Transactions of the Materials Research Society of Japan 27 [2] 459-462 (2002)

Development of Instrument for Evaluating Weakly Caking State of Powder —Evaluation for Caking of Sodium Bicarbonate—

Minoru Kuniyoshi [1], Tugio Murakami [1], Tadato Mizota [2]
1) Process Development, Nanyo Technology Center, Tosoh Corp.,
4560 Kaisei, Shinnanyo, 746-8501, Japan
Fax: 81-0834-63-9836, e-mail: kuniyosi@tosoh.co.jp
2) Department of Advanced Materials Science and Engineering, Faculty of Engineering,
Yamaguchi University, Ube 755-8611, Japan

Caking is defined as a state of powder that sticks into hardened body. Caking of powder-products, if it happens, in food, chemical or medical industries cause serious economic loss. For example, sodium bicarbonate easily forms cakes during the storage and/or the transportation process after the production. Sodium bicarbonate may then loose value as raw materials such as chemicals and medicines. Evaluation techniques of caking must be developed to overcome the problems. Method of characterizing weak caking is therefore required. An instrument equipped with a punching metal cylinder and lifting boards were developed for the characterization of powdering rate of caked clots. The powdering rate evaluated well the weakly caking state in sufficient accuracy. Filling density of powder affects significantly the hardening of the cakes. A new theoretical equation was proposed successfully to simulate powdering rate useful for evaluating the caking state.

Key words: Weak caking, Caking state, Powder, Solidification, Sodium bicarbonate

1. INTRODUCTION

Medical tablets or pellets for industrial catalysts, for example, are sufficiently hard so that the strength measurements have been performed so far by using test machines for breaking strength of specimens[1, 2]. Inducing forces for the testing are classified into nine types such as compression, bending, strain, cracking, impact, wear, vibration, shearing, and push in[3]. All of these types were examined to find out the best method of evaluating the weak caking state.

2. EXPERIMENTAL AND RESULTS

(1) Method of caking

Table 1 shows the particle size distribution and purity of the sodium bicarbonate sample. The cell shown on Fig. 1 is filled up with the sodium bicarbonate powder. Two types of filling methods were adopted such as natural filling and dense filling under 2.6×10^5 Pa loading. Each sample was exposed in the atmosphere at 30℃ and the relative humidity (RH) of 80%. The sodium bicarbonate specimens were then caked for a fixed duration, and are called caked clots thereafter. When the exposure time was longer, a caked clot became harder. The stronger the compression, the specimen was caked harder.

(2) Breaking strength measurement by compression

The strength of caked clots was measured by means of the Kiya type hardness meter[4].

Each caked cylindrical clot was placed vertical on the sample stand of the hardness meter and was loaded gradually with a pushing pin of the diameter of 7mm. Then, caking strength was defined as the load when the sample was cracked.

Table1 Characteristics of sodium bicarbonate sample

Particle size(μm)	Distribution(%)
+150	17
106～150	30
75～106	28
45～ 75	19
－ 45	6
Average particle size : 100 μm	
Purity: over 99.8%	

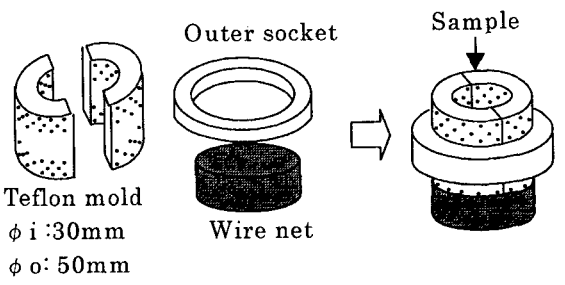


Fig. 1 : Sample molding cell

Figure 2 shows relation between the caking strength and the time for which the sample was held at RH80 %. Although densely filled caked samples were relatively strong, the strength values obtained distributed widely and were thought unreliable. This explains the caking strength obtained from the method due mainly to that of the weakest point, which distributes randomly. The strength of caked sample by the natural filling was equal to or less than 1.0 N, which exceeds the lower limit of measurement value of the Kiya type hardness meter used. The method was identified to be not suitable for the evaluation of such kind of weak strength.

