

Crystal Growth Rate of Calcite in a Constant-Composition Environment

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Abstract—The growth kinetics of calcite crystal was studied in a fluidized-bed crystallizer using a constant-composition method. Several operation variables were investigated, including supersaturation, pH value, activity ratio, ionic strength and superficial velocity. These factors except superficial velocity are significant as far as the influence on the calcite growth rate is concerned in the range of relative supersaturation (σ) between 0.5 and 1.5, pH value between 8.5 and 9.5, ionic strength between 0.004 M and 0.016 M; however, the calcite growth rate remains constant for the ionic strength varying from 0.016 M to 0.028 M. The crystal growth rates were calculated from the consumption rate of titration volume, contrast to the decreasing rate of calcium ion used in the pH-stat method. Then the calcite growth rates were compared with those determined by the pH-stat method.

Key Words : Constant-composition method, Calcite, Crystal growth rate, Solution properties, Fluidized-bed crystallizer

INTRODUCTION

To design a pellet reactor, which is a reactive, fluidized-bed, and growth-type crystallizer, for water softening, the crystal growth rate of calcium carbonate is needed. In 1991 van Dijk and Wilms proposed an empirical model of calcite growth rate as follows:

$$\frac{d[\text{Ca}^{2+}]}{dt} = k_T a ([\text{Ca}^{2+}][\text{CO}_3^{2-}] - K_{sp}), \quad (1)$$

where $(d[\text{Ca}^{2+}]/dt)$ is the depletion rate of calcium ion in the solution, which can be taken as the crystal growth rate of CaCO_3 in moles per unit time; k_T is a constant; a is the specific surface area of seed crystals; K_{sp} is the concentration solubility product of calcite; and $([\text{Ca}^{2+}][\text{CO}_3^{2-}] - K_{sp})$ represents the driving force for crystal growth. Equation (1) is considered as an over-all growth-rate model because it does not take into account the mechanism of crystal growth. Several mechanisms regarding crystal growth have been proposed in the literature (Mullin, 1993), among which the two-step growth model is the most useful one because the parameters in the model can be estimated. The steady-state growth model can be expressed mathematically by the following equations:

$$G = k_d(\sigma - \sigma_i) \text{ mass transfer} \quad (2)$$

$$= k_r \sigma_i^r \text{ surface reaction.} \quad (3)$$

Equation (2) deals with the diffusion rate of solute between the bulk solution and the solution/crystal interface and Eq. (3) takes into account all the steps occurring on the crystal surface, including adsorption, desolvation, surface diffusion, and incorporation. Although this model is a simplified scheme of crystal growth, it reveals a lot of useful kinetic information, which was summarized by Tai (1997). Besides, all the parameters, *i.e.*, r , k_d , and k_r , can be evaluated using a well-suspended fluidized bed for several readily-soluble salts with known diffusivity (Tai, 1999). First, the mass-transfer coefficient, k_d , which is a function of system properties only, can be estimated by the following equation, (Tai *et al.*, 1987)

$$Sh = 0.306 Ga^{1/3} Mv^{1/3} Sc^{1/3}. \quad (4)$$

Then, σ_i 's are calculated from experimental G - σ data using Eq.(2). Finally, according to Eq. (3), k_r and r are evaluated from the interception and slope, respectively, on a plot of $\ln G$ vs. $\ln \sigma_i$. If the diffusivity is not available, such as the sparingly-soluble salts, the surface-reaction order should be determined by other means in order to evaluate the mass-transfer and surface-reaction coefficients (Tai *et al.*, 1999).

Tai *et al.* (1999) studied the growth kinetics of calcite crystals suspended in a fluidized-bed crystallizer, which was maintained at a constant pH instead of constant composition. They found that supersatu-

