CASE 7

Optimization of a Measuring Method for Granule Strength

Abstract: Our company produces granule products from herbal extracts. The granules are produced by a dry granulating process, including press molding, crushing, and classifying. The granules require a certain amount of hardness and fragility to meet the specifications and to make pills easier to swallow. Granules are difficult to dissolve if too hard and become powdery if too soft. In the current measuring method, granules are vibrated; then the powder is separated by screening. The amount of powder remaining on the screen is measured to indicate the hardness. This method is time consuming and the measurement error is high. This case reports the development of a new method with a short measurement time and high precision.

1. Introduction

Our research dealt with granules manufactured from solidified essence that is extracted from herbal medicines. Measurement of the granular strength were taken as follows. After sieving granules and removing fine powders, we forcibly abraded them with a shaker and then got rid of the abraded granules through sieving and suction. Measuring the remainder after suction, we defined the granular strength as

$$\text{granular strength} = \frac{\text{sample weight} - \text{remainder weight)}}{\text{sample weight}} \times 100\% \quad (1)$$

Figure 1 is a schematic of a new measuring instrument. The measuring method is as follows.

1. *Supply of a test sample.* We removed the lid on a test sample (of known weight), placed the sample on a sieve, and replaced the lid.
2. *Abrasion and classification.* By sucking the inside air from an exhaust outlet using a vacuum cleaner, we made air flow out of a rotating slit nozzle. Receiving this flowing-out air, the test sample hits the sieve frame and lid and is thus abraded. The abraded powders were removed from the exhaust outlet through suction.
3. *Measurement of the remainder.* After a certain period, we measured the weight of the sample remaining on the sieve.

This measurement instrument was considered to have two types of functions. The first function was the ratio of the amount of abrasion to the operation time: The shorter the operation time, the less the abrasion; the longer the time, the greater the abrasion. In terms of this function, by focusing not on hardness as a quality characteristic but on pulverization of granules as the measurement instrument's function, we evaluated the function. Considering a time-based change in the number of granules or their shapes, we supposed that like a chemical reaction, the amount of abrasion for the operation

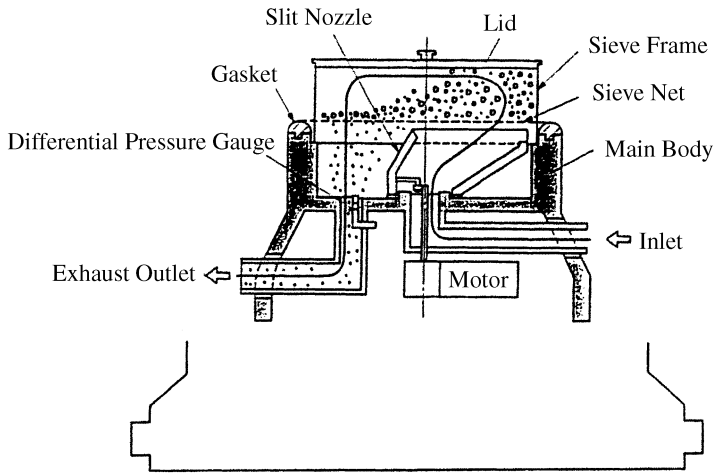

Figure 1
Schematic of new measurement instrument for granular strength

time followed not simple linearity but natural logarithmic proportionality.

The other function was the proportion of the amount of abrasion to the test sample's hardness. For a constant operation time, a test sample with low abrasive resistance (or high hardness) had a smaller amount of abrasion, whereas one with high resistance had more. The true value of hardness discussed here was unknown. However, since we obtained a relative difference by altering manufacturing conditions, we evaluated the difference as a signal.

