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EFFECT OF A LUBRICANT ON WEAR RATE OF TABLETS

Ruey-ching Hwang and Eugene L. Parrott

Division of Pharmaceutics
College of Pharmacy, University of Iowa
Iowa city, Iowa 52242

ABSTRACT

The rate of wear and hardness of tablets of Emcompress and sodium chloride compressed at various applied pressures in a lubricated and an unlubricated die are compared. The effect of applied pressure and concentration of magnesium stearate blended with several direct compression excipients on the wear rate constant, hardness and tensile strength of a tablet is reported. The data allow a comparison of the methods used to express the mechanical strength of tablets.

INTRODUCTION

Studies (1-4) of the effect of lubricants on the strength of tablets have reported a decrease in crushing strength to a constant value, no change of strength, and an increase in strength as the concentration of lubricant is increased. Juslin and Krogerus (5) found that in general increasing the quantity of lubricant results in a reduction of tablet strength. It appears that the effect of a lubricant may depend on the material and the bonding mechanism.

In this investigation the effect of a lubricated and an unlubricated die on the structural strength of a tablet was studied. The prime purpose of this study was to measure the

effect of applied pressure and concentration of magnesium stearate on the structural strength of tablets of several common direct compression excipients. The wear rate constant, hardness, and tensile strength are used as expressions of mechanical strength to allow a comparison of methodologies employed to quantize the resistance of a tablet to processing and handling.

EXPERIMENTAL

The preparation of flatface tablets of Emcompress™ (Dibasic Calcium Phosphate Dihydrate, USP, Edward Mendell Co., Inc., Carmel, NY 10412), Sodium Chloride, USP (Mallinckrodt, St. Louis, MO 63147), Compactrol™ (Calcium Sulfate Dihydrate, NF, Edward Mendell Co., Inc.), Fast-Flo™ lactose (Lactose, USP, Foremost, Bloomington, MN 55420), Anhydrous Lactose for Direct Tableting (Lactose, USP, Sheffield Products, Norwich, NY 13815) and Di-Pac™ (Compressible Sugar, NF, Amstar Co., New York, NY 10020) having a diameter of 1.275 cm has been described (6). Hardness was measured by a Schleuniger 2E hardness tester. If lubricated, the die wall was lubricated by applying a 5% slurry of Magnesium Stearate, NF (Fisher Scientific, Pittsburgh, PA 15219) in ethanol. For blends magnesium stearate that had passed an 80-mesh sieve was blended for 15 minutes in a V-blender with the direct compression excipient. Wearability expressed as a first-order wear rate constant and half life was determined using the rotating wear test method previously described (6).

RESULTS AND DISCUSSION

Friction during compression affects the structural homogeneity of a compressed tablet. Train (7) has shown that the incorporation of lubricants enhances the structural homogeneity of a compact. Lubricants reduce interparticular friction and the friction between the die wall and the material. Although lubricants enhance the structural homogeneity of a compressed tablet, they may reduce the mechanical strength of a compressed tablet by interfering with the bonding between particles.

TABLE 1

Influence of Die Wall Friction on Hardness (kp) of a Tablet

Die Wall	Applied Pressure, kg/cm^2			
	390	785	1175	1565
Emcompress				
Unlubricated	3.0	3.2	3.7	4.5
Lubricated	4.7	7.9	10.0	12.6
Ratio lubricated to unlubricated	1.57	2.44	2.70	2.80
Sodium Chloride				
Unlubricated	5.4	9.6	11.3	14.2
Lubricated	7.9	13.1	16.5	18.3
Ratio lubricated to unlubricated	1.47	1.37	1.47	

Effect of Lubricant on the Die Wall. As models for a brittle fracture material and a plastic material Emcompress and sodium chloride, respectively, were selected. The effect of a lubricated die wall on the hardness of a tablet is shown in Table 1. With no lubricant on the die wall the friction between the material and the die wall expends energy and less pressure is transmitted and utilized to consolidate the tablet. When the die wall is lubricated, friction is reduced, and the greater transmitted pressure consolidates a harder tablet. Die wall friction reduces the hardness of tablets of Emcompress to a greater extent than tablets of sodium chloride. As shown in Table 1 as the applied pressure is increased, the ratio of the hardness of a tablet compressed in a lubricated die to a tablet compressed in an unlubricated die is increased, i.e., the increased applied pressure tends to compensate for the loss of transmitted pressure

due to friction. For sodium chloride the ratio is essentially unchanged presumably due to its plastic behavior.

