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Spray Drying Agglomeration. I. Physicochemical Properties of Agglomerated Synthetic Aluminium Silicate or Magnesium Carbonate

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For the purpose of improving the fluidity of solids for tabletting, various aqueous slurries containing synthetic aluminium silicate or magnesium carbonate and several kinds of binderes were spray dried using a centrifugal wheel atomizer. Gum arabic, gelatin, poly vinyl alcohol, carboxymethylcellulose, methylcellulose, and poly (vinylpyrolidone) were used as the binders. The finely agglomerated powders (10—80μ) obtained by this process showed free flowing properties and could be easily tablated, as opposed to the original powders. The surface of these products were examined by scanning electron microscopy. Important informations were obtained pertaining to surface topography. The geometric mean diameter (D_g) of the agglomerated powder was found to be influenced primarily by the type of binder used and its concentration. Some physicochemical properties of the agglomerated powder, *i.e.*, fluidity and packing, were also examined and the most important factors influencing these properties were found to be particle size and specific surface area.

Spray drying has been utilized for many years as a drying process. In addition, this process is also useful as a method for agglomeration and effecting changes in some of the physicochemical properties of powdered materials. This may result in more desirable characteristics in the dried product. There have been some previous studies on spray drying agglomeration of pharmaceuticals. Riegelman, *et al.*[2] applied spray drying techniques to pharmaceutical powders. Gunsel and Lachman[3] compared the evaluation of tablet formulations prepared from conventionally processed and spray-dried lactose. Robinson[4] described the utility of this process for the preparation of granules, and Kornblum[5] studied sustained-action tablets prepared for granulation by a spray-drying technique. Some spray-dried formulations of sulfaethylthiadiazole for prolonged release medication have been investigated by Asker and Becker.[6] There have been no studies, however, on the physicochemical properties of powders agglomerated by spray drying nor of the effect of the composition of the formulation prior to spray drying on these properties. The purposes of the present study were threefold: to find a method for improving the fluidity of finely powdered, non-free-flowing synthetic aluminium silicate or magnesium carbonate, to determine which of the parameters describing the formulations influence the physicochemical properties of dried products, and to determine the relationship between these properties and micromeritic parameters of the dried products.

Experimental

Preparation of Slurries——Aqueous solutions of binders, gum arabic, gelatin, polyvinyl alcohol, carboxymethylcellulose, methylcellulose, and polyvinylpyrolidone in respective concentrations of 0, 0.5, 1.0,

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2) S. Riegelman, J.V. Swintosky, T. Higuchi, and L.W. Busse, *J. Am. Pharm. Assoc. Sci. Ed.*, **39**, 444 (1950).

3) W.C. Gunsel and L. Lachman, *J. Pharm. Sci.*, **52**, 178 (1963).

4) M.S. Robinson, *J. Am. Pharm. Assoc. Sci. Ed.*, **50**, 76 (1961).

5) S.S. Kornblum, *J. Pharm. Sci.*, **58**, 125 (1969).

6) A.F. Asker and C.H. Becker, *J. Pharm. Sci.*, **55**, 90 (1966).

2.0 and 3.0%(w/v), were prepared. Finely powdered synthetic aluminium silicate (J.P. grade) or magnesium carbonate (J.P. grade) was added to slowly to 1500 ml of each of the binder solutions, using a homogenizer for stirring, until uniform, smooth slurries were obtained. Each batch of slurry thus prepared contained 300 g of solids. The compositions of the slurries are shown in Table I.

Table I. Composition of the Slurries

	Binder 0%	Binder 0.5%	Binder 1.0%	Binder 2.0%	Binder 3.0%
Material (g)	300	300	300	300	300
Binder (g)	0	7.5	15.0	30.0	45.0
Water (ml)	1500	1500	1500	1500	1500

Spray Drying Technique——A laboratory spray dryer, equipped with a centrifugal atomizing wheel driven at about 40000 rpm, was employed in this study. The heated chamber of the spray dryer was maintained at $150 \pm 10°$ and the outlet temperature was held at $100 \pm 10°$. It was necessary to maintain the slurry uniform throughout the spray drying process by the use of a homogenizer. The slurry was fed to the atomizing wheel by gravity and the feed rate was controlled by a glass stopcock. A cyclone collector was used to collect the agglomerated powder. The apparatus is shown in Fig. 1.

