Effect of Water Content on Sticking during Compression

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A rotary type tablet machine (Correct 12 HUK-AW, Kikusui, Co., Ltd.) fitted with flat-faced punches 8 mm in diameter was used to measure the scraper pressure (SCR), a kind of shear stress, as a direct means of evaluating sticking. The shear stress between the tablet and lower punch surface was determined using a system of SCR detection (strain gauge type, Okada Seiko Co., Ltd.).

SCR increased with increasing compression pressure for samples at all water contents tested. The SCR had a tendency to increase up to a water content of 3%, then showed a decrease at a high water content. This suggests that the adhesive force between particles increases with increasing water content, but for high water content, shear stress decreased due to the effect of water as a lubricant. It is assumed that the sticking ratio increases proportionally with water content until about 3%.

Mean surface roughness (R_a) of tablets due to sticking showed the same relationship between SCR and water content. The SCR increased with an increasing value of R_a. Thus, it was suggested that the adhesive force between particles and the punch surface plays an important role in sticking.

Key words sticking; scraper pressure; water content; shear stress; surface roughness

Sticking and picking are phenomena that occur during the manufacture of tablets, in which particles adhere to the punch during compression. These problems can be major obstacles in the production of high quality tablets.

Various factors related to the materials used, *i. e.* melting point, wettability, size and distribution, surface condition and the hardness of particles, may be responsible for sticking and picking. Factors related to machinery and the environment such as the surface condition of the punch, compression force and speed, as well as the temperature and humidity around the machine, may also have an effect. Schmidt *et al.*[1] measured the force of removing the tablet from the punch surface. Naito *et al.*[2-4] reported a means of measuring the slipping force of a tablet surface and the passive pressure of the lower punch during compression. Toyoshima *et al.*[5] studied the relationship between sticking and tablet surface roughness. Sugimori *et al.*[6] found that capping occurs when a tablet is cracked by high residual die wall pressure at the final stage of the decompression process. These studies, however, dealt with capping, and reports of direct measurement sticking and picking are rare.

Measurement of scraper pressure (SCR), a kind of shear stress, would be the most direct means of evaluating sticking and picking. The purpose of this study was to quantify sticking and picking on the basis of SCR. We focused here upon the effects of water content on sticking during compression, because sticking occurs easily with an increase in water content. Measurements were carried out under various conditions of drying of the granules and the results were explained on the basis of water content.

Materials and Methods

Materials The powders used to prepare the granules were lactose (JPXII grade, DMV 200 mesh) and polyvinylpyrrolidone (PVP K-90, BASF).

Granulation Method Granulation experiments were conducted using an extrusion type granulator (Type KAR-130, Tsutsui Rikagakukikai) after 800 g of lactose and 80 ml of granulating fluid (5% water solution of PVP K-90) were kneaded in a kneading machine (Type LK-5, Erweka) for 15—20 min and passed through a 1.0 mm aperture sieve. The wet mass was dried at 110 °C for 30, 60, 120, 180 or 360 min, and water content was calculated from the weight after drying each time.

Measurement of SCR A rotary type tablet machine (Type Correct 12 HUK-AW, Kikusui) fitted with flat-faced punches 8 mm in diameter was used to measure SCR during 5—10 turns of the rotor. The shear stress between the tablet and the lower punch surface was deterimed using a system of SCR detection (strain gauge type, Okada Seiko) placed on the scraper (Fig. 1), and the output value of strain was examined with a compression stress analyzer (Type Daatsu, Okada Seiko).

Detection of Sticking The surface of the tablets was visually observed and the sticking ratio was estimated by the following equation:

$$\text{sticking ratio} = \frac{\text{sticking area (cm}^2\text{) in the tablet}}{\text{flat-faced area (cm}^2\text{) of the tablet}} \quad (1)$$

where the sticking area was measured with a planimeter.

