Chem. Pharm. Bull. 16(8)1433—1439(1968)

UDC 615.453.2.01

Influence of Particle Size on Physicochemical Properties of Pharmaceutical Powders. V.[1] On Fluidity of Sodium Borate and Boric Acid Powders. (2)[2]

Nobuyoshi Kaneniwa and Akiko Ikekawa

School of Pharmaceutical Sciences, Showa University[3]

(Received June 13, 1967)

Rate of discharge of sodium borate and boric acid powders from acrylic and brass funnels were investigated.

Large particles discharge steadily from a funnel but flow behaviour seems to be different in the region below critical size.

Flow rate largely depends on materials, outlet diameter, outlet length and cone angle of the funnel. Angle of repose characterized by particle size, kind of particles and the like, is an important parameter controlling flow rate of particles.

Drained angle is markedly influenced by particle size in the region below critical size just as is the angle of repose and the sliding angle.

Many studies have been reported concerning discharge of powders from an orifice.[4] Smalley, *et al.* investigated variations of the flow rate with particle diameter and found that the flow rate was maximum for a diameter of 150—300 μ, below and above which flow behavior was different.[5,6] Furthermore, Tanaka, *et al.* reported on the correlation between flow rate and angle of repose of powders.[7,8]

In our previous paper, flow rate is found to be a maximum at 160 μ for sodium borate and boric acid powders.[2] We have also reported on the presence of a critical particle size, below which the angle of repose, the sliding angle and the volume per gram of powders in loosest state largely depend on particle size.[2,9,10] Flow rate of powders is probably related to these findings, because the particle size for maximum flow rate, 160 μ, is nearly identical to the critical size.

In this paper, rate of discharge of sodium borate and boric acid powders from an orifice is investigated and parameters for flow rate are discussed.

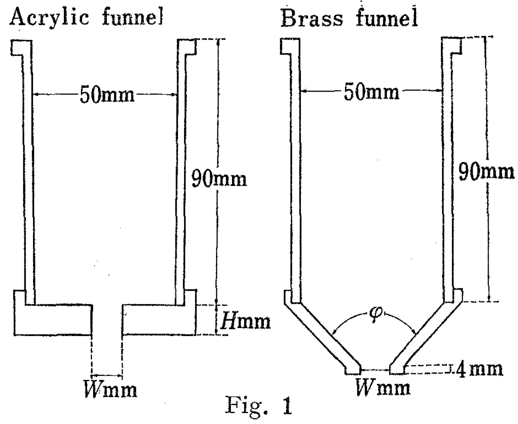

Fig. 1

Experimental

The samples used are the same as referred to in the previous paper.[2]

1) A. Ikekawa and N. Kaneniwa, *Chem. Pharm. Bull.* (Tokyo), **16**, 1174 (1968).
2) N. Kaneniwa, A. Ikekawa, and H. Aoki, *Chem. Pharm. Bull.* (Tokyo), **15**, 1441 (1967).
3) Location: *Hatanodai, Shinagawa-ku, Tokyo.*
4) S. Miwa, "Micromeritics," No. 8, Summer, 1962, p. 25.
5) I.J. Smalley, *Nature*, **201**, 173 (1964).
6) C. Kawashima and N. Murata, *J. Ceramic. Assoc. Japan*, **63**, 88 (1955).
7) T. Tanaka and S. Kawai, *Kagaku Kogaku*, **20**, 144 (1956).
8) H.E. Rose and T. Tanaka, "The Engineer," October 23, 1959, p. 1.
9) A. Ikekawa, H. Aoki, K. Masukawa, and N. Kaneniwa, *Chem. Pharm. Bull.* (Tokyo), **15**, 1626 (1967).
10) A. Ikekawa and N. Kaneniwa, *Zairyo*, **16**, 314 (1967).

Two funnels were used. One is an acrylic cylinder (5 cm in diameter and 9 cm of height) attached to an acrylic disk with an orifice (W mm in diameter and H mm of length) in center. Whilst H is maintained 20 mm, W is varied between 2 and 14 mm, and alternatively whilst W is maintained 4 or 10 mm, H is varied between 2 and 39 mm. The other funnel consists of brass cylinder (5 cm in diameter and 9 cm high) attached to a brass cone, angle of φ, with an orifice (W mm in diameter and 4 mm long) in center. When φ is maintained 60° or 180°, W is varied between 2 and 14, while φ is varied 45° and 180° when W is maintained 4 or 10.

