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Analysis of particle coating by spouted bed process

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Abstract

The coating of nearly spherical porous alumina with a mean diameter, $\bar{d}_p$, of 3.9 mm, with a suspension of sucrose and talc in water, was carried out in a conical–cylindrical spouted bed. The rate of increase in particle mass, K_1, and the mass fraction of atomized solids incorporated by the particles, η, were measured as a function of spouting gas temperature, T_{gi}, mass flow rate of atomized suspension relative to spouting gas, W_s/W_g, and the gas flow rate relative to minimum spouting, Q/Q_{ms}. The spouting gas temperature was varied between 67 and 83°C, W_s/W_g between 0.00263 to 0.00471, and Q/Q_{ms} between 1.5 and 1.7. K_1 and η were found to be insensitive to T_{gi} within the studied range; expressions for their dependence on W_s/W_g and Q/Q_{ms} were derived. Particle mass increased linearly with the coating time. The final product is uniform in shape and conserves a log-normal particle mass distribution. © 1997 Elsevier Science B.V.

Keywords: Coating; Kinetics of growth; Spouted beds

1. Introduction

The coating of particles, agglomerates and compacts is an industrial operation of importance in food, pharmaceutical and chemical industries. Different types of equipment, including rotating pans, fluidized and spouted beds, have been utilized for applying a thin and uniform layer of material to the surface of a body with multivariate purposes (Doane et al., 1977; Porter, 1978; Salman et al., 1989; Watano et al., 1994).

In particular, to the coating in spouted beds have been attributed the advantages of formation of very uniform layers in a relatively short time, due to excellent conditions of heat and mass transfer within the bed (Kmiec, 1980; Kucharski

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Table 1
Mean properties of porous alumina used

$\bar{d}_{p0}$ (cm)	$\bar{m}_{p0}$ (g)	ρ_p (g/cm^3)	$\bar{\phi}$	$\bar{\varepsilon}$
0.394	0.0437	1.386	0.92	0.09

Table 2
Variables and levels investigated

Variables	Levels	Uncertainty
T_{gi} (°C)	67; 75; 83	±0.5
Q/Q_{ms}	1.5; 1.6; 1.7	±0.01
W_s/W_g	0.00263; 0.00367; 0.00471	±0.0002

and Kmiec, 1983, 1988). On the other hand, attrition and elutriation of fines are limitations that severely confine this technique to materials with adequate physical and chemical properties (Pavarini and Coury, 1987). Also, the control of the operation variables is often very strict, in order to avoid bed collapse due to overfeeding or underdrying of the coating material (Ormenese et al., 1989).

In this work, nearly spherical particles of porous alumina were coated with a suspension of sucrose and talc in water so that the effect of spouting gas flow rate, temperature and coating suspension feed flow rate on the process could be evaluated. It aims to contribute to the practical utilization of the spouted bed coating process, proposing an empirical correlation that can be used for design purposes.

2. Materials, equipment and experimental methods

2.1. Materials, equipment and preliminary tests

As coating material, a suspension consisting of 39.8 wt.% sucrose, 29.9 wt.% talc and 30.3 wt.% distilled water was used. The alumina particles used have the approximate shape of a prolate ellipsoid with a fairly narrow size distribution. The mean properties of the alumina particles are shown in Table 1. In this table $\bar{m}_{p0}$ is the initial mean particle mass, $\bar{d}_{p0}$ is the initial mean particle diameter, ρ_p is the particle density, $\bar{\varepsilon}$ is the mean particle porosity and $\bar{\phi}$ is the mean particle sphericity.