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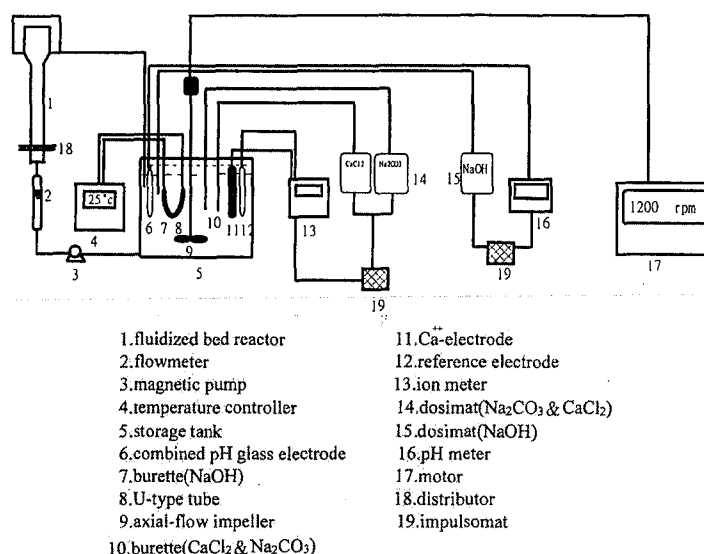


Fig. 1. A constant-composition crystallization system.

ration, pH, and ionic strength were significant variables that affected the growth rate of calcite crystals. In the same study a growth rate equation, which is based on the two-step crystal growth model, was proposed for design purpose. However, there is a disadvantage of the pH-stat method, in which the supersaturation changes during a run and the crystal growth rate is determined from the slope of calcium ion concentration profile of the solution in the crystallizer. The crystal growth rates so obtained are less accurate than those estimated by the constant-composition method, which uses the depletion rate of titration volume to calculate the crystal growth rates.

The aim of this study is to investigate the effects of operation variables, including supersaturation, pH, activity ratio, ionic strength and superficial velocity on the crystal growth rate of calcite in a fluidized-bed crystallizer using the constant-composition method. The growth experiments were performed in the metastable region, which had been identified experimentally by Tai *et al.* (1999), using cured natural calcite in a size range between 390 μm and 921 μm . The crystal size was the average size of the apertures of the two closest ASTM standard sieves. For example, crystal of size 460 μm was the fraction of crystals retained between sieve No.40 and 35, with nominal apertures of 420 and 500 μm . The crystal growth rates were evaluated from the volume depletion rate of titration solution. The results were then compared with those obtained by the pH-stat method.

EXPERIMENTAL

Crystallization system

The crystallization apparatus containing a lab-

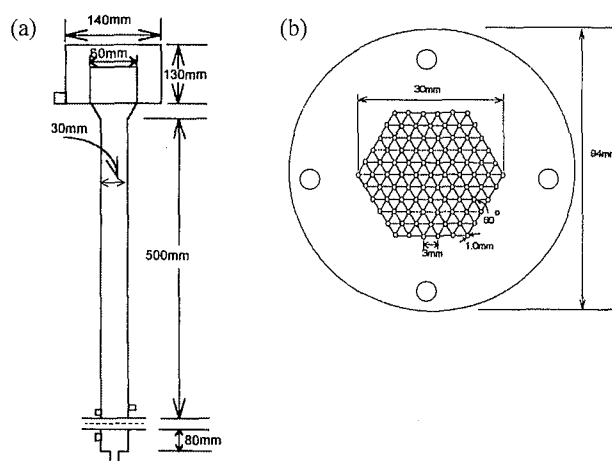


Fig. 2. Specification of fluidized-bed, (a) fluidized-bed column; (b) distributor.

scaled fluidized-bed crystallizer, a storage tank, and a composition control system, is shown in Fig. 1. A detailed construction of the fluidized bed, including the distributor which was designed to obtain an even flow in the fluidized-bed, is illustrated in Fig. 2. The composition control system consists of two autotitrators; one for adding reaction solutions and the other for adding sodium hydroxide solution. At the beginning of an experiment, a supersaturated solution of 6 L, prepared by mixing CaCl₂ and Na₂CO₃ solutions, with a desired concentration within the metastable region (Tai *et al.*, 1999) and a suitable pH, was charged into the storage tank and another 0.4 L of the same solution was fed into the fluidized bed. Then the solution in the storage tank was pumped through a flow meter and then into the crystallizer. After the solution passed through the crystallizer column, it overflowed to the storage tank. When the flow rate, pH, and composition of the solution became steady, the pump was stopped and approxi-

