2. Stress distribution in tablets and capping phenomena

2. 1 Analysis of primary factors influencing capping

Capping phenomena have been studied by Train\(^1\), Long\(^2\), Shotton\(^3\), and other workers for many years. However, few of their fruits can be applied to practical production of a tablet which is performed by rotary tableting machine within such a short period of time as 0.02 to 0.1 seconds. The author has investigated the primary factors influencing capping which are listed in Table 1. A part of the results will be described below.

(a) Iterative compression

Compressive strength and capping generation of tablets were determined by two different compressing methods. Two tableting machines (A and B) were used which showed different characteristics in tableting. One comprises the iterative compressing formation which was carried out by A-equipment first and then by B-equipment, while the other comprises the converse procedure. The results obtained in this test are listed in Table 2. It can be estimated that capping would not take place in a compressing stage but in an ejecting one since the same operating conditions were employed in both stages. The difference in tableting performance between these two equipments might possibly result from the slight difference in mechanism of the ejecting stage.

(b) Ejection under pressure

The author has devised a specific rotary tableting machine with pressurized ejection mechanism, which is schematically illustrated in Fig. 1. This machine allows tablets to be discharged out of a die in keeping application of compression pressure from 1 to 10 MPa with upper and lower punches after the main compression. The compressing force during ejection can be adjusted by sliding wedge-type pressure adjuster and by moving guide rail of the lower punch up and down.

Figure 2 shows a drilling load measuring apparatus which consists of a load cell \(1\), a sample holder \(2\), elevating equipment \(3\), a drill bit \(4\), and a recorder \(5\). Typical drilling load distributions of the tablets obtained by the machine shown in Fig. 1 are illustrated in Fig. 3 where the result of the pressurized ejection is compared with that of the non-pressurized ejection. It is found that crack occurred at 2 mm depth from the surface.

Summarizing the results obtained from these findings:

1) The primary factors influencing capping of
Table 1 Factors related to capping in compressed tablets

| Physical process | Capping factor                                      | Binding force of powder | Crack of compact |
|------------------|-----------------------------------------------------|-------------------------|------------------|
| Compression stage| Pre-compression                                    |                         |                  |
|                  | Degree of compression                               |                         |                  |
|                  | Pre-compressing period                              |                         |                  |
|                  | Maximum compressing force                           |                         |                  |
|                  | Compressing period                                  |                         |                  |
|                  | Increasing rate of compressing force                |                         |                  |
|                  | Decreasing rate of compressing force                |                         |                  |
|                  | Stress distribution in compact                      |                         |                  |
|                  | Displacement and displacing speed of upper and lower punch |                         |                  |
|                  | Friction between compact and die wall               |                         |                  |
| Ejection stage   | Ejection of compact                                 |                         |                  |
|                  | Displacement and displacing speed of upper and lower punch |                         |                  |
|                  | Friction between compact and die wall               |                         |                  |

Table 2 Compressive strength and capping percentage

| 1st compression | 2nd compression | Compressive strength | Capping percentage |
|-----------------|-----------------|----------------------|--------------------|
| A               | B               | 8.3 N                | 100%               |
| B               | A               | 50.8 N               | 0%                 |

the compacts produced by a rotary tabletting machine are involved in the ejection stage.

2) The reduction of capping can be effectively achieved by the method in which the tablets are discharged out of a die under the remaining compression of 1 to 10 MPa with an upper and lower punch.

2. 2 Stress analysis in capping generation

(a) Compressive stress during compression

Generation of capping may be considered to
be due to the stress condition during decompression. The author measured the compressive stresses in the die shown in Fig. 4 at the upper punch and the die wall during compression and decompression periods. As shown in Fig. 5, the stress $q_r$ at the die wall still exists after complete release of the pressure applied by the upper and lower punch.

Figure 6 shows the effect of the curvature radius of a concave tablet on the residual stress at the die wall. As can be seen from Fig. 6, the residual stress at the die wall increased as the ratio $h/h_E$ increased, that is, the radius of curvature decreased. It has been empirically known that capping increases as the curvature radius decreases. In view of these facts, it may be considered that capping is closely related to the residual stress at a die wall.

(b) Residual stress at die wall $q_r$ and extent of capping

If the compressive stress at a die wall is different from the compressive stress of an upper punch, a shear stress is generated in the body of the tablet. The maximum shear stress in the tablet may be given by

$$
\tau_{max} = \frac{(\text{Compressive load applied by upper punch}) - (\text{Stress at die wall})}{2}
$$
The value of $\tau_{\text{max}}$ during decompression was examined for two materials: caffeine and ethoxybenzamide, which are regarded capping-poor and capping-rich, respectively. The results are shown in Fig. 7. $\tau_{\text{max}}$ becomes $\eta_{r}/2$ when the compressive force of the upper punch is completely released.

