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RATE OF WEAR OF PHARMACEUTICAL TABLETS

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ABSTRACT

Several methods are presented to quantitatively measure the wear of compressed tablets. The natural logarithm of the weight fraction, W/W_o, decreases linearly with increasing wear time. Thus, the wearability of a tablet may be described by a first-order rate constant or its half life. As the wear process continues there may be more than a single rate constant depending on the geometry and structural homogeneity of the tablet.

INTRODUCTION

Tablets must possess sufficient strength to withstand processing (coating, pneumatic conveyance and packaging) and subsequent wear in a partially filled container as the consumer uses the medication. Mechanical strength has been measured by various methods such as tensile (1, 2) hardness (3, 4), impact-wear (5), fatigue creep (6), flexal (7), impact-rebound (8) and friability (9) tests. The data from these tests can seldom be correlated as mechanical strength is the response to stress, which is dependent on the test method.

The wear process is a gradual loss of material from the surface of a tablet caused by a continuous stress of a frictional and/or abrasive nature. As wear proceeds the weight of the tablet decreases. The purpose of this study was to design and compare several wear tests and to attempt to express wear in terms of a rate constant.

EXPERIMENTAL

Preparation of Tablets. Tablets of direct compression excipients were prepared using 80/100-mesh size fractions of Emcompress™ (Dibasic Calcium Phosphate, USP, Edward Mendell Co., Inc. Carmel, NY 10512), Fast-Flo™ lactose (Lactose, NF, Foremost, Bloomington, MN 55420), Anhydrous Lactose for Direct Tableting (Lactose, NF, Sheffield Products, Norwich, NY 13815), and Di-Pac™ (Compressible Sugar, NF, Amstar, New York, NY 10020) and 45/60-mesh size fraction of Compactrol™ (Calcium Sulfate Dihydrate, NF, Edward Mendell Co., Inc.). Approximately 0.8 g of material was weighed on an analytical balance and compressed in a Carver hydraulic press for 15 seconds. A set of flat-faced punches and die with a diameter of 1.275 cm was used. The die wall was lubricated prior to each compression with a slurry of 5% magnesium stearate in ethanol.

Measurement of Hardness and Tensile Strength. The hardness of the tablets was measured after 48 hours had elapsed by a Schleuniger 2E hardness tester. The radial tensile strength of the tablets was determined by the Hounsfield tensiometer (2).

Wear Tests. The rotating wear apparatus consists of a stainless steel cylinder that is 6.2 cm in diameter and 10.7 cm in height mounted centrally to a shaft connected to a variable speed motor and rotated end-over-end at 40 rpm. Twenty tablets were placed in the cylinder. The tablets were weighed at various intervals after dedusting by a stream of air. Initially the procedure was repeated until 50% of the original weight was worn or for 1000 minutes.

The shaking wear apparatus consisted of a 20-mesh sieve on which the tablets were localized by a cylindrical plastic cover that was 8.0 cm in diameter and 2.0 cm in height. The sieve was placed on a Tyler sieve shaker and by means of a sieve cover the plastic cover was fastened to the center of the sieve. After shaking the tablets were removed and weighed at various intervals.

The fluidizing wear apparatus consisted of a vertically mounted glass cylinder 3.5 cm in diameter and 40 cm in height. The tablets were fluidized by compressed air at 10 psi. Tablets were removed and weighed at various intervals.

RESULTS AND DISCUSSION

As the wear tests proceeded the tablets were weighed at various intervals of time, and the weight fraction, W/W_o, of the worn tablets was calculated. The weight fraction decreased with the passage of time. First-order mathematics may be applied to the interpretation of the wear process of compressed tablets. The first-order wear rate may not be the same for the entire tablet because of geometric and structural characteristics of the tablet matrix. To analyze and interpret the wear process a plot of ln W/W_o against time was analyzed by piecewise linear regression based on least square method resulting in several linear segments. The transition point between each linear segment of the plot was determined by applying the likelihood ratio tests to the two adjacent regression lines (10). Each segment reflects a difference in the geometry or structure of the tablet. The first-order wear rate constant of the *i*-th linear period of wear is designated as k_i, and is evaluated from the slope of the plot of ln W/W_o against time. The wear half life, $t_{\frac{1}{2}}$, was determined from the linear equation of the regression line of the major first-order wear period which passed the 50% wear point.