mately 25 g of seed crystals were introduced into the crystallizer. Then the pump was restarted and the growth experiment began. In this experiment the solution superficial velocity was set lower than the particle terminal velocity to give the bed a clear solution-suspension boundary, *i.e.*, the bed was operated in a dense mode. The composition and pH were maintained constant during the operation, using two sets of autotitrator to control the added amount of reaction solutions (0.1 M for both CaCl_2 and Na_2CO_3 aqueous solutions added simultaneously) and 0.1 M NaOH solution. The volume of titration solution added to the crystallizer was recorded automatically. The titration curve was then used to calculate the crystal growth rate. During an experimental run the chloride ion and the sodium ion would accumulate. The accumulation of both ions increased the ionic strength of the solution, which might affect the crystal growth rate (Tai *et al.*, 1999). However, the increment of ionic strength in this experiment was less than 5%, which cause little change in the crystal growth rate.

Determination of crystal growth rate

The crystal growth rate in $\text{kg/m}^2\text{s}$ can be expressed as

$$R_g = \frac{1}{A} \frac{dW}{dt}, \quad (5)$$

where A and W are the total mass and surface area of crystals, which are related to the crystal size by the following forms,

$$W = n\rho_p\alpha L^3, \quad (6)$$

$$A = n\beta L^2, \quad (7)$$

where ρ_p is the crystal density; α and β are the volume and surface area shape factor, respectively; and n is the number of crystals. Substituting Eqs.(6) and (7) into Eq.(5) to give

$$R_g = \frac{3\alpha\rho_p}{\beta} \frac{dL}{dt} = \frac{3W}{LA} \frac{dL}{dt}. \quad (8)$$

Then Eq.(5) and Eq.(8) can be combined to give the linear crystal growth rate in m/s

$$G = \frac{dL}{dt} = \frac{L}{3W} \frac{dW}{dt}. \quad (9)$$

In a constant-composition operation, the solution volume increases during a run. The change in volume should be taken into consideration in the determination of crystal growth rate. Thus,

$$\frac{dW}{dt} = M \frac{d(V_o[\text{Ca}^{2+}]_o + V_a[\text{Ca}^{2+}]_a - (V_o + V_a)[\text{Ca}^{2+}]_t)}{dt}, \quad (10)$$

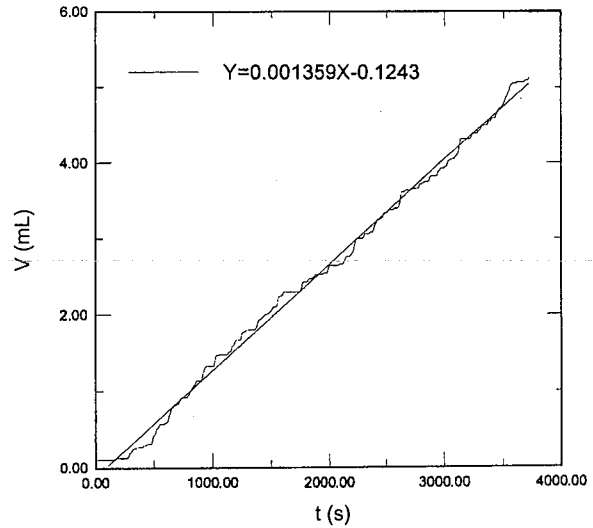


Fig. 3. A typical titration curve; $\sigma=1$, $\text{pH}=9.0$, $R=2.0$, $I=0.018\text{ M}$.

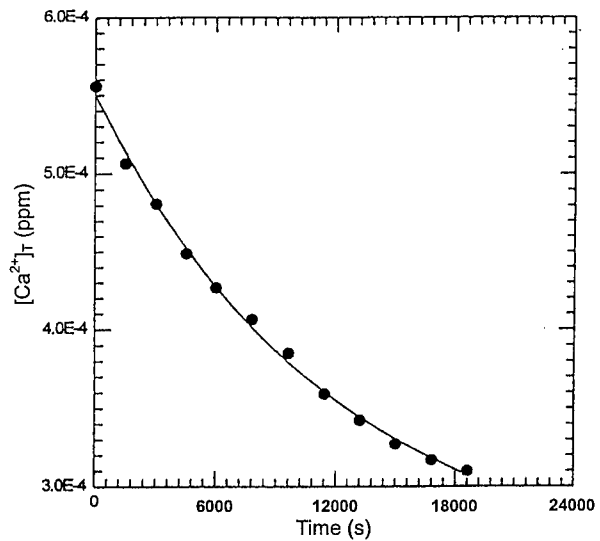


Fig. 4. A typical trend showing the change of total calcium concentration during a pH-stat operation; $L=651\text{ }\mu\text{m}$, $[\text{Ca}^{2+}]_T=6\times 10^{-4}\text{ M}$, $[\text{CO}_3^{2-}]_T=5.55\times 10^{-5}\text{ M}$, $\text{pH}=9.5$, $T=25^\circ\text{C}$. (Tai *et al.*, 1999). (•) experimental data; (—) fitted curve.