The average surface roughness, R_a (μm), was measured by scanning laser microscopy at a magnification of 20 (Type 1LM21, Laser Tex) at five points on the tablet surface. R_a was calculated by the following equation:

$$R_a = (1/N) \sum_{i=1}^{N} |z_i - \bar{z}| \quad (2)$$

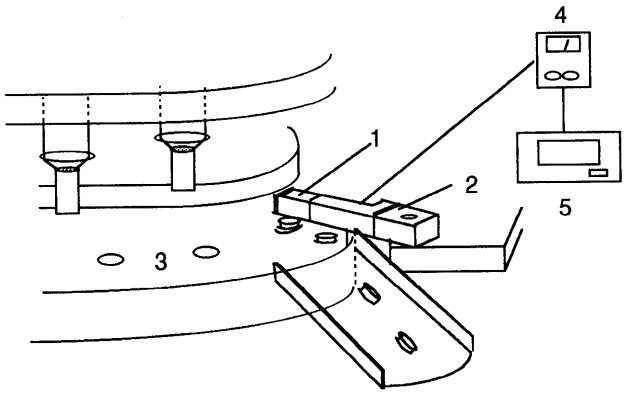

Fig. 1. Schematic Diagram of the Device Used to Measure SCR

1, unit of SCR gauge; 2, scraper; 3, turntable; 4, amplifier; 5, computer.

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where N is the total pixel number, z_i (μm) is the height of i, $\bar{z}$ (μm) is the mean of the height.

Granule Strength (S_t) Measurement S_t was measured with a hardness tester (Type Grano, Okada Seiko), and values are shown as averages of 30 particles. S_t (mN/m^2) was estimated by the following equation[7]:

$$S_t = 2.8P/(\pi d^2) \quad (3)$$

where P (g) is the crushing load of the granule and d (μm) is the granule diameter.

Tablet Strength Measurement The diametral compression test for tablets was performed at least 24 h after compression. The method of measurement of tablet tensile strength, σ_t, has been described elsewhere.[8] σ_t (N/m^2) was calculated using the following equation[9]:

$$\sigma_t = 2F/(\pi Dt) \quad (4)$$

where F (kg) is the maximum load when the tablet fractured, D (cm) is the tablet diameter, and t (cm), the tablet thickness, as measured with a micrometer (IDC-1012M; Mitsutoyo Co., Ltd.). The mean value of 10 tablets was determined.

Results and Discussion

Changes in Water Content of Granules during Drying Ten-gram samples of granules were reweighed after drying at 110 °C for various periods. Figure 2 shows the relationship between drying time and the water content of the granules (dry base). The water content of the granules was estimated from the ratio of the granule weight before and after drying. Water content decreased suddenly until 30 min between drying time, and the decreasing tendency was shown by slow degrees afterwards, as shown in Fig. 2.

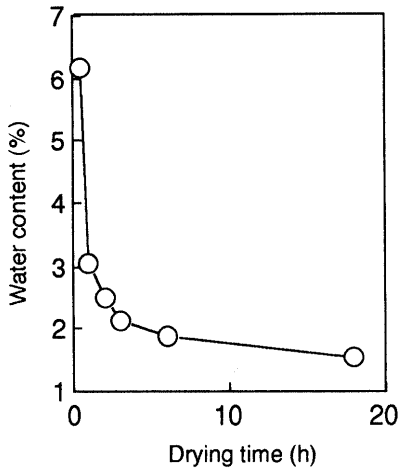

Fig. 2. Relationship between Drying Time and Water Content

Effect of Water Content on Granule Strength (S_t) Previously, we showed that the adhesive force between particles was found to be greatly affected by water adsorption.[10] In this study, we examined the effect of

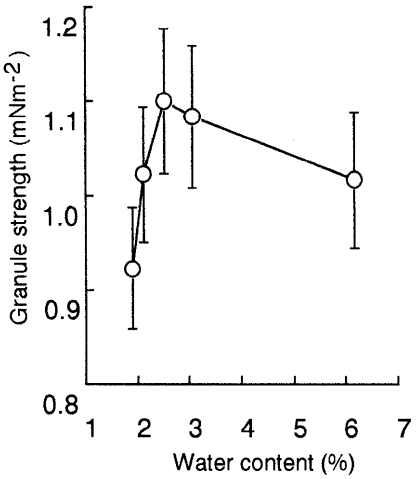

Fig. 3. Effect of Water Content on Granule Strength

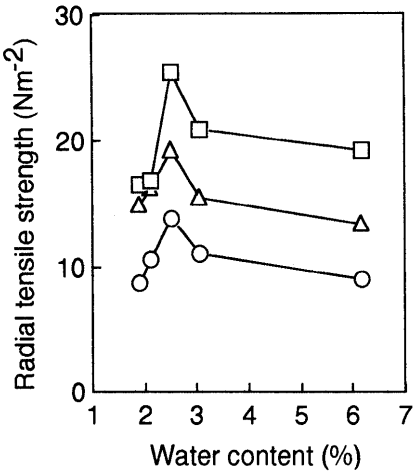

Fig. 4. Effect of Water Content on Radial Tensile Strength
○, 100 (MPa); △, 150 (MPa); □, 200 (MPa).

