

An Apparatus and Method for the Coating of Solid Particles

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A method for the coating of discrete solid particles and a diagram of the apparatus used is presented. In this process, solid particles are educted from a fluidized bed by a turbulent gas jet, separated into discrete units, and transported upward through a converging cone into a coating application zone. In this zone, the solid particles are contacted by discrete liquid droplets comprising the coating material. These liquid droplets are entrained in a downward moving airstream which has a greater velocity than the upward moving solid particles. The velocity differential removes the wetted solid particles from the coating zone and returns them to the fluid bed. The coating liquid is spread over the surface of the solid particles in this bed prior to their reeducation into the turbulent gas jet. The cycle is repeated until coatings of the desired kind and thickness are obtained.

COATING PHARMACEUTICAL DOSAGE forms of their component particles is an old and extensive practice. The purpose of coating has been to modify chemical or physical properties, such as taste, appearance, solution rate, reactivity, sensitizing or irritation potential, flowability, compressibility, dusting propensity, *etc.* Methods or apparatus used in the pharmaceutical industry to coat solid particles include rotating pans (1), fluid and spouted beds (2, 3), and tablet presses (4). Coacervation (5), spray-drying (6-8), and emulsification (9) have been used to entrap or encapsulate solid particles.

The vast majority of coating is done in rotating pans. This predilection for one apparatus is due, in part, to tradition and to this industry's large investment in pan coating equipment and technology. It is also because the more esoteric processes are often complex, costly, and in some instances have minimal advantage over pan coating. This combination of factors has resulted in a tendency for the pharmaceutical industry to coat only those particles which can be handled in a rotating pan. This is unfortunate because it limits the obvious utility of coating to large monosized particles, and limits the coating materials to a few nonadhesive substances. It appeared desirable to develop a simple, inexpensive apparatus and method capable of coating most of the solid particles used in the pharmaceutical industry with a wide variety of coating materials. This report describes and outlines the method of operation for a coating device developed in this laboratory (10). This device has been used to coat solid particles that have mean diameters ranging from about 10 to $10^3 \mu$ with materials having widely different properties. It is simple, inex-

pensive, and appears to have desirable characteristics not possessed by other coating equipment.

Subsequent reports will present an evaluation of coated particles prepared using this device and illustrate the utility of using coated particles in the formulation of pharmaceutical dosage forms.

DISCUSSION

Description of the Coating Apparatus—A schematic drawing of a laboratory size particle coating unit is shown in Fig. 1. It consists of a conical base (A), vertical casing (B), expanded upper section (C), and a gas-vapor exhaust system (D). The bottom of the unit contains a receptacle for air inlet nozzles (E) of varying cross-section and design. A converging tube (F) is centered in the vertical column and this tube can be moved vertically up or down. An air ring (G) with a series of holes on its inner surface is located between the conical base and vertical casing; the air inlet holes are slightly above the base of the converging tube. A vertically adjustable tube (H) is centered in the expanded upper section and this tube contains gas and liquid feed lines. The lower end of tube H is designed to accept atomizing nozzles of varying design. The center of the atomizing nozzle is in precise alignment with the centers of E and F. The gas vapor exhaust system

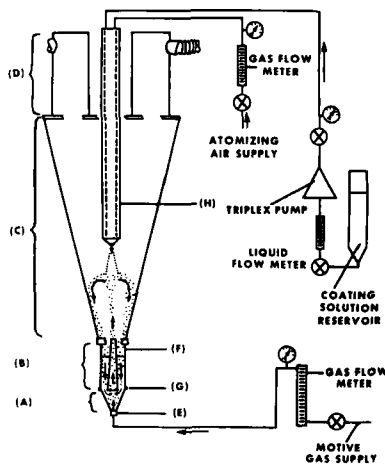


Fig. 1—A schematic representation of the particle coating device.

