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Evaluation of the Strength of a Scored Oblong Tablet

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In order to evaluate the resistance of an oblong tablet with a center groove to breaking into halves, various tests for measuring the tablet breaking load were performed. The results were compared with those of a shock test which was considered to simulate the impacts occurring in the processes of packaging, handling and transport. It was found that the hardness values obtained from the Monsanto hardness tester were independent of the shock test data. This may be due to the difference in mechanism of tablet fracture between the two tests. In the bending test, all the test tablets cleanly fractured into halves. However, the values of breaking load obtained from a Kiya hardness tester were, on the whole, too small to be useful for distinguishing between tablets having different strengths. Since this result seemed to be caused by the high loading rate in this test, the influence of the loading rate was examined by the use of a material testing machine. When the rate of loading was sufficiently slow, higher breaking load values were obtained and differences in mechanical strength between the different tablets were easily detected. The flexure breaking loads obtained from the tester with a spring balance were in good agreement with the corresponding shock test data. In this test, the actual loading rate may be reduced by the buffer action of the spring, resulting in both higher breaking load values and greater apparent differences in strength between the test tablets. It is thought that this instrument is suitable as a conventional device for evaluating the fracture resistance of this type of tablet.

Keywords——scored oblong tablet; spring balance; shock test; bending test; loading rate; breaking load; Monsanto hardness; tablet strength

Compared with tablets having regular shapes, the oblong tablet with a center groove is convenient when a part of a tablet is to be dosed. On the other hand, this type of tablet is prone to breakage in the processes of packaging, handling and transport.

While there have been many reports[1-7] concerning the breaking strength of round-shaped tablets, few reports have appeared on oblong tablets,[8,9] and very little work has been done on the mechanical strength of scored tablets.

In the present investigation,compression tests and bending tests for several kinds of scored oblong tablets were undertaken and the results were compared with those of shock tests. As regards the bending test a detailed study was made on the measuring apparatus and operating conditions. It was found that the bending test with the use of a spring balance and the shock test are apparently suitable for use as conventional methods for evaluating the mechanical strength of scored oblong tablets.

Experimental

Material——Nalidixic acid (Toyo Jozo Co.), microcrystalline cellulose (Asahi Chemical Industry Co.), potato starch (Nichiden Co.), acacia(Nihon Powder Co.), polyoxyl 40 stearate (Nikko Chemicals Co.) and magnesium stearate (Taihei Chemical Co.) were all of J.P. X grade. Polyvinylpyrrolidone (PVP K-30, Mitsubishi Chemical Co.) was used as received.

Preparation of Granules——a) Wet Granulation Method: Two thousand grams of nalidixic acid (or lactose–potato starch mixture (6:4)) and 180 g of microcrystalline cellulose were blended in a twin shell type blender. The powder mixture was wetted with 30 vol % of aqueous ethanol solution containing 20 g of polyoxyl 40 stearate, 12 g of acacia and 8 g of PVP, using a Henshell type mixer (Daiwa Kakoki Co.) or a kneading type mixer (Sanei Seisakusho Co.). The wet granules were passed through an 840 μm sieve (Oscillator No. 34c-2, Kikusui Seisakusho Co.) and dried at 40°C for 10 h in a forced air oven. The dried granules were again passed through a 840 μm sieve.

b) Fluidized bed granulation method: The same solution as described for the wet granulation method was sprayed into a fluidized powder mix in a fluidized bed granulator (ST-1, Fuji Sangyo Co.) maintained at 40°C. The granules produced were passed through an 840 μm sieve. Table I lists the five preparations investigated.