The weight of powders discharging from a funnel in a fixed time was measured.

Results and Discussion

The rate of discharge of sodium borate and boric acid powders from an orifice is independent on the height of powders above the aperture. This fact is also confirmed by many other cases with a few exception.[6,7] Large paricles discharge steadily from a funnel, but sodium borate (137 μ and 115 μ in diameter) and boric acid (96.5 μ) begin to discharge steadily, only by thrusting powders with a stick, and smaller particles stop discharging. Thus flow behaviour seems to be different above and below the critical size found for angle of repose, and so on.[2,9,10]

Effect of Orifice Diameter

Fig. 2, 3, 4 and 5 show the influence of orifice diameter on flow rate. Flow rate is large for small particles, and this tendency decreases as the orifice diameter increases.

Brown, *et al.*[11,12] studied the mechanism of discharge of powders from a hopper and found that particles did not pass within 0.15 cm from the edge of the outlet and the effective orifice diameter was equal to $(W-k_w)$. On the basis of this fact the following equation was proposed between flow rate, v, and orifice diameter, W,

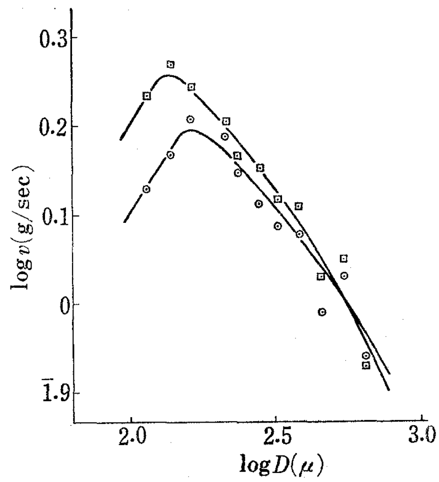

Fig. 2. Influence of Particle Size on Flow Rate of Sodium Borate Powders from Brass Funnel with 4.2 mm of Outlet Diameter

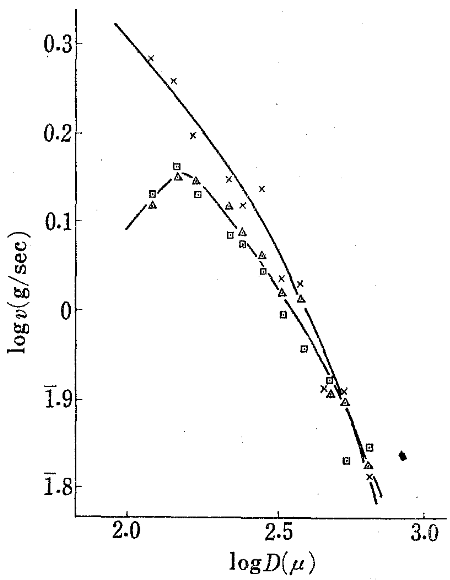

Fig. 3. Influence of Particle Size on Flow Rate of Sodium Borate Powders from Funnel with 4 mm of Outlet Diameter and 180° of Cone Angle

11) T. Tanaka, *Kagaku Kōgaku*, **26**, 835 (1962).
12) R.L. Brown and J.C. Richards, *Trans. Inst. Chem. Engrs.* (London), **38**, 243 (1960).

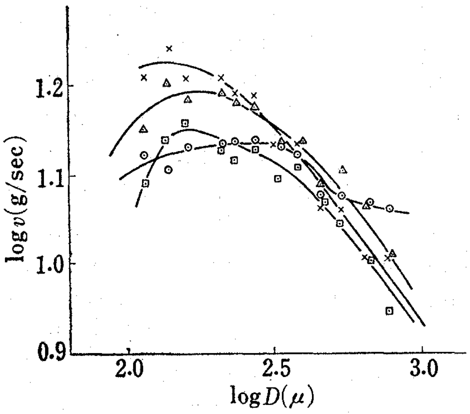

Fig. 4. Influence of Particle Size on Flow Rate of Sodium Borate Powders from Funnel with 10 mm of Outlet Diameter

—⊙— brass, φ: 60°, H: 4 mm
—⊡— brass, φ: 180°, H: 4 mm
—△— acrylic, φ: 180°, H: 4 mm
—×— acrylic, φ: 180°, H: 20 mm