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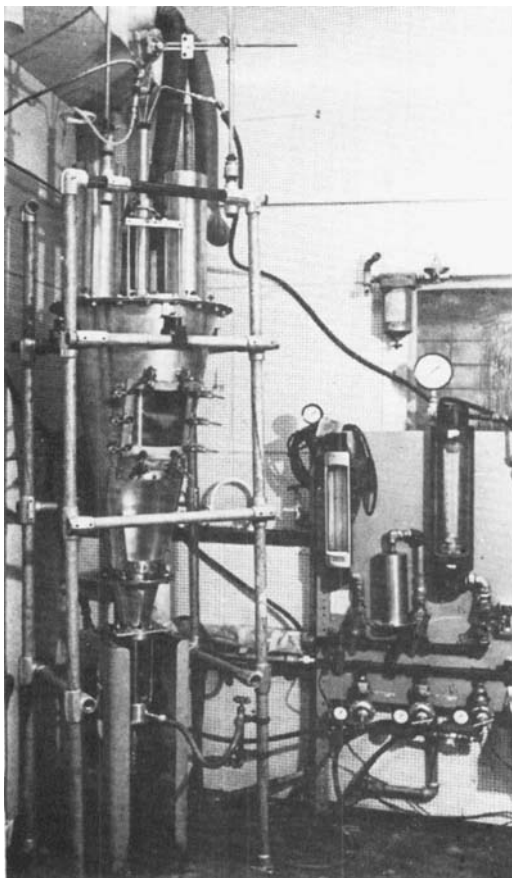


Fig. 2—The laboratory model particle coating device.

contains two frame-supported cloth bags enclosed in cylindrical caps. These caps contain outlets which are connected with flexible hoses to an exhaust blower. Combination sight and fill ports are located in the top and side of the expanded upper section. Air actuated vibrators¹ are attached to B and C. All metal parts are fabricated from stainless steel.

The gas and liquid feed systems are also shown in Fig. 1. Liquid wetted parts are either glass or stainless steel. Compressed air is obtained from a compressor² capable of supplying 100 s.c.f.m. at 100 psia. Air entering the device is filtered³ and dried to a dew point of -40°C . by passing it through a silica gel column.⁴ Gases other than air, such as nitrogen or carbon dioxide, are obtained from pressurized cylinders. A thermostatically controlled heater⁵ having a range of $150\text{--}550^{\circ}\text{F}$. is used to heat the gas when indicated. Solutions of the coating material are pumped to the atomizing nozzle by a triplex metering pump.⁶ Flowmeters⁷ are used to measure both gas and liquid flow and this flow is controlled by conventional valves and pressure gauges. Photographs of a laboratory and pilot unit are shown in Figs. 2 and 3.

¹ Vibrolator, Martin Engineering Co., Neponset, Ill., and Piston Vibrator, Branford Vibrator Co., New Britain, Conn.

² Gardner Denver, Quincy, Ill.

³ Dollinger, Rochester, N. Y.

⁴ Oriad, C. M. Kemp Mfg. Co., Glen Burnie, Md.

⁵ Chromalox, Edwin L. Weigand Co., Pittsburgh, Pa.

⁶ Wallace and Tiernan, Belleville, N. J.

⁷ Fischer-Porter, Warminster, Pa.