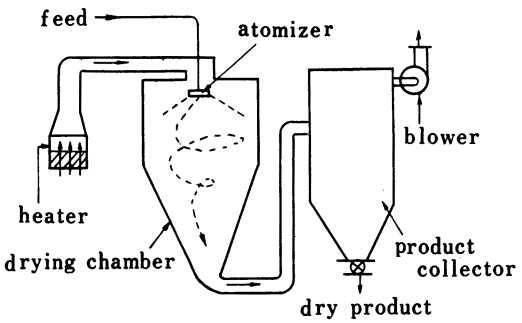


Fig. 1. Apparatus for Spray Drying

drying chamber diameter: 95 cm
drying chamber height: 150 cm
atomizer diameter: 4 cm
inlet temp.: 300°
chamber temp.: $150 \pm 10°$
atomizer rotation: 4×10^4 rpm

Size Distribution of Agglomerated Powder Particle——Size distribution of agglomerated powders was determined by a photographic counting procedure. Representative samples were photographed at convenient magnification. About 1000 particle for each sample were counted and the particles diameters were determined by a Zeiss particle size analyzer.

Scanning Electron Microscopy of Particles——Electron photomicrograms of magnesium carbonate or aluminium silicate products from slurries containing 3% gelatin and 1% methylcellulose were taken with scanning electron microscope Model JSM-2 (Nihon Denshi) in the Research Laboratory of the Kao Soap Co. Specimens were vapor-deposited to coat a thin-layer of gold. Magnifications were from 300 to 3000.

Measurement of Physicochemical Properties——The angle of repose was determined by measuring the poured angle of repose using a plate of 10 cm diameter. Packing density was obtained after tapping the samples contained in a glass cylinder (diameter 1.5 cm). The tapping rate was 150 (tap/min). Bulk density at the loosest packing was measured in air by the funnel method. Specific surface area was measured by the adsorption method (BET method) or by the liquid permeability method[7] using alcohol. Moisture content was obtained by measuring the weight loss drying to constant weight. True density was measured by the Beckmann air comparison pycnometer.

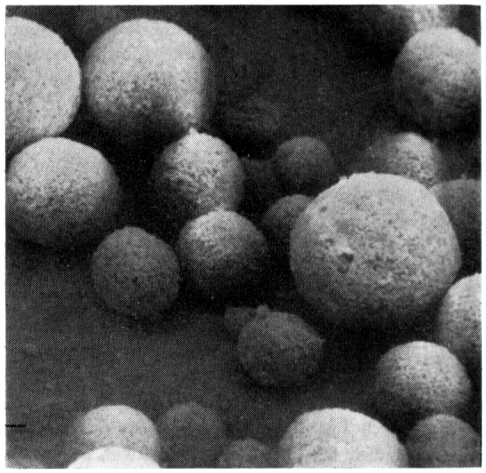
Tablet Manufacture from Agglomerated Powder——Agglomerated powders were compressed easily into tablets using a Korsch machine employing 8 mm flat bevel-edge punches at the rate of thirty tablets per minute. The dimensions of the tablets were measured by a micrometer.

Result and Discussion

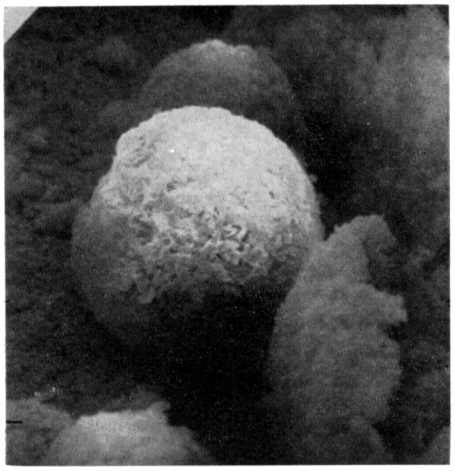
Properties of Agglomerated Powders

The agglomerated products were fine powders having a diameter of 10—80μ compared with 1—8μ for the original samples. Almost all of the agglomerates were free flowing.

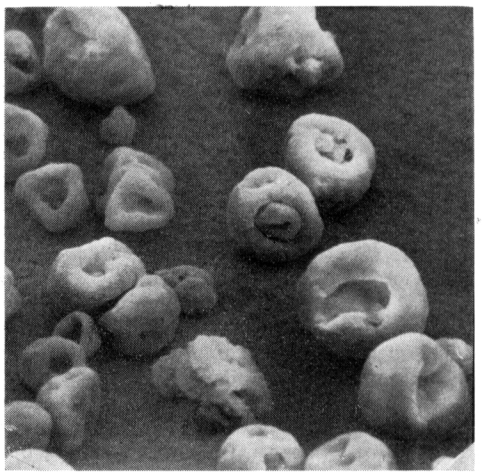
7) H. Sunada, *J. Res. Assoc. Powder Technol.*, **7**, 3 (1970).



