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RESEARCH PAPER

Evaluation of the Tablet Coating by the Conventional Spouted-Bed Process

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ABSTRACT

The purpose of this paper was to present an analysis of the tablet coating by the conventional spouted-bed process. To analyze the equipment performance, the rate of increase of the tablets mass, K_1*, and the adhesion coefficient* η *were determined as a function of the feed flow rate of coating suspension* W_s*; of the Reynolds number* R_{ep}*; of the flow rate of atomizing gas* W_{at}*, and of the cone base angle* γ*. To analyze the product quality, the uniformity of coating mass deposition onto the tablet's surface was used. Three different procedures for description of kinetics growth, weighing method, image analysis, and measurements with a micrometer were used to verify the validity of the commonly used weighing method. Comparison between experimental results of kinetics growth with estimates obtained by a literature model was also performed. A tendency toward an increase in* K_1 *and in* η *with the feeding flow rate of coating suspension* W_s *was detected. The weighing method can be used for the process analysis. The kinetics of growth can be described by the growth model used. The variable that produce more pronounced effect on* K_1 *and* η *was the feed flow rate of coating suspension, the weighing method describes very well the increase of particle diameter with coating time, the growth model can be used for the describe the kinetics of growth during the coating operation, and the coating does not deposit uniformly onto the tablet's surface.*

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INTRODUCTION

Studies presently reported have the purpose of development and optimization of the methods for coating application in agrochemical, chemical, and pharmaceutical products such as seeds, pesticides, fertilizers, nuclear compounds, tablets, pills, capsules, and granules. The objective is apply a thin, uniform layer of one material onto the surface of a body to serve multiple purposes (1–7). The equipment usually used in these operations include the coating pans and spouted and fluidized beds (2,8).

The advantage of coating by spouted beds lies in the formation of a uniform coating layer in a short processing time. The technique can be applied to the materials with distinct physical chemical properties except friable and brittle materials. The control of operational conditions should be very strictly maintained to avoid the blocking of the process to caused by excessive humidity inside the bed.

Knowledge of the effect of operational conditions on the spouted-bed coating process is important for optimal design, construction, and operations of this type of equipment. To obtain more information about such operations, this study was developed with the objective of analyzing effects of operational conditions in spouted-bed coating operations. Sugar-base suspension were used because they permit higher growth rates in reduced experimental time to be obtained.

EXPERIMENTAL PROCEDURES

Materials

Placebo tablets with a concave cylindrical shape were used for the coating application. The mean physical properties of the tablets are presented in Table 1, where d_{p0} is the mean particle diameter, m_{p0} is the mean particle mass, ρ_p is the particle density, and ϕ is the shape factor.

The mean particle mass was determined by weighting a sample of 20 tablets. The particle and coating density, shape factor, and mean diameter were estimated by two different procedures: direct measurement and photographic analysis. In the first procedure, measurement of the three characteristic dimensions of samples of tablets were performed with a micrometer. Figure 1(a) shows a scheme of characteristic dimensions used. The second procedure is based on image analysis of photography of tablets samples, as shown in Figure 1(b). These analyses were performed with the aid of a system for image analysis (9).

Table 1.

Mean Physical Properties of Tablets Used

d_{p0} (mm)	m_{p0} (mg)	ρ (g/cm^3)	ϕ (−)
7.10 ± 0.1	250.0 ± 11.0	1.23 ± 0.03	0.90 ± 0.0005

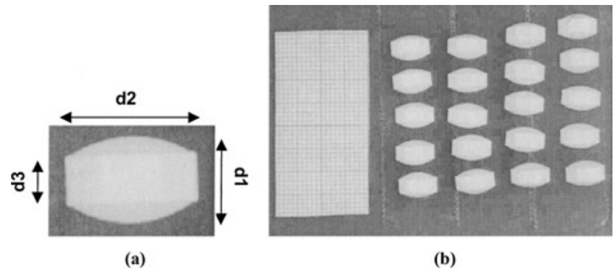

Figure 1. Characteristic dimensions and photography in scale of samples of tablets, Figure (a) and (b), respectively.

A suspension containing (by weight) 38.7% sucrose, 20.0% talc, 32.6% distilled water, 4.7% titanium dioxide, and 4.0% carbowax 6000 and dye was used as coating material (in weight). Density, ρ_s; surface tension, δ_s; and viscosity, μ_s of this suspension were determined by picnometry, tensiometer (Fisher Scientific, Surface Tensiomatic 21), and Brookfielf bob and cup rheometer (DV3 model). Table 2 shows the results obtained.