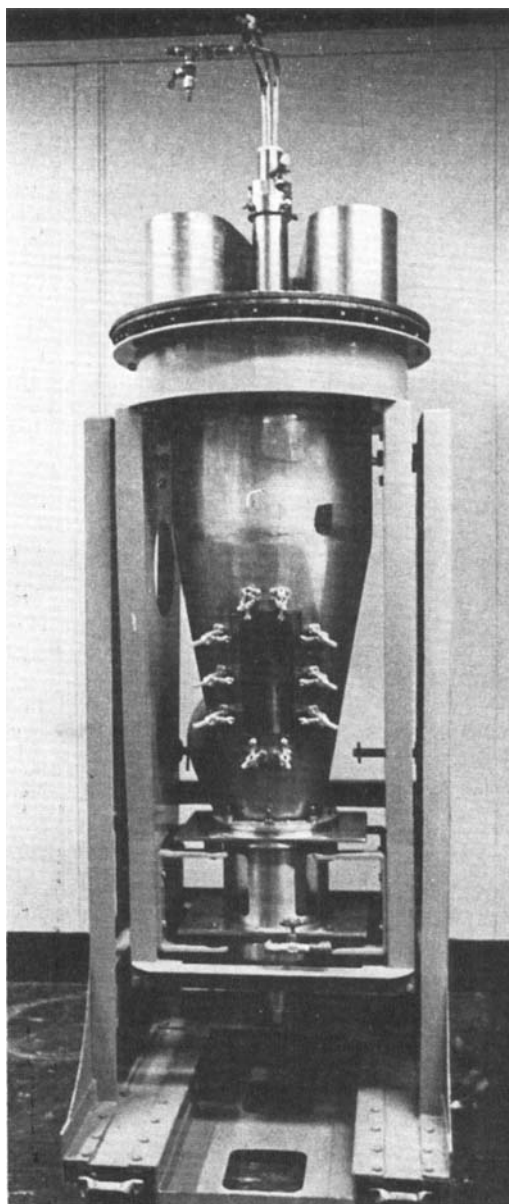


Fig. 3—The pilot model particle coating device.

Operating Procedure—The coating process can be subdivided into distinct phases. These phases include: (a) separating of the particle assemblage into discretely separate units of the desired size, (b) applying of liquified coating material onto the surface of these units, (c) spreading of the coating material uniformly over the surface of the units, (d) continuing of phases a through c until the required coating thickness is obtained, and (e) solidifying of the coating and collecting of the coated particles. The flow of particles in the coating device is shown schematically in Fig. 1. Particles introduced through an inlet port form a dense phase bed in the annular space between the walls of the vertical column B and the converging tube as shown in Fig. 4. The particles are fluidized by the action of the vibrators and this fluid state can be augmented by

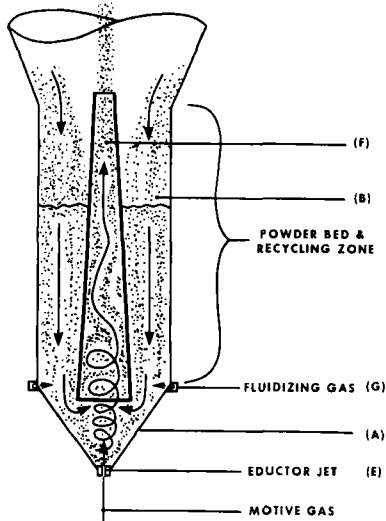


Fig. 4—A schematic representation of the particle collection and separation section.

passing gas through the holes in the air ring G. A jet of gas moving through nozzle E educts particles from the base of this bed as distinctly separate units and transports these units upward through the converging tube F. The frequency and amplitude of vibration, gas velocity, and particle flow rate are adjusted to provide particles of the desired size. The volume of gas moving through nozzle E is also adjusted so that the particle velocity in F exceeds the saltation or choking velocity but does not reduce the solid volume concentration below about 1.5%.

A solution of coating material and entraining gas moves under pressure through the tubing in H. The coating solution is atomized by a nozzle at the tip of H, as shown in Fig. 5. The gas velocity is adjusted so that it exceeds the velocity of the droplets and the spray of particles issuing from the outlet of tube F. The spray angles and distance between F and the liquid atomizing nozzle are adjusted to give maximum particle to droplet contact. The velocity of

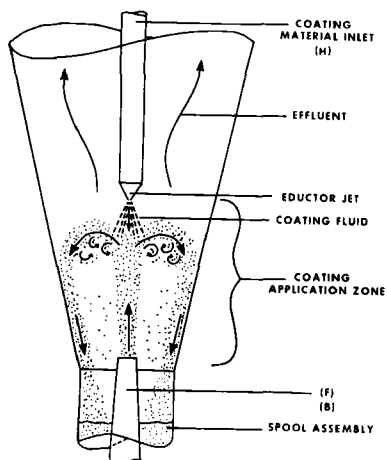


Fig. 5—A schematic representation of the coating application section.

the opposed gas streams creates a zone of intense mixing in which there is essentially no solidification of liquid particles. The velocities of the two opposed gas streams are controlled so that the downward motive force is greater. This results in the particles being scrubbed out of the upwardly moving gas stream and returned to the dense phase fluid bed. As the wetted particles move downward through this bed, they are again deagglomerated and the liquified coating is spread over the surface of the separated particles.