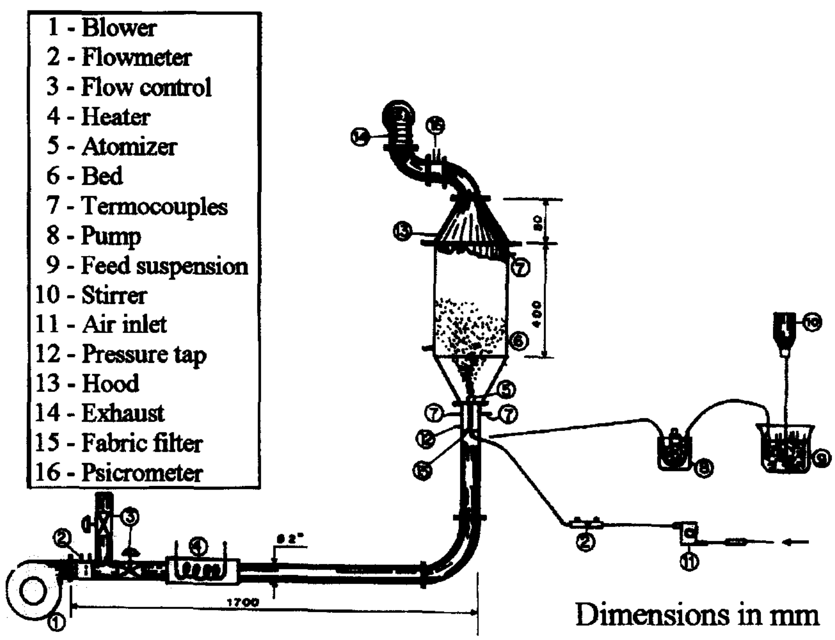

Fig. 1. Scheme of the equipment utilized in this work.

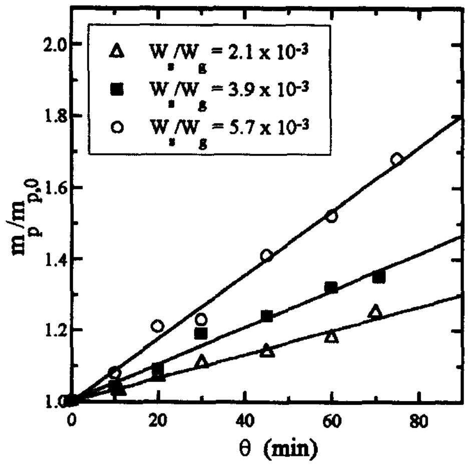

Fig. 2. Mean particle mass relative to the initial mass ($\bar{m}_p/\bar{m}_{p0}$) as a function of coating time, θ, for three values of W_s/W_g, with $Q/Q_{ms} = 1.6$ and $T_{gi} = 75$°C.

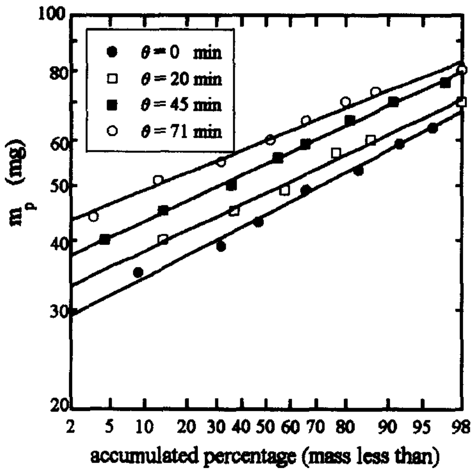

Fig. 3. Typical particle mass distribution in the bed for various coating times ($W_s/W_g = 0.0039$; $Q/Q_{ms} = 1.6$; $T_{gi} = 75$°C).

Table 3
Experimental results of the rate of increase in the particle mass, K_1, and of the fraction of solids incorporated by the particles, η

Run No.	Q/Q_{ms}	W_s/W_g	T_{gi} (°C)	$K_1 \times 10^3$ (min^{-1})	$\eta \times 10^2$
1	1.5	0.00263	67	3.7	90
2	1.5	0.00263	75	3.5	85
3	1.5	0.00263	83	3.1	75
4	1.5	0.00367	67	5.3	86
5	1.5	0.00367	75	5.0	85
6	1.5	0.00367	83	5.5	96
7	1.5	0.00471	67	6.8	92
8	1.5	0.00471	75	6.8	92
9	1.5	0.00471	83	6.7	91
10	1.6	0.00263	67	3.7	84
11	1.6	0.00263	75	3.6	82
12	1.6	0.00263	83	3.8	86
13	1.6	0.00367	67	5.4	88
14	1.6	0.00367	75	5.6	91
15	1.6	0.00367	83	5.4	88
16	1.6	0.00471	67	7.2	91
17	1.6	0.00471	75	7.0	96
18	1.6	0.00471	83	6.9	88
19	1.7	0.00263	67	4.0	85
20	1.7	0.00263	75	3.4	73
21	1.7	0.00263	83	3.6	77
22	1.7	0.00367	67	5.6	85
23	1.7	0.00367	75	6.4	96
24	1.7	0.00367	83	5.2	79
25	1.7	0.00471	67	7.1	89
26	1.7	0.00471	75	7.5	89
27	1.7	0.00471	83	7.4	88

