Development of a Novel Compression Tester and Rheo-Mechanical Properties of Wet-Mass Powder. II —Effect of Moisture Content on the Rheo-Mechanical Properties—

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In our previous paper [Watano S., *et al.*, *Chem. Pharm. Bull.*, 51(7), 747—750 (2003)], a novel compression tester, which enables the vertical and radial pressure transmission measurement, has been developed and quantitative analysis of rheo-mechanical properties of wet-mass powder prepared by different kneading times was conducted. In this study, the compression test was conducted by using the kneaded wet-mass powders prepared by different moisture contents. Pressure transmission characteristics and rheo-mechanical properties have been investigated to characterize the wet-mass powders. The relationship between these parameters and granule physical properties was also investigated.

Key words compression tester; wet-mass powder; rheo-mechanical property; kneading; moisture content

Kneading and extrusion are one of the fundamental processes in the manufacturing of pharmaceutical and agriculture formulations, foods, ceramics, chemicals and *etc.* It is because their operations are simply with low cost and uniform physical properties (size, density, main ingredient content and *etc.*) can be obtained. However, their mechanisms have not been well studied yet, although several trials have been made by measuring the extrusion pressure[1,2] and by several experiment approaches.[3,4]

In the previous paper,[5] we have developed a novel compression tester with vertical and radial pressure transmission measurements and then proposed a practical method to probe rheo-mechanical properties of wet-mass powder. In conclusion, the rheo-mechanical properties of wet-mass powders prepared by different kneading times have been cleared up. However, the effects of moisture content on the rheo-mechanical properties as well as on the granule physical properties have not been studied yet.

This paper deals with further investigation on the rheo-mechanical properties of wet-mass powders prepared by different moisture contents.

Experimental

Powder Samples Powder samples and binder used here were the same as previously reported.[5] Pharmaceutical excipient consisted of lactose, cornstarch and crystallinecellulose (mixing mass ratio was 67.2 : 28.8 : 4.0), and 3 wt% of dry hydroxypropylcellulose (HPC-L) as binder were mixed together, and then kneaded with purified water for predetermined time. The water content varied from 21 to 27%.

Equipment and Procedures For wet kneading, a five litter planetary mixer with twin top-drive paddles (Twin-servo, Shinagawa Manufacturing Co., Ltd.) was used. The revolution and turning speeds were 10 rpm and 4.025 rpm, respectively. The kneaded masses were extruded through an extrusion granulator (DG-L1, Fuji Paudal, Co., Ltd.), then dried in a fluidized bed (NQ-125, Fuji Paudal, Co. Ltd.).[6] These experimental equipments and procedures were the same as previously reported.[5]

A compression test of the kneaded mass was conducted using a developed compression tester as previously descried.[5] The moving speed of upper punch was set at 50 mm/min. As described in our previous paper,[5] Young's modulus, E, the pressure transmission, G (ratio of vertical upper to lower pressure), Rankin factor, K (ratio of vertical to radial pressure) and effective internal friction ($\tan \phi$ were calculated based on the compression test).

Results and Discussion

Solid–Liquid Occupation Diagram Figure 1 shows relationship between kneading torque and water content (this is so called "solid–liquid mixing diagram"). According to the literature,[7] the solid–liquid mixing diagram can be divided into four states; pendular, funicular, capillary and slurry. In our powder system, pendular state should be the optimum moisture content to produce granules (pellets) suitable for pharmaceutical usage. Also, seen from Fig. 1, 30 wt% of the moisture content was the pendular limit, thus, we determined to vary initial moisture content between 21 and 27 wt% in the following.

Figure 2 indicates the relationship between the critical pressure and moisture content. This critical pressure is the point where the pressure transmission begins to increase awfully in the plastic deformation region. It shows that the water completely distributes among the powder bed.[5] Seen from Fig. 2, the critical pressure has a liner correlation with moisture content. This implies that the critical pressure can be used to analyze the kneading condition in case of varying moisture content. Considering the results obtained in the previous paper[5] (we have shown that the critical pressure can be used in case of changing the kneading time), the critical pressure can be used universally for analyzing the kneading condition (state).