Rotating Wear Test. For the rotating wear test a plot of ln W/W_o against wear time for flat-faced tablets of Emcompress compressed at 390 kg/cm^2 is shown in Figure 1a. Initially for a small portion (2-3%) of the flat-faced tablet there is a rapid wear of the sharp edges (see Table 1). After the sharp edges have been worn, there follow one or several periods of first-

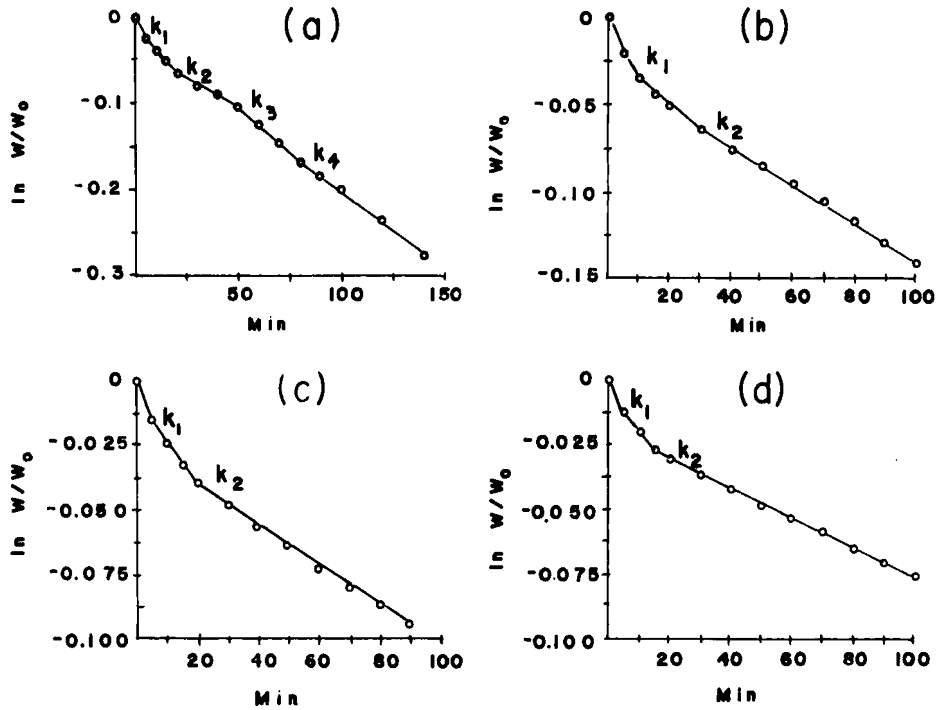

FIGURE 1

Relationship of Natural Logarithm of Weight Fraction Remaining to Wear time of Rotating Wear Test of Tablets of Emcompress Compressed at (a) 390; (b) 785; (c) 1175; and 1565 kg/cm^2.

order kinetics depending on the geometry and homogeneity of the tablets.

In Figure 1a four first-order wear rate constants (k_1 = 24.69 X 10^{-4}, k_2 = 13.16 X 10^{-4}, k_3 = 21.31 X 10^{-4} and k_4 = 17.30 X 10^{-4} min^{-1}) were identified. The constants k_1, k_2 and k_3 reflect the wear rounding process, and k_4 describes the major first-order wear process. Usually k_{i+1} is smaller than k_i because as demonstrated by Train (11) the core of a compressed tablet is stronger (has a greater density due to differences in the transmission of compressional force) than the peripheral regions. Visually the lower surface of a flat-faced tablet wore faster than the upper

TABLE 1
Wear Characteristics of Flat-Faced Tablets Compressed at Various Applied Pressures as Measured by Rotating Wear Test