The experiments were carried out in a spouted bed that consisted of a conical base with an inlet orifice diameter of 52 mm, connected to a cylindrical column with a diameter of 200 mm and a height of 400 mm. All parts were made of stainless steel. Two different base conical angle were used (40 and 60°, respectively). A double fluid atomizer was installed in the bottom of the bed, and the coating suspension was fed concurrently with the spouting air. A peristaltic pump was used to feed the mass flow rate of coating material to the equipment. Figure 2 shows an schematic diagram of experimental set-up.

Experimental Procedure

The coating operation started with the introduction of a load of tablets into the bed. The spouting of this load was promoted by air injected at the base of the bed. As

Table 2.

Physical Properties of Coating Suspension Used

ρ_s (g/cm^3)	δ_s (dyn/cm^2)	μ_s[a] (g/cm · s)
1.48 ± 0.03	61.2 ± 0.1	0.685 ± 0.0014

[a] Plastic behavior with $\tau_0 = 6.4 \pm 0.2$ dyn/cm^2.

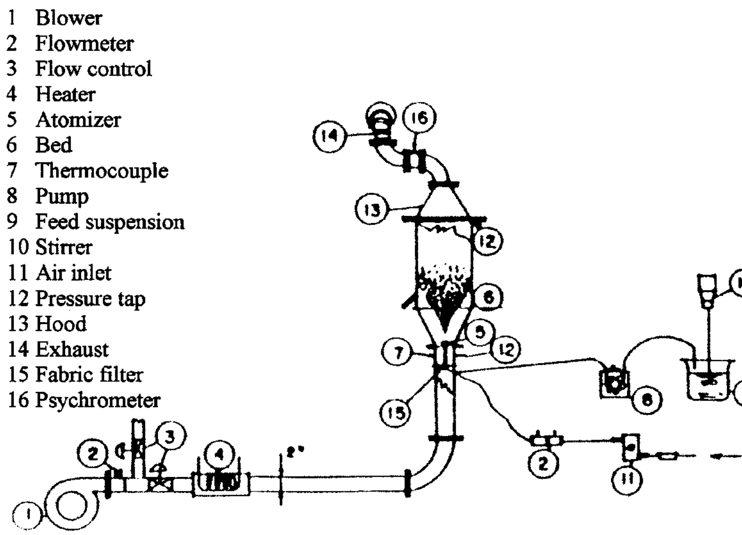

Figure 2. Schematic diagram of the experimental set-up.

soon as spouting was established, the air was heated to the desired temperature. After reaching thermal equilibrium, feeding of coating suspension with a preset flow rate and ambient temperature, as well as the atomizing air, were started. Samples of tablets were withdrawn from the bed, in the spout zone, at regular intervals before and after the process started. The samples were dried in an oven at 85°C and weighed so that the mean particle mass as a function of process time, m_p, could be determined.

The experimental data of mean particle mass as a function of processing time thus determined should be corrected to compensate for the deviation caused by the samples withdrawn. For this, the following equation was used (10).

$$m_p = m_{p0} + \frac{1}{n_0} \cdot \sum_{i=1}^{N} \left(m_{p(i)} - m_{p(i-1)}\right) \cdot n_{(i)} \tag{1}$$

In Eq. (1), n_0 is the initial number of particles in the bed and i is the sample number. Using the corrected data, the experimental value of increase rate of particle mass K_1, the adhesion efficiency η, and the increase of particle diameter as a function of processing time d_p can be estimated by Eq. (2) to (4), respectively (2,10,11).

$$K_1 = \frac{m_p - m_{p0}}{\theta \cdot m_{p0}} \tag{2}$$

$$\eta = \frac{n_0 \cdot (m_p - m_{p0})}{W_s \cdot C_s \cdot \theta} \tag{3}$$

$$\frac{d_p}{d_{p,0}} = \left[\frac{6 \cdot m_p}{\pi \cdot \rho_{coat} \cdot d_{p,0}^3} + \left(1 - \frac{\rho_p}{\rho_{coat}}\right)\right]^{1/3} \tag{4}$$

To use Eq. (4), it is only necessary to determine the values of mean particle mass as a function of processing time, knowing the value of value of coat density ρ_{coat}. In this work, this method was called the weighing method. A comparison between estimates obtained by Eq. (4) with measurements of particle diameter obtained by image analysis and by measurements with a micrometer was made to verify its validity for description of real particle diameters.