2. SN Ratio

Considering the above as functions of a new measuring instrument, we developed a zero-point proportional equation based on two types of signal factors: operation time and abrasive resistance (hardness) of a test sample. Since it was difficult to collect and measure the abraded powders, we measured primarily the weight of a test sample (W_1), and secondarily, gauged the weight of the remainder left on the sieve net (W_2). The remaining rate was defined as

Table 1
Remaining fractions for experiment 1

Abrasive Resistance to Test Sample	Operation Time		
	T_1 (15 s)	T_2 (30 s)	T_3 (60 s)
M_1 (hard)	0.992	0.991	0.989
M_2 (medium)	0.990	0.988	0.986
M_3 (soft)	0.988	0.986	0.980

Table 2
Logarithmized values for experiment 1

Abrasive Resistance of Test Sample	Operation Time			Linear Equation
	T_1 (15 s)	T_2 (30 s)	T_3 (60 s)	
M_1 (hard)	0.00783	0.00944	0.01065	1.03961
M_2 (medium)	0.00984	0.01186	0.01389	1.33681
M_3 (soft)	0.01207	0.01388	0.02039	1.82062

$$\text{fraction remained} = \frac{W_2}{W_1} \tag{2}$$

The principle of abrasion was grounded on collision among granules; therefore, we focused on the same principle as that of a chemical reaction. Using this idea, we proposed that the remaining fraction of a test sample after an operation time of T seconds could be expressed by the following exponential equation of an operation time T:

$$\frac{W_2}{W_1} = e^{-\beta T} \tag{3}$$

$$\ln \frac{W_2}{W_1} = -\beta T \tag{4}$$

$$y = -\ln \frac{W_2}{W_1} \tag{5}$$

$$y = \beta T \tag{6}$$

Table 3
Control factors and levels

Control Factor		Level		
		1	2	3
A:	water content in granule	Low	Mid	High
B:	humidity of sucked air (RH %)	$L_1 - 10$	L_1	$L_1 + 10$
	A_1	$M_1 - 10$	M_1	$M_1 + 10$
	A_2	$H_1 - 10$	H_1	$H_1 + 10$
	A_3			
C:	mesh size (mm)	0.297	0.355	0.500
D:	suction pressure (Pa)	L_2	1.5 L_2	2 L_2
	F_1	M_2	1.5 M_2	2 M_2
	F_2	H_2	1.5 H_2	2 H_2
	F_3			
E:	lid height (mm)	57	42	27
F:	amount of test sample	Low	Mid	High
G:	distance between slit nozzle and sieve net	4	9	12

In short, this is identical to the idea of a chemical reaction.

As a test sample, we prepared granules of a certain size or larger obtained from an actual pretreated product. In analyzing experimental data, by selecting a natural-logarithmized remaining fraction as output, we calculated an SN ratio and sensitivity based on the ideal relationship $y = \beta T$. Table 1 shows the remaining fraction for the data in the first row of an L_{18} orthogonal array. In addition, Table 2 summarizes the output computed through natural logarithmization of the remaining rates.

Total variation:

$$S_T = 0.00783^2 + 0.00944^2 + \cdots + 0.02039^2 = 0.001448 \qquad (f = 9) \tag{7}$$

Effective divider:

$$r = 15^2 + 30^2 + 60^2 = 4725 \tag{8}$$

Linear equations:

$$L_1 = (0.00783)(15) + (0.00944)(30) + (0.01065)(60) = 1.03961$$
$$L_2 = 1.33681, \qquad L_3 = 1.82062 \tag{9}$$