where M is the molecular weight of CaCO_3 ; V_o and V_a are the volume of the original solution and added solution, respectively; $[\text{Ca}^{2+}]_o$ and $[\text{Ca}^{2+}]_a$ are the Ca^{2+} concentration of the original solution and added solution, respectively. Equation (10) is substituted into Eq.(9) to yield

$$G = \frac{LM}{3W} ([\text{Ca}^{2+}]_a - [\text{Ca}^{2+}]_o) \frac{dV_a}{dt}. \quad (11)$$

The volume of titration solution was recorded automatically as shown in Fig. 3. The titration curve was fitted by a straight line. The slope of the straight line was used to calculate the crystal growth rate according to Eq.(11).

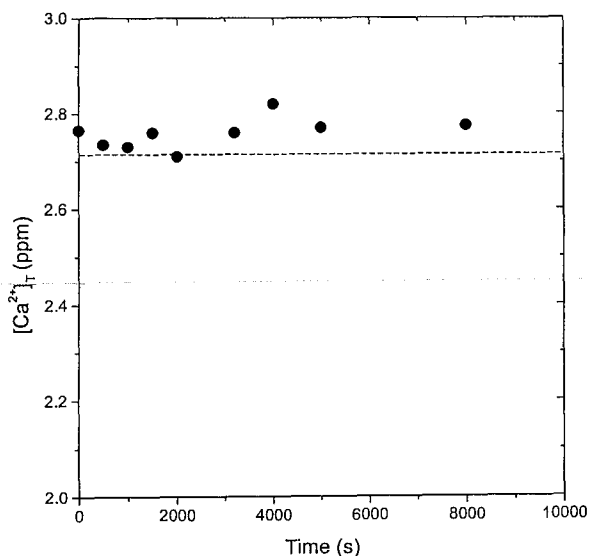


Fig. 5. A typical trend showing the concentration of total calcium during a constant-composition operation controlled by a calcium-ion selective electrode.

The technique used here is different from that used in the pH-stat method, in which the concentration of Ca^{2+} decreased when seed crystals grew. The solution was sampled and the Ca^{2+} concentration was determined by an atomic absorption spectrometer. A typical calcium-ion concentration profile is shown in Fig. 4 (Tai *et al.*, 1999). Then the concentration profile was fitted by a polynomial. Taking derivative of the polynomial and then substituted into the following formula to give the crystal growth rate.

$$G = \frac{LMV}{3W} \left(-\frac{d[\text{Ca}^{2+}]}{dt} \right). \quad (12)$$

Since the Ca^{2+} concentration is decreasing, the crystal growth rate so obtained is less accurate than that obtained by the constant-composition method.

RESULTS AND DISCUSSION

Assurance of constant composition

Although this experiment was designed to operate at constant composition, the setting point might run away due to unknown reasons. To check the constant-composition of the system, solution samples were withdrawn intermittently by using a syringe fitted with a 0.22- μm filter, and the total calcium-ion concentration was determined by an atomic absorption spectrometer. A typical plot of total calcium ion concentration up to the operation time of 8000 s (2.2 h), which was longer than most of the experimental runs, is shown in Fig. 5. Although the total concentration fluctuated around the setting point, the maximum deviation was less than 5%. We concluded that