Table 4
Analysis of variance for the rate of increase in the particle mass, $K_1 \times 10^3$

Variable	Sum of squares	Degrees of freedom	Mean square	F_{CALC}
T_{gi}	0.11	2	0.06	0.46
$T_{gi(\ell)}$	0.08	1	0.08	0.67
$T_{gi(s)}$	0.03	1	0.03	0.25
W_s/W_g	53.56	2	26.78	223.17[a]
$W_s/W_{g(\ell)}$	53.39	1	53.39	444.92[a]
$W_s/W_{g(s)}$	0.17	1	0.17	1.42
Q/Q_{ms}	0.81	2	0.41	3.38[c]
$Q/Q_{ms(\ell)}$	0.80	1	0.80	6.67[b]
$Q/Q_{ms(s)}$	0.01	1	0.01	0.08
Iteration				
$T_{gi(\ell)}$ $W_s/W_{g(\ell)}$	0.05	1	0.05	0.42
$T_{gi(s)}$ $W_s/W_{g(\ell)}$	0.05	1	0.05	0.42
$T_{gi(\ell)}$ $W_s/W_{g(s)}$	0.01	1	0.01	0.08
$T_{gi(s)}$ $W_s/W_{g(s)}$	0.12	1	0.12	1.00
$T_{gi(\ell)}$ $Q/Q_{ms(\ell)}$	0.00	1	0.00	0.00
$T_{gi(s)}$ $Q/Q_{ms(\ell)}$	0.13	1	0.13	1.08
$T_{gi(\ell)}$ $Q/Q_{ms(s)}$	0.01	1	0.01	0. 08
$T_{gi(s)}$ $Q/Q_{ms(s)}$	0.01	1	0.01	0.08
$W_s/W_{g(\ell)}$ $Q/Q_{ms(\ell)}$	0.08	1	0.08	0.67
$W_s/W_{g(s)}$ $Q/Q_{ms(\ell)}$	0.00	1	0.00	0.00
$W_s/W_{g(\ell)}$ $Q/Q_{ms(s)}$	0.03	1	0.03	0.25
$W_s/W_{g(s)}$ $Q/Q_{ms(s)}$	0.01	1	0.01	0.08
Error				
W_s/W_g T_{gi} Q/Q_{ms}	0.94	8	0.12	—
Total	55.92	26	—	—

Subscript ℓ and s are the linear and square effects.
[a] Term is significant at 0.01 level.
[b] Term is significant at 0.05 level.
[c] Term is significant at 0.10 level.

The tests were carried out in the rig shown in Fig. 1. The bed had a cylindrical section 140 mm in diameter and 400 mm in height, and included an angle of conical base of 60° with an inlet orifice of 39 mm. A two-fluid atomizer was placed in the inlet of the bed and the coating suspension was fed concurrently with the spouting air. To feed the mass flow rate of the coating material to the equipment, a peristaltic pump was used.

A series of preliminary tests was performed to establish the range of the bed temperature, mass flow rate of atomized suspension and gas flow rate for stable spouting. Table 2 lists the variables, their ranges and also the uncertainty associated to each measurement, estimated with the procedure described by Holman (1978). Also, the sampling procedure and sample size were defined after preliminary tests. No effect of sampling position in the results was detected and sampling from the bed top was adopted. The sample size was of 126 particles, determined after a number of runs, and the experimental error due the sampling was 3%. A detailed description of the experimental set-up and preliminary tests can be seen elsewhere (Oliveira, 1992).