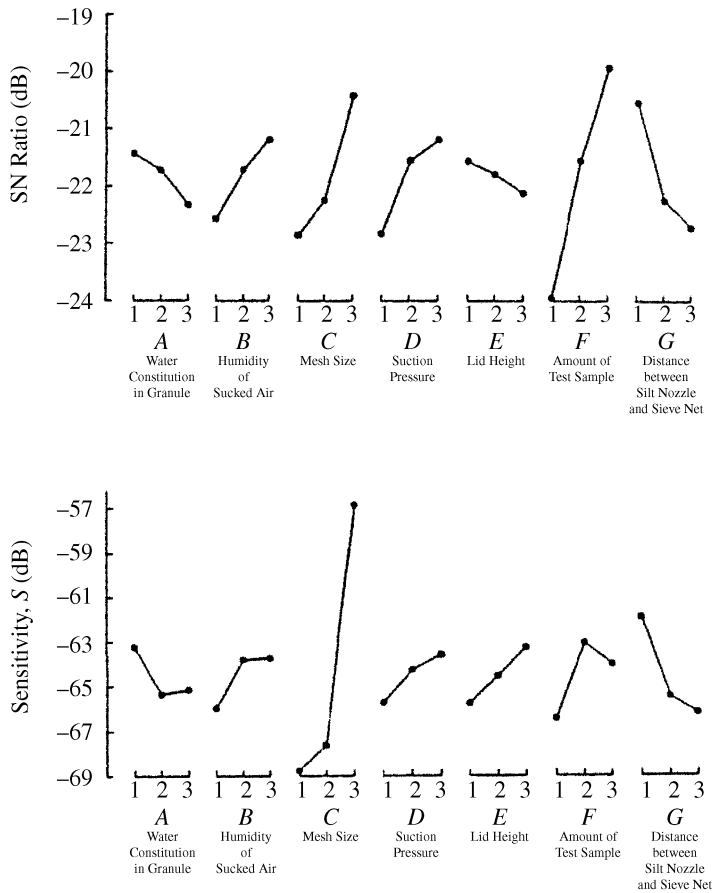

Figure 2
Response graphs for determining good pulverization conditions

Variation of proportional term:

$$S_\beta = \frac{(1.03961 + 1.33681 + 1.82062)^2}{(3)(4725)} = 0.001243 \qquad (f = 1) \tag{10}$$

Variation of differences between hardness between samples:

$$S_{M\beta} = \frac{1.03961^2 + 1.33681^2 + 1.82062^2}{4725} - 0.001243 = 0.000066 \qquad (f = 2) \tag{11}$$

Error variation:

$$S_e = 0.001448 - 0.001243 - 0.000066 = 0.00014 \qquad (f = 6) \tag{12}$$

Error variance:

$$V_e = \frac{0.00014}{6} = 0.000023 \tag{13}$$

Analysis for Finding Good Pulverization Conditions

SN ratio:

$$\eta = 10 \log \frac{(1/3r)(S_\beta - V_e)}{V_e} = 10 \log \frac{[1/(3)(4725)](0.001243 - 0.000023)}{0.000023} = -24.32 \text{ dB} \tag{14}$$

Sensitivity:

$$S = 10 \log \frac{1}{3r}(S_\beta - V_e) = 10 \log \frac{1}{(3)(4725)}(0.001243 - 0.000023) = -70.65 \text{ dB} \tag{15}$$

Analysis for Increasing Detectability of Difference in Pulverization

SN ratio:

$$\eta' = 10 \log \frac{[1/(3r)\ (S_{M\beta} - V_e)}{V_e} = 10 \log \frac{[1/(3)(4725)](0.000066 - 0.000023)}{0.000023} = -38.90 \text{ dB} \tag{16}$$

Sensitivity:

$$S = 10 \log\ (1/3r)(S_{M\beta} - V_e) = 10 \log\ [1/(3)(4725)](0.000066 - 0.000023) = -85.23 \text{ dB} \tag{17}$$

3. Optimal Configuration and Confirmatory Experiment

As control factors, we selected *A* to *G*, shown in Table 3. In addition, for two pairs of factors that were supposed to have mutual interactions: between *A* (water content in granule) and *B* (humidity of sucked air) and between *D* (suction pressure) and *F* (amount of test sample), we used the sliding-level technique.

After allocating the seven control factors in Table 3 to an L_{18} orthogonal array, we conducted an experiment. The resulting response graphs of SN ratios (η, η^*) and sensitivities (S, S^*) are shown in Figures 2 and 3. Basically, from the response graphs we selected levels with a high SN ratio as optimal. Then, for control factor *C*, mesh size, we chose level 3 (mesh size = 0.500 mm), which had the highest SN ratio. However, when we used this level, the meshes became clogged when granules were sieved. We judged, therefore, that it would be difficult to use this level in actual production because proper abrasion would be unlikely. Thus, as an optimal level, we chose level 2, which had the second-highest SN ratio. In contrast, the lowest-SN-ratio level was set as the worst. Since there was no current level because a new measuring method was being studied, each of the second level of control factor was selected as the comparative level.