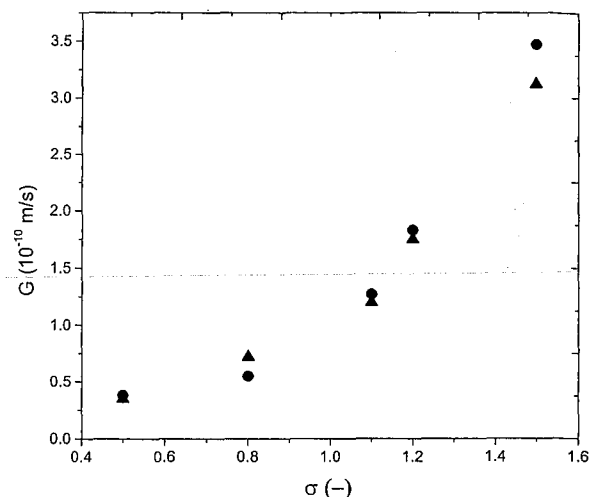


Fig. 6. Reproducibility of calcite growth rate data. Other operation variables kept constant are: pH=8.5, $I=0.018\text{ M}$, $R=5.54$, $v=0.047\text{ m/s}$, $L=774\text{ }\mu\text{m}$, $T=25^\circ\text{C}$. (●) and (▲) indicate the data of two different runs.

the concentration of calcium ion can be kept nearly constant during a long period of operation.

Reproducibility of experimental data

To test the reproducibility of experimental data obtained by the constant-composition method, two sets of experimental data at relative supersaturations between 0.5 and 1.5, were collected at the beginning and the end of this research, while keeping other operation variables constant, including pH, ionic strength, concentration ratio of components, superficial velocity, crystal size, and temperature, which were identified by Tai *et al.* (1999) as operation variables that might affect growth rates. The two sets of growth rate data were plotted in Fig. 6. The difference between the two sets of data are within 15%, except the data at $\sigma=0.8$ with a difference of 23.6%. In our experience on the measurement of crystal growth rate, the consistency of the results is acceptable.

Effects of operation variables

The effects of five operation variables, including supersaturation (σ), pH, ionic strength (I), activity ratio (R), and superficial velocity were investigated in this experiment. The superficial velocity chosen was lower than the terminal velocity of crystals so that the fluidized bed was operated in a dense mode, *i.e.*, a clear boundary existing between the solution and suspension. The effect of superficial velocity can be explained; using the relative velocity between the crystals and solution; however, the effects of solution properties, including σ , I , and R , remains unclear.

The supersaturation σ was defined by Nielson

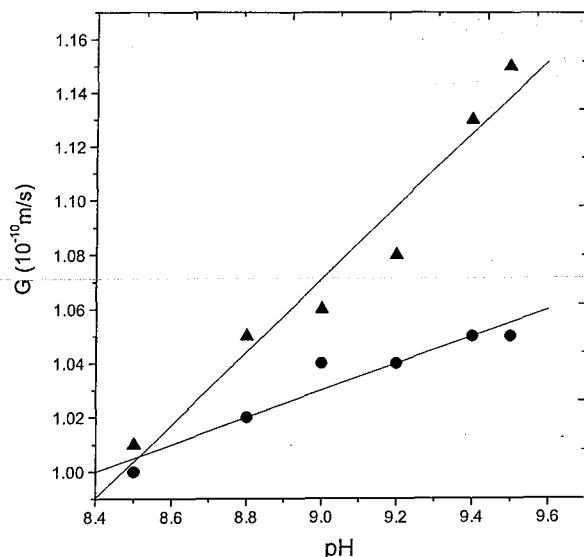


Fig. 7. Growth rate of calcite as a function of pH for two values of R ; $R=5.54$ (▲), and $R=11.5$ (●). Other variables kept constant are: $\sigma=1.0$, $I=0.018$ M, $L=651$ μm , $\nu=0.035$ m/s.

and Toft (1984) as:

$$\sigma = (K_{ip} / K_{sp})^{1/2} - 1, \quad (13)$$

where K_{ip} is the ionic product and K_{sp} is the solubility product of CaCO_3 . A computer program was used to calculate σ (Nancollas, 1966; Tai *et al.*, 1993). Figure 6 shows the influence of supersaturation on calcite growth rate, which increases with an increase in supersaturation. This is true for all kinds of crystal, since supersaturation is the driving force for crystal growth.