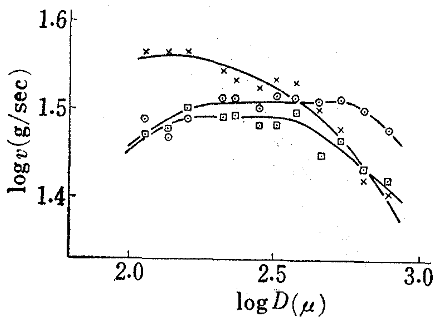

Fig. 5. Influence of Particle Size on Flow Rate of Sodium Borate Powders

—⊙— brass, φ: 60°, W: 14 mm, H: 4 mm
—⊡— brass, φ: 180°, W: 14 mm, H: 4 mm
—×— acrylic, φ: 180°, W: 14 mm, H: 20 mm

$$v^{2/5}=K(W-k_w) \quad (1)$$

k_w, K : constant

Equation (1) can be well applied to our experimental data, as shown in Fig. 6, although the numerical value of k_w is negative for small particles.

Table I shows the influence of particle size on k_w and K. These numerical values increase with increase of particle size and depend on the kind of samples, and outlet length, cone angle or material of the funnel.

Table I

Sample	Sodium borate						Boric acid					
Funnel	A		B		C		A		B		C	
Particle diameter	K	k_w	K	k_w	K	k_w	K	k_w	K	k_w	K	k_w
96.5 μ							0.240	−0.18	0.243	−1.02	0.271	−0.49
115	0.278	−1.00	0.266	−0.05	0.296	−0.03	0.274	0.15	0.269	−0.59	0.273	−0.03
137	0.284	−0.02	0.278	−0.03	0.296	−0.02	0.286	0.30	0.250	−0.07	0.280	−0.07
163	0.284	−0.02	0.293	0.00	0.300	0.00	0.278	0.20	0.270	−0.08	0.283	0.00
214	0.292	0.08	0.290	0.20	0.299	0.10	0.290	0.49	0.269	0.21	0.282	0.32
235	0.292	0.08	0.289	0.25	0.301	0.31	0.290	0.49	0.269	0.24	0.281	0.20
279	0.290	0.04	0.289	0.25	0.301	0.30	0.289	0.33	0.275	0.25	0.286	0.48
324	0.302	0.56	0.292	0.50	0.302	0.54	0.286	0.55	0.273	0.51	0.282	0.60
385	0.303	0.65	0.300	0.70	0.298	0.49	0.293	0.75	0.281	0.78	0.289	0.70
460	0.302	0.69	0.286	0.70	0.294	0.90	0.297	0.81	0.273	0.87	0.290	0.86
545	0.301	0.90	0.300	1.10	0.294	0.96						
650	0.301	0.90	0.279	0.77	0.294	1.25						
775	0.309	1.30	0.291	1.26	0.287	1.20						

A; brass, : 60°, H: 4 mm
B; brass, : 180°, H: 4 mm
C; acrylic, : 180° H: 20 mm

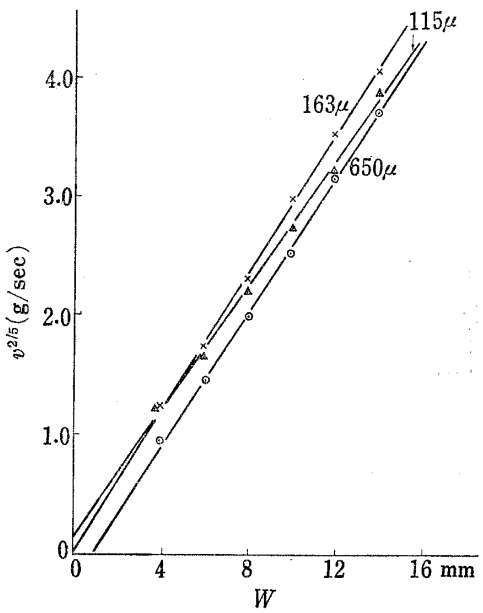

Fig. 6. Application of Equation (1) to Flow Rate of Sodium Borate Powders from Brass Funnel (φ:180°)

In Fig. 3 and 4, the flow rate of sodium borate powders through an acrylic funnel is larger than through a brass funnel if W is 10 mm, but flow rate is independent of the material of the funnel if W is 4 mm. Fig. 2, 3, 4 and 5 also suggest the following facts. The flow rate of large particles is independent of outlet length, but that of small particles from the funnel with 10 mm of outlet length is larger than that from the funnel with 4 mm of outlet length. When W is maintained 4 mm, essentially no decrease of flow rate of large particles occurs by decreasing cone angle from 60° to 180°, but the flow rate of small particles from the funnel with 180° of cone angle is larger than that from the funnel with 60° of cone angle. On the other hand, when W is 10 or 14 mm, flow rate increases by decreasing cone angle from 180° to 60° and this tendency is more remarkable in the case of large particles.