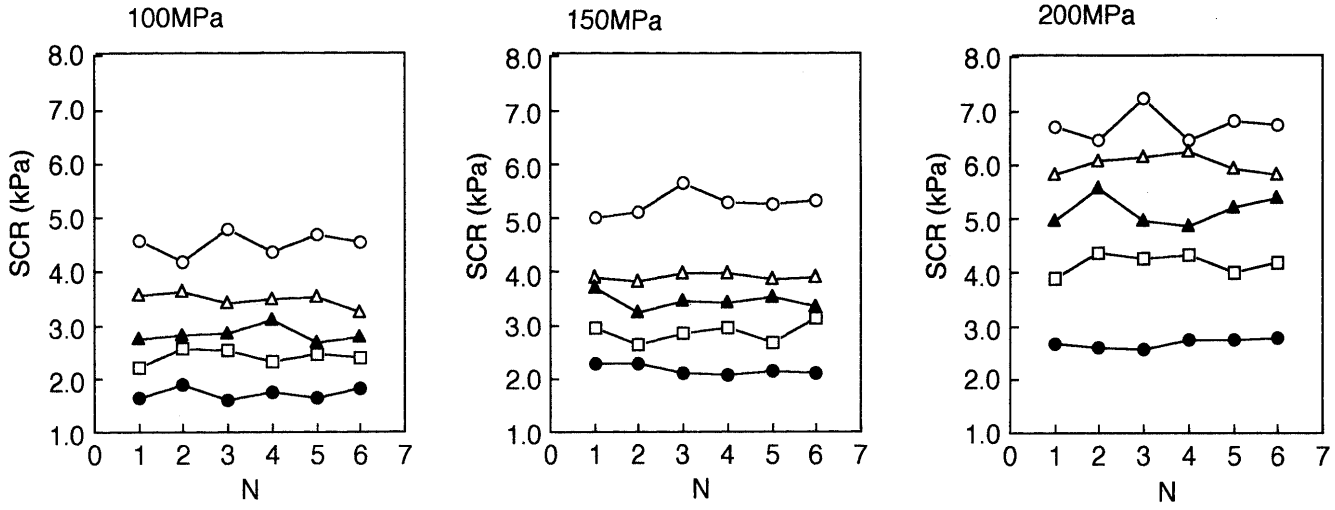

Fig. 5. Relationship between Punch Number and SCR
□, 1.9%; ▲, 2.1%; △, 2.5%; ○, 3.1%; ●, 6.2%.

water content on S_t. S_t tended to increase until a water content of about 3%, and showed a decrease at high water content. At a lower water content, S_t had a low value caused by the small adhesive force between particles, while at a high water content, S_t decreased due to the surface energy being lowered by the adsorption of water molecules[10,11] and due to particles slipping.

Effect of Water Content on Tablet Strength Figure 4 shows the effect of water content on the radial tensile strength of tablets, σ_t, for three levels of tableting pressure. For each tableting pressure, σ_t tended to increase with water content increase until reaching a water content of 2.5%, and showed a decrease at a high water content. These results indicate the same relationship between S_t and water content.

Effect of Water Content on SCR Sticking occurs when the punch surface is not smooth. We considered that the SCR represented the shear stress between the tablet surface and punch surface. Therefore, we previously examined the relationship between SCR and shear strength by per-

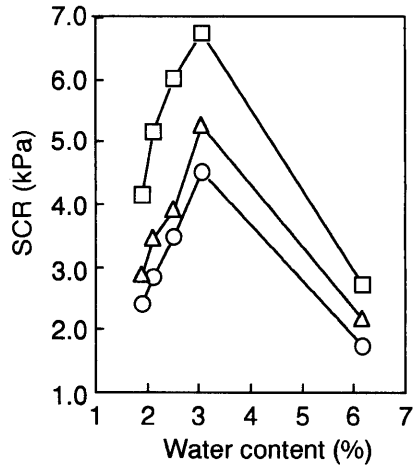

Fig. 6. Effect of Water Content on SCR
○, 100 (MPa); △, 150 (MPa); □, 200 (MPa).