The pH effect on the calcite growth rate is shown in Fig. 7 for two different activity ratio, that is, $R = 5.5$ and 11.5 . The growth rate increased by 15% for $R = 5.5$ and slightly increased for $R = 11.5$, when the pH changes from 8.5 to 9.5. Note that the data shown in Fig. 7 were taken at $\sigma = 1$, at which Tai *et al.* (1999) found that the crystal growth rate did not change much in the same pH range; however, the difference in growth rate was significant at either higher or lower supersaturation. It is interesting to see the pH effect at $\sigma = 0.5$ or $\sigma = 1.5$.

The activity ratio of component R is defined as,

$$R = a_{\text{Ca}^{2+}} / a_{\text{CO}_3^{2-}} \\ = [\text{Ca}^{2+}] \cdot \gamma_{\text{Ca}^{2+}} / [\text{CO}_3^{2-}] \cdot \gamma_{\text{CO}_3^{2-}}, \quad (14)$$

where $\gamma_{\text{Ca}^{2+}}$ and $\gamma_{\text{CO}_3^{2-}}$ are the activity coefficients of calcium ion and carbonate ion respectively. The effect of activity ratio was investigated at two levels of pH values, *i.e.*, pH=9.0 and 9.5. In both cases, a maximum of crystal growth rate was found at R between 2 and 3. Figure 8 shows the effect of activity ratio at pH=9.5. In the range of R from 1 to 18, a

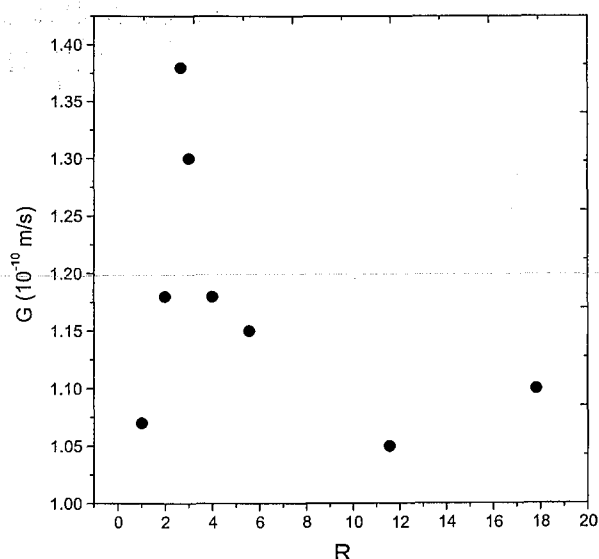


Fig. 8. Growth rate of calcite as a function of activity ratio, $\sigma=1.0$, pH=9.5, $I=0.018$ M, $L=651$ μm , $\nu=0.035$ m/s.

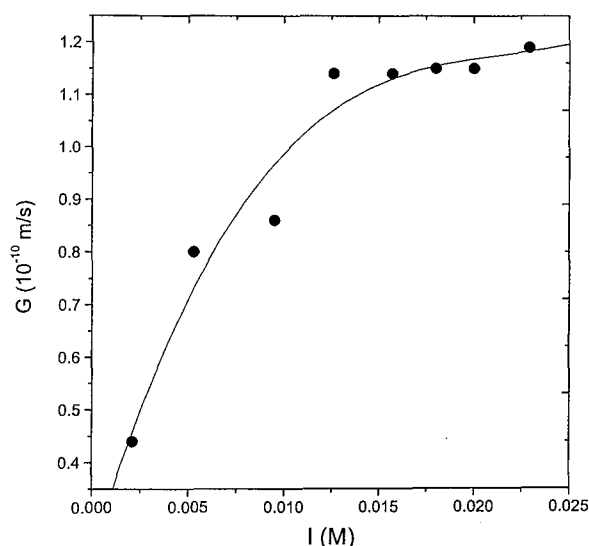


Fig. 9. Growth rate of calcite as a function of ionic strength, $\sigma=0.8$, pH=9.5, $R=5.54$, $L=651$ μm , $\nu=0.035$ m/s.

maximum difference in growth rate around 35% is observed. It is obvious that a maximum value exists.