Applied Pressure, kg/cm^2	Wear Time, min	Percent Remaining	r^2	Wear Process	10^4 Wear Rate Constant, min^{-1}	Half Life, min
Emcompress						
390	5-20	97.4-93.8	0.997	k_1	24.69	
	20-50	93.8-90.1	0.997	k_2	13.16	
	50-80	90.1-84.5	0.999	k_3	21.31	
	80-380	84.5-50.3	1.000	k_4	17.30	385
785	10-30	96.6-93.8	0.994	k_1	14.42	
	30-680	93.8-49.0	1.000	k_2	9.94	660
1175	5-20	98.5-96.1	0.997	k_1	16.45	
	20-1000	96.1-50.2	1.000	k_2	6.565	1006
1565	5-15	98.8-97.4	0.994	k_1	14.27	
	15-1000	97.4-59.8	0.999	k_2	4.89	1360
Compactrol						
390	4-20	95.3-83.1	0.998	k_1	83.58	94
	20-80	83.1-55.1	1.000	k_2	68.80	
785	4-10	97.4-95.0	1.000	k_1	41.58	
	12-18	94.4-92.6	1.000	k_2	32.09	
	18-300	92.6-50.7	0.999	k_3	21.96	298
1175	10-20	97.1-95.6	0.999	k_1	15.57	
	20-800	95.6-50.8	1.000	k_2	8.035	817
1565	10-20	97.4-96.1	0.998	k_1	13.44	
	20-70	95.1-91.4	0.994	k_2	7.26	
	70-300	91.4-80.8	0.999	k_3	5.77	
	300-1000	80.8-61.4	0.999	k_4	3.845	1534

surface. After the wear had rounded the tablet, weight was lost mainly from the side of the tablet in the major first-order wear period. The thickness at the center of the tablet did not decrease during the testing period. In the rotating wear test of flat-faced tablets of Emcompress compressed at 785, 1175 and 1565 kg/cm^2 only two constants were observed; k_1 represents the wear rounding process and k_2 represents the major first-order wear process.

Similar data in Table 1 was obtained for flat-faced tablets of Compactrol compressed at various applied pressures. The tablets compressed at 390 kg/cm^2 were too soft for their hardness to be measured. When applied to these soft tablets the rotating wear test provided two first-order wear periods. When the applied pressure was increased to 1565 kg/cm^2, four first-order wear rate constants were seen.

As the applied pressure is increased, the portion of the weak edges is reduced. For example with flat-faced tablets of Emcompress compressed at 390, 785, 1175 and 1565 kg/cm^2, the major first-order wear began after 15.5, 6.2, 3.9 and 2.6%, respectively, of the tablet has been worn. As the applied pressure is increased, the wear rate constant is decreased due to the greater strength. As seen in Table 1 for Emcompress as the applied pressure is increased from 390, 785, 1175 and 1565 kg/cm^2 the first major-order rate constants are 17.30×10^{-4}, 9.937×10^{-4}, $6.565\ 10^{-4}$, and $4.892 \times 10^{-4} min^{-1}$, respectively, and the wear half lives are 385, 660, 1006 and 1360 minutes, respectively.

The relationship between the logarithm of the major first-order rate constant and applied pressure, hardness and radial tensile strength is shown in Figure 2. Similar results were obtained for tablets of Compactrol.

<u>Shaking Wear Test</u>. Plots of ln W/W_o against shaking wear time of flat-faced tablets of Emcompress, Compactrol and Fast-Flo

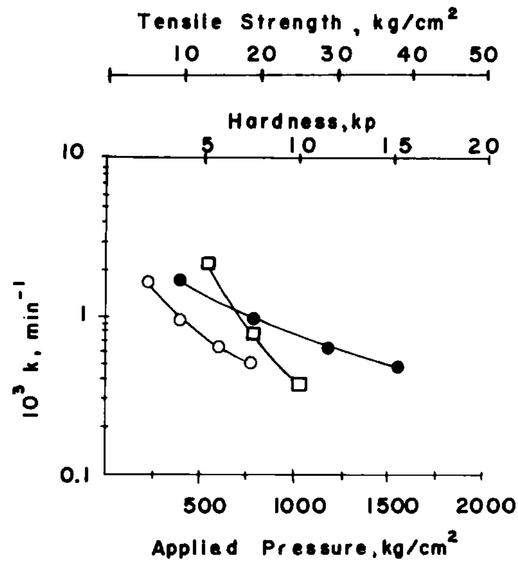

FIGURE 2

Relationship of Logarithm of Major Wear Rate Constant of Rotating Wear Test of Emcompress Tablets to Applied Pressure (●); Hardness (□); and Tensile Strength (○).

lactose were drawn, and the first-order shaking wear rate constants and wear half-lives shown in Table 2 were determined. The shaking wear test provided the tablets with sufficient attrition against the sieve so that the upper and lower surface were worn, and the thickness of the tablets decreased during testing. Tablets wore faster in the shaking wear test than in the rotating test as the movement was more vigorous and the sieve provided greater attrition.