Effect of Outlet Length[13]

As shown in Fig. 7 and 8, flow rate gradually increases with increase of outlet length and the tendency is more remarkable for small particles. The data obtained by the funnel with 4 mm of outlet diameter will be discussed in detail, since the experimental error for 10 mm of outlet diameter is too large compared to the difference caused by H.

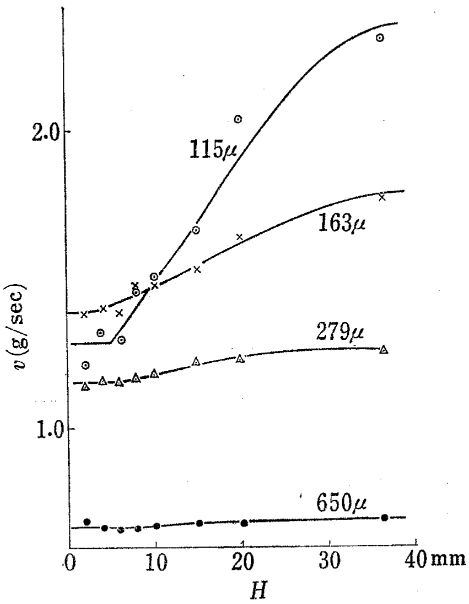

Fig. 7. Influence of Outlet Length on Flow Rate of Sodium Borate Powders from Acrylic Funnel (W:4 mm)

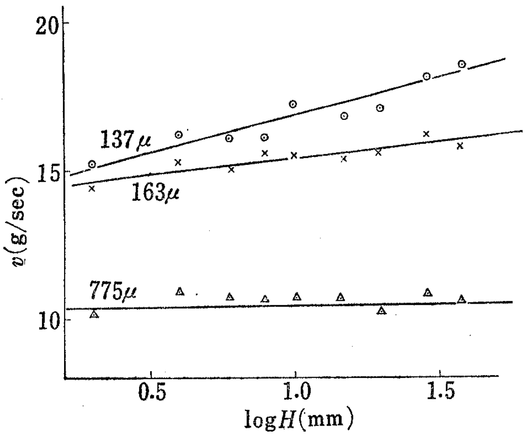

Fig. 8. Influence of Outlet Length on Flow Rate of Sodium Borate Powders from Acrylic Funnel (W:10 mm)

Outlet length has only a small effect on flow rate when H is smaller than 6 mm, but when H is larger than 6 mm, straight lines are obtained by the plot of v *vs.* log H as shown in Fig. 9.

13) T. Uchiyama and S. Taneya, *Zairyo*, **15**, 142 (1966); N.R. Zaporin, *Inzh. Fiz. Zh. Akad. Nauk Belorussk SSR*, **4**, [33], 18 (1960).

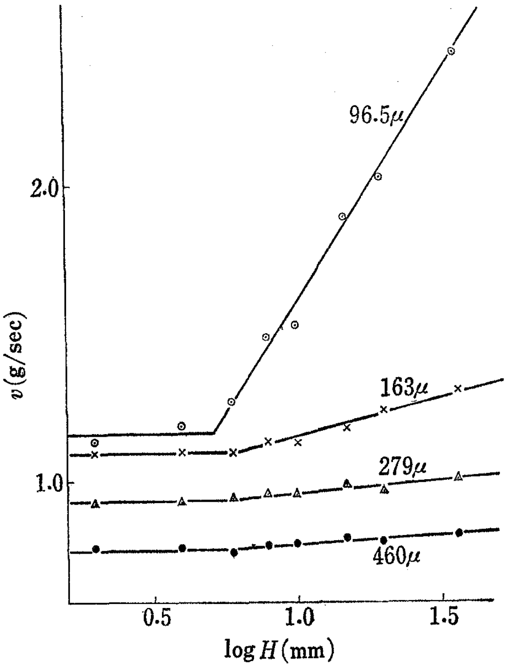

Fig. 9. Relation between Outlet Length and Flow Rate of Boric Acid Powders from Acrylic Funnel (W:4 mm)