At various applied pressures the natural logarithm of weight fraction, W/W_o, of tablets plotted against wear time demonstrated a first-order process, and the wear rate constant was evaluated from the slope (6). The wear rate constants of tablets compressed in a lubricated and an unlubricated die are compared in Table 2. At applied pressures of 390, 785, 1175 and 1565 kg/cm^2, the major first-order wear rate constants of tablets of Emcompress compressed in an lubricated die are smaller (k_{lub}/k_{unlub} is 0.36, 0.41, 0.48 and 0.55, respectively) than those compressed in a unlubricated die; and the half lives of tablets of Emcompress compressed in a lubricated die are longer ($t_{\frac{1}{2},lub}/t_{\frac{1}{2},unlub}$ is 2.9, 2.2, 2.0 and 1.8, respectively).

At applied pressures of 390, 785, 1175 and 1565 kg/cm^2, the major first-order wear constants for tablets of sodium chloride compressed in an unlubricated die were approximately 1.2 that of tablets compressed in a lubricated die; and the wear half lives of tablets compressed in an unlubricated die were approximately 0.8 that of tablets compressed in a lubricated die.

For Emcompress and sodium chloride tablets as the applied pressure is increased, the rate of wear is slower for tablets compressed in a lubricated and an unlubricated die as shown in Figure 1. For tablets of Emcompress k_{lub}/k_{unlub} is increased as the applied pressure is increased; however, for sodium chloride the ratio is not changed significantly by increased pressure. The relationship of wear rate constant to hardness is shown in Figure 2 for tablets compressed in a lubricated and an unlubricated die.

Effect of Lubricant Blended in Formulation. Emcompress, Compactrol, Fast-Flo lactose, Anhydrous Lactose for Direct Tableting, and Di-Pac were chosen for investigation because they are widely used direct compression excipients. For these

TABLE 2

Wear Rate Constants* and Wear Half-Lives of Tablets Compressed at Various Applied Pressures in a Lubricated and an Unlubricated Die

Die Wall	Applied Pressure, kg/cm^2			
	390	785	1175	1565
Emcompress				
Unlubricated				
$10^3 k_1$, min^{-1}	8.356	1.737	0.937	0.720
$10^3 k_2$, min^{-1}	4.800	2.300	1.355	0.895
$t_{1/2}$, min	132	301	538	773
Lubricated				
$10^3 k_1$, min^{-1}	2.460	1.442	1.645	1.427
$10^3 k_2$, min^{-1}	1.316	0.994	0.656	0.489
$10^3 k_3$, min^{-1}	2.131			
$10^3 k_4$, min^{-1}	1.730			
$t_{1/2}$, min	385	660	1006	1360
k_{lub}/k_{unlub}	0.36	0.43	0.48	0.55
Sodium Chloride				
Unlubricated				
$10^3 k_1$, min^{-1}	9.668	4.469	2.693	1.791
$10^3 k_2$, min^{-1}	4.840	2.458	1.865	1.301
$t_{1/2}$, min	112	254	350	476
Lubricated				
$10^3 k_1$, min^{-1}	5.753	2.856	2.139	1.705
$10^3 k_2$, min^{-1}	4.485	2.039	1.567	1.131
$t_{1/2}$, min	150	329	434	599
k_{lub}/k_{unlub}	0.93	0.83	0.84	0.87

*k_2 is the major first-order wear constant except for k_4 for Emcompressed compressed at 390 kg/cm^2