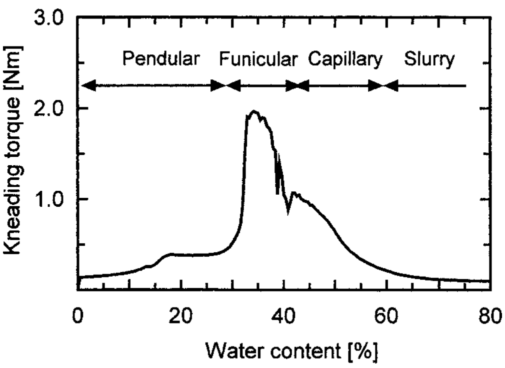

Fig. 1. Phase Diagram of Solid–Liquid Mixture

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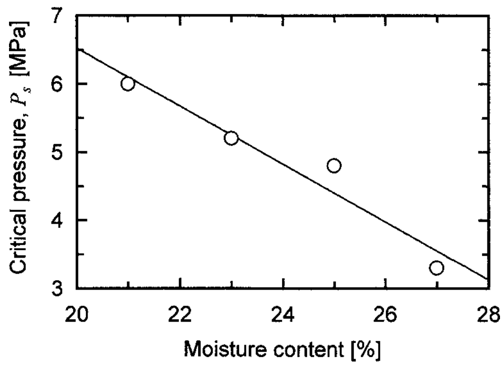

Fig. 2. Relationship between Critical Pressure and Moisture Content

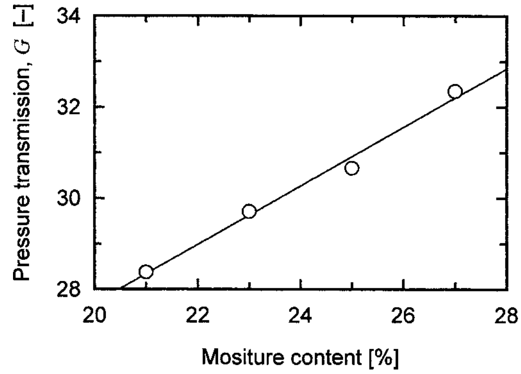

Fig. 5. Pressure Transmission *vs.* Moisture Content

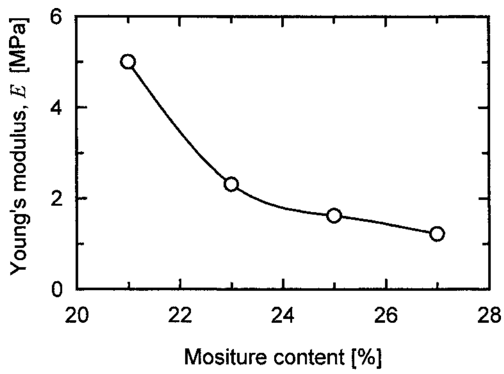

Fig. 3. Young's Modulus *vs.* Moisture Content

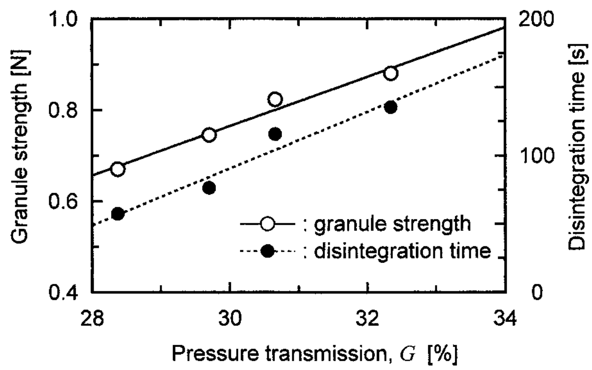

Fig. 6. Relationship between Pressure Transmission and Granule Physical Properties

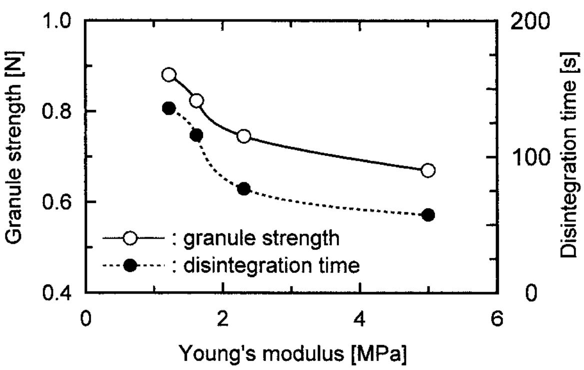

Fig. 4. Relationship between Young's Modulus and Granule Physical Properties

Mechanical Properties of Wet-Mass Powder Figures 3 and 4 investigate the relationship between Young's modulus and moisture content, and granule physical properties (granule strength and disintegration time) and Young's modulus. Seen from these figures, Young's modulus gradually decreases with moisture content and granule strength and disintegration time become small with Young's modulus. This is because the wet-mass powder becomes soft with moisture content, leading to increase the plastic deformation characteristics. This also contributes to produce hard pellets, because the wet-mass powder is compressed well when it goes through the extruder's screen. However, contrary to the previous paper,[5] which investigated the same characteristics when varying kneading time under the same moisture content, the granule physical properties and Young's modulus does not show any linear relationships. These results conclude that the Young's modulus cannot be used to predict granule physical properties, in case of changing moisture content.