2.2. Experimental procedure

A full factorial planing at three levels was used to analyze the effect of spouting gas temperature, T_{gi}, mass flow rate of atomized suspension relative to spouting gas, W_s/W_g, and the gas flow rate relative to gas flow at minimum spouting, Q/Q_{ms},

Table 5
Analysis of variance for the fraction of solids incorporated by the particles, η

Variable	Sum of squares	Degrees of freedom	Mean square	F_{CALC}
T_{gi}	34.30	2	17.15	0.46
$T_{gi(\ell)}$	26.89	1	26.89	0.73
$T_{gi(s)}$	7.41	1	7.41	0.20
W_s/W_g	369.41	2	184.71	4.99[a]
$W_s/W_{g(\ell)}$	346.72	1	346.72	9.37[a]
$W_s/W_{g(s)}$	22.69	1	22.69	0.61
Q/Q_{ms}	76.08	2	38.04	1.03
$Q/Q_{ms(\ell)}$	53.39	1	53.39	1.44
$Q/Q_{ms(s)}$	22.69	1	22.69	0.6
Iteration				
$T_{gi(\ell)}$ $W_s/W_{g(\ell)}$	21.33	1	21.33	0.58
$T_{gi(s)}$ $W_s/W_{g(\ell)}$	28.44	1	28.44	0.77
$T_{gi(\ell)}$ $W_s/W_{g(s)}$	32.11	1	32.11	0.87
$T_{gi(s)}$ $W_s/W_{g(s)}$	19.59	1	19.59	0.53
$T_{gi(\ell)}$ $Q/Q_{ms(\ell)}$	6.75	1	6.75	0.18
$T_{gi(s)}$ $Q/Q_{ms(\ell)}$	10.03	1	10.03	0.27
$T_{gi(\ell)}$ $Q/Q_{ms(s)}$	10.03	1	10.03	0.27
$T_{gi(s)}$ $Q/Q_{ms(s)}$	3.34	1	3.34	0.09
$W_s/W_{g(\ell)}$ $Q/Q_{ms(\ell)}$	3.00	1	3.00	0.08
$W_s/W_{g(s)}$ $Q/Q_{ms(\ell)}$	2.78	1	2.78	0.08
$W_s/W_{g(\ell)}$ $Q/Q_{ms(s)}$	2.78	1	2.78	0.08
$W_s/W_{g(s)}$ $Q/Q_{ms(s)}$	1.81	1	1.81	0.05
Error				
W_s/W_g T_{gi} Q/Q_{ms}	296.08	8	37.01	—
Total	917.86	26	—	—

Subscripts ℓ and s are the linear and square effects.
[a] Term is significant at 0.01 level.

on coating operation. The static bed height was maintained constant at 11 cm, and the feed flow rate of atomizing air at 0.0167 kg/min and 1.0 kgf/cm^2. The air flow rate at minimum spouting, Q_{ms}, was determined through measurements of pressure drops in the particles bed as a function of gas flow rate introduced (Mathur and Epstein, 1974). These measurements were made without atomization of the coating suspension in the bed and with an inlet gas temperature of 25°C. The value of 0.93 m^3/min for Q_{ms} was obtained.

The coating operation started with the introduction of a determined load of alumina particles into the bed. The spouting of this load was promoted by air injected at the base of the bed. As soon as spouting was established, the air was heated to the desired temperature. After reaching thermal equilibrium, the feeding of the coating suspension with a pre-set flow rate and ambient temperature, as well as the atomizing air, were started. Samples of coated particles were withdrawn from the bed, in the spout zone, at regular intervals. The samples were dried in an oven at 85°C and weighed so that the mean particle mass as a function of process time, $\bar{m}_p$ could be determined.

The experimental values of the rate of increase in particle mass, K_1, the mass fraction of atomized solids incorporated by the particles, η, and the particle diameter as a function of process time were estimated by Eqs. (1)–(3), respectively:

$$K_1 = \frac{\bar{m}_p - \bar{m}_{p0}}{\theta \bar{m}_{p0}} \quad (1)$$

$$\eta = \frac{n_0(\bar{m}_p - \bar{m}_{p0})}{W_s C_s \theta} \quad (2)$$

$$\frac{\bar{d}_p}{\bar{d}_{p0}} = \left[\frac{6\bar{m}_p}{\pi\rho_{coat}\bar{d}_{p0}^3} + \left(1 - \frac{\rho_p}{\rho_{coat}}\right)\right]^{1/3} \quad (3)$$

where C_s is the mass fraction of solids in the coating suspension, n_0 the initial number of particles in the bed, Θ the coating time and ρ_{coat} the density of the coating layer.