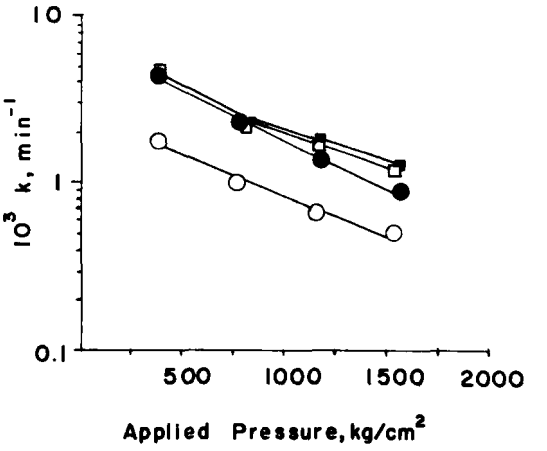

FIGURE 1

The relationship of applied pressure to wear rate constant of tablets. Key: Emcompress, lubricated (◯) and unlubricated (●) die; and sodium chloride, lubricated (□) and unlubricated (■) die.

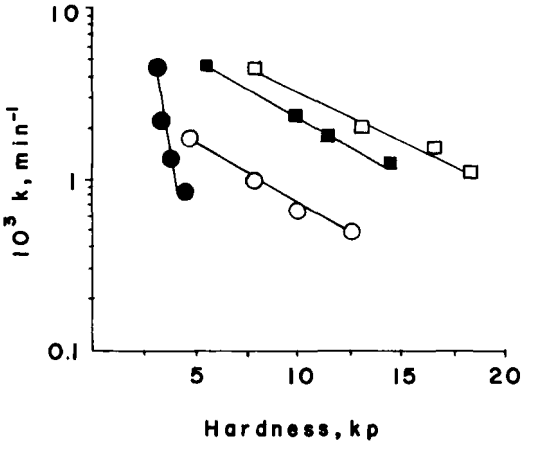

FIGURE 2

The relationship of wear rate constant to hardness. Key: Emcompress, lubricated (◯) and unlubricated (●) die; and sodium chloride, lubricated (□) and unlubricated (■) die.

TABLE 3

Influence of 1% Magnesium Stearate on Major Wear Constant, Hardness and Tensile Strength

Characteristics	Applied Pressure, kg/cm^2					
	155	390	785	1175	1565	1960
Emcompress						
No lubricant						
Hardness, kp	-	4.7	7.9	10.0	12.6	13.1
Tensile strength, kg/cm^2	-	5.2	9.6	14.6	19.8	26.1
$10^3 k$, min^{-1}	-	1.730	0.994	0.656	0.489	-
Lubricant						
Hardness, kp	-	4.3	6.0	8.3	10.1	11.2
Tensile strength, kg/cm^2	-	5.2	9.0	12.9	16.0	20.4
$10^3 k$, min^{-1}	-	2.948	1.258	0.937	0.489	-
k_{lub}/k_{unlub}	-	1.70	1.27	0.70	1.00	-
Compactrol						
No lubricant						
Hardness, kp	-	-	5.2	7.6	10.1	12.7
Tensile strength, kg/cm^2		3.7	8.0	13.6	16.8	23.0
$10^3 k$, min^{-1}	-	6.89	2.196	0.803	0.385	-
Lubricant						
Hardness, kp	-	3.3	5.9	8.5	10.8	12.8
Tensile strength, kg/cm^2	-	4.2	8.9	13.7	17.5	23.0
$10^3 k$, min^{-1}	-	4.150	2.410	1.538	1.203	-
k_{lub}/k_{unlub}	-	0.60	1.09	1.91	3.12	-

excipients compressed at various applied pressures into tablets containing no lubricant and containing 1% magnesium stearate blended with the granules the hardness and tensile strength are compared in Table 3 and 4. As the applied pressure is increased, the hardness and tensile strength are increased as shown in Figure 3.

