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Technical note

A new practical index to predict capping occurring during the tableting process

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1. Introduction

When pharmaceutical powders are compacted into tablets, cracking, usually termed capping, sometimes occurs inside the tablets. Many studies have been done from practical and theoretical viewpoints to overcome this problem [1–3]. The mechanism of capping is, however, not fully understood. Although some indices were proposed to estimate capping tendency [4–6], it is difficult to estimate capping quantitatively with any type of pharmaceutical powders by means of those indices.

The present authors described capping as a cracking produced inside the tablet and was induced by residual die wall pressure applied at the final stage of the decompression process [7]. The capping ratio (tendency of capping) of a tablet was represented by $(F_u\text{-}F)/F_u$, where F is the crushing strength of a tablet prepared by the normal method and F_u is that of an ideally compacted tablet without capping using a loaded-ejection-type compaction apparatus [7]. The value of the capping ratio varies from zero (without capping) to unity (completely capped). The authors have proposed a capping index (CI) defined by Q_r/P_c, where Q_r is the residual die wall pressure which is evaluated by extrapolating a linear relationship between the die wall pressure and the axial pressure at the final stage of decompression process to zero of axial pressure and P_c is the axial crushing strength of a cylindrical compact. A good correlation was found between the two parameters. Capping always occurred when the capping index exceeded unity and where the residual die wall pressure exceeded the crushing strength. When the capping index was approximately unity, the tablets were partially capped. It was possible to predict capping occurring during tableting by this index.

Thus the capping index is very useful for predicting capping during tableting. However, it is difficult to measure the residual die wall pressure unless a special die with a strain gauge is used [8]. In this study, the physical meaning of the capping index was rediscussed more quantitatively and a new useful practical method to find this index was investigated by estimating the residual die wall pressure (Q_r) from a crushing test of a cylindrical compact.

2. Materials and methods

2.1. Materials [7]

Caffeine (Shizuoka, Japan), cornstarch (Japan Cornstarch, Japan) and a mixture (2:1) of lactose (DMV, Holland) and cornstarch were used as reference powders without capping tendency. Ethoxybenzamide (ethenzamide, Yoshitomi, Japan), lactose, hydroxy-

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buthylphenetidin (bucetin, Yamato, Japan) and the granule prepared from ethenzamide (45%), bucetin (45%) and a-starch (10%, Nichiden, Japan) by wet massing were used as powders with capping tendency.

2.2. Tablet preparation [7]

Tablets were prepared by using a die with 8.5 mm internal diameter, punches with either flat face or concave face (6.5 mm radius of curvature) and a compacting test apparatus (Autograph, Shimadzu Seisakusho, Japan). The die was lubricated by puffing a very small amount of powdered magnesium stearate (Sakai, Japan) prior to every compaction.

2.3. Measurement of residual die wall pressure [8] and crushing strength [7]

Die wall pressure was measured using a specially designed die with a strain gauge attached to the outer surface of the die. The crushing strength of the compacted powder was measured by compressing axially a flat-faced cylindrical compact (1000 mg weight, 11.3 mm diameter, 7–8.5 mm thickness).

3. Results and discussion

3.1. Physical meaning of the capping index ($CI = Q_r/P_c$)

Fig. 1 (upper) shows the stresses applied on a tablet in a die at the final stage of the decompression process and its Mohr's circle. The tablet is compressed by low axial pressure (P) and high radial pressure (Q). Shear stresses (τ) and compressive stress (s) in the tablet are expressed by a Mohr's circle which passes through (P,0)