(3) Development of the instrument for measuring strength of the weak caking

The average strength as a whole sample must be measured to evaluate the strength of caked clots. The impact or the abrasion method may therefore be suitable. Several equipments for the methods were tested to evaluate caking strength, by using cylindrical drums. A 40.0g of sodium bicarbonate sample was caked into a clot by the method described in 2(1).

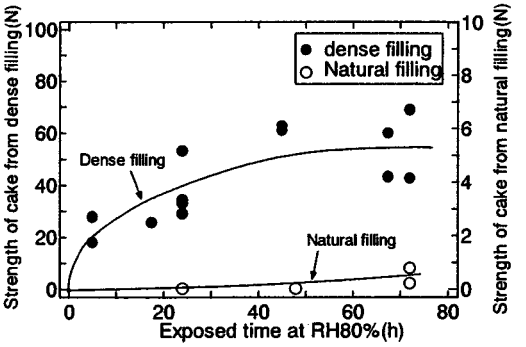


Fig. 2 : Caking strength measured by means of the Kiya type hardness meter
Caking condition : 30℃,RH80%
Sample : Sodium bicarbonate
Dense filling =density1.27×10^{3}kg/m^3
Natural filling=density1.09×10^{3}kg/m^3

The cylinders with a caked clot was rotated at the constant rate(0.367s^{-1}) for a fixed duration at the room temperature to measure the weight of powder produced (Fig. 3). Four Types of cylinders as shown in Fig. 4 were tested for the abrasive method.

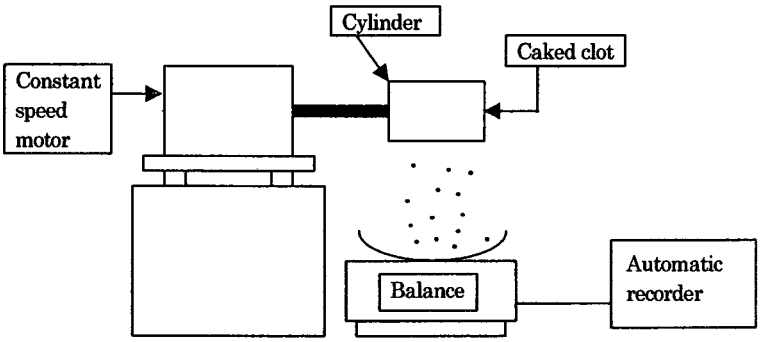


Fig. 3 The equipment for powdered percentage measurement

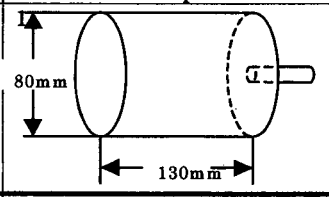
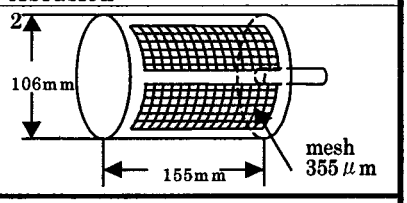
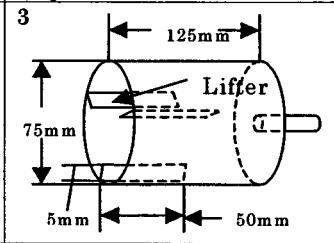
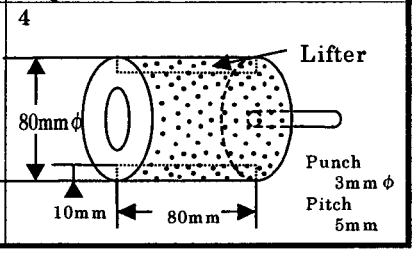
Type of cylinder	Glass circular cylinder	Circular cylinder with sieve
	Abrasion + Impact	Abrasion
Shape	1 	2 
Type of cylinder	Glass circular cylinder with three lifters	Punching metal circular cylinder with two lifters
	Impact	Impact
Shape	3 	4 