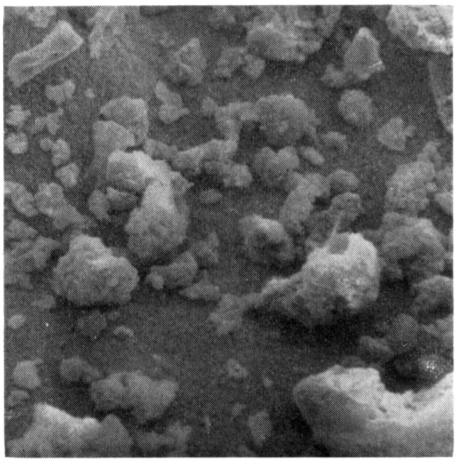
(a) length: 1 cm=45.6 μ
width: 1 cm=43.9 μ



(b) length: 1 cm=13.7 μ
width: 1 cm=13.1 μ



(c) length: 1 cm=45.6 μ
width: 1 cm=43.9 μ



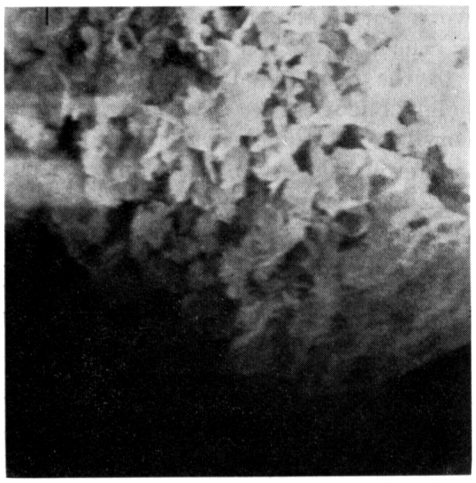
(d) length: 1 cm=45.6 μ
width: 1 cm=43.9 μ

The products from slurries containing 3% gelatin (G-3.0), 1% carboxymethylcellulose (CMC-1.0), and 1% methylcellulose (MC-1.0) showed the greatest free flowing tendencies. Magnesium carbonate products were fairly uniform spherical particles. On the other hand, aluminium silicate products tended to show void spaces in the particles when viewed by an optical microscope.

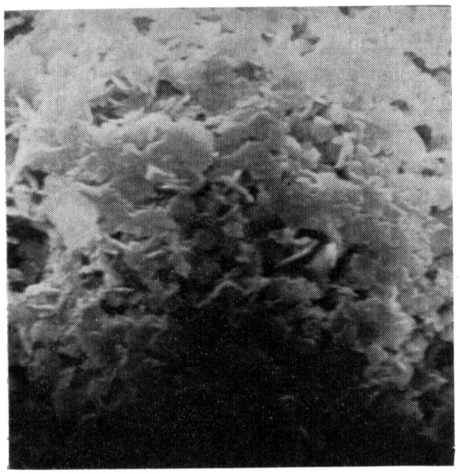
Johari and Bhattacharyya[8] studied the application of scanning electron microscopy for the characterization of powders and described that scanning electron microscopy was used to obtain information pertaining to shape, surface topography, texture and pore shape. The scanning electron microscopy photographs of MC-1 and G-3 products are shown in Fig. 2.

At the magnification of 300 and 1000, the scanning electron microscopy photographs in Fig. 2(a)—(d) show a large amount of information on shape revealed by its three dimen-

8) O. Johari and S. Bhattacharyya, *Powder Technol.*, **2**, 335 (1968/69).

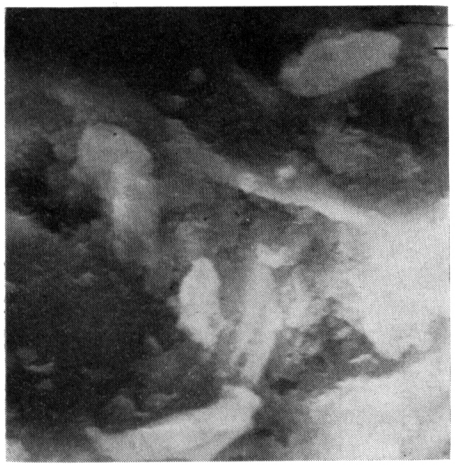


(e) length: 1 cm=4.56 μ
width: 1 cm=4.39 μ



(f) length: 1 cm=4.56 μ
width: 1 cm=4.39 μ

(g) length: 1 cm=4.56 μ
width: 1 cm=4.39 μ



(h) length: 1 cm=4.56 μ
width: 1 cm=4.39 μ

Fig. 2. Scanning Electron Microscopy Photographs of Agglomerated Products

(a) magnesium carbonate, MC-1 (300×)
(b) magnesium carbonate, G-3 (1000×)
(c) aluminium silicate, MC-1 (300×)
(d) aluminium silicate, G-3 (300×)
(e) magnesium carbonate, MC-1 (3000×)
(f) magnesium carbonate, G-3 (3000×)
(g) aluminium silicate, MC-1 (3000×)
(h) aluminium silicate, G-3 (3000×)