By substituting Eq. (3) in Eq. (4), the following model to describe the particle diameter can be obtained:

$$\frac{d_p}{d_{p,0}} = \left(1 + \frac{\eta \cdot W_s \cdot C_s \cdot \theta}{n_0} \frac{\rho_p}{\rho_{coat}}\right)^{1/3} \tag{5}$$

The uniformity of coating deposition onto tablets surface was determined via measurement of the dimensions of the coated and noncoated tablets. Two characteristics were used in this analysis, the variation of shape factor ϕ, and the uniformity ratio R, defined by the following:

$$\phi = \frac{\text{surface area of equivalent sphere}}{\text{particle surface area}} \tag{6}$$

$$R = \frac{d_{1,f} - d_{1,0}}{d_{2,f} - d_{2,0}} \tag{7}$$

A value near 1 represents a uniform deposition of coating material on the whole surface of the tablets. Table 3 lists the parameters and operational ranges investigated.

Table 3.

Parameters and Operational Ranges Investigated

Parameters	Symbol	Ranges
Feed flow rate of coating suspension (mL/min)	W_s	2.0–8.5
Atomizing air pressure (kgf/cm^2)	P_{at}	1.5
Atomizing air flow rate (l/min)	W_{at}	9.1–26
Spouting gas temperature (°C)	T_{gi}	65
Air flow rate relative to minimum spouting* (−)	Q/Q_{ms}	1.3–1.7
Particle Reynolds number (−)	R_{ep}	256.3–405.2
Initial bed mass (kg)	M_0	0.5–1.0
Concentration of coating suspension (kg/kg)	C_s	0.674
Cone base angle (degree)	γ	40–60

RESULTS AND DISCUSSION

For all tests performed, a linear increase in tablet mass with the coating time was observed. It is illustrated in Figure 3, where we can observe that the increase rate of particle mass is strongly dependent of feed flow rate of coating composition. However, there is a limiting feed flow rate of coating suspension above which collapse of the process caused by excessive tablet surface humidity can occur.

Using the procedure presented in section 2, the experimental values of increase rate of particle mass K_1 and of adhesion efficiency η were determined for all conditions analyzed. Figures 4 to 6 and 5 to 7, respectively, show the experimental results of K_1 and η as a function of the Reynolds number, R_{ep}; the conical base angle, γ; and the feed flow rate of atomizing gas, W_{at}, having the feed flow rate of coating suspension as a parametric index, W_s.

Confirming previous results (11), we can see that both K_1 and η show a tendency to increase proportionally with feed flow of coating suspension, W_s. This behavior is expected because an increase in W_s increases the humidity inside the equipment as well as the humidity with which the atomized material reaches the tablet surface, leading to an increase in the adhesion forces particle coat. An increase in Rep did not significantly affect K_1. However, the adhesion efficiency showed a more complex behavior with this parameter, presenting a maximum value dependent on the feed flow rate of the coating suspension, W_s

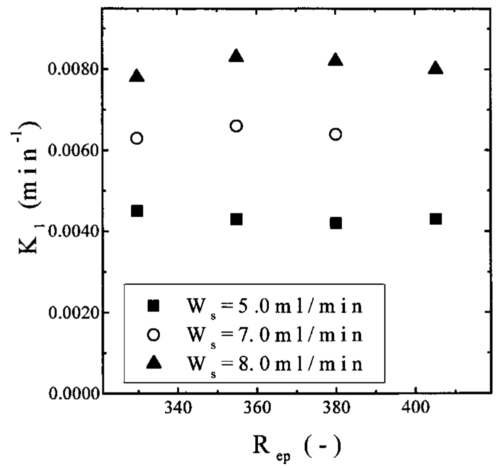

Figure 4. K_1 as a function of the R_{ep}, with W_s as parametric index ($W_{at} = 15.0$ L/min, $\gamma = 60°$, $M_0 = 1.0$ kg).