The particles are continuously cycled in this manner until the desired amount of coating has been applied. The liquid feed is then stopped and additional drying gas admitted through the air ring C. When the solvent has evaporated the particles can be removed through a port in the base section A. At this point, it is also possible to continue coating with a second material if desired.

A powder may be added during the coating cycle. This is accomplished by adding the solids to the top of the fluid bed during coating, or by mixing them with the liquified coating material, and then reducing the particle-separating forces to a level at which the added particles are not separated in the coating cycle.

Comparison of Coating Processes—A major difference between this coating method and others (rotating pan, fluid, and spouted bed) is the range and control of the particle-separating forces. The need for range and control is most clearly seen when the factors associated with particles separation are considered. To separate a particle from an assemblage requires that the separating force exceed the forces holding the assemblage together. In a bed of particles, the force resisting separation is the sum of the particle(s) weight plus the interparticle adhesive forces. The latter are related to properties of the particles and of the coating material. In the absence of coating material, particles agglomerate primarily because of van der Waals' attractive forces and/or adsorbed fluid. Electrostatic bonding forces are usually negligible (11, 12). These bonding forces act at points of particle contact and the area of contact depends on bed density, particle size, and particle shape. When an agglomerate has been separated into units of the desired size, and sufficient coating material applied to entirely cover the surface of the particle, the adhering force is proportional to the tensile strength of the liquified coating film. Depending on the properties of the coating material, this film tensile strength may increase or decrease as it solidifies. From this generalized description, it can be seen that the separating force needed to coat particles must vary considerably and will depend on the properties of substance to be coated and the coating materials.

The limitation of space does not permit a complete presentation of the theory and supporting experimental data associated with particle separation in this or other coating processes. Factors associated with particle separation in fluid beds have been reviewed in detailed by Zenz and Othmer (13). These factors will also be discussed in subsequent publications, in which theory and experimental data associated with solid particle coating in rotating pans and in this particle coating device will be presented. Differences in particle separation in various coating devices can be illustrated by comparing the relation-

ship between particle weight (G_p) and the interparticle adhesive forces (ψ). In a fluid or spouted bed a major force for particle separation is the drag resulting from the interaction among the particle and upward flowing gas. This drag force is given (12) by

$$F_d = \alpha^{1/2} C_d \rho_f A_p V_f^2 \quad (\text{Eq. 1})$$

For particle separation to occur

$$F_d = G_p + \psi \quad (\text{Eq. 2})$$

The superficial gas velocity (V_f) to provide this drag then is

$$V_f = \left(\frac{G_p + \psi}{\alpha^{1/2} C_d \rho_f A_p} \right)^{1/2} \quad (\text{Eq. 3})$$

However, at the required superficial gas velocity the ability to fluidize or spout a bed of particles and the quality thereof is determined by the relative magnitudes of G_p and ψ (12). When ψ is large relative to G_p the bed may not fluidize or spout because there is insufficient particle flow to break up the channels created by the upwardly moving gas stream. Alternatively, particle flow may be of a stick-slip type which results in an erratic, surging, slugging bed; this type of bed is poorly adapted for coating. Under this limiting condition ($\psi \gg G_p$), there will also be considerable elutriation of particles from the bed, because the superficial gas velocity required for particle separation must be greater than that required for conveying. Since G_p is proportional to particle size (D_p) there is a limit below which particles cannot be coated in either a fluid or spouted bed coater. This lower limit varies with the material used (both particle and coating), but in the authors' experience, the limiting value for D_p is near $10^3 \mu$.