By replacing Eq. (1) on Eq. (3), the following model to describe the particle diameter can be obtained:

$$\frac{\bar{d}_p}{\bar{d}_{p0}} = (1 + K_2\theta)^{1/3} \quad (4)$$

where:

$$K_2 = \rho_p/\rho_{coat}K_1 \quad (5)$$

3. Results and discussion

A linear increase in particle mass with the coating time was observed in all tests and is illustrated in Fig. 2. Also, the particle mass maintained its log-normal distribution (Fig. 3) during the coating. These trends, together with stable operation of the spouted bed, are indicators of the feasibility of the spouting technique for particle coating.

Table 3 shows the experimental results of the rate of increase in the particle mass, K_1, and of the fraction of solids incorporated by the particles, η. An analysis of variance for these results, using the F-test, was performed and the interaction between the three variables studied was used to estimate the individual significance (Montgomery, 1976; Box and Draper, 1987). The analysis of variance results are summarized in Tables 4 and 5, for K_1 and η, respectively. It reveals that the process was insensitive to spouting gas temperature, T_{gi}, and that the mass flow rate of atomized suspension relative to spouting gas, W_s/W_g, was the variable exerting more influence in the coating operation (significance level smaller than 0.01). The experimental results of the rate of increase in the particle mass, K_1, and of the fraction of solids incorporated by the particles, η, as a function of W_s/W_g, having Q/Q_{ms} as a parameter, is shown in Figs. 4 and 5, respectively, for the three temperatures studied.

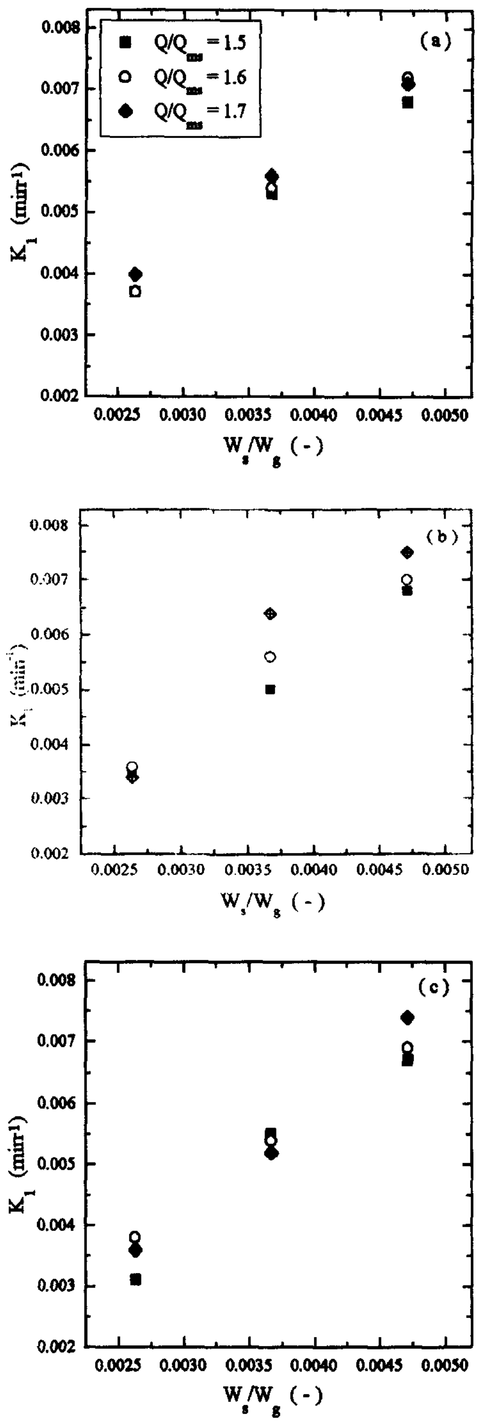

Fig. 4. Rate of increase in particle mass, K_1, as a function of W_s/W_g, having Q/Q_{ms} as a parameter with T_{gi} equal to (a) 67, (b) 75 and (c) 83°C.