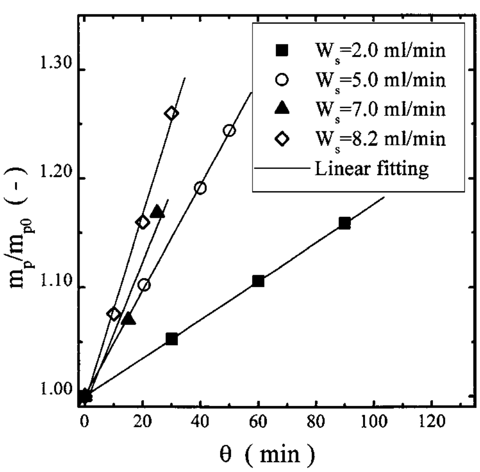

Figure 3. Increase in tablet mass as a function of coating time.

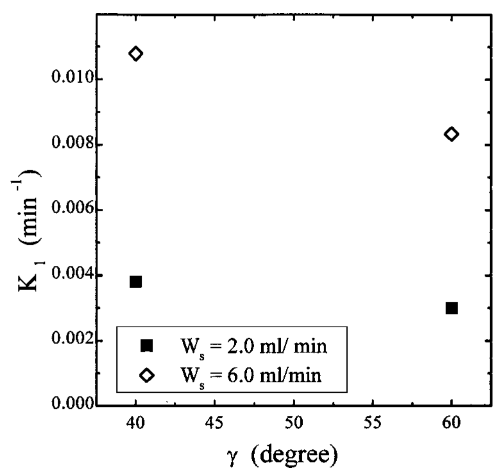

Figure 5. K_1 as a function of the of γ, with W_s as parametric index ($W_{at} = 15.0$ L/min, $R_{ep} = 292.5$, $M_0 = 0.5$ kg).

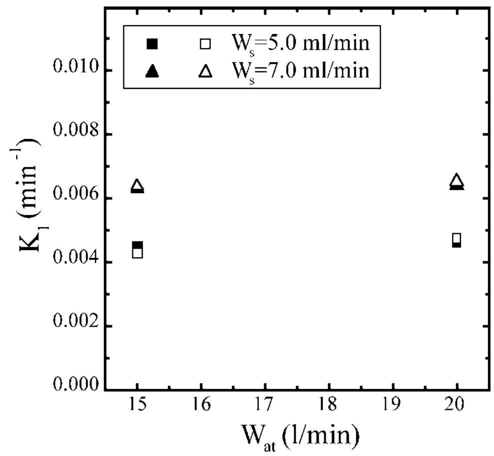

Figure 6. K_1 as a function of W_{at}, with W_s as parametric index ($\gamma = 60°$, $M_0 = 1.0$ kg. Solid symbol: $R_{ep} = 329.7$; open symbols: $R_{ep} = 380$).

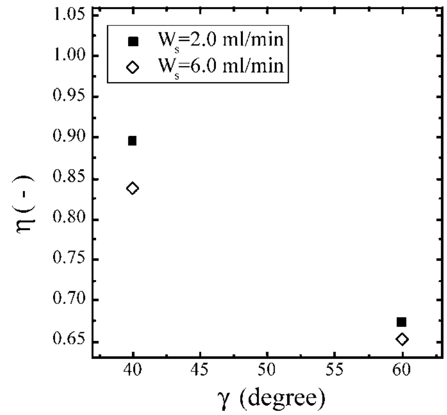

Figure 8. η As a function of γ, with W_s as parametric index ($W_{at} = 15.0$ L/min, $R_{ep} = 292.5$, $M_0 = 0.5$ kg).

(see Fig. 4). Reduction of the base conical angle produced an increase of K_1 and adhesion efficiency. A plausible explication for this behavior could be related to particle concentration in the front of the atomizing zone facilitating direct interception of droplets of the coating material. Both K_1 and η showed a slight tendency to increase with increased feeding flow rate of atomizing gas. Higher values of W_{at} produce small atomized droplets of coating material. Small droplets can be distributed more easily to the particle surface and can also facilitate the direct interception by the particles, leading to an increase on K_1 and η in the experimental range. The experimental results of adhesion coefficient obtained in this work were compared with the correlations presented by Kucharski and Kmiec (2) and by Oliveira et al. (11).

Unrrealistic estimates of adhesion efficiency were obtained by the correlation proposed by Kucharski and Kmiec (2). This is an indication that this Eq. is rather limited to the experimental conditions used by this author. In contrast, the comparison between the estimates obtained from correlation proposed by Oliveira et al. (11) shows good agreement with most experimental data with a mean deviation of 8.74%. Figure 10 shows the results of this comparison.