Fig. 4 Four types of evaluation instrument for powdering of caked clots

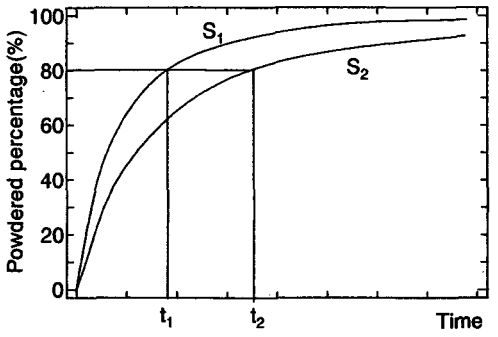


Fig. 5 Powdering patterns

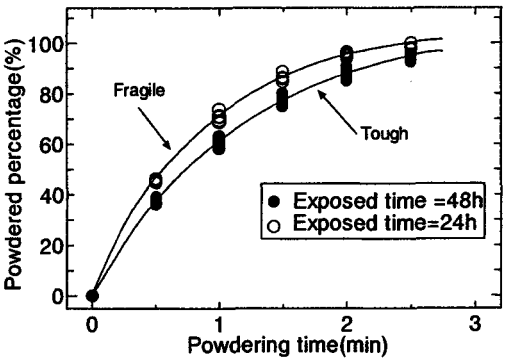


Fig. 6 Measurement of the caking strength using the punching metal cylinder with two lifters rotated at 0.367s^{-1}.
Sample clots were prepared without compression at 30℃,RH80%, and exposed for 24h or 48h giving fragile or tough nature respectively.

Obtained powder samples were collected in two methods as follows. 1) For cylinders 1 and 3, pieces of the clot with produced powder were gently transferred from the cylinder to a sieve with the opening of 1mm to separate the smaller portion. 2) For cylinders 2 and 4, powder generated by the rotation motion is sieved automatically through punched holes of the drum surface and drop down onto the dish of the balance. The weight of powder was measured by means of an electric balance.

The caking strength was evaluated as the powdering rate. The powdering pattern is expressed as the dependence between powdered percentage and time as lines drawn in the Fig. 5.

The powdered percentage P was calculated by a relation, $P(\%)=w/(w_0)\times100$, where w is the powdered weight of the clot and w_0 is the original weight of the clot.

Pattern S_1 indicates the larger powdering rate than that of S_2, and thus the smaller caking strength relative to that of S_2. The strength can also be evaluated as the 80% powdered time for each sample as shown t_1 or t_2 in the figure, that is, $t_1 < t_2$.

As a result, the punching metal cylinder with lifters was the most suitable for powdering rate reproducibility for the weakly caking strength measurement (Fig. 4-4). Figure 6 shows the result of measurement of the caking strength using the punching metal cylinder with two lifters. The cylinder has many punching holes on the surface with two lifters inside. The motion of the cylinder continuously exhausts the powder generated from impact given by the lifters. The measurement precision as the 80% powdered percentage of the caked clots is within 1.1 % (standard deviation), and 1.5 % (change coefficient, CV) when it is tested at 30℃ and RH80% for 24h. The caking strength could thus be evaluated quantitatively as the 80 % powdered time.

(4) Evaluation of caking strength by means of the punching metal cylinder with two lifters

Caking strength was evaluated for the sodium bicarbonate sample of 38.0g in the Table 1.

The clot density could be controlled by changing the feeding height of the powder-funnel above the sample cell at 4-levels of 1, 3, 5, and 7 cm (Table 2). The powder is filled naturally without compression and kept for 15 h at 30℃ and RH80%. The result is shown in Fig. 7. The clot density increases positively with the 80% powdered time. Densely caked clots increased the contact surface area among particles and resulted in the increased caking strength.