TABLE I. Preparations Investigated

Sample	Main ingredient	Method of granulation: Granulator	Method of granulation: Rotational speed rpm
H	Nalidixic acid	Henschel type mixer	200
K-1	Nalidixic acid	Kneading type mixer	60
K-2	Nalidixic acid	Kneading type mixer	40
F	Nalidixic acid	Fluidized bed granulator	—
FLS	Lactose/Potato Starch (6/4)	Fluidized bed granulator	—

Preparation of Tablets——Before compression, magnesium stearate (0.9%) was added to the granules. Compression was effected by utilizing a rotary tabletting machine (RT F-9-2, Kikusui Seisakusho Co.) with 17.3×6.8 mm oblong dies and shallow concave punches. In order to prepare scored tablets, upper punches with ridges were used. In this study, the tablet weight was fixed at 560 mg and the tablet thickness was adjusted by changing the pressure settings. Figure 1 gives the shape and dimensions of the tablet.

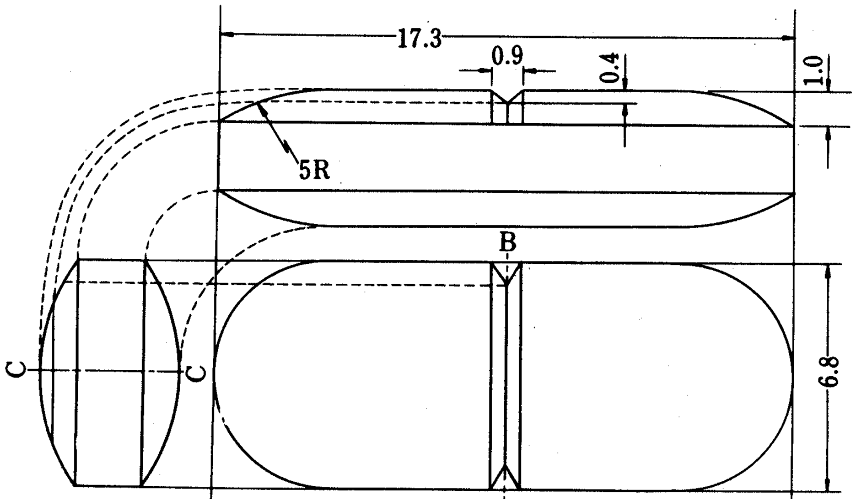

Fig. 1. Dimmensions of a Tablet in mm

Tablet Thickness——Tablet thickness was measured with a micrometer (Mitsutoyo Co.)

Shock Test——The glass tube shaking method[7] was employed for the shock test of tablets. Two tablets were placed in each of five stoppered glass tubes, 20 cm long and 2 cm in diameter.

The glass tubes were shaken at a frequency of 200 cycles per minute through a distance of 15 cm. After 5 min of operation, the number of tablets broken into halves was counted.

Tablet Strength Measurement——a) Compression Test: A Monsanto hardness tester was used to determine the tablet crushing strength. b) Bending Test: A plunger and fulcrum pieces were used for the bending test (Fig. 2). A test tablet is placed on the fulcrums and pressed with a plunger until the tablet breaks into halves. For measuring the breaking load, the following instruments were employed; (1) Kiya hardness tester (Kiya Seisakusho Co.), (2) spring balance with a sensitivity of 1.35 cm per kg and capable of taking a maximum

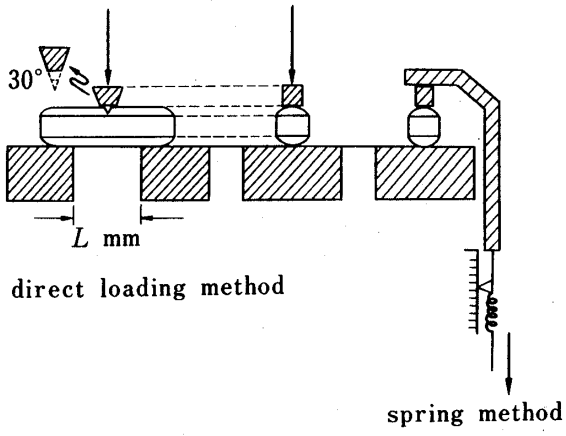

Fig. 2. Methods of Determining the Breaking Load by Bending Test

load of 5 kg (Ohoba Seisakusho Co.), (3) material testing instrument (TOM 5000, Shinko Tsushin Co.). In the former two methods, the load was applied manually. The distance between fulcrums (L) is of importance in this type of test. In order to split the test tablet cleanly into halves, it is required to satisfy the following equation.[10)]