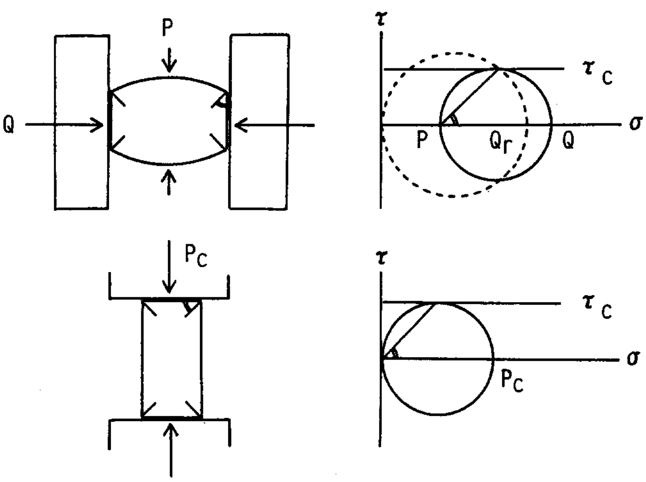

Fig. 1. Stress and Mohr's circle for a tablet (upper) and for a cylindrical compact (lower).

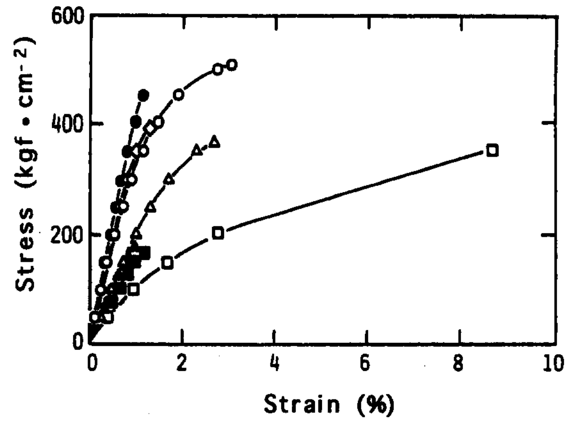

Fig. 2. Stress–strain curves on crushing cylindrical compacts (1 kgf/cm^2 = 98.07 kPa) [1]. ○, caffeine; △, lactose and cornstarch mixture; □, cornstarch; ◇, ethenzamide and bucetin granule; ●, ethenzamide; ▲, lactose; ■, bucetin.

and (Q,0), and shear stress (τ) reaches the maximum (($Q-P$)/2) acting in a direction of 45 degrees. If the shear stress (τ) overcomes the shear strength of the compacted powders (τ_c), the tablet may crack in the direction shown in the figure. The Mohr's circle of the tablet at the extrapolated end stage of decompression process is represented by the dotted line, passing through (0,0) and (Q_r,0), where Q_r is the residual die wall pressure and $Q_r/2$ is the maximum shear stress.

The shear strength (τ_c) of compacted powder was estimated from the axial crushing strength of (P_c) a cylindrical compact. Mohr's circle of the compact when crushed is shown in Fig. 2 (lower), which passes through (0,0) and (P_c,0). The shear stress in the compact becomes maximum at a direction of 45 degrees and the compact is cracked in this direction. Therefore the capping index (Q_r/P_c) is described in terms of the ratio of diameter of solid lined Mohr's circle to dotted line, that is the ratio of maximum shear stress ($Q_r/2$) and shear strength of compacted powder ($P_c/2$).

3.2. Estimation of residual die wall pressure

The results of the crushing test and the residual die wall pressure measurement on a corresponding flat-faced tablet, which were reported in a previous publication [7] are represented in Fig. 2 and Table 1.

The powders with lower residual die wall pressure are assumed to indicate higher strain on the axial crushing test of a cylindrical compact. The maximum strain in the crushing test (S_{max}) was compared to the residual die wall pressure of a flat-faced tablet (Q_{rf}). The relationship between the residual die wall pressure and the reciprocal of maximum strain is shown in Fig. 3. The residual die wall pressure was approximately proportional to the reciprocal of the maximum strain. This means that the residual die wall pressure depends on

Table 1
Crushing strength of a cylindrical compact and residual die wall pressure of a flat-faced tablet [7]

Material	Crushing strength (kgf/cm^2)[a]	Maximum strain (%)	Residual pressure (kgf/cm^2)
Caffeine	505	3.11	95
Lactose and cornstarch mixture	365	2.68	90
Cornstarch	350	8.68	5
Ethenzamide and bucetine granule	390	1.32	220
Ethenzamide	450	1.16	240
Lactose	175	0.97	195
Bucetine	165	1.19	165

[a] The unit 1 $kgf/cm^2 = 98.07$ kPa

plastic and elastic properties of the powder. Plasticity increases while elasticity decreases residual die wall pressure. The following empirical relationship was obtained between the two parameters.

$$Q_{rf} = 236/S_{max} \quad (r = 0.91) \tag{1}$$

Eq. (1) means that it is possible to estimate the residual die wall pressure on the cylindrical compact using the crushing test. This is a very practical method to estimate the residual pressure.