sional character, and surface of products. In Fig. 2(a), magnesium carbonate products (MC-1) show well rounded spheres and a number of micropores on their surface. A few of fractured ones are seen also in Fig. 2(b). In contrast to magnesium carbonate products, aluminium silicate products show a hollow, nearly spherical particle in Fig. 2(c) and fractured, shrivelled ones in Fig. 2(d). Diameters of hollows are about 15—20μ. At the magnification of 3000 the scanning electron microscopy photographs in Fig. 2(e)—(h), show a large amount of surface topography in detail. In Fig. 2(e) and (f), the surface of magnesium products seems to consist of a large number of thin crusts of 4—6.5 μ and small pores. Pore size is estimated to be between 0.5 and 1.3 μ and the very dark nature of the pore indicates that they are deep. Fig. 2(g) shows that the depth and volume of the hollow are so deep and large that it may be

the like of a crater which was shown by Charlesworth and Marshall[9] in the experiment of evaporation from drops containing dissolved solids. The outer surface of particles of aluminium silicate products is rather smooth compared to the magnesium product in Fig. 2(g) and (h). Fig. 2(h) shows that the surface has no hollow but it is very dimpled and wrinkled.

It is difficult to estimate the mechanism of the drying or agglomeration process from these figures but these scanning electron microscopy photographs suggest that the kind of materials or binders affect these processes and the physicochemical properties of products. Because of their characteristic surface topography, it was expected that aluminium silicate products would show a larger specific surface area than magnesium carbonate products. In fact, aluminium silicate products showed surface area of about 150—300 m^2/g, while that of magnesium carbonate products was about 15—30 m^2/g when determined by the BET method. On the other hand, the liquid permeability method gave similar values for both products, 1—3.5 m^2/g for aluminium silicate products and 1—3 m^2/g for magnesium carbonate products (Table IV).

Table II. Analysis of Variance for Geometric Mean Diameter (D_g)

Source of variation	s.s.	d.f.	m.s.	F_0
Main effect				
Material	8930	1	8,930	3.47
Binder	144187	3	48,062	18.69[a]
Conc.	32583	1	32,583	12.67[a]
Interaction				
Material × binder	28030	3	9,343	3.63
Binder × conc.	21755	3	7,252	2.82
Material × conc.	26730	1	26,730	10.39[a]
Error	7717	3	2,572	
Total	269932	15		

a) significant at 5% level

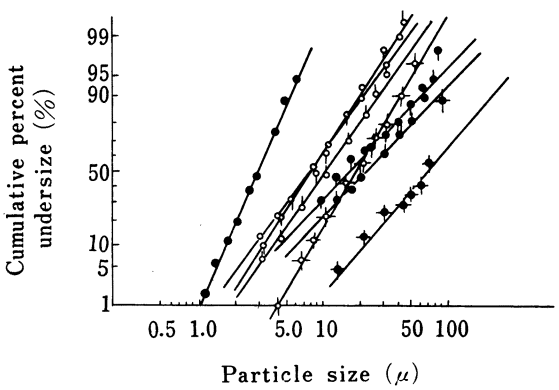


Fig. 3. Particle Size Distributions by Number of Several Spray-dried Agglomerates using Gelatin

magnesium carbonate: ●, G-0.5, ●, G-1.0, ●, G-2.0, ◆, G-3.0
aluminium silicate: ○, G-0.5, ○, G-1.0, ○, G-2.0, ◇, G-3.0

The particle size distribution of almost all the agglomerated products was found to have log normal distributions (Fig. 3). As may be seen in Table II, the geometric mean diameter (D_g) of the particles is affected mainly by the kind of binder used and the binder concentration, and the kind of solid material is also of some importance (Table II).

The smallest particles in terms of volume–surface diameter (D_{vs}) were produced from binder-free formulations. Thus the effect of binders on agglomeration is obvious. Upon increasing the binder concentration, D_{vs} is increased in gum arabic and gelatin product (Table III).

The viscosity and the surface tension of the formulations prior to spray drying were measured (Table III). Aluminium silicate products showed a tendency towards increased D_{vs} with increasing viscosity or surface tension. Magnesium carbonate products showed no such tendency.

9) D.H. Charlesworth and W.R. Marshall, Jr., *A.I.Ch. E, Journal*, **6**, 9 (1960).