TABLE 4

Influence of 1% Magnesium Stearate on Hardness and Tensile Strength

Fast-Flo Lactose						
No lubricant						
Hardness, kp	-	5.0	8.3	10.6	12.8	13.9
Tensile strength, kg/cm^2	-	4.1	7.7	12.5	17.6	23.5
Lubricant						
Hardness, kp	-	1.7	4.9	6.8	8.5	11.2
Tensile strength, kg/cm^2	-	2.4	4.7	8.8	16.1	19.3
Anhydrous Lactose for Direct Tableting						
No lubricant						
Hardness, kp	6.4	10.4	15.2	19.2	-	-
Tensile strength, kg/cm^2	6.8	11.1	20.9	31.2	42.3	-
Lubricant						
Hardness, kp	6.6	9.3	15.1	18.4	-	-
Tensile strength, kg/cm^2	8.4	11.5	22.6	31.3	41.0	-
Di-Pac						
No lubricant						
Hardness, kp	4.4	8.4	13.4	16.8	-	-
Tensile strength, kg/cm^2	4.0	7.7	16.1	22.5	-	-
Lubricant						
Hardness, kp	4.1	8.0	12.2	16.3	-	-
Tensile strength, kg/cm^2	4.0	7.8	15.4	20.7	28.4	-

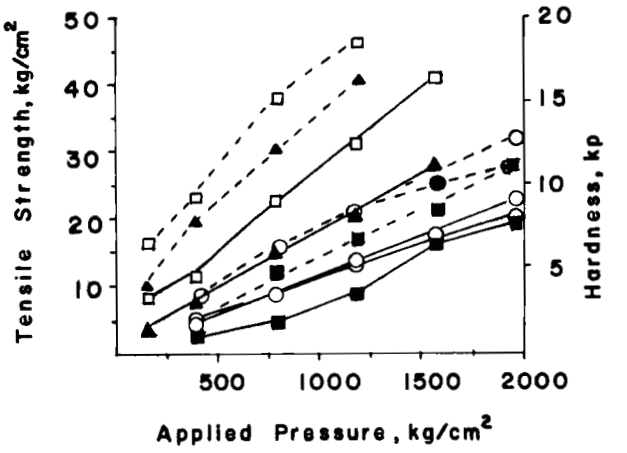

FIGURE 3

The relationship of applied pressure to tensile strength and hardness. Key: tensile strength (———); hardness (----); Emcompress (●); Compactrol (○); Fast-Flo Lactose (■); Anhydrous Lactose for Direct Tableting (□); and Di-Pac (▲).

Usually the incorporation of magnesium stearate reduced the hardness and tensile strength of the tablet; however, the hardness and tensile strength of tablets of Compactrol were slightly increased by the incorporation of 1% magnesium stearate. For anhydrous lactose containing magnesium stearate the decrease of thickness with increased applied pressure was more rapid than the change of crushing strength under diametral compression with increased pressure, and consequently the radial tensile strength was increased by the addition of 1% magnesium stearate.

The $\ln W/W_o$ of tablets of Emcompress and Compactrol containing 1% magnesium stearate was plotted against wear time in the rotating wear test, and the first-order wear rate constants were determined from the slope and are given in Table 3.

With the incorporation of magnesium stearate there is a more rapid wear of the sharp edge of the Emcompress tablet. The tablet containing the lubricant is more homogeneous than the unlubricated tablet as there is only one wear rate constant for tablets compressed at pressures from 390-1175 kg/cm^2. The addition of magnesium stearate to Emcompress increases the magnitude of the wear rate constant, and the ratio k_{lub}/k_{unlub} varies from 1.70, 1.27, 0.70 and 1.00 for applied pressures of 390, 785, 1175 and 1565 kg/cm^2, respectively.

Similarly the sharp edge of a Compactrol tablet containing magnesium stearate wore more rapidly than an unlubricated tablet. For tablets of Compactrol containing 1% magnesium stearate compressed at 390, 785, 1175 and 1565 kg/cm^2, the major first-order wear rate constant occurred when 17.6, 27.0, 27.1 and 32.1%, respectively, of the tablet was worn. This indicates that the portion of the tablet constituting the weak edge is increased by the addition of magnesium stearate, and the effect becomes more pronounced as the applied pressure was increased. Hence the addition of magnesium stearate produces a tablet that wears more rapidly than an unlubricated tablet.