Analysis for Finding Good Pulverization Conditions

Optimal configuration:	$A_1B_3C_2D_3E_1F_3G_1$
Worst configuration:	$A_3B_1C_1D_1E_3F_1G_3$
Comparative configuration:	$A_2B_2C_2D_2E_2F_2G_2$

Analysis for Increasing Detectability of Differences in Pulverization

Optimal configuration:	$A_1B_3C_2D_2E_3F_2G_1$
Worst configuration:	$A_2B_1C_1D_1E_2F_1G_3$
Comparative configuration:	$A_2B_2C_2D_2E_2F_2G_2$

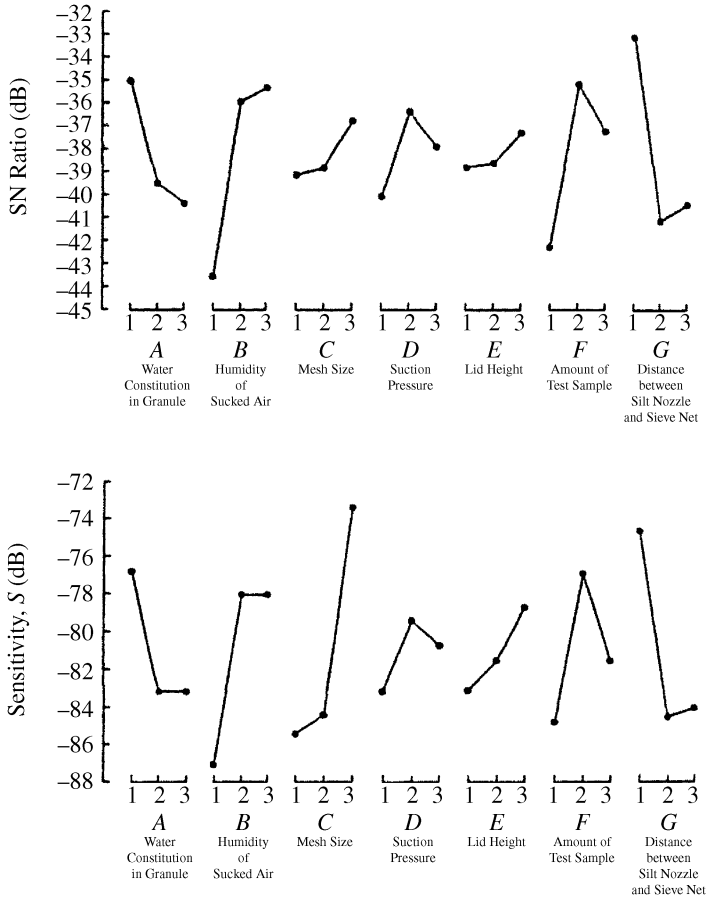

Figure 3
Response graphs for increasing detectability of differences between materials

Table 4
Confirmation of SN ratio and sensitivity (dB)

Configuration	SN Ratio		Sensitivity	
	Estimation	Confirmation	Estimation	Confirmation
Optimal	−18.38	−13.13	−63.54	−56.23
Worst	−26.79	−21.54	−73.61	−75.63
Comparative	−22.09	−16.71	−67.05	−68.38
Gain between optimal and worst	8.41	8.41	10.07	19.40
Gain between optimal and comparative	3.71	3.58	3.51	12.15

Confirmatory Experiment

To conduct a confirmatory experiment, comprehensively judging all configurations obtained from the two types of analyses, we selected the following optimal, worst, and comparative configurations:

Optimal configuration:	$A_1B_3C_2D_3E_3F_3G_1$
Worst configuration:	$A_3B_1C_1D_1E_1F_1G_3$
Comparative configuration:	$A_2B_2C_2D_2E_2F_2G_2$

As a result of the confirmatory experiment, we calculated them as shown in Table 4. Comparing the estimation and confirmation, we could see that the reproducibility in gain of the SN ratio was fairly good, whereas that of sensitivity had a disparity of 9 dB. The reason was assumed to be that the sieving of test samples had been conducted improperly as a pretreatment in the confirmatory experiment. Therefore, in the future we must sieve test samples accurately during pretreatment.

By adopting the measuring method under the optimal configuration, we could detect granular strength precisely for each prescription in parallel with maintaining the same measurement accuracy as that of the current procedure. We reduced measurement time to 1/30 that of the current level, thereby improving productivity.

Reference

Yoshiaki Ohishi, Toshihiro Ichida, and Shigetoshi Mochizuki, 1996. Optimization of the measuring method for granule fragility. *Quality Engineering,* Vol. 4, No. 5, pp. 39–46.

This case study is contributed by Yoshiaki Ohishi and Shigetoshi Mochizuki.