3.3. Shape correction factor of residual die wall pressure

Usually capping takes place more frequently in convex-faced tablets than flat-faced tablets, convex-faced tablets showed higher residual die wall pressure than flat-faced [7,8]. A thinner tablet showed higher residual die wall pressure and higher capping ratio. The authors described the following relationship between residual die wall pressure and tablet shape [7].

$$(Q_{rc} - Q_{rf})/Q_{rf} = 0.7(T_v - T_e)/T_e \quad (r = 0.97) \tag{2}$$

where Q_{rc} and Q_{rf} are the residual die wall pressures of convex-faced and flat-faced tablets, T_v is the thickness of the flat-faced tablet having the same volume as a convex-faced tablet and T_e is the edge thickness of a convex-faced tablet as illustrated in Fig. 4. The shape correction factor (F_s) of residual die wall pressure was defined by Eq. (3).

$$F_s = Q_{rc}/Q_{rf} = 0.3 + 0.7T_v/T_e \tag{3}$$

The residual die wall pressure of a convex-faced tablet can be estimated with the value of a flat-faced tablet by using Eq. (4).

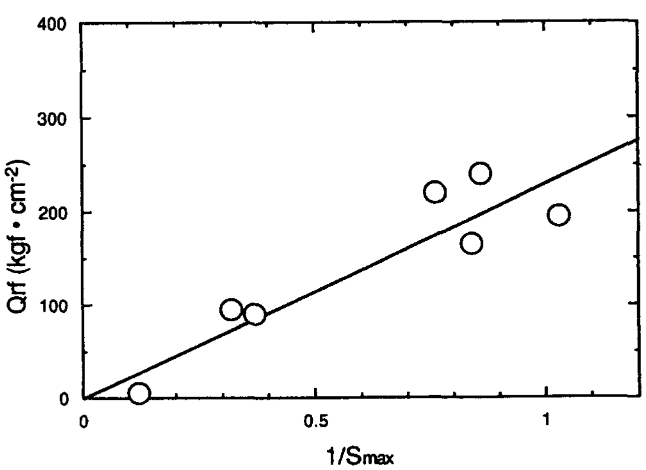

Fig. 3. Relationship between residual die wall pressure and maximum strain (1 $kgf/cm^2 = 98.07$ kPa).

3.4. Estimation of capping index

It is possible to calculate the capping index (CI) of various shaped tablets without measuring die wall pressure by combining Eqs. (1) and (2) as follows.

$$CI_{calc} = (235/P_cS_{max})(0.3 + 0.7T_v/T_e) \quad (r = 0.94) \tag{4}$$

The capping indices of various shaped tablets were calculated using the crushing test results of a cylindrical compact (P_c and Smax) and tablet shape (T_v and T_e). The calculated indices were compared with the observed indices which were evaluated using directly measured residual die wall pressure (Fig. 5). A good correlation was obtained between the two indices. This means that it is possible to predict the capping tendency of tablet from the strength and the strain data of a cylindrical compact crushing test. This is a very convenient method to predict compressing behavior of powders and very useful for the practical study of tablet formulation.