The ionic strength of a solution is calculated by the following equation

$$I = \frac{1}{2} \sum_{i=1}^n Z_i^2 C_i, \quad (15)$$

where Z_i is the valence of i species and C_i includes the concentration of H^+ , OH^- , Ca^{2+} , CaOH^+ , CaHCO_3^+ , HCO_3^- , CO_3^{2-} , Na^+ , and Cl^- (Tai *et al.*, 1993). In this experiment, the ionic strength was adjusted by adding NaCl to the solution because Na^+ and Cl^- were already in the solution. Figure 9 shows the effect of ionic strength, indicating a profound in-

Table 1. Growth rates of calcite at various superficial velocities with other operating variables kept constant: $\sigma=1.0$, $\text{pH}=8.5$, $R=5.54$, $I=0.018$ M, $L=651$ μm , $w=25$ g, $[\text{Ca}^{2+}]_T=5.9\times 10^{-4}$ M, $[\text{CO}_3^{2-}]_T=5.9\times 10^{-4}$ M, $\text{NaCl}=0.016$ M.

v (m/s)	ε	dV/dt (mL/s)	$G\times 10^{10}$ (m/s)
0.014	0.50	0.001463	1.27
0.024	0.58	0.001370	1.19
0.028	0.64	0.001474	1.28
0.035	0.71	0.001509	1.31
0.047	0.79	0.001336	1.15

Table 2. Comparison of calcite growth rates obtained by pH-stat and constant- composition methods. Operating variables kept constant are: $\text{pH}=9.5$, $L=650$ μm , $I=0.0025$ M.

Supersaturation σ	Calcite Growth Rate (10^{-11} m/s)		Difference (%)
	pH-stat method	constant-composition method	
1.05	13.4	12.0	10.4
0.87	10.1	9.52	5.7
0.77	8.40	7.25	13.7
0.65	6.02	6.24	3.7
0.55	5.21	5.67	8.1

fluence on the calcite growth rate which is almost tripled when the ionic strength varied from 0.0021 M to 0.0157 M. After that the growth rate levels off. This results is similar to that reported by Tai *et al.* (1999) using the pH-stat method. They measured the calcite growth rate for I from 0.0025 to 0.0340 M. The growth rate increased from low ionic strength, but remained constant after I was greater than 0.0185.

The growth rates of calcite were measured at various superficial velocities, that is, 0.014, 0.024, 0.028, 0.035, 0.047 m/s, with other operation variables fixed as shown in Table 1. Although the bed voidage changes a great deal, from 0.50 to 0.79, the calcite growth rates are rather constant. The result is consistent with that reported by Tai *et al.* (1999) in a pH-stat experiment. An explanation on the constant growth rates using the two-step growth model is as follows: both mass-transfer coefficient and surface-reaction coefficient are constant. For the same crystal size, it is not surprising to see that the solution velocity does not influence the surface-reaction step, because the particles are gently suspended in a dense fluidized-bed. For the mass-transfer coefficient, it is a constant for a lean fluidized bed, which is operated at a superficial velocity near the terminal velocity of crystals, as derived and verified by Tai *et al.* (1987). When the superficial velocity is reduced, the bed voidage and thus the cross-sectional area for the passage of solution become smaller. Thus, it is possible that the relative velocity, which is the velocity that determines the mass-transfer coefficient, in a dense bed is almost equal to the terminal velocity of crystals regardless of bed voidage. As a result, the mass-transfer coefficient in a dense bed is a constant and is identical to that in a lean bed. The constant mass-transfer coefficient is also supported by the evidence in crystal dissolution experiments. Tournie *et al.*

(1979) analyzed extensive dissolution data in the literature for both lean and dense fluidized beds, and they concluded that the mass-transfer coefficient was practically independent of liquid solution and particle size.

Comparison of calcite growth rates obtained by two methods

The calcite growth rate obtained by the pH-stat and constant composition methods were compared under similar operation conditions except the ionic strength, which varied within 5%. This difference in ionic strength should cause no effect on the calcite growth rate. Table 2 compares the calcite crystal growth rates obtained by the two methods, when the supersaturation varies from 0.55 to 1.05, the maximum difference is 13.7% at $\sigma=0.77$, and the average difference is 10.6%. The results from both methods check each other, although the technique to determine the crystal growth rate is quite different.