When particles are coated in a rotating pan, there is a similar relationship between G_p and ψ . When a bed of particles is rotated in a pan, that part of the bed adjacent to the wall is carried to a given height by wall friction or baffles. At this height the particles will flow or fall back into and through the bed. The particles closest to the wall will travel the greatest distance and will return to the bed under essentially free fall conditions. It is this condition of particles falling on and through the bed that provides, to a considerable extent, the mixing and separation required for coating. The size of the units which separate at a given height depends on the relationship between G_p and ψ . If the weight of a single particle is greater than ψ , the particles separate and flow through the bed as individual units. If it is not, the particles will separate as agglomerates, which will either move to the front of the pan or be compacted to form aggregates. These aggregates will increase in size until G_p is larger than ψ . There are also separating forces in a coating pan which are associated with (a) the weight of the bed above a given assemblage of particles and (b) the centrifugal force (torque) which induces the particles in a bed to rotate. Although related to particle weight (size) and ψ , these separating forces are probably small when ψ is large relative to G_p . In the writers' experience, the limiting value for D_p in a coating pan is about two to three times that of a fluid or spouted bed coater.

The relationship between G_p and ψ is considerably less critical for the particle coating device (PCD). When an assemblage of particles is introduced into

this unit, it will break up into units with dimensions that are dependent on the magnitude of G_p and ψ . If ψ is large relative to G_p , these units will be agglomerates. As shown in Fig. 4, these units fall into and form a dense phase bed, whose mass (essentially plug) flow is controlled by the frequency and amplitude of vibration, use of fluidizing gas, and the cross-sectional area of the outlet. Vibration or fluidization may augment particle separation but the major separating force is the turbulent jet into which these particles are educted. Gas flow from this jet can be adjusted to provide the velocity needed for particle transport and separation (Eq. 3). The conveying gas velocity is relatively uniform in the converging nozzle, because the flow is turbulent. The volume flow of conveying gas is controlled by the cross-sectional area (or design) of the inlet nozzle (E). By controlling gas velocity and bed flow it is possible to reduce agglomerates to particles having a D_p as small as 5μ , and to transport the deagglomerated particles upward through the converging tube without saltation, choking, or slugging. The motion of a particle as it leaves the converging nozzle is described by the relationship

$$\text{inertial force} + \text{drag forces} = -\text{gravitational force} \quad (\text{Eq. 4})$$

It is obvious from the foregoing that the magnitude of these forces is associated with the relationship between G_p and ψ . In addition to the force of gravity, the upward motion of the particles is opposed by the downward motion of the coating liquid droplets and their entraining gas stream. The inertial force (F_i) for both particles and droplets is given by

$$F_i = M \frac{dv}{dt} \quad (\text{Eq. 5})$$

A droplet inertial force greater than that of the solid particle can be obtained by control of droplet size and velocity. This is done in the PCD by proper selection and operation of the atomizing nozzle. For example if a two-fluid educting-type nozzle is used, droplet size and velocity are related to gas velocity and liquid feed rate (14). It is also possible to vary the gas velocity flowing from tube H, and thus create a net downward drag on the wetted particles in the area of opposing gas flow. Through control of these variables, upward particle motion is arrested and redirected towards the wall of the expanded chamber, as illustrated in Fig. 5. However, there is always a net upward flow of gas plus vapor to the exhaust. The drag created by this upward flow is sufficient to elute particles having low values of G_p from the particle assemblage as it flows towards the wall. These particles are either suspended in the expanded section or carried upward into the bag collectors; they are not coated. In practice, it has been found that this loss is significant only when the majority of particles have D_p less than about 5μ , probably because there is considerable agglomeration of particles in the coating zone, which reduces elutriation and increases G_p .