$$L > a \sin(\varphi/2) + 2h \cos(\varphi/2)$$

where a is a major axis of the tablet (17.3 mm), h is the tablet thickness and φ is the angle of the plunger tip (30°, see Fig. 2). When h is 4.75 mm, L should be wider than 13.7 mm. Unless otherwise specified, 14 mm was used in a series of measurements.

Five replicate measurements were made for each test and the mean and standard deviation (S.D.) of the tablet breaking load were calculated.

Results and Discussion

Shock Test by the Glass Tube Shaking Method

The results of the shock test are shown in Table II (3rd column). Some of the tablets prepared from the granules made in the kneading type mixer (K-1 and K-2) fractured into halves along the groove.

Compression Test by Monsanto Hardness Tester

Tablets were compressed through the axes B-B and C-C (Fig. 1) and the breaking loads were measured. The results of the test are shown in the 4th and 5th columns of Table II. No correlation was found between the results of the shock test and the Monsanto hardness, and it

TABLE II. Results of Shock Test and Breaking Loads measured by Various Methods

Sample	Tablet thickness mm	Number of broken tablets in shock test[a)]	Breaking load kg±SD[b)] Monsanto hardness tester (Compression test) B-B axis[c)]	C-C axis[c)]	Kiya hardness tester (Bending test)[d)]	Spring balance (Bending test)[d)]
H	4.75	0	14.0±0.4	9.0±0.4	0.8±0.2	1.65±0.09
	4.65	0	16.8±0.3	10.4±0.4	0.9±0.1	1.90±0.08
	4.55	0	16.9±0.6	12.1±0.7	1.0±0.1	2.20±0.10
K-1	4.75	4	16.6±0.7	8.6±0.2	0.8±0.1	0.75±0.04
	4.65	3	18.5±0.4	9.4±0.3	0.9±0.2	0.98±0.05
	4.55	4	18.0±0.4	8.2±0.7	0.8±0.4	1.17±0.14
K-2	4.75	2	10.6±0.3	6.4±0.3	0.9±0.3	1.17±0.11
	4.65	0	10.6±0.4	7.4±0.4	0.7±0.2	1.50±0.09
	4.55	0	11.5±0.2	9.2±0.6	0.9±0.2	1.93±0.04
F	4.75	0	12.3±0.3	9.1±0.5	0.8±0.2	1.88±0.07
	4.65	0	14.1±0.4	10.2±0.6	0.7±0.1	1.92±0.09
	4.55	0	17.3±0.4	11.4±0.5	0.8±0.1	2.02±0.04
FLS	4.45	0	15.6±0.4	11.5±0.3	0.7±0.1	1.78±0.06
	4.35	0	21.0±0.7	12.3±0.5	0.7±0.2	1.96±0.07
	4.25	0	25.5±0.6	12.4±0.5	0.9±0.2	2.13±0.09

a) Results for 10 tablets.
b) Standard deviation, based on 5 determinations.
c) Direction of compression (see Fig. 1).
d) Distance between fulcrums was 14mm, and tablets were tested with the scored face down.

was apparent that the hardness obtained by this method could not be used as a measure of the ease of breakage of this type of tablet. This may be because the mechanism of tablet breakage is different from that in the shock test.

Bending Test by Direct Loading with a Plunger

In the bending test, the tablet was placed on fulcrums with the scored face down and a load was applied from above, which caused the tablet to break into halves. The breaking loads obtained from the Kiya hardness tester are given in the 6th column of Table II. The results did not appear to be related to those of the shock test.