Evaluation of the kinetics of growth by comparison of three different techniques to measure the particle diameter

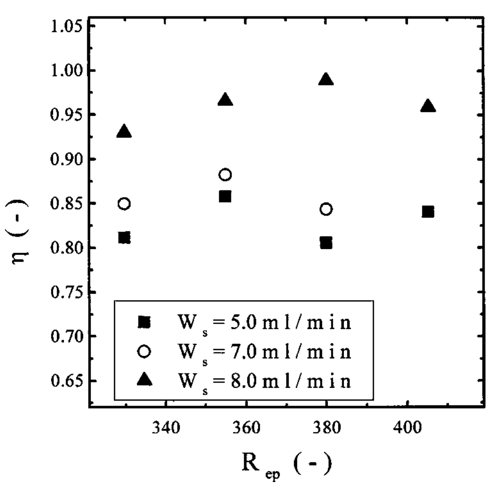

Figure 7. η As a function of the R_{ep}, with W_s as parametric index ($W_{at} = 15.0$ L/min, $\gamma = 60°$, $M_0 = 1.0$ kg).

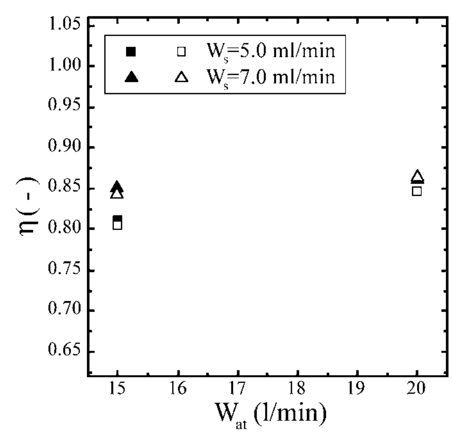

Figure 9. η As a function of W_{at}, with W_s as parametric index ($\gamma = 60°$, $M_0 = 1.0$ kg. Solid symbol: $R_{ep} = 329.7$; open symbols: $R_{ep} = 380$).

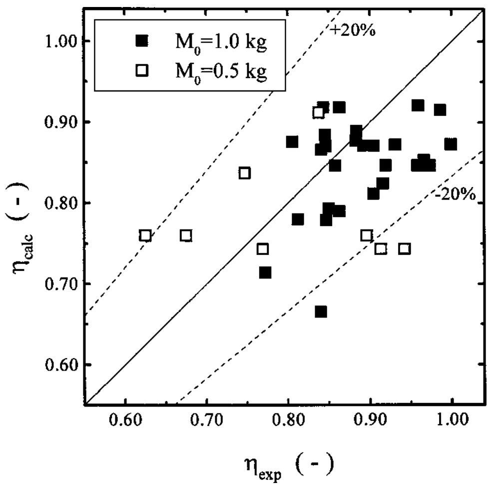

Figure 10. Comparison of experimental and calculated value adhesion efficiency.

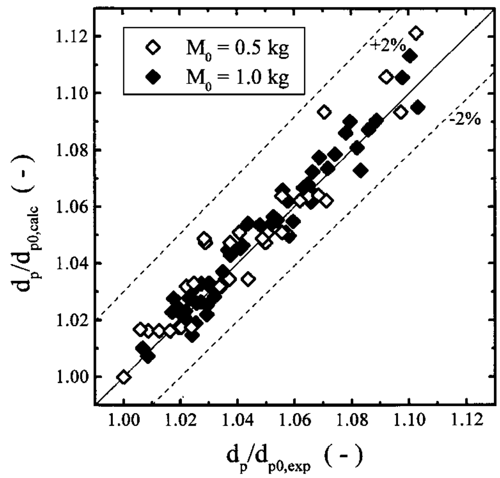

Figure 12. Comparison of experimental mean mass diameter with values obtained by the growth model (Eq. 5).

(image analysis, measurement with a micrometer, and weighing method) was carried out to validate the utilization of Eq. (4) to describe the particle diameter. The results of these comparison are presented in Figure 11, in which it can be observed that the increase in particle diameter with coating time was well represented by the weighing method, the differences with image analysis being nonsignificant (<0.5%). In contrast, a difference of approximately 2% between the image analysis and weighing method in one side and measurements with a micrometer on the other was observed. Thus, it may be concluded that the weighing method can be used to estimate the increase in particle diameter as a function of coating time.