TABLE III. Particle Diameter of Agglomerates and Viscosity and Surface Tension of Slurry Prior to Spray Drying

Sample symbol	Geometric mean diameter (μ)	Geometric standard deviation	Volumesurface diameter (μ)	Surface tension (dyne/cm)	Viscosity (centipoise)
a) Magnesium carbonate					
G-0.5	3.0	0.197	4.9	61.1	450
G-1.0	17.0	0.391	55.5	59.0	380
G-2.0	22.5	0.425	58.2	58.7	359
G-3.0	66.0	0.384	77.8	56.2	362
GA-0.5	8.2	0.302	24.4	57.7	19
GA-1.0	9.2	0.260	25.7	52.9	10
GA-2.0	6.2	0.370	29.1	54.7	17
GA-3.0	17.0	0.362	53.9	52.5	25
CMC-0.5	9.4	0.670	62.7	65.5	348
CMC-1.0	35.0	0.254	59.7	67.2	1025
MC-0.5	7.8	0.503	55.3	53.2	1905
MC-1.0	36.0	0.454	63.5	61.0	4550
PVA-1.0	8.0	0.355	35.3	75.3	1273
PVP-1.0	4.5	0.301	12.8	68.5	759
No bind.	1.54	0.325	6.1	68.3	473
Original	3.20	0.143	4.1		
b) Aluminium silicate					
G-0.5	8.0	0.335	23.8	67.0	118
G-1.0	8.3	0.302	20.3	59.5	55
G-2.0	10.5	0.318	23.1	55.2	9
G-3.0	17.5	0.268	35.0	54.3	9
GA-0.5	12.0	0.315	29.2	68.7	6
GA-1.0	7.1	0.422	21.4	70.8	7
GA-2.0	14.5	0.336	36.0	68.9	9
GA-3.0	20.0	0.290	39.0	72.0	10
CMC-0.5	18.0	0.304	40.7	72.3	99
CMC-1.0	28.0	0.273	53.5	72.6	698
MC-0.5	42.0	0.410	64.2	53.4	158
MC-1.0	40.0	0.374	62.5	56.9	863
PVA-0.5	12.0	0.361	30.2	66.1	2
No bind.	5.1	0.379	17.5	71.5	2
Original	3.1	0.250	7.7		

The angle of repose of agglomerated products is shown in Table IV. As the concentration of binders increases, the angle of repose decreases. Generally speaking, in this study, the aluminium silicate products showed smaller angles than the magnesium carbonate products. The angle of repose of the product was found to be most strongly affected by the binder concentration. The kind of binder and the kind of solid material had less effects (Table V). In order to investigate methods of influencing angle of repose, several micromeritic parameters of the products were determined and correlated with the angle of repose. The results are shown in Table VI.

The correlation between bulk density and micromeritic parameters was examined. In this study, D_{vs} and S_{wp} were found to be correlated with the bulk density for only magnesium carbonate products. The relationship among the angle of repose, bulk density and particle diameters and specific surface area determined by the liquid permeability method are shown in equations (1) to (3).

For magnesium carbonate products:

$$\text{angle of repose} = -0.0137(D_{vs}) + 7.18(S_{wp}) + 25.4 \quad (1)$$

TABLE IV. Angle of Repose and Micrometric Parameters

Sample symbol	Angle of repose (degree)	D_{vs} (μ)	ρ (g/cm^3)	ρ_b (g/cm^3)	S_{wb} (m^2/g)	S_{wp} (m^2/g)	M.C. (w%)	C.R. (—)
a) Magnesium carbonate								
G-0.5	46.9	4.9	2.20	0.126	20.8	3.11	0.4	5.59
G-1.0	34.3	55.5	2.21	0.180	13.7	1.45	0.4	9.59
G-2.0	32.3	58.2	2.14	0.197	14.3	1.20	0.4	24.79
G-3.0	30.4	77.8	2.08	0.199	15.2	1.05	1.0	28.36
GA-0.5	48.0	24.4	2.29	0.121	20.3	3.10	0.6	28.90
GA-1.0	40.2	25.7	2.25	0.136	18.4	2.28	0.8	22.0
GA-2.0	37.1	29.1	2.18	0.152	17.7	1.58	0.6	16.7
GA-3.0	35.1	53.9	2.14	0.186	17.9	1.16	0.4	22.2
CMC-0.5	35.4	62.7	2.27	0.199	18.6	1.18	1.0	27.9
CMC-1.0	29.8	59.7	2.21	0.220	21.1	1.06	0.6	23.5
MC-0.5	39.3	55.3	2.16	0.127	28.9	1.87	0.4	37.2
MC-1.0	31.8	63.5	2.20	0.181	18.1	1.01	0.9	23.5
Original	51.0	4.1	2.29	0.188	20.8	5.96	2.3	9.3
b) Aluminium silicate								
G-0.5	39.4	23.8	3.22	0.216	292	3.33	8.7	42.5
G-1.0	35.0	20.3	3.17	0.213	279	3.49	5.0	37.4
G-2.0	32.1	23.1	2.84	0.232	234	1.69	8.7	18.5
G-3.0	32.0	35.0	2.75	0.274	198	1.09	6.8	17.5
GA-1.0	36.7	29.2	3.25	0.253	330	2.61	8.6	41.2
GA-2.0	33.7	36.0	3.00	0.251	226	2.45	7.6	44.1
GA-3.0	32.7	39.0	2.82	0.272	230	1.88	7.0	34.4
CMC-0.5	35.4	40.7	3.19	0.225	298	3.31	9.7	71.6
CMC-1.0	30.1	53.5	3.10	0.253	281	0.963	12.3	26.6
MC-0.5	30.9	64.2	2.99	0.214	261	1.16	8.0	37.0
MC-1.0	29.2	62.5	2.72	0.248	250	0.883	10.6	21.2
Original	47.9	7.7	2.29	0.227	288	10.60	15.4	30.9