Examples of Coated Particles—Through control of particle separation and transport, it is possible to coat particles having a large range of G_p and ψ , and also to use coating materials having large differences in film tensile strength. The utility of the PCD is illustrated by the following examples of coated solid

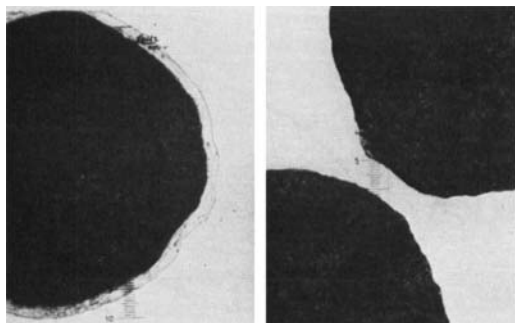


Fig. 6—Uncoated and ethylcellulose coated sugar pellets.

particles. Photomicrographs of sugar pellets ranging in D_p from 710–840 μ which have been coated with ethylcellulose⁸ are shown in Fig. 6. The solution rate of sugar from these pellets is shown in Table I. Solution rates were determined using the method of Souder and Ellenbogen (15).

TABLE I—DISSOLUTION OF SUGAR FROM ETHYLCELLULOSE-COATED PELLETS

Run No.	% Coating	% Dissolved, hr.		
		0.5	2	4.5
1	10.4	24	65	81
2	10.3	20	65	81
3	10.2	19	66	80
4	10.7	—	—	81
5	10.7	—	—	80
6	10.6	—	—	81

Photomicrographs of acetylsalicylic acid crystals coated with cellulose acetate phthalate⁹ are shown in Fig. 7. The solution times for these enteric-coated crystals are given in Table II. Figure 8 shows the rate of CO_2 evolution when NaHCO_3 par-

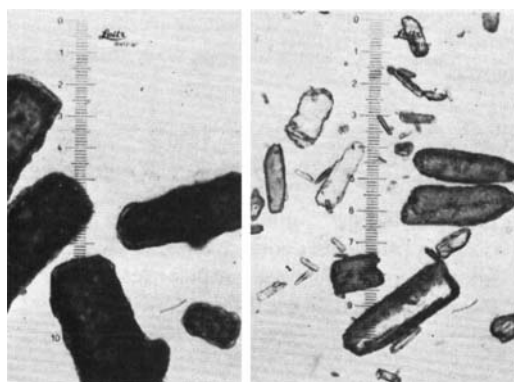


Fig. 7—Uncoated and C-A-P coated acetylsalicylic acid crystals.

ticles coated with hydrogenated castor oil¹⁰ are intensively agitated in 0.1 N HCl solution. The acetylsalicylic acid and NaHCO_3 crystals were obtained from commercially available powders meeting

⁸ Ethocel, Std. Ethoxy 10 cps. Dow Chemical Co., Midland, Mich.

⁹ C-A-P, Distillation Products, Rochester, N. Y.

¹⁰ Castor Wax, Baker Castor Oil Co., Bayonne, N. J.

TABLE II—SOLUTION TIMES FOR ENTERIC-COATED ACETYLSALICYLIC ACID CRYSTALS

Run No.	% Coating	% Dissolved	
		USP Gastric Fluid after 4 hr.	USP Intestinal Fluid after 15 min.
1	36.6	9	99
2	37.2	4	99
3	38.4	7	98

USP standards. Photomicrographs of acetaminophen particles coated with 12-hydroxystearyl alcohol¹¹ are shown in Fig. 9. These closely sized particles were obtained by spray drying and had $D_p \approx 15 \mu$ prior to coating. The solution rate of acetaminophen in USP intestinal fluid at 37°C. is shown in Fig. 10. The size distributions for micronized saccharin and for saccharin coated with 12-hydroxystearyl alcohol are shown in Fig. 11. The dissolution of the coated saccharin particles in distilled water at 25°C. is shown in Fig. 12.