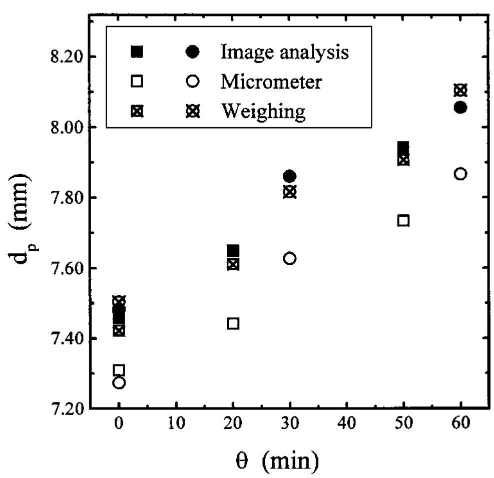

Figure 11. Comparison of the experimental values of particle diameter as a function of processing time obtained by the image analysis technique, by measurement with a micrometer, and by weighing ($W_s = 5.0$ mL/min; $W_{at} = 20$ L/min; $\gamma = 60°$; $M_0 = 1.0$ kg. Square: $R_{ep} = 380.0$; circle: $R_{ep} = 329.7$).

Values of coating density were estimated through weighing and dimension measurements of samples of coated and uncoated tablets. A mean value of 1.28 ± 0.34 was obtained. The values of particle diameter obtained by the weighing method were compared with estimates obtained by the growth model, using the Eq. proposed by Oliveira et al. (11) to describe the adhesion efficiency. Figure 12 showing this comparison indicates that the estimates obtained by the model used are in excellent agreement with experimental data, with the maximum deviation being $< \pm 2\%$ for all runs. Thus, Eq. (4) belonging with the correlation proposed by Oliveira et al. (11) can be used to estimate the experimental results of kinetics of growth obtained in this work.

To verify the uniformity of coating deposition onto a tablet's surface, the shape factor, ϕ, and the uniformity ratio, R, were determined. Figure 13 shows a typical result of shape factor, ϕ, as a function of process time. It can see that ϕ present a tendency to increase during the spouted-bed coating process. This indicates nonuniform coating deposition onto the tablet's surface. To confirm this, the uniformity ratio, R, was calculated for the experiments presented in Figure 13. Uniformity values equal to 1.75 and 1.57 were obtained, confirming the view that the coating material does not distribute uniformly over the whole tablet surface, with the extremities receiving less coating material than the upper and lower central parts. This information should be considered in the development of special applications such

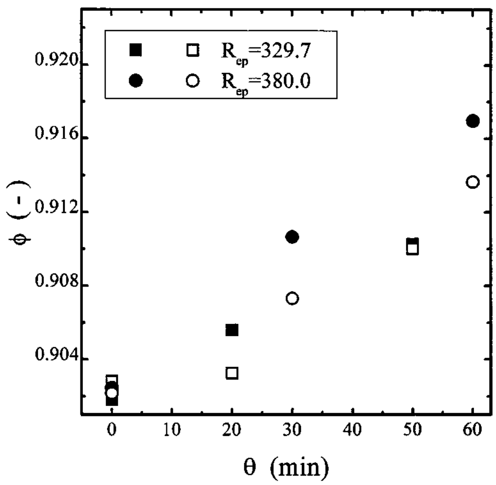

Figure 13. Variation of shape factor estimated by the image analysis and by measurement with a micrometer evaluated as a function of process time (closed and open symbols, respectively: $W_s = 5.0$ mL/min; $W_{at} = 20$ L/min; $\gamma = 60°$; $M_0 = 1.0$ kg).

as gastric resistant- and retard-release coatings, which are extremely dependent of the thickness of the coating barrier.

CONCLUSIONS

The following can be shown by this work:

1. K_1 and η show a tendency to increase with W_s;
2. An increase in R_{ep} did not significantly affect K_1;
3. η Showed a maximum, dependent on R_{ep} and the feeding flow rate of coating suspension;
4. K_1 and η tend to decrease conversely with the conical base angle, γ;
5. The feed flow rate of atomizing gas produced an increase in K_1 and η;
6. The experiment shows good agreement with the previously published work of Oliveira et al. (11);
7. The weighing method describes very well the increase in particle diameter with coating time;
8. The growth model can be used for the describe the kinetics of growth; and
9. The coating does not deposit uniformly onto the tablet's surface, with the extremities receiving less coating material, increasing the shape factor, ϕ, and the uniformity value, R.

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