ρ: true density ρ_b: bulk density S_{wb}: specific surface area by the BET method S_{wp}: specific surface area by the permeability method[7] M.C.: moisture content C.R.: coefficient of rugosity

TABLE V. Analysis of Variance for Angle of Repose

Source of Variation	s.s.	d.f.	m.s.	F_0
Main effect				
Material	41.93	1	41.93	14.98[a]
Binder	238.81	3	79.60	28.43[a]
Conc.	178.89	1	178.89	63.89[b]
Interaction				
Material × binder	51.67	3	17.22	6.15
Binder × conc.	41.83	3	13.94	4.98
Material × conc.	11.39	1	11.39	4.07
Error	8.39	3	2.80	
Total	572.91	15		

a) significant at the 5% level
b) significant at the 1% level

For aluminium silicate products:

$$\text{angle of repose} = -0.0404(D_{vs}) + 1.97(S_{wp}) + 30.8 \quad (2)$$

For magnesium carbonate products:

$$\rho_b = 0.000102(D_{vs}) - 0.0366(S_{wp}) + 0.225 \quad (3)$$

TABLE VI. Correlation Coefficients between Angle of Repose and Micromeritic Parameters

Material	D_{vs}	ρ	ρ_b	*M.C.*	*C.R.*	S_{wp}
Magnesium carbonate	$-0.898^{a)}$	$0.586^{b)}$	$-0.922^{a)}$	-0.486	0.496	$0.969^{b)}$
Aluminium silicate	$-0.740^{a)}$	0.261	-0.446	$-0.614^{b)}$	0.259	$0.868^{a)}$

a) significant at the 1% level
b) significant at the 5% level
ρ: true density ρ_b: bulk density M.C.: moisture content C.R.: coefficient of rugosity S_{wp}: specific surface area by the permeability method

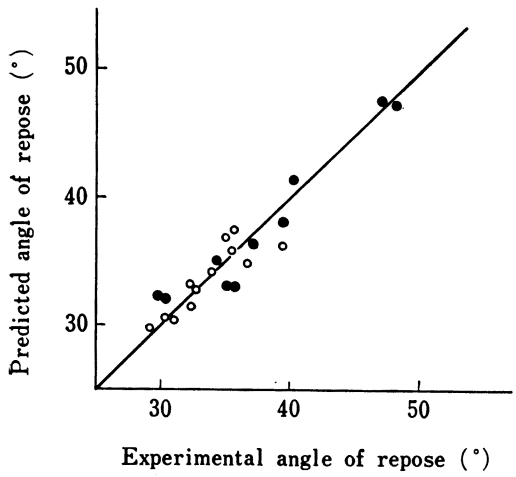


Fig. 4. Comparison of Experimental with predicted Angle of Repose
●: magnesium carbonate
○: aluminium silicate

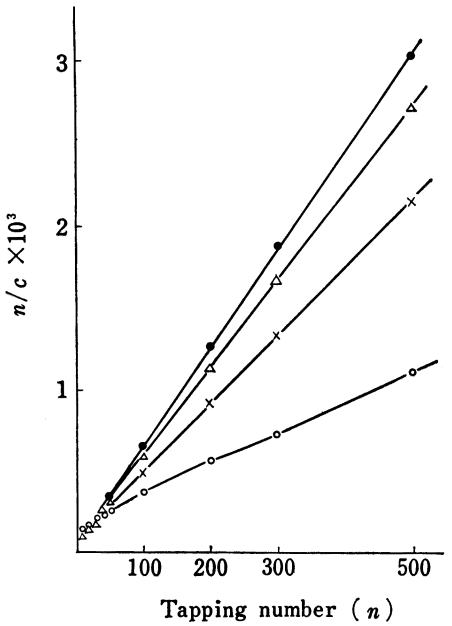


Fig. 5. Decrease in Specific Volume with Tapping *n*/*c* *vs* *n*
magnesium carbonate
○: gelatin-0.5 ×: gelatin-1 △: gelatin-2 ●: gelatin-3

Angles of repose calculated by eq. (1) and (2) agree fairly well with the observed values (Fig. 4).