For flat-faced tablets of Compactrol compressed at 390, 785, 1175 and 1565 kg/cm^2 the major first-order wear started after 16.9, 8.4, 7.8 and 7.4%, respectively, was worn. The relationship between the logarithm of the major first-order shaking wear rate constant and the applied pressure, hardness and radial tensile strength of flat-faced tablets of Compactrol is shown in

TABLE 2

Wear Characteristics of Flat-Faced Tablets Compressed at Various Applied Pressures as Measured by Shaking Wear Test

Applied Pressure, kg/cm^2	Wear Time, min	Percent Remaining	r^2	Wear Process	10^4 Wear Rate Constant, min^{-1}	Half Life, min
			Emcompress			
390	2-95	96.9-49.2	0.999	k_1	72.24	92
785	2-25	98.3-90.6	0.999	k_1	34.81	
	25-240	90.6-49.8	1.000	k_2	28.06	236
1175	5-70	98.0-87.6	0.998	k_1	17.03	
	70-460	87.6-48.9	0.999	k_2	14.77	453
1565	5-30	98.4-94.8	0.998	k_1	14.64	
	30-280	94.8-72.5	0.998	k_2	10.73	
	280-500	72.5-60.2	0.998	k_3	8.39	723
			Compactrol			
390	4-25	95.6-83.1	0.999	k_1	66.93	
	30-120	81.0-49.5	0.998	k_2	56.09	115
785	4-15	96.3-91.6	0.997	k_1	45.47	
	15-250	91.6-48.4	0.999	k_2	26.03	241
1175	8-25	95.5-92.2	0.999	k_1	20.83	
	25-120	92.2-81.5	0.998	k_2	12.99	
	120-500	81.5-55.9	1.000	k_3	9.93	604
1565	15-30	94.6-92.6	1.000	k_1	14.20	
	30-220	92.6-76.8	0.999	k_2	9.70	
	220-500	76.8-61.7	0.999	k_3	7.83	766
			Fast-Flo Lactose			
390	0-110	100-50.2	1.000	k_1	62.72	111
785	0-130	100-74.4	0.999	k_1	22.25	
	130-340	74.4-49.8	1.000	k_2	18.96	337
1175	0-100	100-89.8	0.999	k_1	10.61	
	100-500	89.9-69.3	0.999	k_2	6.54	991
1565	5-40	99.5-97.6	0.996	k_1	5.37	
	40-500	97.6-82.0	1.000	k_2	3.75	1821

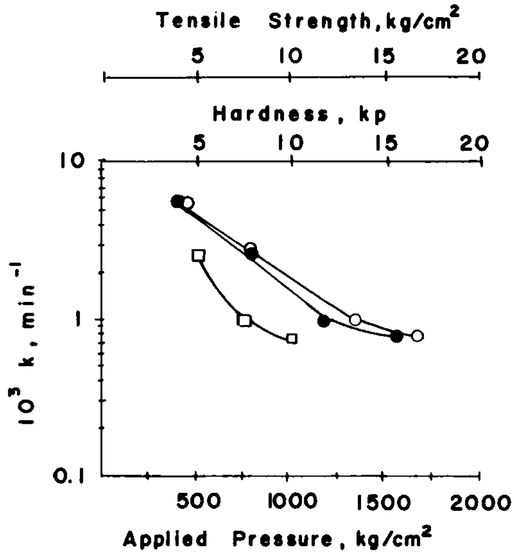

FIGURE 3

Relationship of Logarithm of Major Wear Rate Constant of Shaking Wear Test of Compactrol Tablets to Applied Pressure (●); Hardness (□); and Tensile Strength (○).

Figure 3. There was a similar relationship for tablets of Emcompress and Fast-Flo lactose.

Fluidizing Wear Test. Plots of ln W/W_o against fluidized wear time of flat-faced tablets of Emcompress, Anhydrous Lactose DT and Di-Pac were drawn, and the first-order fluidizing wear rate constants and wear half-lives shown in Table 3 were determined. The first period of fluidizing wear is relatively slow compared to the following period and is not a first-order process. Initially the tablets did not move rapidly, but as wear progressed and the weight of the tablets decreased, the tablets moved more vigorously resulting in a faster wear.