Effect of Concentration of Lubricant. Emcompress compressed at 390 kg/cm^2 was used to study the effect of concentration of magnesium stearate from 0.5-10% on the wearability, hardness and tensile strength of the tablets. The hardness and tensile strength were essentially unchanged from 0.5-2% magnesium stearate, but as the concentration increased from 3-10%, the hardness and tensile strength increased as shown in Figure 4.

The ln W/W_o of tablets of Emcompress containing from 0.5-10% magnesium stearate was plotted against wear time, and the first-order wear rate constant was determined from the slope. The addition of 0.5-1.5% magnesium stearate increased the structural homogeneity of the tablets as only one wear rate constant was detected in contrast to three wear rate constants

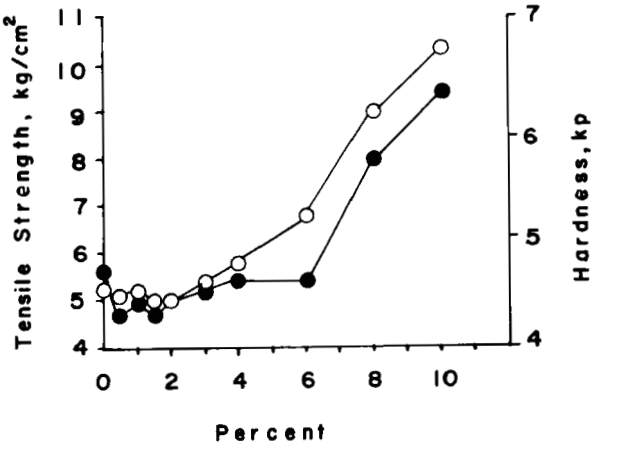

FIGURE 4

The influence of concentration of magnesium stearate on the hardness and tensile strength of tablets of Emcompress. Key: hardness (●); and tensile strength (○).

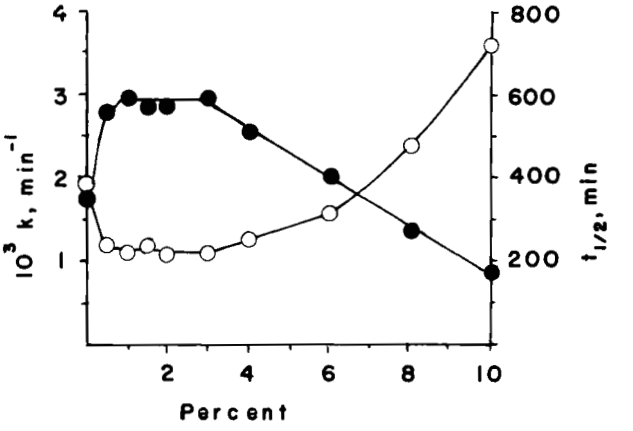

FIGURE 5

The influence of concentration of magnesium stearate on the wear rate constant and half life of tablets of Emcompress. Key: wear rate constant (●); and half life (○).

shown by unlubricated tablets. For concentrations from 2-10% magnesium stearate a wear rate constant corresponding to the rounding process and a major wear rate constant were observed. The influence of concentration of magnesium stearate on the wearability of the tablets is shown in Figure 5 in terms of the wear rate constant and the time, $t_{\frac{1}{2}}$, required to wear the tablet to 50% of its initial weight. At concentrations exceeding 6% the tablets are more resistant to wear. Magnesium stearate melts at 88°C. According to the Skotnicky equation (8) the pressure at the points of contact will lower the melting point, and when the pressure is relieved, the magnesium stearate will congeal. This suggests that at high concentrations there may be sufficient magnesium stearate to form more bridges between the particles as it congeals than at low concentrations resulting in a stronger consolidated matrix.

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