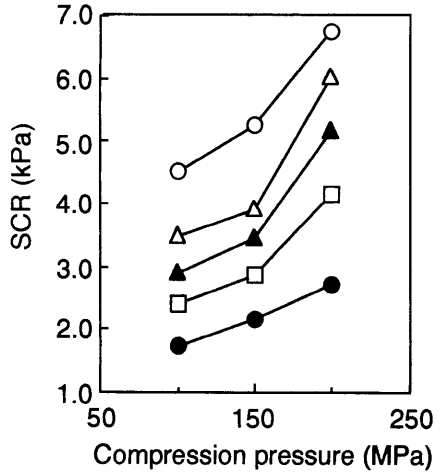

Fig. 7. Effect of Compression Pressure on SCR
□, 1.9%; ▲, 2.1%; △, 2.5%; ○, 3.1%; ●, 6.2%.

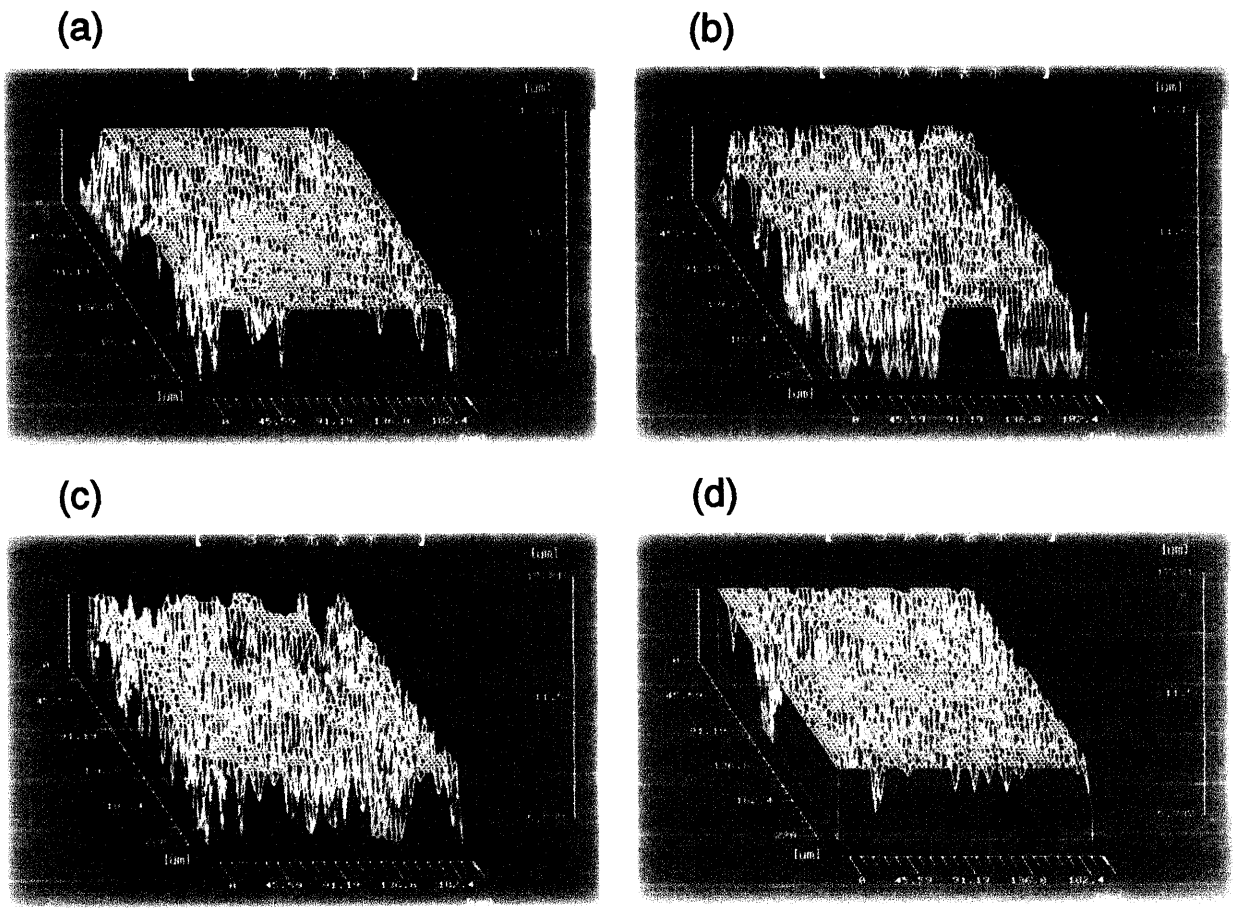

Fig. 8. Three-Dimensional Indication of Tablet Surface
(a) 1.9%; (b) 2.1%; (c) 3.1%; (d) 6.2%.

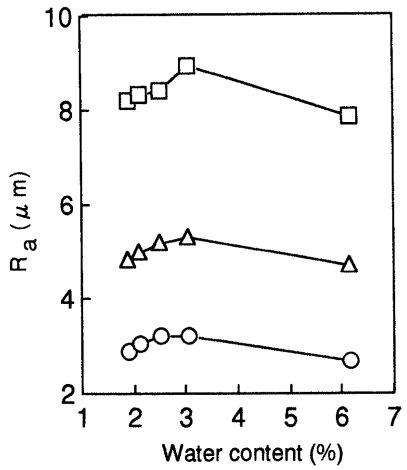

Fig. 9. Relationship between Water Content and Surface Roughness (R_a)

○, 100 (MPa); △, 150 (MPa); □, 200 (MPa).

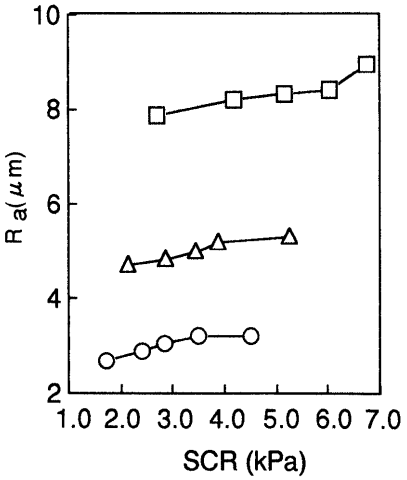

Fig. 10. Relationship between SCR and R_a

○, 100 (MPa); △, 150 (MPa); □, 200 (MPa).

forming shear tests at various temperatures.[12] These results showed a good correlation. Figure 5 shows the change in SCR value with water content for each punch, N (N is the punch number), and each measurement point is the average of six tablets. These results suggest that the conditions of all punch surfaces are almost the same because no scattering of SCR value was seen for each compression pressure. However, the SCR value changed with the water content of the granules.

Figure 6 shows the effect of water content on SCR. The SCR tended to increase until reaching a water content of 3%, and showed a decrease at 6% for each punch pressure tested. The adhesive force between particles and the punch surface was considered to increase with increasing water content, while at a high water content, this force decreased due to the action of water as a lubricant.

Figure 7 shows the changes in SCR with compression pressure for each water content value. SCR value increased with increasing compression pressure. The shear force between the lower punch and the tablet surface was considered to increase with increasing compression pressure, due to the increase in true contact area between the lower punch and the tablet surface with increasing compression pressure.

Relationship between SCR and Sticking At a high compression pressure and at a proper water content, SCR became large, and under these conditions the probability of sticking was high. Thus, we observed the surface of tablets using a scanning laser-microscope to determine whether sticking actually occurred, and examined the SCR value in relation to water content. Figure 8 shows the tablet surface measured by scanning laser microscopy for a load of 100 (MPa). We observed the surface roughness of tablets due to particles of the tablet surface adhering to the punch surface, which will cause sticking. These results indicate that R_a increased with increasing water content until 3%.

Figure 9 shows the effects of water content on the surface roughness, R_a. R_a increased with increasing water content for each compression pressure. This result shows the same relationship between SCR and water content.

Figure 10 shows that R_a increases in proportion to SCR. Thus, it was suggested that the measurement of SCR allows estimation of the amount of sticking which will occur in tablet production.

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