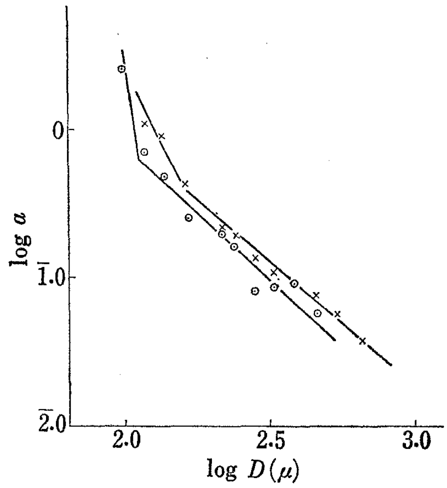

Fig. 10. Influence of Particle Size on Tangent a
—×— sodium borate —⊙— boric acid

The tangent of the line, a, increases gradually with the decrease of particle diameter, as shown in Fig. 10. The tangent, a, shows a dependence similar to angle of repose, sliding angle, the volume per gram of powders in loosest state and so on,[2,9,10] that is, the rate of increase of the tangent (a) by decrease of particle size is larger for particles below critical size. Thus it is suggested that a is the dependent value of internal friction and cohesive force between particles of powders.

According to several studies on movement of particles,[11] discharge of granular materials from an orifice is controlled by two different movement of particles, the primary movement and the secondary movement. The primary movement is a vertical movement resulting from the weight of particles themselves and the weight imposed on the particles by the other particles, and close packing is caused by this movement. The secondary movement consists of inclination and rotation of particles, shortening the distance between the horizontal particles, and mixing particles with each other. If outlet diameter becomes larger and is identical with the funnel diameter, particles flow out of the funnel only by the primary movement. If orifice length is larger than a certain value, the outlet will serve as a cylinder fixed below the funnel and promote the primary movement. The increase of flow rate of powders with the increase of outlet length will be due to the above facts.

Effect of Cone Angle

Fig. 11 shows that flow rate is maximum at 60° and decreases gradually with increase of cone angle, and is minimum at $\varphi_{\min}$ and increases with the increase of φ again. The numerical values of $(90°-\varphi_{\min}/2)$ are identical with angles of repose obtained by Nogami–Sugiwara's method, that is, 30° for sodium borate ($D \geq 160$ μ) and boric acid) $D \geq 100$ μ), 40° for sodium borate (137 μ) and boric acid (96.5 μ), and 50° for sodium borate (115 μ). When outlet diameter is maintained 10 mm in Fig. 12, the numerical values of $(90°-\varphi_{\min}/2)$ are also identical with angles of repose (α). It is suggested from these findings that angle of repose is an important parameter controlling discharge of powders from an orifice.

The above tendency is also reported in several studies.[4] Deming, *et al.*[14] found that flow rate of glass and some other powders increased with the dercease of φ, satisfying equation (2), and further Franklin and his coworkers[15] reported that equations (2) and (3) applied for the relation between φ and flow rate of large particles, where c_1, c_2, c_3 and c_4 are constants.

$$\varphi<180°-2\alpha \qquad v=1/(c_1+c_2\sin\varphi/2) \tag{2}$$

$$\varphi>180°-2\alpha \qquad v=c_3+c_4\sin\varphi/2 \tag{3}$$

Tanaka, *et al.*[11] also found the same tendency and proposed the following euqations.

$$\varphi\geqq180°-2\alpha \qquad v=c_5 \tag{4}$$

$$\varphi<180°-2\alpha \qquad v=c_6(\tan\varphi/2)^{-c_7} \tag{5}$$

c_5, c_6, c_7 : constants

On the other hand, Kehemah, *et al.*[4,16] reported that minmum flow rate was obtained for 45° of cone angle. This tendency is also shown for small particles of sodium borate and boric acid powders.