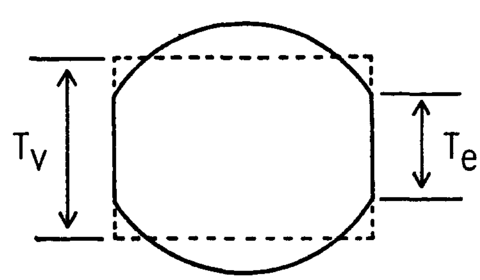

Fig. 4. T_v and T_e of a tablet (T_v is the thickness of the flat-faced tablet having the same volume of a convex-faced tablet)

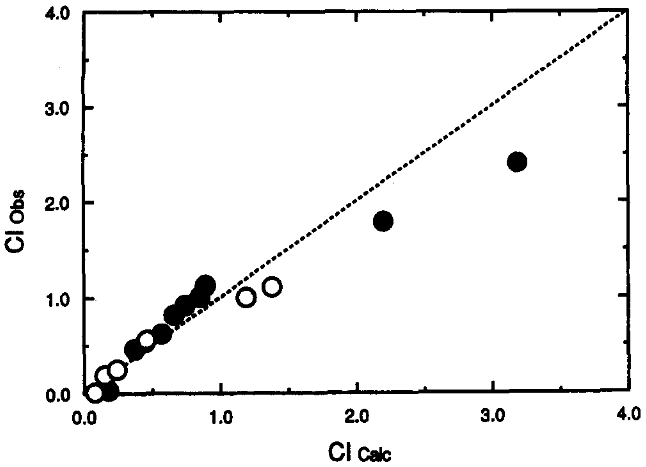

Fig. 5. Relationship between calculated capping index and observed capping index. ○, flat-faced tablet; ●, convex-faced tablet

3.5. Prevention of capping

Eq. (4) suggests a way to prevent capping. Increasing P_c decreases capping index, leading to reduced capping. The addition of binder with fine particle size or in a state of solution effectively increases P_c [9]. Increasing S_{max} is also effective in reducing capping. The powders of polymer materials, such as starch or cellulose, show higher S_{max}. The addition of these powders is effective in prevention of capping [10]. The shape of a tablet is another important factor in preventing capping. Considering convex-faced tablets, tablets without capping may be obtained if punches with larger radii of curvature are employed because this decreases the value of T_v/T_e. Thus tablets without capping can be successfully formulated or designed using Eq. (4).

References

[1] P.J. Rue, P.M.R. Barkworth, P. Ridgway-Watt, P. Rough, D.C. Sharland, H. Seager, H. Fisher, Analysis of tablet fracture during tableting by acoustic emission techniques, Int. J. Pharm. Tech. Prod. Mfr. 1 (1979) 2–5.

[2] G.E. Amidon, D.P. Smith, E.N. Hiestand, Rotary press utilizing a flexible die wall, J. Pharm. Sci. 70 (1981) 613–617.

[3] L. Casahoursat, G. Lemagnen, D. Larrouture, The use of stress relaxation trials to characterize tablet capping, Drug Dev. Ind. Pharm. 14 (1988) 2179–2199.

[4] P.J. Jarosz, E.L. Parrot, Factors influencing axial and radial tensile strengths of tablets, J. Pharm. Sci. 71 (1982) 607–614.

[5] A.B. Bangudu, N. Pilpel, Effects of composition, moisture and stearic acid on the plasto–elasticity and tableting of paracetamol–microcrystalline cellulose mixtures, J. Pharm. Pharmacol. 37 (1985) 289–293.

[6] W.E. Jetzer, Measurement of hardness and strength of tablets and their relation to compaction performance of powders, J. Pharm. Pharmacol. 38 (1986) 254–258.

[7] K. Sugimori, S. Mori, Y. Kawashima, Introduction of a new index for the prediction of capping tendency of tablets, Chem. Pharm. Bull. 37 (1989) 458–462.

[8] K. Sugimori, S. Mori, Y. Kawashima, Characterization of die wall pressure to predict capping of flat- or convex-faced drug tablets of various sizes, Powder Technol. 58 (1989) 259–264.

[9] K. Sugimori, S. Mori, Y. Kawashima, The role of binders in the prevention of capping within a tablet, Chem. Pharm. Bull. 37 (1989) 1064–1067.

[10] K. Sugimori, S. Mori, Y. Kawashima, Application of a newly defined capping index in evaluation of the compressibility of pharmaceutical powders, Adv. Powder Technol. 1 (1990) 25–37.