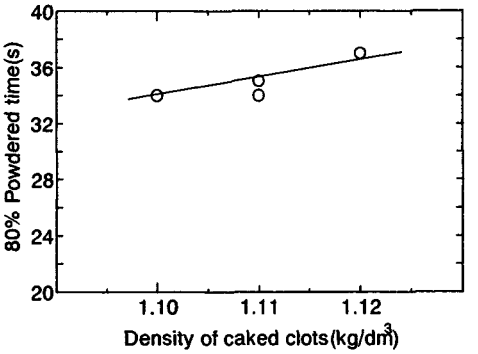


Fig. 7 Density of caked clots and 80% powdered time relations.
Measurement cylinder : puching metal cylinder with two lifters
Sample : sodium bicarbonate, Filling : without compression
Rotation rate=0.367s^{-1}
Caking condition : 30℃,RH80%,15h

Table 2 The feeding height and the the density of caked clots relations

Feeding height (cm)	1	3	5	7
Density of caked clots(kg/m^3)	1.10×10^3	1.11×10^3	1.11×10^3	1.12×10^3

3. DISCUSSION

A basic equation was proposed relating the powdering time with the weight change of caked clots in the cylinder and was derived from the total change of the positional energy for the sample clot. The equation(II) and the equation(III) are given when K is supposed constant. The equation(III) is obtained by integrating the equation(I). The equation(II) reveals well the experimental results and thus confirmed valuable (Fig. 8).

$$-\frac{dM}{dt} = \frac{KMghr}{S \cdot E} \quad \cdots (\mathrm{I})$$

$$M(t) = M_0 e^{-\frac{Kghr}{S \cdot E} t} \quad \cdots (\mathrm{II})$$

$$\therefore S \cdot E = \frac{Kghr \cdot t}{\log M_0 - \log M(t)} \quad \cdots (\mathrm{III})$$

M : caked clot weight (kg)
K : powdering constant (kg/m^2)
g : gravitational acceleration(m/s^2)
h : fall distance (m)
r : number of fall times (s^{-1})
M_0 : initial weight of caked clot (kg)
S : contact surface area among powder particles (m^2/m^2)
E : The caking energy per contact surface area among particles (J/m^2)

The equation (IV) is derived taking the $M(t)$ as the 80% powdered weight and t_{80} as 80% powdered time for the equation(III)

$$S \cdot E = \frac{Kghr \cdot t_{80}}{\log M_0 - \log(M_0 \times 0.2)} = \frac{Kghrt_{80}}{\log 5} (J/m^2) \quad \cdots (\mathrm{IV})$$

Supposing the constant K, the t_{80} is independent upon the initial weight of the clot, The term t_{80} is proved to be a measure of the caking energy per unit area.

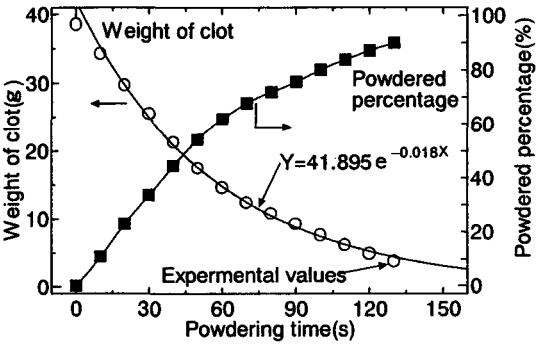


Fig. 8 The change in weight of caked clot by powdered percentage measurement

References
[1] H. Nakata, I. Sekiguchi, H. Tohata: Powder Science and Engineering, April, p.23(1978)
[2] The Society of Powder Technology, Japan(ed.): Powder Technology Handbook, Nikkan Kogyo Shimbun Ltd., p.138(1986)
[3] T. Kanazawa, M. Chikazawa: Powder Science and Engineering, August, p.65(1986)
[4] T. Kanazawa(ed.): Caking phenomenon of powder and measures for preventing caking, p.115(1996)
[5] M. Kuniyoshi, H. Saitoh : Japan Patent, Application number 2000-231371
[6] M. Kuniyoshi and T. Murakami: Journal of the Mining and Materials processing Institute of Japan, 117, 869-874(2001)

(Received December 20, 2001; Accepted March 18, 2002)