Pressure Transmission Characteristics Figures 5 and 6 examine the relationship between pressure transmission and moisture content, and granule physical properties (granule strength and disintegration time) and pressure transmission. Both figures show linear relationships; with an increase in moisture content, water distribute/penetrate among the powder bed well, resulting into the larger pressure transmission. As discussed earlier,[5] pressure transmission of wet-mass powder, in case of changing kneading time with keeping the same moisture content, showed linear relationships with kneading time and pellet's physical properties. Therefore, it can be said that the water distribution directly affects the pressure transmission for both kneading time and moisture content change. In addition, this pressure transmission can predict granule physical properties (granule strength and disintegration time) with a good accuracy.

Rheological Properties of Wet-Mass Powder Figures 7 and 8 investigate the relationship between effective internal friction and moisture content, and granule physical strength (granule strength and disintegration time) and effective internal friction. Seen from Fig. 7, effective internal friction linearly decreases with moisture content, showing that the large quantity of water between particles reduces the friction.

Also, kneading with higher torque (as shown in Fig. 1) results in a large granule strength and disintegration time. Since the granule physical properties (strength and disinte-

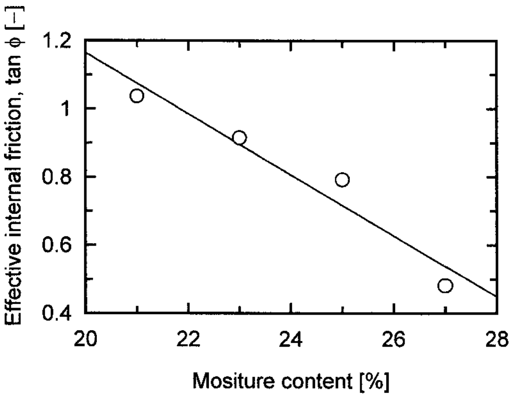

Fig. 7. Effective Internal Friction *vs.* Moisture Content

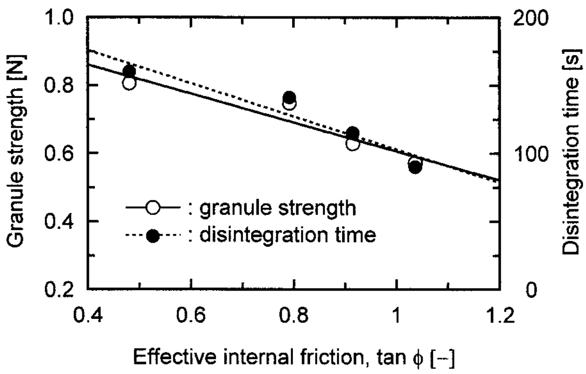

Fig. 8. Relationship between Effective Internal Friction and Granule Physical Properties

gration time) and effective internal friction show linear relationship, the effective internal friction can predict granule physical properties well. This tendency is contrary to the previous study,[5] where kneading was conducted under various kneading times. In the case where the rheologocal properties change awfully such as moisture content change, the effective internal friction can be used to analyze the kneading condition, as well as to predict physical properties of extruded pellets.

Conclusions

Developed compression tester, which can measure both vertical and radial pressure transmissions, was used to analyze the rheo-mechanical properties of wet-mass powders prepared by different moisture contents. It was found that the mechanical property (Young's modulus) has no linear relationship between granule physical properties, though smaller Young's modulus resulted in larger granule strength and disintegration time. Contrary to Young's modulus, vertical pressure transmission and effective internal friction had linear relationships with granule physical properties. These parameters well predicted physical properties of granules prepared by different moisture contents.

By generalizing the present results with the previous achievements,[5] it can be concluded that the mechanical property of wet mass powder described by using Young's modulus can be used to predict granule physical properties where the elasticity of wet-mass powder changed awfully. The effective internal friction can be used to predict granule physical properties where the rheological properties changed greatly. In addition, pressure transmission can be used to predict granule physical properties where the mechanical as well as the rheological properties changed. Depending on the wet-mass powder's properties, optimum parameters was found to exist for analyzing the wet-mass powder properties as well as predicting the granule physical properties.

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