In this test, it is probable that the breaking load depends to some extent on the loading rate. As regards the influence of loading rate on the breaking behavior of brittle materials, there are a few published studies[11-14] in field other than pharmaceutical technology. The results reported are quite varied, depending on the characteristics of the test material as well as the test conditions. Recently, Degennaro and Geiger[15] reported that, in a diametral compression test with the use of some commercially available hardness testers, the hardness increased with increasing loading rate.

In order to examine the effect of loading rate on the breaking load of oblong tablets, measurements were made of several samples by utilizing a material testing machine. In this method, the speed of the plunger was kept constant. Figure 3 shows the recorder tracings for one of the samples, FLS, and Fig.4 shows the relationship between the plunger speed and the breaking load. In general, the tablets broke at a higher load when the plunger speed was lower. The fact that the mechanical strength is higher when the loading rate is lower may presumably be explained as follows. When the load is applied at a slow speed the contact area between the tablet and the plunger may increase owing to deformation of the contact point, resulting in stress dispersion.[16] Thus, a greater load may be required to cause breakage.

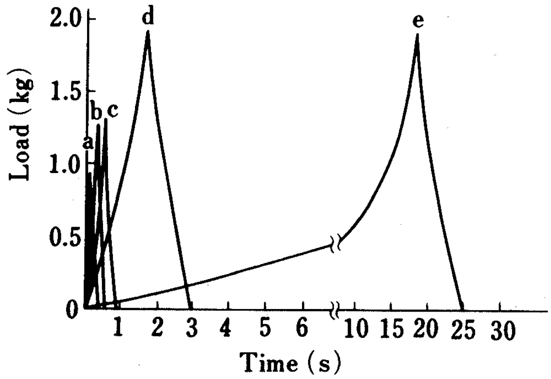

Fig. 3. Recorder Tracings for Bending Test (Direct Loading Method)

Tablet: FLS (thickness, 4.25 mm).
Plunger speed: a, 100 mm/min; b, 50 mm/min; c, 20 mm/min; d, 5 mm/min; e, 0.5 mm/min.

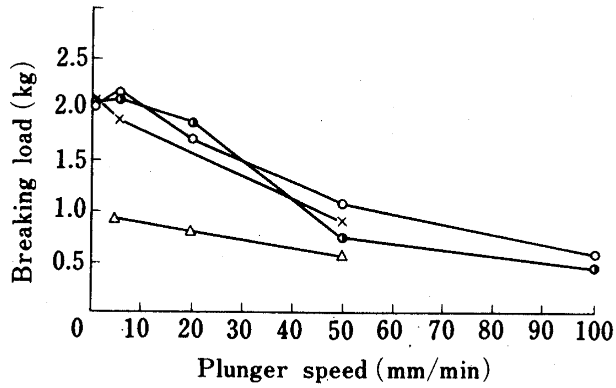

Fig. 4. Relationship between Plunger Speed and Breaking Load for Bending Test (Direct Loading Method)

×: Tablet H (thickness, 4.55 mm).
△: Tablet K-1 (thickness, 4.75 mm).
○: Tablet F (thickness, 4.55 mm).
◑: Tablet FLS (thickness, 4.25 mm).

In Fig.4, it is noteworthy that the sample K-1, which was easily split into halves by the shock test, has a mechanical strength that is less dependent on the loading rate when compared with those of other samples. These results suggest that it may be possible to distinguish the fracture resistance of oblong tablets by means of a bending test at a low loading rate. On the other hand, the plunger travel speed in the Kiya hardness tester was found to be 45 ± 5 mm/min when operated under usual conditions. Hence, the values obtained from this tester (6th column in Table II) were too small to be useful for detecting the difference in breaking strength between different tablets.