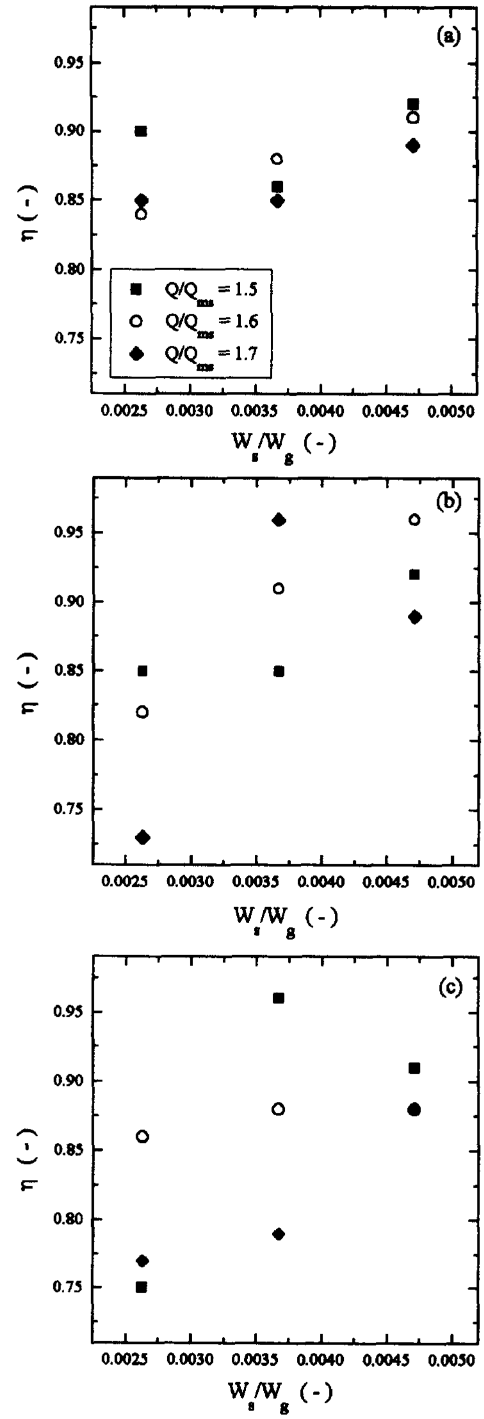

Fig. 5. Fraction of solids incorporated by the particles, η, as a function of W_s/W_g, having Q/Q_{ms} as a parameter, with T_{gi} equal to (a) 67, (b) 75 and (c) 83°C.

As expected, K_1 increases with W_s/W_g for all temperatures. A slight increase in K_1 with Q/Q_{ms} is observed (except for $T_{gi} = 83$°C), and this may be due to an increase in the inertial interception of atomized droplets by the bed particles. The inlet air temperature, T_{gi}, seems to have no effect on K_1, confirming the analysis of variance result.

As for η, a more complex behavior of the coating process is apparent: at $T_{gi} = 67$°C, η shows a certain increase with W_s/W_g, and a slight tendency to decrease with Q/Q_{ms}. For higher temperatures, these trends progressively vanish. This reveals a complex balance between the mechanisms of particle growth (adhesion of droplets), particle erosion (abrasion and bouncing) and reentrainment of solids (eroded from bed particles and dried material from the feed suspension), and all are probably affected by the bed temperature. The increase in the bed temperature increases the drying rate and, consequently, the overall bed mobility (as the average bed moisture decreases), favoring the dispersion of the feeding suspension. This would increase bed circulation and η, as the number of cycles of particle through the atomizing zone increases. On the other hand, the drying of droplets of feeding suspension before reaching the particle surface increases with bed temperature, thus decreasing its collection by the growing particle. These competing effects may explain the observed behavior for η shown in Fig. 5.

The experimental results of K_1 and η were correlated with W_s/W_g and Q/Q_{ms} by statistical methods, and the best fits were:

$$K_1 = 0.143\left(\frac{W_s}{W_g}\right)^{0.440}\left(\frac{Q}{Q_{ms}}\right)^{0.255} - 0.00817 \quad (6)$$

$$\eta = 6.050\left(\frac{Q}{Q_{ms}}\right) - 1.944\left(\frac{Q}{Q_{ms}}\right)^2 + 174.156\left(\frac{W_s}{W_g}\right) - 17977.0\left(\frac{W_s}{W_g}\right)^2 - 4.204 \quad (7)$$

Fig. 6 shows a comparison between the experimental and calculated values of K_1 and η, having W_s/W_g and Q/Q_{ms} as parameters in Fig. 6(a) and (b), respectively. The average deviation between the experimental data and the estimates obtained by equations adjusted was less than 3.9% for K_1 and for η, whereas the maximum deviation does

