined by Damronglerd but the influence of Re number on the mass transfer was greater in this study.

Moreover, the values of Sh number were about three to five times lower than those which could be extrapolated from Damronglerd's correlation. According to the different conditions, the Re number is lower than 1 and Sc number greater than 40,000 while the work of Damronglerd's has Re number greater than 45 and Sc number lower than 2000 giving different arbitrary constant and the different exponent.

Conclusion

This study tried to present briefly some general aspects of crystallization from solution. The main point of the experiment is to show that the growth rate of monohydrate citric acid crystals in fluidized bed is entirely diffusion controlled, which is an extremely rare case in crystallization processes.

Notations

A : total surface area of crystals in the bed (m^2)
a_c : surface area of a crystal (m^2)
B_1, B_2 : complex temperature dependent constants (°C)
C : solution concentration (kg hydrate per kg water)
C^* : solubility of citric acid in water (kg hydrate per kg water)
$(C-C^*)_m$: log mean of concentration difference in the bed (kg hydrate per kg water)
g : order of the growth rate
k_d : mass transfer coefficient i for dissolution (kg hydrate/h.m^2. (kg hydrate/kg water)
k_g : overall coefficient for growth (kg hydrate/h.m^2. (kg hydrate/kg water)
k_r : surface reaction constant
L : characteristic size (cm)
L_1, L_2 : characteristic size at the beginning and at the end of the run (cm)
M : total mass of crystals in the bed (kg)
m : exponent of U
m_c : mass of a crystal (kg)
N : number of crystals in the bed
n : exponent of L
r : order of the surface reaction
R_g : growth rate define by equation 1 (kg of hydrate/h.m^2.)
T : temperature (°C)
t : time, duration of a run (h)
α : volume shape factor
β : surface shape factor
ΔC : supersaturation (kg of hydrate/kg of water)
ΔM : mass variation during a run (kg)
ρ_c : density of crystals (kg/cm^3)
σ : degree of supersaturation ($\sigma = \frac{C - C^*}{C}$)

Ga : Galileo number $\left(\frac{L^3\rho(\rho_c - \rho)g}{\mu^2}\right)$

Re : Reynolds particle number $\left(\frac{LU\rho}{\mu}\right)$

Sc : Schmidt number $\left(\frac{\mu}{\rho D}\right)$

Sh : Sherwood number $\left(\frac{kL}{\frac{\rho L}{i+c} D}\right)$

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บทคัดย่อ

เทคนิคของฟลูอิไดเซชั่นแบบของเหลว ให้ประโยชน์ต่อขบวนการตกผลึกเป็นอย่างมาก ผลึกมีการเคลื่อนไหวอย่างสม่ำเสมอ ภายในหอทดลอง และไม่เกิดการชนกันอย่างรุนแรงระหว่างผลึกด้วยกันเอง ผลึกมากกว่าหนึ่งหมื่นผลึกถูกนำมาใช้เลี้ยงให้โตขึ้นในหอทดลองที่โปร่งแสง มีขนาดเส้นผ่าศูนย์กลาง 94 มม. ขนาดของผลึกที่ใช้มีหลายขนาด อยู่ในช่วงระหว่าง 1.12 มม. ถึง 4.62 มม. อุณหภูมิในแต่ละครั้งพยายามรักษาให้อยู่ที่ 25° ซ.

ผลการทดลองทั้งหมดได้คำนวน และรวบรวมอยู่ในสูตร

$$Sh = 0.21\ Re^{0.18}\ Ga^{0.31}\ Sc^{1/3}$$

อัตราการเติบโตของผลึกพบว่าเป็นสัดส่วนโดยตรงกับค่าของความเข้มข้นเกิน (supersaturation) ในลักษณะอันดับที่หนึ่ง