Bending Test by the Use of a Spring

Increased breaking loads were anticipated when the load was applied through a spring since relatively slow transmission of the load to the tablet could be obtained. The results obtained with a hardness tester using a spring balance are shown in the last column of Table II. The values obtained here are not only higher than those with the Kiya hardness tester, but are superior in reproducibility. It was found that the hardness values of the samples which showed marked fragility in the shock test were significantly lower than those of other samples. When this apparatus was operated in the usual manner, it took 2—2.5 s in the case of sample K-1 and 3—4 s in the case of sample H to break the tablets. Therefore, the order of the loading rate is considered to be comparable to a plunger speed of less than 5 mm/min in bending tests by direct loading. In order to examine the influence of the spring pulling rate, measurements were made using a material testing machine for the sample FLS. As shown in Fig. 5, the load increased linearly with time, *i.e.*, the load was applied at a uniform rate. The breaking load appeared to be constant within the range of pulling rate used in the present tests.

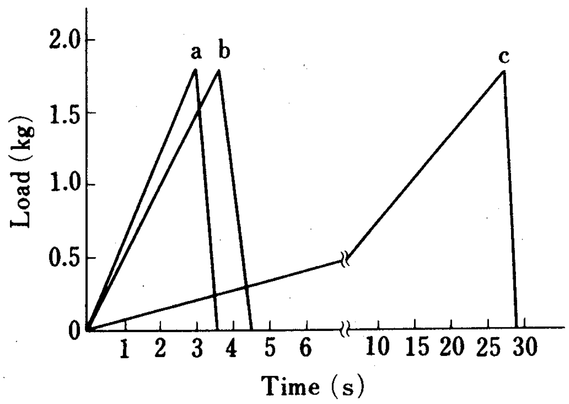

Fig. 5. Recorder Tracings for Bending Test (Spring Method)

Tablet: FLS (thickness, 4.25 mm).
Pulling speed: a, 500 mm/min; b, 100 mm/min; c, 50 mm/min.

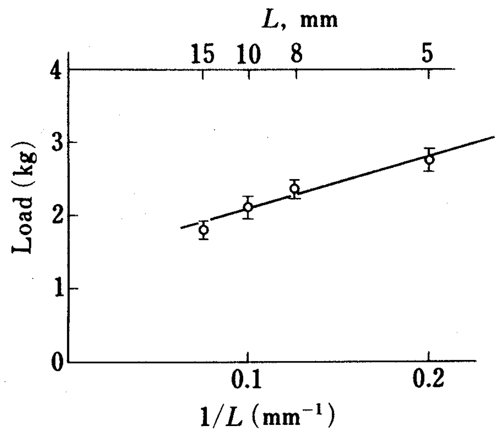

Fig. 6. Plot of Breaking Load (Spring Balance Method) against Reciprocal of Distance between Fulcrums

Effect of Distance between Fulcrums on Breaking Load

The effect of the distance between fulcrums on the breaking load was examined by the spring balance method using FLS as the test sample. The breaking load (W, kg) decreases with increasing distance between fulcrums (L, mm), and a plot of W against $1/L$ was linear, as shown in Fig. 6.

TABLE III. Effect of the Position of Scored Face on the Breaking Load measured by the Spring Balance Method for Sample FLS (Tablet Thickness : 4.45 mm)

Distance between fulcrums mm	Breaking load, kg±S.D.[a]		
	Up[b]	Down[b]	t-Statistics[c]
10	2.40±0.18	2.10±0.13	4.273[d]
14	2.13±0.26	1.78±0.10	3.973[d]

a) Standard deviation, based on 10 determinations.
b) Position of scored face.
c) Student's t-test.
d) Difference significant at 0.1 % level.

For a beam of rectangular cross-section subjected to bending, it can be shown theoretically that the bending load is inversely proportional to the distance between fulcrums.[3,9] For the present data, the straight line in Fig. 6 does not pass through the origin. Thus, the following empirical equation is applicable.

$$W=7.12\ (1/L)+1.37 \qquad r=0.984$$

Effect of Groove Position of Breaking Load in the Spring Balance Method

Higher values of breaking load were obtained when the tablet was tested with the scored face up than when it was tested with the scored face down (Table III). However, the tablet with the scored face down showed a cleaner breakage into halves.

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