Tablets wear rapidly in the fluidizing wear test, and the wear process is less well defined than with the other wear tests studied, because the sharp edges rounded so rapidly that the

TABLE 3
Wear Characteristics of Flat-Faced Tablets Compressed at Various Applied Pressures as Measured by Fluidizing Wear Test

Applied Pressure, kg/cm^2	Wear Time, min	Percent Remaining	r^2	Wear Rate Constant[a], min^{-1}	Half Life, min
		Emcompress			
390	6-9	76.7-43.6	0.997	0.188	8.3
785	12-18	72.2-48.4	0.999	0.066	17.6
1175	18-30	76.4-46.1	0.997	0.041	28.4
1565	26-42	71.6-48.8	0.998	0.024	41.4
		Anhydrous Lactose DT			
155	9-20	90.0-49.2	1.000	0.055	19.8
390	14-45	90.2-48.6	1.000	0.020	43.5
785	20-70	93.3-49.0	0.999	0.013	68.5
1175	40-100	85.9-48.6	1.000	0.0095	97.0
		Di-Pac			
155	10-16	82.1-47.8	0.999	0.091	15.5
390	20-30	75.9-49.7	0.999	0.042	30.0
785	25-50	85.9-49.4	1.000	0.022	49.6
1175	35-75	86.0-49.7	1.000	0.0014	74.6

[a] major first-order wear period

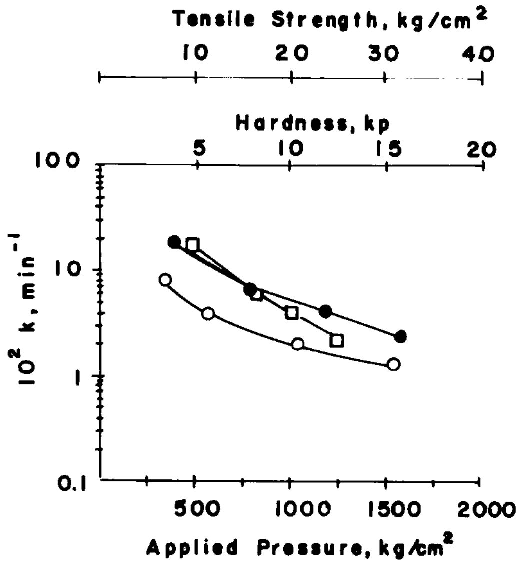

FIGURE 4

Relationship of Logarithm of Major Wear Rate Constant of Fluidizing Wear Test of Emcompress Tablets to Applied Pressure (●); Hardness (□); and Tensile Strength (○).

weight change could not be conveniently followed. The fluidizing wear test was the most time saving; however, it was not satisfactory as weak tablets would break, and it did not characterize the wear process as well as the other methods. The relationship between the logarithm of the major first-order fluidizing wear rate constant and applied pressure, hardness and radial tensile strength is shown in Figure 4.

CONCLUSION

Rotating, shaking and fluidized wear tests were described and employed to measure the wearability of tablets of several direct compression tablet excipients compressed at various applied pressures. Wear rate constants were calculated. A typical comparison of the wear rate constants to hardness, tensile strength and friability is shown for Emcompress in Table 4.

Table 4
Comparison of Mechanical Characteristics of Tablets of Emcompress Compressed at Various Applied Pressures as Measured by Various Tests

Characteristics	Applied Pressure, kg/cm^2			
	390	785	1175	1565
Hardness, kp	4.7	7.9	10.1	12.6
Tensile Strength, kg/cm^2	5.2	9.6	14.6	19.8
Friability, %	2.8	2.2	1.5	1.2
Rotating Wear Test				
$10^4 k^*$, min^{-1}	17.30	9.94	6.56	4.89
$t_{1/2}$, min	385	660	1006	1360
Shaking Wear Test				
$10^4 k^*$, min^{-1}	72.24	28.06	14.77	8.39
$t_{1/2}$, min	92	236	453	723
Fluidizing Wear Test				
k^*, min^{-1}	0.188	0.066	0.041	0.024
$t_{1/2}$, min	8.3	17.6	28.4	41.4

*Major first-order wear rate constant

Hardness and radial tensile strength express tablet strength as a resistance to failure under diametrically applied compressive force. Friability is a single period determination (4 minutes). The rotating wear test expresses the wearability of a tablet and may be used to interpret the tablet strength in terms of its geometry and structural homogeneity. The shaking wear test can be used to interpret the tablet structure emphasizing the wear on the upper and lower faces. The fluidizing wear test measures the wearability of a tablet but provides limited information of the tablet structure.

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