On the other hand, Fig. 8 shows the compressive stress-compressive strain curves of the cylindrical tablets of caffeine and ethoxybenzamide, which have been prepared by tableting each 1 gram of the powders by a circular die of 1 cm$^2$ cross sectional area and a flat punch. From the curve in Fig. 8, the maximum compressive strength of the tablets can be determined as $P_b$ and then the maximum shear stresses in the tablets at fracture as $P_b/2$. By comparing the value of $P_b/2$ with those of $\eta_{r}/2$, the maximum shear stresses in the tablets under complete decompression give the relations:

$$\frac{\eta_{r}}{P_b} = 1 \quad \text{for ethoxybenzamide}$$
$$\frac{\eta_{r}}{P_b} < 1 \quad \text{for caffeine}$$

These findings have proven well that the capping tendency of ethoxybenzamide is high and that of caffeine low.

(c) Capping tendency and $q_r/P_b$

Consider the ratio of the compressive strength of the tablet obtained by ejection under no pressure, $H_r$, to the one under a pressure, $H_0$, as the specific strength, $H/H_0$. The relationship between $H/H_0$ and $q_r/P_b$ for several materials are shown in Fig. 9. It is found that the specific strength varied largely at $q_r/P_b = 1$. This implies that the degree of capping can be estimated by the value of $q_r/P_b$. It is emphasized, therefore, that a practical design for a tablet requires a suitable selection of powder prescription and tablet shape.

3. Powder coating granulation and packing structure of solid-liquid system

3. 1 Necessary condition for powder coating granulation

The conditions required for powder coating granulation are considered to be:

1) Mixing and dispersion of solids and liquids
2) Optimization of moisture content
3) Consolidation by tumbling

The first is how uniformly coating liquids and powders can be dispersed in the bulk of solids; the second is the ratio of liquid to solid...
Table 3 Processes, equipment, and operating conditions required for powder coating granulation

| Required item                  | Required process          | Equipment                  | Operating condition                      |
|--------------------------------|---------------------------|----------------------------|------------------------------------------|
| Mixing and dispersion of liquid| Feed of small liquid droplets | Spraying equipment        | Liquid pressure                          |
|                               |                           |                            | Liquid/air flow rate                      |
|                               |                           |                            | Position of nozzle                        |
|                               | Large shearing force      | Coating granulator         | Quantity fed                             |
|                               | Uniform shearing          |                            | Rotating speed                           |
|                               | – without stagnation zone |                            |                                          |
|                               | – without local excessive shearing |                      |                                          |
|                               | Large mixing speed        |                            |                                          |
| Mixing and dispersion of powder| Shear mixing              | Powder and liquid feeding system | Control program                         |
|                               | { as above                |                            |                                          |
|                               | Circulating flow formation|                            |                                          |
| Optimization of moisture content| Programmed feeding of powder and liquid | Coating granulator | Control program                         |
|                               | Program control for moisture content of particle surface |                            |                                          |
| Tumbling compaction           | Moderate tumbling compaction | Coating granulator        | Quantity fed                             |
|                               |                           |                            | Rotating speed                           |

in the bulk of solids; the third is an importance of tumbling for suitable consolidation. The required equipments and conditions are listed in Table 3.

3. 2 Development of coating granulator

(a) Coating granulator

A configuration of the granulator which has been developed after trial and error is schematically illustrated in Fig. 10. This configuration was found to provide an excellent performance on smooth convective mixing without any local stagnation in the granulator, which was called CF equipment by the author. Typical mixing characteristics are shown in Fig. 11, which indicates that the mixing was finished within 20 to 30 seconds.

(b) Spraying of coating liquids

The ratio of air to liquid flow rates is of great importance for spray coating liquids with concentric two-fluid nozzle system. It is because as the droplet diameter is larger, the generation rate of particle coagulation becomes larger. As can be seen from the relationship

![Fig. 10 Centrifugal fluidization-type coating equipment (CF equipment)](image)

![Fig. 11 Mixing characteristics of CF-1000 equipment](image)
between the air-to-liquid ratio and the generation rate of coagulated particles shown in Fig 12, the decrease in droplet size requires an increase in air-to-liquid ratio.