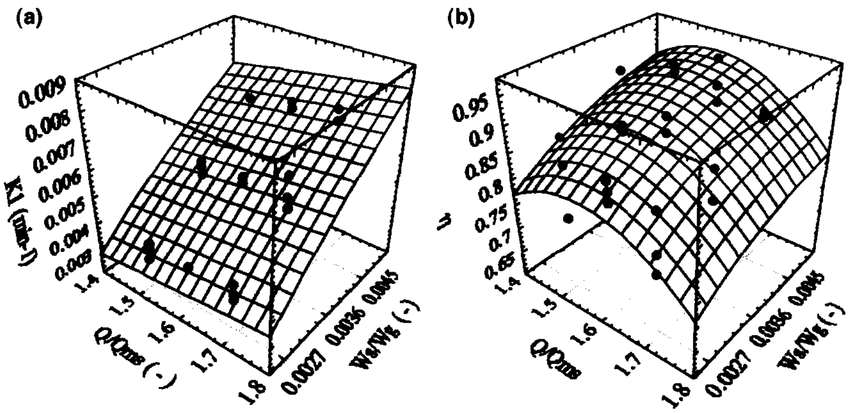

Fig. 6. Comparison between the experimental and calculated values of K_1 and η, having (a) W_s/W_g and (b) Q/Q_{ms} as parameters.

not exceed 11.2% for all experiments realized. Eqs. (6) and (7) are valid in the following ranges: $1.5 \le Q/Q_{ms} \le 1.7$; $67.0°C \le T_{gi} \le 83.0°C$; $0.00263 \le W_s W_g \le 0.00471$.

No reliable correlation for K_1 and for η in terms of the inlet gas temperature, T_{gi}, could be derived as the process was quite temperature-independent, within the studied range.

The last stage was the verification of the validity of Eq. (4) to describe the kinetics of growth, using Eqs. (5) and (6) to estimate K_2. The experimental value of particle diameter was calculated by Eq. (3) (mean mass diameter). The value of ρ_{coat} was determined by measurements of thickness and weight of the coating layer of a sample of particle using a micrometer and a analytical balance, respectively. The value of 1.33 g/cm^3 was obtained. Fig. 7 shows the comparison of the experimental values of particle diameter relative to initial diameter with estimates obtained by Eqs. (5) and (6). From this figure we can be see the good agreement between the experimental and calculated values, with deviations less than $\pm 3\%$ for all experiments realized. This confirm the applicability of the proposed model to estimate the kinetics of growth during spouted bed coating of particles.

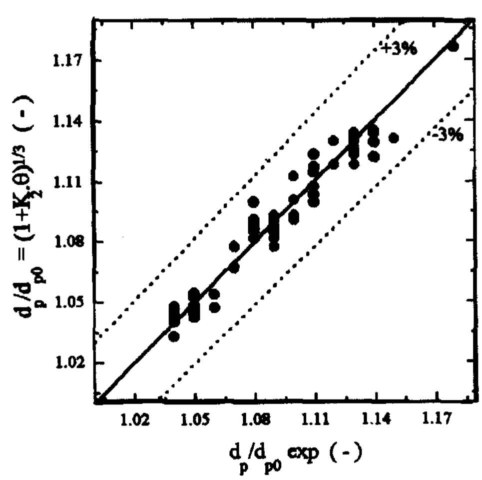

Fig. 7. Comparison of the experimental values of particle diameter relative to initial diameter with estimates obtained by Eqs. (4)–(6).

4. Conclusions

(1) Particle mass increased linearly with coating time, maintaining the log-normal distribution of the original alumina particles.

(2) The rate of increase in the particle mass and fraction of solids incorporated by the particles varied significantly with flow rate of spouting gas and with feed mass flow rate of the coating suspension.

(3) The experimental results of the rate of increase in the particle mass and of the fraction of solids incorporated by the particles obtained in this study can be estimated by Eqs. (6) and (7), respectively.

(4) In the ranges analyzed, the coating process, as a whole, was insensitive to inlet gas temperature.

(5) The proposed model to estimate the kinetics of growth during spouted bed coating of particles showed good agreement with experimental data.

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