CONCLUSION

A fluidized bed crystallizer controlled at constant-composition was successfully operated by using two set of autotitrators, one with a calcium-ion electrode and the other one with a pH electrode. The titration volumes of reagents recorded automatically were used to calculate the crystal growth rates. Several operation variables were investigated in this study, including supersaturation, pH value, activity ratio, ionic strength, and superficial velocity. These factors except superficial velocity were significant as far as the influence on the calcite growth rate was concerned in the range of relative supersaturation (σ)

between 0.5 and 1.5, the pH value between 8.5 and 9.5, the ionic strength between 0.0021 M and 0.0157 M; however, the growth rate remained constant for the ionic strength varying from 0.0157 M to 0.0340 M. The effect of superficial velocity, which did not influence the calcite growth rate, was explained by the constant relative velocity between the crystals and solution at various superficial velocities. However, the explanation on the effects of pH value, ionic strength, and activity ratio needs to be explored. Finally, the growth rate data obtained in this study using a constant-composition method were compared with those obtained by a pH-stat method. The average difference of five data was only 10.6%, although the technique to evaluate the crystal growth rate was quite different.

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NOMENCLATURE

A	surface area of crystals, m^2
a	specific surface area, m^2/m^3
C_i	concentration of i species, M
$[\text{Ca}^{2+}]$	calcium-ion concentration, mol/m^3
$[\text{Ca}^{2+}]_a$	calcium-ion concentration of titration solution, mol/m^3
$[\text{Ca}^{2+}]_o$	calcium-ion concentration of original solution, mol/m^3
$[\text{Ca}^{2+}]_T$	total calcium concentration, mol/m^3 or ppm
$[\text{CO}_3^{2-}]$	carbonate-ion concentration, mol/m^3
$[\text{CO}_3]_T$	total carbonate concentration, mol/m^3
G	linear crystal growth rate, m/s
Ga	Galileo number
I	ionic strength, M
K_{ip}	ionic product, $(\text{kmol})^2/\text{m}^6$
K_{sp}	concentration solubility product, $(\text{kmol})^2/\text{m}^6$
k_d	mass-transfer coefficient, m/s
k_r	surface-reaction coefficient, m/s
k_T	proportional constant in Eq. (1)
L	crystal size, m
M	molecular weight of calcium carbonate
M_v	density number, $(\rho_p - \rho)/\rho$
n	number of crystals
R	activity ratio
R_g	crystal growth rate, $\text{kg}/\text{m}^2\cdot\text{s}$
r	surface reaction order

Sc	Schmidt number
Sh	Sherwood number
t	time, s
V_a	titration volume, m^3
V_o	volume of original solution, m^3
v	superficial velocity, m/s
W	crystal weight, kg
$\gamma_{\text{Ca}^{2+}}$	activity coefficient of calcium ion
$\gamma_{\text{CO}_3^{2-}}$	activity coefficient of carbonate ion
z_i	valence of i species

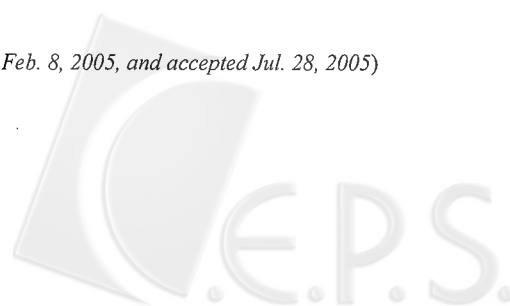
Greek symbols

α	volume shape factor of crystal
β	surface area shape factor of crystal
ε	void fraction of fluidized bed
ρ	solution density, kg/m^3
ρ_p	crystal density, kg/m^3
σ	relative supersaturation
σ_i	interfacial supersaturation

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方解石晶體在定組成環境下之成長速率

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摘 要

本文以定組成方法研究方解石晶體在流化床結晶槽中之成長動力學，所探討之操作變數包括過飽和度、酸鹼值、活性比、離子強度及表面流速。在以下操作變數之範圍內，如相對過飽和度 (σ)，介於 0.5 與 1.5 間，pH 值介於 8.5 與 9.5 間，離子強度介於 0.004 M 及 0.016 M 間，除表面流速外，這些變數對方解石之成長都有顯著影響。但是在離子強度介於 0.016 M 至 0.028 M 間，成長速率並無太大變化。晶體成長速率是以滴定溶液消耗速率計算而得，與酸鹼恆定方法之鈣離子消耗速率有所不同。然後將定組成法與酸鹼恆定法所得之方解石晶體成長速率作一比較。