These equations predict that the products will be free flowing if the particle diameter is large and specific surface area is low. The relationship between angle of repose and bulk density was found for only magnesium carbonate products in equation (4).

$$\text{angle of repose} = -161.6(\rho_b) + 64.2 \tag{4}$$

Packing of Agglomerated Products

The packing of agglomerated powders was examined and the decrease in specific volume with tapping is shown in Fig. 5. It is apparent that this experimental value is consistent with Kawakita's[10] equation (5).

$$n/c = 1/ab + n/a \tag{5}$$

where $c = (V_o - V_n)/V_o$, a and b = constants, and n = number of taps.

Constant a is the ratio of the difference between the specific volume at the loosest packing and at the closest packing to the loosest one, that is, $a = (V_0 - V_\infty)/V_0$. Constant b is the re-

10) K. Kawakita, *Zairyo*, 13, 421 (1964).

TABLE VII. Correlation Coeffcients between Constants a and b and Micromeritic Parameters

Material	D_{vs}	ρ	ρ_b	$M.C.$	$C.R.$	S_{wb}
Constant a						
Magnesium carbonate	$-0.945^{a)}$	0.374	$0.908^{a)}$	0.287	−0.481	$0.929^{a)}$
Aluminium silicate	$-0.930^{a)}$	0.026	$0.678^{a)}$	0.331	0.259	$0.876^{a)}$
Constant b						
Magnesium carbonate	$0.866^{a)}$	−0.464	$0.910^{a)}$	−0.080	0.369	$0.889^{a,b)}$
Aluminium silicate	$0.930^{a)}$	0.070	$0.690^{a)}$	−0.299	−0.262	$0.873^{a,c)}$

a) significant at the 1% level
b) $(S_{wp})^{-1.30}$
c) $(S_{wp})^{-1.45}$
ρ: true density ρ_b: bulk density M.C.: moisture content C.R.: coefficeint of rugosity S_{wp}: specific surface area by the permeability method

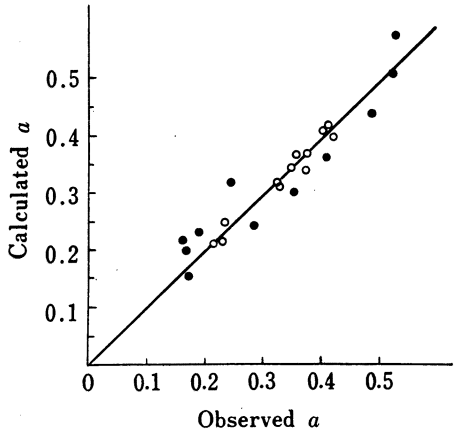


Fig. 6. Comparison between Observed and Calculated Values of Constant a

●: magnesium carbonate
○: aluminum silicate

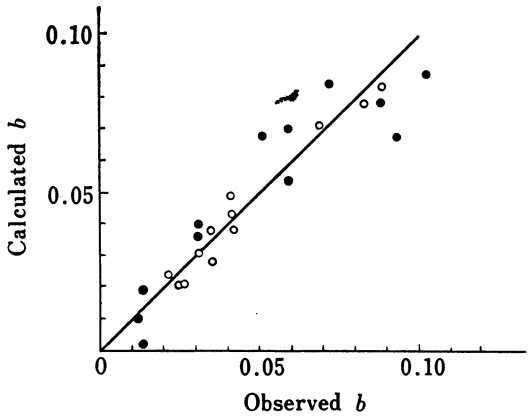


Fig. 7. Comparison between Observed and Calculated Values of Constant b

●: magnesium carbonate
○: aluminium silicate

ciprocal of the number of taps at which the specific volume is equal to one-half of sum of the loosest and the closest packed volumes, that is, $b=1/n$ at $V_n=(V_0+V_\infty)/2$. As would be expected, constants a and b are correlated to the angle of repose. The relationship between them, together with the correlation coefficient, r, are shown below in equations (6) to (9). The R test shows that the correlation coefficients are significant at the 1% level.
For magnesium carbonate products:

$$a=-0.377+0.0184(\text{angle of repose}) \qquad r=0.924 \qquad (6)$$

$$b=0.222-0.00459(\text{angle of repose}) \qquad r=-0.887 \qquad (7)$$

For aluminium silicate products:

$$a=-0.242+0.0164(\text{angle of repose}) \qquad r=0.824 \qquad (8)$$

$$b=0.211-0.00472(\text{angle of repose}) \qquad r=-0.740 \qquad (9)$$

It is clear that powders having a low angle of repose show ease of packing and no sticking properties. The micromeritic parameters affecting these constants, a and b, were found to be mainly the particle diameter (D_{vs}), specific surface (S_{wp}) and bulk density (ρ_b). (Table VII). The relationship between these constants and particle diameter (D_{vs}) or specific surface (S_{wp}) is expressed by equations (10) to (13).

TABLE VIII. Physical Specification and Properties of Tablets prepared from the Agglomerated Powder

Sample symbol	Diameter (mm)	Thickness (mm)	Weight (mg)	Hardness (kg)	Density $_{ap.}$
a) Magnesium carbonate					
MC-0.5	8.103	3.563	103.4	0.59	0.563
MC-1.0	8.135	3.616	143.8	0.98	0.765
CMC-0.5	8.143	3.686	158.4	0.93	0.825
CMC-1.0	8.159	3.721	178.5	2.38	0.917
GA-0.5	8.090	3.306	88.8	0.50	0.523
GA-1.0	8.103	3.421	106.7	0.56	0.605
GA-2.0	9.104	3.534	124.6	0.61	0.683
GA-3.0	8.171	3.619	152.6	0.84	0.804
G-0.5	8.163	3.333	92.5	0.44	0.530
G-1.0	8.145	3.574	145.2	0.90	0.780
G-2.0	8.159	3.674	164.2	2.55	0.855
G-3.0	8.124	3.712	167.1	3.77	0.849
b) Aluminium silicate					
GA-1.0	8.265	6.752	225.3	0.66	0.622
GA-2.0	8.268	6.728	239.5	0.75	0.663
GA-3.0	8.295	6.800	251.3	0.63	0.684
G-0.5	8.297	6.802	208.2	0.50	0.566
G-1.0	8.300	6.990	203.8	0.50	0.538
G-2.0	8.289	7.087	214.2	0.52	0.560
G-3.0	8.328	7.142	230.6	0.56	0.593

TABLE IX. Analysis of Variance for Apparent Density of Tablet prepared from Spray-dried Agglomerates

Source of variation	s.s.	d.f.	m.s.	F_0
For magnesium carbonate				
Concentration	48985	1	48985	14.32[a)]
Binder	101058	3	33686	9.85[a)]
Error	10261	3	3420	
Total	160304	7		
For aluminium silicate				
Concentration	3429	2	1715	37.3[a)]
Binder	12881	1	12881	280.0[b)]
Error	92	2	46	
Total	16402	5		

a) significant at the 5% level
b) significant at the 1% level

For magnesium carbonate products:

$$a=-0.00330(D_{vs})+0.084(S_{wp})+0.318 \quad (10)$$

$$b=0.000461(D_{vs})+0.0744(S_{wp})^{-1.30}-0.0183 \quad (11)$$

For aluminium silicate products:

$$a=-0.00324(D_{vs})+0.0235(S_{wp})+0.400 \quad (12)$$

$$b=0.00104(D_{vs})+0.0185(S_{wp})^{-1.45}-0.00409 \quad (13)$$

Values calculated by equations (10) to (13) agree fairly well with the observed values (Fig. 6 and 7). These equations predict that particles having a large D_{vs} will show small a and large b, that is, ease of packing and lack of cohesive properties.

The relationship between these constants and bulk density may be expressed by equations (14) to (17).
For magnesium carbonate products:

$$a=-3.46(\rho_b)+0.890 \quad (14)$$

$$b=0.806(\rho_b)-0.0840 \quad (15)$$

For aluminium silicate products:

$$a=-1.44(\rho_b)+0.684 \quad (16)$$

$$b=0.487(\rho_b)-0.0745 \quad (17)$$

Tablet Manufacture from Agglomerated Powder

The original samples proved difficult to be fed into the die due to their cohesive tendencies but the spray-dried agglomerated powders could be easily tableted as the result of improved fluidity. Physical specifications and properties of the tablets prepared from the spray-dried powder are shown in Table VIII. Fifty tablets were prepared and the properties were measured with 25 representative tablets selected at random. Tablets prepared from formulations containing a high concentration of binders showed the highest apparent density and hardness. The apparent density of the tablets was affected mainly by the binders and their concentration (Table IX).

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