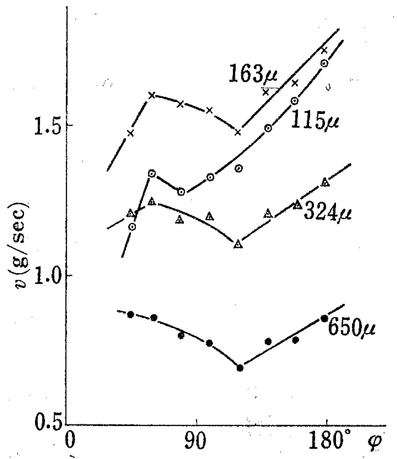

Fig. 11. Influence of Cone Angle on Flow Rate of Sodium Borate Powders from Brass Funnel (W: 4.2 mm)

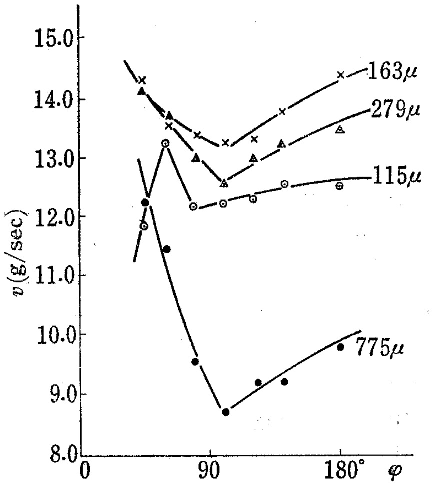

Fig. 12. Influence of Cone Angle on Flow Rate of Sodium Borate Powders from Brass Funnel (W: 10 mm)

Then the relation between angle of repose and flow rate is investigated. In Fig. 13, v is plotted *vs* tan $(90-\varphi/2-\alpha)$, where α is the angle of repose by Nogami–Sugiwara's method. Three kinds of tangents of the straight lines in Fig. 13 decrease with decrease of particle size, respectively, and it is suggested, by the plot of these tangents *vs* log D, that the rate of decrease of them becomes larger at the region below critical size referred to in the previous papers.[2,9,10]

Drained Angle

Measurement of drained angle was made using acrylic and brass funnels with 180° of cone angle. Angles were independent of the outlet diameter, if W was larger than 6 mm. In Fig. 14, the drained angle shows the same tendency as the angle of repose and the sliding angle,[2,10] that is, it increases gradually with decrease of particle size, and the rate of increase

14) W.E. Deming and A.L. Mehring, *Ind. Eng. Chem.*, **21**, 661 (1929).
15) F.C. Franklin and L.N. Johanson, *Chem. Eng. Sci.*, **4**, 119 (1955).
16) F.E. Keneman, *et al.*, *Inzh.–Fiz. Zh.*, *Akad. Nauk Belornssk SSR*, **3**, 69 (1960).

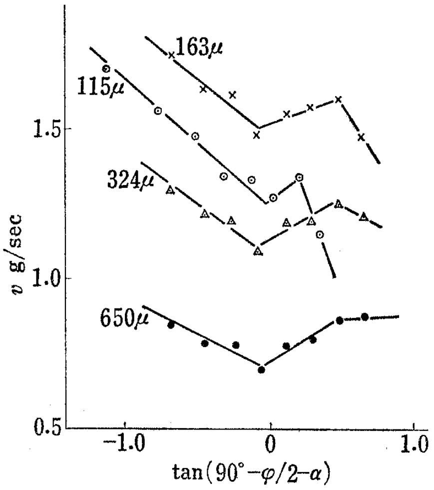

Fig. 13. Relation between Cone Angle and Flow Rate of Sodium Borate Powders from Brass Funnel (*W*: 4.2 mm, H: 4 mm)

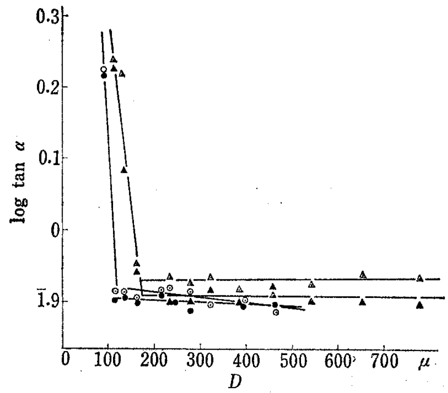

Fig. 14. Influence of Particle Size on Drained Angle

—△— sodium borate, brass
—▲— sodium borate, acrylic
—⊙— boric acid, brass
—●— boric acid, acrylic

of the angle is more remarkable in the region below 160 μ for sodium borate and below 100 μ for boric acid. Drained angles of large particles are a little larger than the angles by Nogami–Sugiwara's method, and similar to those obtained by Neumann's or Nelson's method. But drained angles of the particles below critical size are much larger than angles of repose and nearly identical with sliding angles.