(c) Control system of moisture content on particle surface

To keep the mixture at its optimum wet condition, the granulator is required to be equipped with the specific control system which consists of measuring moisture content of particle surface and determining the feed rate of spraying liquid or powder. It is well known that powder bed becomes an electrical conductor to some extent if spraying liquid is an aqueous solution. Hence the electrical conductivity of the powder bed was used as a measure of moisture content of the particle surface. The system established is schematically indicated in Fig. 13.

3. 3 Powder coating granulation

Experimental data with the granulation established by the author and operated under the automatically controlled conditions are listed Table 4.

3. 4 Optimum ratio of solid to liquid

Another experiment using this granulator was also conducted to determine the optimum ratio of liquid to solid for practical operations.

The liquid used was a saturated syrup solution and the result is listed in Table 5.

3. 5 Occupation ratio of liquid among particles and optimum ratio of solid to liquid

The specific volume of wet compact $\varphi_0$ was determined by gradually compressing the particulate material which had been preliminarily mulled in a 100 cm$^3$ container with liquid at a given liquid-to-solid ratio of 1.5 kg/cm$^2$. Its porosity $\varepsilon_0$ is calculated by the following form:

$$\varepsilon_0 = \frac{L + S \frac{1}{\rho_p}}{S + L \rho_s} \frac{1}{\varphi_0}$$

where $L$ : Feed of syrup [cm$^3$]  
$S$ : Feed of powder [g]  
$\rho_p$ : True density of powder [g/cm$^3$]  
$\rho_s$ : Density of syrup [g/cm$^3$]

The ratio of a void filled with water among particles of a wet material in consideration of the solubility, $\Psi$, is expressed as

$$\Psi = \frac{L(1 + \lambda \frac{1}{\rho_p})}{(S + L \rho_s) \varphi_0 \varepsilon_0 + L(1 + \lambda \frac{1}{\rho_p})}$$

$$= \frac{L(1 + \lambda \frac{1}{\rho_p})}{S - \lambda L} \frac{1}{\varphi_0 \varepsilon_0} + \frac{L(1 + \lambda \frac{1}{\rho_p})}{S - \lambda L}$$

(2)
Table 4 Experimental data of size growth for reproducibility

| No. | Feed of nucleus | Sprayed amount of powder | Sprayed amount of binding liquid | Good-quality percentage | Average diameter | Particle distribution σ | Nucleus |
|-----|----------------|--------------------------|---------------------------------|-------------------------|------------------|------------------------|---------|
|     | [g]            | [g]                      | [cm³]                           | [%]                     | [µm]             | [µm]                   |         |
| 1   | 2250           | 1960                     | 865                             | 98.75                   | 760              | 60                     |         |
| 2   | 2250           | 1960                     | 865                             | 98.15                   | 770              | 50                     |         |
| 3   | 2250           | 1960                     | 855                             | 98.58                   | 760              | 50                     |         |
| 4   | 2250           | 1960                     | 870                             | 99.52                   | 750              | 50                     |         |
| 5   | 2250           | 1960                     | 865                             | 99.19                   | 760              | 60                     |         |
| Ave. |                |                          | 865                             | 98.84                   | 760              | 54                     |         |
| σ   |                |                          | 4.5                             | 0.48                    | 3                | 2                      |         |

Table 5 Optimum ratio of liquid to solid of several powders

| Powder | Powder sugar | Cornstarch | Ascorbic acid | Nicotinic acid | Fine crystallized cellulose |
|--------|--------------|------------|---------------|----------------|-----------------------------|
| Optimum ratio of liquid to solid | 0.17 | 0.52 | 0.16 | 0.18 | 9.9 |

As seen from Eq. (2), the occupation ratio \( \Psi \) can be calculated from the specific volume \( \varphi_{01} \) and the \( \varphi_{01} \) obtained from Eq. (1). Plots of the occupation ratio against the ratio \( L(1 + \lambda/\rho_p)/(S - \lambda L) \) are shown in Fig. 14, in which the optimum granulating condition is expressed as the symbol \( \Phi \).

It is obvious from this figure that the optimum condition would be involved within 55 to 65% of \( \Psi \). The author could verify this tendency irrespective of the kind of liquid or powder materials. This suggests that powder coating granulation could be optimized in actual operation for most cases if the occupation ratio \( \Psi \) is kept within 55 to 65%.

References

1) Train, D.: *J. Pharm. Pharmacol.*, 8, 745 (1956).
2) Long, W. M.: *Powder Met.*, 6, 73 (1960).
3) Shotton, E.: *J. Pharm. Pharmacol.*, 25, suppl., 202S (1971).