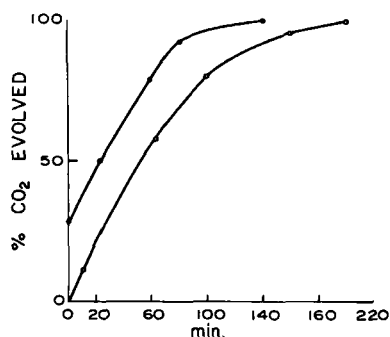
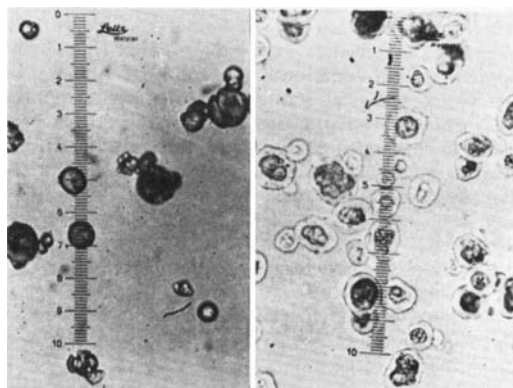
Fig. 8—Rate of CO_2 evolution from NaHCO_3 crystals coated with hydrogenated castor oil. Key: NaHCO_3 : HCO , ●—●, 2:1; ○—○, 1:1.

Fig. 9—Uncoated and coated particles of acetaminophen.

These few examples illustrate typical particles which have been coated using the particle coating device. They are representative of the majority of the solid particles found in the pharmaceutical industry. It is interesting that this flexibility was obtained with a single, simple device without the need to employ a variety of complex, expensive coating procedures.

¹¹ Adol 45, Ashland Chemical Co., Minneapolis, Minn.

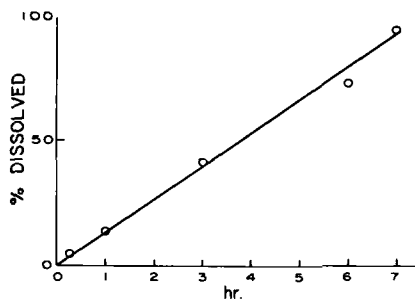


Fig. 10—Solution rate of coated acetaminophen particles in USP artificial intestinal fluid.

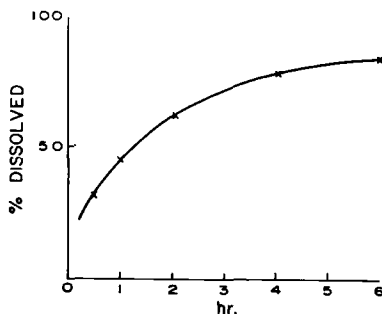


Fig. 12—Solution rate of coated saccharin particles in distilled water.

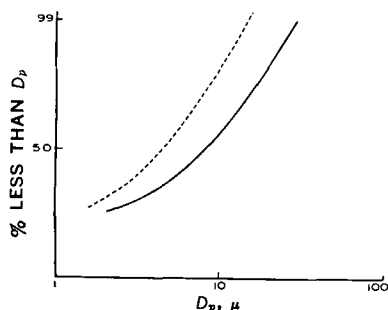


Fig. 11—Size distribution of micronized and coated saccharin particles. Key: ---, uncoated; —, coated.

SUMMARY

The description and method of operation for a solid particle coating device has been presented. It appears that, by control of particle separation and transport, it is possible to coat particles ranging in size from about 10 to 10³ μ in diameter with a variety of coating materials. It is believed that this development should contribute to coating technology in the pharmaceutical industry. Subsequent reports will present an evaluation of coated particles prepared in this coating device and illustrate the utility of using coated particles in the formulation of pharmaceutical dosage forms.

NOMENCLATURE

A_p = effective particle cross-sectional area
 C_d = drag coefficient
 D_p = representative particle diameter
 F_d = drag force
 F_i = inertial force

G_p = particle weight
 M = mass
 t = time
 v = velocity
 V_j = superficial velocity
 α = constant
 ρ_f = density of the gas
 ψ = effective interparticle force

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Keyphrases

Particle coating—apparatus, method
 Coating processes—comparison
 Photomicrographs—coated particles
 Diagram—particle coating apparatus